School of Science and Engineering

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Design of a biodiesel production pilot unit: Upgrade and automation

By

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Capstone Report

Student Statement:

I, Oumaima Hachimi, hereby affirm that I have applied ethics to the design process and in the selection of the final proposed design. And that I have held the safety of the public to be paramount and have addressed this in the presented design wherever may be applicable.

Approved by my supervisor

________________________
Dr. Abdelghani El Asli
Acknowledgments

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Abstract

Our planet Earth has lately been significantly and rapidly changing due to climate change. This change is highly impacting our lives and health, causing an increase in carcinogenic diseases and other serious health problems. Besides that, it is changing the ecosystems in various ways. For instance, habitat loss is forcing species to migrate, relocate, or die. Moreover, climate change is considered a significant threat to endangered species, reducing their chance of survival. To fight this alarming situation, policymakers, environmentalists, and engineers in all over the world are promoting environmental awareness and integrating new strategies to reduce GHGs emissions. In Morocco, renewable energy systems have been lately gaining great interest as a solution to reduce emissions and meet energy needs. When most of Morocco’s projects deal with solar and wind energies, our capstone project tackles the biodiesel production from waste cooking oil as another prominent method. Since the day Ifrane was selected the cleanest city in Africa, Al Akhawayn University has been promoting this image by being involved in sustainable development projects. This capstone highlights the importance of biodiesel over conventional diesel. It also consists of upgrading and automating the design of the existing biodiesel production pilot plant at AUI. For that, Edraw and Automgen have been used as tools to come up with adequate design/system that is ecofriendly while minimizing human intervention throughout the process.

Keywords: Biodiesel, waste cooking oil, transesterification, glycerol, methanol recovery, water washing purification, design, automation.

Résumé

Notre planète Terre a récemment subi des changements importants et rapides dus au changement climatique. Ce changement a un impact dangereux sur nos vies et notre santé, provoquant une augmentation de maladies cancérogènes et d’autres problèmes de santé graves. En plus, il modifie les écosystèmes de différentes manières. Par exemple, la perte d'habitat oblige les espèces à migrer, se déplacer ou mourir. De plus, le changement climatique est considéré comme une menace importante pour les espèces en danger, réduisant ainsi leurs chances de survie. Pour lutter contre cette situation alarmante, les décideurs, les environmentalistes et les ingénieurs du monde entier s’emploient à promouvoir la sensibilisation à l’environnement et à intégrer de nouvelles stratégies de réduction des émissions de GES. Au Maroc, les systèmes d’énergie renouvelable ont récemment suscité un vif intérêt en tant que solution pour réduire les émissions et répondre aux besoins énergétiques. Lorsque la plupart des projets marocains portent sur les énergies solaire et éolienne, notre projet s’attaque à la production de biodiesel à partir d’huiles de cuisson usées. Depuis le jour où Ifrane a été choisie la ville la plus propre d’Afrique, l’Université Al Akhawayn promeut cette image en s’impliquant dans des projets de développement durable. Cette pierre angulaire souligne l’importance du biodiesel par rapport au diesel conventionnel. Elle consiste également à moderniser et à automatiser la conception de l’usine pilote de production de biodiesel existante à AUI. Pour cela, Edraw et Automgen ont été utilisés comme des outils permettant de concevoir un système adéquat en minimisant les interventions humaines tout au long du processus.

Mots-clés : Biodiesel, huile de cuisson usée, transestérification, glycérol, récupération de méthanol, purification par lavage à l'eau, conception, automatisation.
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List of Acronyms and Abbreviations

AC: Activated carbon
ASTM: American Society for Testing and Materials
B5: Blend of 5% biodiesel and 95% conventional fuel
B10: Blend of 10% biodiesel and 90% conventional fuel
B20: Blend of 20% biodiesel and 80% conventional fuel
B100: Pure biodiesel
BTE: Brake Thermal Efficiency
BSFC: Brake-Specific Consumption
CE: Circular Economy
CI: Compression-Ignition
CO: Carbon Monoxide
CO2: carbon dioxide
EN: European Standard
FAME: Fatty Acid Methyl Ester
FFAs: Free Fatty Acids
GHGs: Greenhouse Gases
IX: Ion-exchange resin
KOH: Potassium hydroxide
MD: Molecular Distillation
NOx: Nitrogen Oxides
nPAH: nitrated PAH
PAH: polycyclic aromatic hydrocarbons
THC: total hydrocarbons
WCO: Waste cooking oil
I. Introduction:

Energy has always been one of the most essential and fundamental resources that endorses the survival, progress and evolution of human species. Throughout history, from the long span of prehistory era where human species relied on both somatic and extra somatic energy types; to our modern societies where there has been a massive reliance and consumption of nuclear and fossil fuels, the shifting of energy has always been a great challenge for human societies to provide their basic daily life needs. As a matter of fact, this shifting of energy has contributed to create a modern world where development has been one of the key achievements of this era. The development seen in many sectors and aspects in our lives, such as in economy, industry and transportation, has contributed to a dramatic and rapid population and economic growths, resulting in an increase demand of primary energy. The following figures shows how dramatic the world population growth is, with a rate of 1.07%/year [1][2].

Figure 1: Increase of the World population [1]
According to the Energy Information Administration (EIA), fossil fuels are the mostly used energy supplies in our industrialized modern world. In fact, crude oil, natural gas and coal supply the vast energy demands and make approximately 80% of the global energy supply [3]. Although fossil fuels are the oldest and most consumed source of energy to provide heat for cooking and space heating and produce mechanical and electrical powers in transportation and industrial sectors, the consequences of their consumption are uncountable. In fact, the burning of fossil fuels results in high emissions of harmful and toxic substances that affect both the environment and the human health.

![Figure 2: The world population yearly growth rate (%) [2]](image1)

![Figure 3: World energy consumption by energy source [4]](image2)
Moreover, the burning of fossil fuels is the main reason behind climate change. As a matter of fact, fossil fuels have always been behind the relatively high emissions of the greenhouse gases, mainly SO$_x$, NO$_x$, CO, and CO$_2$. According to the Intergovernmental Panel on Climate Change (IPCC), the anthropogenic drivers are the main reason behind climate change [5]. IPCC considers the GHGs resulting from industrial activities and the combustion of fossil fuels the dominant factor of climate change being anthropogenic [5]. Furthermore, the other effects of the fossil fuels emissions can be seen in biodiversity. In fact, the GHGs can make the vegetation more likely susceptible to frost damage and diseases, which result in reduced growth and crop yields. Drawback of the reliance on fossil fuels as an energy source is their depletion and exhaustion due to their over-production and overuse to fulfill the high energy demand. Studies have shown that fossil fuels reserves are subjected to a serious shortage. This decline is at an alarming rate, somewhere in the range of 40% to 60% in the upcoming years [6]. The following graph can illustrate this significant decrease in fossil fuels reserves.

![Figure 4: The future of the fossil fuels reserves](image-url)

*Figure 4: The future of the fossil fuels reserves [7]*
The effects of these emissions are not only limited to the environment but can also be extended to harm the public health. According to the National Institute of Environmental Health Sciences, the changes in the amount of the GHGs and other drivers being the result of burning fossil fuels can bring about pernicious human health consequences [8]. See level rise, significant precipitation changes, heat waves, hurricanes, and other environmental impacts of climate change can directly and indirectly alter all of the physical, social, and psychological human health.

Since the beginning of the 21st century, there has been a growing consensus among engineers, environmentalists, politicians and business leaders who affirm that integrating new emissions policies, using renewable energies, as well as developing ways and techniques to use conventional fossil fuels are all essential to induce greenhouse gas emissions reductions and mitigate climate change. Many international organizations, agencies and protocols have been created and committed to develop, promote, and accelerate the shift toward sustainability and renewable energies. As a matter of fact, this clean source of energy is undeniably considered the ultimate solution to both the energy crisis the world has been facing lately and the climate change effects on both the Earth’s ecosystems and human health. This growing interest in renewable energy systems and technologies is due to so many reasons. These systems are first economically safe as they are completely independent of fossil fuel supply and price variables. Moreover, renewable energy technologies have shown a similar efficiency rate as conventional energy sources, but with zero environmental effects, especially toxic and GHGs emissions. Another important reason is the fact that renewable energy resources are geographically more uniformly distributed than fossil or nuclear fuels, which can provide indigenous energy resources to all the countries, especially the fuel-poor ones.
The context of this capstone project is, then, clear. When most of Morocco’s recent projects focus on solar and wind energy, our project focuses only on the production of biodiesel from waste cooking oil. In fact, our project seeks to upgrade an existing biodiesel production pilot unit at Al Akhawayn University. Our project puts sustainable development practices among its top priorities, and for that, we decided to make our biodiesel production pilot unit 100% sustainable.

This capstone adopts as its motivation the intent to increase awareness on the importance of circular economy and biodiesel production in climate change mitigation and adaptation. Moreover, our study seeks to increase the biodiesel production in Morocco starting by the region of Ifrane. As this latter has been selected to be the cleanest city in Africa and Middle East, our project aims to be blended with the context and inspire the rest of the community to take big steps toward sustainability.

II. Literature Review

1. Energy in Morocco

Morocco makes a good example of fuel-poor countries. With approximately no identified fossil fuels, Morocco is heavily dependent on its imports to meet more than 90% of its energy requirements, which makes the country one of the largest energy importers in the MENA region. Since the 20th century, and as a developing country, Morocco has faced a significant steady growth in its energy demand, due to the soared-up living standards, economic development, and industrialization, which have been the reason behind a demand increase of about 6% to 7% per year over the last 30 years. According to the EcoMena, Morocco spent in 2012 somewhere around 10 billion dollars on its energy imports [9]. Furthermore, electricity demand increased by an average of up to 7 percent annually since 2002, with the industrial and residential sectors increasing
by 8% and 7.4% annually. Together, they represent more than 75% of the kingdom’s total electricity consumption [10]. Another point worth mentioning is the fact that, in 2008, the GHGs emissions from fossil fuels amounted to approximately 42.1 metric tons of Carbon Dioxide, CO2, and by 2020 are projected to more than double [9][10].

Figure 5: Increase of energy consumption in Morocco [11]

For the above stated pressing reasons, Morocco has set ambitious renewable energy goals. The Moroccan government has adopted a new strategy aiming at lowering its energy bill by investing in renewable energy technologies and sustainable development. In fact, the country invested more than 20 billion dollars in implementing renewable energy projects to meet its energy demands and promote the use of clean energy. Moreover, in 2009, the kingdom created the “Moroccan Renewable Energy Agency (MASEN)”, which is responsible of developing renewable energy technologies, mainly solar, wind, and hydro powers. By 2020, this new national energy strategy aims to increase the renewable energy resources in the total installed power generation capacity to 43% [12].
Considering its geographic location and diversity, Morocco has promising climate environments for solar and wind energies. Starting with solar energy, the *Noor Solar Plant Project*, located in *Ouarzazate*, makes a great example of the ambition of the national energy policy. This solar power station is considered one of the world’s largest solar energy complexes. Once completed, it will provide more than 580 MW of electric energy [14]. This project is divided into four main phases. The construction of *Noor I*, the project’s first phase, has officially started in 2013 and finished in 2016. Right after, the construction of the second and third phases of the project complex were launched [15]. As a matter of fact, the first two project phases, Noor I and Noor II, use CSP, concentrated solar power, whereas Noor III has employed a solar tower to present a wide variety of the CSP technology. As for the fourth phase, a technological variation of photovoltaic technology will be later introduced [15].

Wind energy plays a key role in the Moroccan renewable energy national plan as well. Since 2007, the Moroccan government has been working on the implementation of wind farms projects.
Amogdoul wind farm in Essaouira and Abdelkhaled Torres in Tangier are two valid examples of the wind power projects on the run in Morocco, with a total energy capacity of 65 MW and 50 MW, respectively [16]. Unfortunately, the Moroccan national energy strategy does not include exploiting the biomass energy. There are, however, several potential projects that can promote the biomass energy sector in Morocco, like biofuels, biogas, and waste-to energy power plants from various abundant feedstock such as crop waste, industrial waste, or solid waste. Moreover, agronomic research has shown that new energetic plants can be adapted to arid zones and that these plants could be grown in the country and be exploited as a green barrier against desertification as well as producing biofuels [17]. Similar to wind and solar energies, the biomass energy sector in Morocco needs investment and full support from both the government and private sector.

2. Circular economy

When talking about sustainability development, circular economy will always be mentioned first. As a matter of fact, the concept of circular economy has lately gained wide attention and interest from business leaders and policymakers within the sustainability framework. Since 1970s, the concept of circular economy has been discussed as a sustainable substitution of the linear production and consumption model of economy, which consists of producing, selling, and using goods, then tossing them aside as waste. This old model of economy, which has dominated the global industrial economy for decades, is leading our planet to a very critical unsustainable situation.

Sometimes, the CE concept is interpreted and understood as efficient recycling and reduction of waste. While these practices could enhance and add up to the results of the CE model, they are in no way a real and appropriate definition of the concept. Accordingly, the CE can be defined as a regenerative concept wherein the commodity input, emissions, and waste are significantly
minimized by shrinking and closing the material loops. According to the *Circular Europe Network*, the circular economy system consists of not solely recycling but rather of the 3Rs (Reduce, Reuse, Recycle) and even go well beyond that to solving the problem of waste [18]. In other words, circular economy involves life cycle loops of materials in a way that they are used and reused over time and their consumption is significantly reduced [18].

![Figure 7: Difference between Linear Economy and CE](image)

A comprehensive review of the literature showed that there is a general agreement that the roots of the concept of CE are directly related to the industrial ecology as well as to the environmental and ecological economics [19]. Some scholars clearly stated that the CE concept cannot be directly traced back to one particular scholar or a school of thought. While others argued that the CE concept’s uptake can be however linked back to the environmental economists Pearce and Turner, as they were the first who introduced the concept on their theoretical framework based on previous ecological studies [20] [21] [22]. Those scholars also added that Pearce and Turner cannot be recognized as the founders of the concept. According to Ellen MacArthur Foundation, an international organization mainly focused on teaching and promoting worldwide efforts in the CE, stated that the CE concept is deeply rooted in history and philosophy and it can be traced back in
many ancient philosophy schools [23]. The foundation added that the concept has known a revival in industrialized countries right after the World War II only when the studies conducted on the linear systems revealed that this latter explicitly created the complex and chaotic world we live in [23]. Moreover, the Ellen MacArthur Foundation argued in its book that there are many schools of thoughts and recent theories which have significantly helped in developing the model of CE, for instance, the Functional Service Economy, aka. Performance Economy, by Walter Stahel, the Benyus’s Biomimicry philosophy, the Cradle to Cradle design theory by McDonough, and last but not least, the Hunter Lovins ‘ and Paul Ha’s Natural Capitalism and Blue Economy [23] [24].

3. Biodiesel Production

3.1 Definition of biodiesel

Because of its countless environmental benefits related to its use, biodiesel is nowadays a prominent fuel as a clean, non-toxic and biodegradable substitute of petrodiesel. Studies have shown that biodiesel is considered superior to conventional fuel with regard to “exhaust emissions, cetane rating, flashpoint, and lubricity characteristics, without any significance difference in heat of combustion of these fuels” [25]. Chemically known as long chains of mono alkyl ester \((C_{19}H_{36}O_2)\), biodiesel is defined as a mixture of fatty acid alkyl esters which can be generally obtained from feedstocks and renewable resources such as vegetable oils; which can be edible, non-edible, or waste cooking oils WCOs, animal fats; which can be edible or waste fats, and algae [25]. In the most cases, biodiesel is produced from a transesterification reaction of the triglycerides. However, in some cases, biodiesel or free fatty methyl ester (FAME) can be derived from a combination of two chemical reactions: esterification and transesterification. In other words, it can be made from an acid esterification prior to a transesterification reaction of the FFAs. Both chemical processes react with alcohol in the presence of an appropriate catalyst [25].
Figure 8: Schematic of the two different biodiesel production processes [26]
Biodiesel can be used in compression-ignition engines either as a pure form called B100, or as an additive to make biodiesel-petrodiesel blends. As a matter of fact, a great number of vehicles users have started nowadays using blends of biodiesel and petrodiesel in order to decrease the environmental impacts of conventional fuels. The B factor is used to refer to the percentage of biodiesel in the fuel blends. The name of the blend refers to the percentage of biodiesel used as an additive. For instance, B20 refers to blending 20% of biodiesel along with 80% of conventional fuel, while B5 refers to adding 5% of biodiesel to 95% of petrodiesel.

3.2 Historical Background of Biodiesel:

Obtaining fuel from a fat is not a 21st Century invention. In fact, the first attempt to make biofuel was back in 1853, when scientists E. Duffy and J. Patrick performed the first vegetable oil transesterification reaction, 37 years before the invention of the diesel engine by Rudolf Diesel [28]. Then in 1900, at the Paris Exposition, Rudolf revealed the first diesel engine ran on peanut oil, which is a vegetable oil’s first known use as a diesel engine fuel [28] [29]. However, given the
cheap availability of petroleum fuels at the time, very few people were interested in biofuels [29]. Some years later, there was a growing interest among scientists in separating fatty acids from glycerin in vegetable oil to generate a petroleum-like product [29]. The vegetable oil fuel had gained more interest during the World War II. Countries such as India, Japan, China, and Brazil had used vegetable oil as a fuel when petroleum supplies were drastically cut down. Then after 1945, when the war ended, and oil prices went down again, vegetable oil fuel was completely left behind. 25 years later, the Oil Embargo incident had pushed scientists from the USA, Austria, and many other affected countries to develop the use of vegetable oil as a diesel fuel engine. However, the damage caused by the viscosity of the vegetable oil was a to scientists. In 1984, the word “biodiesel” was first used when scientists found out a way to convert vegetable oil into biodiesel [29]. On the other hand, scientists were concerned about the biodiesel uneven quality, until 2001 when the ASTM International released the biodiesel standard; the ASTM D6751 Standard, which has assured investors and car manufacturers that biodiesel would meet their quality requirements [29].

3.3 Petrodiesel

Petrodiesel, also known as fossil fuel, is defined as the fuel used to run CI engines. Petroleum diesel is the result of exposing crude petroleum to fractional distillation at atmospheric pressure along with very high temperatures, ranging somewhere between 200°C and 350°C [30]. Petrodiesel consists of long chains of carbon, each molecule contains from 8 to 21 atoms of carbon [30]. Petrodiesel is known to be a toxic and carcinogenic fuel. In fact, its emissions are causing pernicious effects on both the environment and humans. Scientists have defined more than 50 toxic and carcinogenic components in petrodiesel. The following figure depicts this harmful side through an illustration of a petrodiesel soot, also known as articulate matter.
3.4 Biodiesel vs Petrodiesel

To assess the potential of biodiesel in being a prominent source of energy, it is imperative to compare it to the conventional fuel, petrodiesel. Many scientific studies have revealed similarities and differences between petrodiesel and biodiesel. A scientific research [32] investigated the thermodynamics behind the use of biodiesel in CI engines and compared it to those of petrodiesel. The study showed that biodiesel performs the same as petrodiesel in CI engines, and sometimes results are even better when using a high compression ratio [32]. The following figure depicts the performance of petrodiesel, B10 blend, and pure biodiesel.
Another study by [33] analyzed the two fuels performance on a diesel engine. The results showed that using biodiesel significantly reduced brake thermal efficiency (BTE) as well as the emissions of GHGs and pollutants to the air but on the other hand increased the brake-specific consumption (BSFC)\[Z\]. As a matter of clarification, BTE is the ratio linking energy input (fuel injection) with the output while BSFC evaluates the efficiency of the fuel in the regard of internal combustion. It is directly related to some properties such as the heating value, viscosity, as well as the density [27].

![Figure 12: BTE and BSFC in D (diesel), B20, B10, B5, and in pure biodiesel at full load [34]](image)

In addition, the results of a scientific research by [32] have revealed that the low vapor pressure makes the biodiesel cavitate less than conventional fuel and that its higher viscosity decreases the flow efficiency as well as the velocity of injection. Moreover, studies have demonstrated that using biodiesel in engines evokes lubricity. In fact, the oxygen atoms in the biodiesel, which do not exist in petrodiesel, generates polarity which is the dominant factor affecting lubricity [35]. As far as the environmental impact is concerned, numerous studies have investigated the behavior of biodiesel
towards the environment. A scientific research by [36] studied biodiesel emissions and compared them to the ones of petrodiesel. Results have shown that GHGs and pollutants were reduced up to 83%, except for NOx. In fact, using biodiesel in engines has an opposite effect on NOx emissions, an increase of up to 12% can be seen.

![Figure 13: Comparison of pollutant emissions obtained when using B100 and B20 fuels [37]](image)

3.5 Waste Cooking Oil

The recycling of waste cooking oil is nowadays gaining wide interest and attracting important investments from international companies involved in managing this waste as well as making profit out of it. Waste cooking oil is the vegetable oil used in cooking and frying food. Due to its high free fatty acid (FFA) content, repeated usage of the frying oil makes the vegetable oils inedible. As a matter of fact, dumping waste cooking oil as waste creates uncountable problems for both the environment and human health. Pouring waste cooking oil without any treatment into the sink has a high pollutant potential, it can ruin the public sewers, more than that, it disturbs the aquatic
ecosystems after being dumped into streams and rivers by causing a drastic drop in oxygen levels, which eventually results in the eutrophication of water [38]. Therefore, instead of dumping it to the environment and then ruining many ecosystems, waste cooking oil can be used as an alternative feedstock for biodiesel production as it is highly efficient as well as cost effective [39]. The conversion of waste cooking oil into biodiesel is thus a winning approach to manage and minimize domestic waste while helping to solve the global energy challenge [39]. In addition to producing biodiesel, waste cooking oil could be used in producing soap as well as additives for making lubricant oils [39]. Furthermore, using waste cooking oil to produce biodiesel decreases the need for crops production and solves the food security issue.

The chemical and physical characteristics of waste cooking oil differ from those of vegetable oil. In fact, this difference is due to the chemical reactions which occur between the oil and food, such as oxidation, hydrolysis, and polymerization [38]. These properties might change based on many conditions, such as the frying time and temperature [39]. As a matter of fact, the frying process leads to converting the triglycerides in the vegetable oil to monoglycerides, diglycerides, and FFAs [38]. Moreover, an increase in the values of both saponification and viscosity can be significantly noticed in the WCO due to the polymerization and oxidation processes compared. Furthermore, because of the transfer of matter between the food and vegetable oil, there is an increase of water in the WCO [38]. Not to forget the fact that the volume of water and amount of heat during the cooking process tends to rise the hydrolysis process of triglycerides, resulting in FFAs growth in the waste cooking oil [38].
Experiments have shown that the presence of water in the WCO usually leads to the hydrolysis reaction during the transesterification process, while high levels of saponification and FFA results from saponification reaction [38] [39]. The following table sums up the main properties of the waste cooking oil.

**Table 1: WCO Properties [42]**

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Density (g/cm³)</td>
<td>0.91 – 0.924</td>
</tr>
<tr>
<td>Kinematic Viscosity (40°C) (mm²/s)</td>
<td>36.4 - 42</td>
</tr>
<tr>
<td>Saponification</td>
<td>188.2 - 207</td>
</tr>
<tr>
<td>Acid value</td>
<td>1.32 – 3.6</td>
</tr>
<tr>
<td>Iodine number</td>
<td>83 – 141.5</td>
</tr>
</tbody>
</table>
Before using WCO in the process of producing biodiesel, it is very important to know and study these chemical and physical characteristics as they are a critical key issue in determining many criteria such as the engine adequacy, quality management and control, etc. [43].

### 3.6 Pretreatment of WCO:

Prior to proceeding for the biodiesel production reaction, WCO has to go through a pretreatment process. First, WCO has to be filtered to get rid of any food chunks. This can easily be achieved using a cloth filter [44]. Studies have shown that preheating WCO will result in higher yields. That being said, WCO has to be preheated at somewhere between 50°C and 60°C to enhance the WCO properties [44]. A study [45] compared WCO properties as well as biodiesel yield during preheating and non-preheating WCO. Results showed that preheating the WCO at 60°C for 30min resulted in a 98% biodiesel yield. It also lowered the water content in the WCO, saponification, as well as the FFAs concentration [45].

### 3.7 Transesterification of WCO:

Transesterification is considered the most known process to produce biodiesel. During this reaction, the triglyceride molecules of vegetable oil react with an alcohol (ethanol, methanol, etc.) in the presence of a catalyst (KOH, NaOH, etc.) to produce biodiesel (esters) and glycerin [46]. The following figure shows the transesterification reaction schematic, where $R$ represents a long chain of hydrocarbons.
According to numerous studies, it has been demonstrated that this reaction largely relies upon numerous parameters including “the reaction temperature, time, pressure, alcohol to oil molar ratio, agitation rate, the type and concentration of the catalyst as well as the FFA concentration in the WCO” [47]. Methanol is mostly used as an alcohol in the transesterification of the WCO for its good efficiency, whereas ethanol and isopropyl alcohols can be used during the transesterification of animal fats [47]. As for the catalysts, the mostly used ones are the homogeneous alkali catalysts, which are soluble and can be either liquid or gaseous [39]. This type of catalysts is widely used for their availability, effectiveness and time efficiency in a way that they can catalyze the reaction at a low temperature and pressure and achieve a high conversion in a less time period [47]. The homogeneous catalysts are two types of these catalysts: alkaline and acidic. The alkaline homogeneous catalysts such as KOH and NaOH are the ones used during the transesterification reaction while the acidic ones are used during the esterification [39][46]. However, the efficient use of the alkaline homogeneous catalyst is solely restricted to the WCO with an FFA percentage below 0.5%, or in other words, an acid value below 1mg KOH/g [39]. Furthermore, after the end of the reaction, separating the catalysts from the biodiesel requires washing this latter with water which could lead to a high energy consumption, generation of a significant amount of waste water,
and in some cases, a loss of fatty acid alkyl esters [48]. Moreover, as the catalyst is so challenging to recover, it could lead to a reactor corrosion, which increases the total biodiesel production costs [48]. The second type of catalysts is the heterogenous catalysts. This type of catalysts is solid, so it is insoluble in the reaction. Metal oxides such as KBr, CaO, SrFe$_2$O$_4$, SiO$_2$-SO$_3$H, and chitosan are the most used ones [39]. From an economic perspective, heterogeneous or solid catalysts can be favored over the homogeneous ones as they can be easily separated from the final products, reused over time, and provide a better final products quality [48]. Moreover, these catalysts do not require a washing stage and can tolerate a high FFA percentage. Nonetheless, heterogeneous catalysts have many inconveniences as well, mainly being the extreme reaction conditions such as higher reaction temperatures, pressure, concentration and time compared to the ones required for the homogeneous catalysts [48]. Moreover, saponification is seen in some cases when using the solid catalysts; thus, a purification process is required to meet the biodiesel standard quality [48].

The third and last type of catalysts is called the enzymatic catalysts. As a matter of fact, this type of biodiesel production catalysts has become the most promising ones when considering an economic standpoint, in a way that they can catalyze the reaction at lower temperature and pressure and tolerate a high FFA percentage [39]. However, the use of an enzyme catalyst requires a much longer reaction time compared to the homogeneous and heterogeneous catalysts.

### 3.8 Esterification of WCO:

As stated before, a high FFAs percentage in the WCO has the potential to cause many problems, mainly an engine damage. The esterification process can be then used to bring the FFAs content in the WCO below 2% [49]. The following figure shows a schematic representation of the reaction.
Figure 17: Schematic of the esterification reaction [49]

Esterification process can also be used to produce an ester prior to the transesterification reaction to reduce the amount of FFAs in the presence of an acid homogeneous catalyst such as Sulfuric acid, sulfonylic acid, and hydrochloric acid [48]. Acid heterogeneous catalysts such as SrFe2O4 can be as well used to catalyze the esterification process and be separated easily through a magnetic field [39].

3.9 Methanol recovery

As it is previously stated, methanol is widely used as a primary alcohol in producing biodiesel from WCO due to its numerous advantages compared to other alcohols. In fact, methanol has a high reactivity, prevents soap formation, and can be easily recovered as it does not form azeotrope [50]. During the transesterification reaction, using an excess of methanol is required to speed up the reaction to completion and maintain an efficient alcohol to oil molar ratio [50]. This being said, the methanol recovery process can enhance both the environmental and economic efficiency of the biodiesel production unit since it helps meet the international biodiesel quality standards and reduces the biodiesel production process cost.

After the end of the transesterification reaction, the unreacted methanol would be then transferred to the product mixture. This mixture, which is the biodiesel and glycerol, is usually
separated using gravity, as the two products have two different densities [51]. The methanol recovery phase can be done either before or after the separation phase. However, a comprehensive review of the literature has shown that a methanol recovery phase is preferred to occur before the separation, as the excess amount of methanol would behave as a phase stabilizer which on the other hand reduces the separation rate and increases the load at the downstream unit [52]. There exist many approaches to recover methanol from a biodiesel production unit, but many studies shed light on the use of a distillation column. As a matter of fact, this approach works in a way that the product mixture is heated up to somewhere above 64.7°C (148.5°F), the boiling point of methanol, then air bubbles are created in the mixture to enhance the vaporization of methanol, finally, the vaporized excess of methanol is recovered through a distillation column [52].

### 3.10 Biodiesel Purification:

After the end of the transesterification reaction, the product mixture is let to settle down. In most cases, the product mixture is divided into two layers based on a density difference, biodiesel is always the upper layer while glycerol is the lower one. However, in the case of using an ionic liquid catalyst, the product mixture is then divided into three layers [53]. The biodiesel purification unit can be done at various phases of the overall production process. In fact, a comprehensive review of the literature has stated that a purification unit can be done either before or after the separation phase [53]. However, recent studies have revealed more effective and efficient advantages of purifying the biodiesel after separating it from glycerol [52]. With this being said, the crude biodiesel produced after the separation from glycerol contains impurities, contaminants and volatile matter which highly effect the performance of biodiesel [53]. As a matter of fact, the biodiesel performance is significantly related to its storage stability, purity and cleanliness. The biodiesel stability is measured by exposing the biodiesel to high temperatures and oxygen,
evaluating color degradation, and analyzing viscosity changes [54]. The following table highlights the effects of impurities on both the performance and properties of biodiesel.

**Table 2: Impurities effects on biodiesel [54]**

<table>
<thead>
<tr>
<th>Impurity</th>
<th>Effect</th>
</tr>
</thead>
<tbody>
<tr>
<td>Free Fatty Acid (FFA)</td>
<td>Corrosion, low oxidative stability</td>
</tr>
<tr>
<td>Water</td>
<td>Formation of FFA, corrosion, bacterial growth (filter blockage)</td>
</tr>
<tr>
<td>Methanol</td>
<td>Low density and viscosity, low flash point, corrosion</td>
</tr>
<tr>
<td>Glycerides</td>
<td>High viscosity, injector deposits, crystallization</td>
</tr>
<tr>
<td>Metals (soap, catalyst)</td>
<td>Injector deposits, high sulfated ash (filter blockage), abrasive engine deposits</td>
</tr>
<tr>
<td>Free Glycerin</td>
<td>Settling problems, Increases in aldehydes and acrolein exhaust emissions</td>
</tr>
</tbody>
</table>

There are numerous techniques and approaches to purify the methyl ester from all its impurities and contaminants. Moreover, processing this crude product and removing the impurities and volatile matter from it will allow manufacturers to meet the ASTM or EN biodiesel quality standards. The following table shows the ASTM standards for the produced biodiesel.

**Table 3: ASTM standards of biodiesel produced from WCO [55]**

<table>
<thead>
<tr>
<th>Property</th>
<th>Standard Value</th>
<th>Unit</th>
</tr>
</thead>
<tbody>
<tr>
<td>Specific gravity</td>
<td>0.87–0.89</td>
<td>g/cc</td>
</tr>
<tr>
<td>Viscosity</td>
<td>1.9–6.5</td>
<td>mm²/s</td>
</tr>
<tr>
<td>Calorific Value</td>
<td>&gt; 35</td>
<td>kJ/g</td>
</tr>
<tr>
<td>Cetane number</td>
<td>&gt; 47</td>
<td>No unit</td>
</tr>
<tr>
<td>Acid value</td>
<td>≤ 0.50</td>
<td>mg KOH/g</td>
</tr>
<tr>
<td>Cloud point</td>
<td>-3 to 12</td>
<td>°C</td>
</tr>
<tr>
<td>Pour point</td>
<td>-15 to 10</td>
<td>°C</td>
</tr>
<tr>
<td>Saponification value</td>
<td>&lt; 312</td>
<td>mg KOH/g</td>
</tr>
<tr>
<td>Iodine value</td>
<td>&lt; 120</td>
<td>g I²/100 g oil</td>
</tr>
</tbody>
</table>
3.10.1 Water washing method

The water washing method is the most used one in the purification of crude methyl ester (biodiesel). As its name implies, wet washing relies on adding water to the crude biodiesel followed by separating the aqueous phase from the organic one to remove all the soluble impurities, be they contaminants, excess production chemicals or volatile matter.

![Diagram of biodiesel water washing process](biopowered.co.uk)

*Figure 18: Diagram of biodiesel water washing process [55]*

A study by Aswati and Suprihastuti [56] analyzed the use of water in purifying the crude biodiesel. The study showed that a 20 min wet washing conducted by adding half the biodiesel volume of water lowered the free glycerol concentration from 0.9331% to 0.09% [56]. The same study showed that the free glycerol content was seen to be less than 0.05% with a pH of approximately 7.3 when 300% biodiesel volume of water was added for wet-washing [52]. Moreover, the study demonstrated that, in order for the glycerol concentration in the biodiesel to be less than 0.02%, the crude biodiesel has to go through multi wet washes [55]. Furthermore,
Aswati and Suprihastuti [56] tackled in their research the effects of both temperature and water to biodiesel volume ratio on the mass transfer from biodiesel to water of the glycerol. In fact, the study showed that the higher the temperature and the water volume ratio to biodiesel resulted in a higher mass transfer rate [56]. In other words, as we increase the wet washing temperature, the diffusivity of impurities in crude methyl ester is significantly raised, which results in a higher mass transfer coefficient, and as the water is more added to the crude biodiesel, the mass transfer area is significantly increased, therefore, the volumetric mass transfer coefficient will be much higher [56]. A study conducted by Berrios [54] investigated biodiesel water washing using deionized water and acidified water. The study’s results showed that acidified water performed better in removing soap but requires many washes to remove other impurities [54]. Another study compared hot water washing to acidified water washing using phosphoric acid. After the two methods were evaluated, the results showed that significant changes on the biodiesel properties were met using hot water washing compared to acidified water washing [57].

3.10.2 Dry washing methods

Biodiesel dry washing is the process of refining the crude biodiesel from all its impurities and contaminants without using water. There exist several methods and techniques to make a biodiesel dry washing unit, being dry washing columns and media [54]. The dry washing technique has numerous advantages. First, as the name implies, it is waterless, which protects the water commodity, meets the ASTM D6751 water content (< 500 ppm), and cancels out both the water washing waste and time [54]. Moreover, the dry washing process is easy to integrate, minimizes space and tank’s capacity [55]. In addition, crude biodiesel dry washing has been proved to reduce the biodiesel production cost as well as time [55]. Studies conducted on dry washing techniques all agreed that this method can achieve a total removal of glycerin and soap, however, it does not
remove the traces of methanol [54]. Some dry washing techniques can remove a small amount of methanol, but certainly not enough to meet the ASTM or EN standards [54][55].

3.10.2.1 Ion-exchange resins:

An ion exchange resin, also called ion-exchange polymer, is another biodiesel purification technique under the dry washing method. The ion exchange resin is defined as a resin which functions as a physical interchange medium of ions. It is made of organic polymer substrates with very small beads whose radius varies between 0.25 mm and 0.5 mm [57]. Usually, the beads of the ion exchange resin are porous and have a wide surface area [57]. The resin matrix consists of ion interchange sites, where the polymer system attracts the functional groups of anions and cations [54]. The ion exchange resins work in a way that they enhance ions exchanges through a reversible chemical reaction which occurs by replacing the dissolved ions with others of a similar electrical charge [57]. In other words, the purification process consists of adsorbing the ions from the ion exchange fluid and replacing them by ions of the same charge. The following figure depicts an example of this reversible mechanism. The figure illustrates an ion exchange adsorption between sodium ion Na\(^+\) and hydrogen ion H\(^+\) where the ion interchange particle is surrounded by a film diffusion layer. In this case, the ion exchange adsorption process works in a way that sodium ions are passed from the fluid to the ion exchange particle through the diffusion film layer while the hydrogen ions are diffused from the diffusion film to the fluid through the same diffusion film layer [58].
The manufactured ion exchange resins are available in five different types, strong and weak acid cation exchange resins, strong and weak base anion exchange resins, and Chelating resins. For the biodiesel purification process, the type used is the strong acid cation type (SAC) [58]. This type of ion exchange resins is produced through the sulphonation process of styrene-divinyl benzene copolymers [58]. The strong acid cation type is mainly manufactured in two different forms, the microporous resin and gel [58]. Gel resins, produced from Purolite, Rohm and Haas, and Thermax, have a luminous appearance and low cross-link bonds [54]. On the other hand, macroporous ion exchange resins have an opaque appearance and higher cross-link bonds [54].
When most of the ion exchange polymers use a resin with small porous microbeads, some employ resins with sheet meshes [54]. Based on their application, the ion exchange resin beads can either have a Gaussian size distribution, or a uniform particle dimension, depending on their application [57]. For instance, the translucent IX resin beads are the most used ones because of their great efficiency and capacity while microporous resin beads, which can be white or yellowish, are used for more demanding conditions because of their extremely higher chemical stability and resistance [58]. The ion exchange resins can be used in many applications, namely purifying the crude biodiesel from its impurities after settling and removing the glycerol. As a matter of fact, literature have shown that after the bed reaches equilibrium, ion exchange resins can work extremely well at purifying the methyl ester from glycerin, soap, water, excess catalyst and other impurities [58]. That being said, the ion exchange resins can be used to absorb the impurities for an infinite number of cycles [58]. On the other hand, a comprehensive review of the literature showed that, in practice, overusing the resin for an infinite series of purification cycles is not effective as it reduces its absorptive potential [58]. Moreover, the unsaponifiable content in the methyl ester highly affects the absorptive capacity of resins [54]. A study [61] investigated the effect of ion exchange resins on the properties of the methyl ester, mainly the concentration of the
methanol, soap, and glycerol, oxidative stability and acid. The study tested two different ion exchange resins, Purolite and Rohm and Haas. It was found that the two different resins performed similarly. The study also showed that after reaching equilibrium, the two resins significantly lowered soap and glycerol concentrations of the crude biodiesel, while they did not influence much the methanol concentration [59]. Moreover, it was found that the methyl ester purification using the two ion exchange resins increased the acid value of the biodiesel, while it did not have any effect on the oxidative stability [59].

3.10.2.2 Activated Carbon:

Since ancient times, carbon had been used as a purifying agent by many civilizations for its useful adsorptive properties. For instance, Pharos used this adsorptive material for purification and medicinal purposes, while ancient Hindu societies used it for water purification [59]. Today, multiple activation and carbonization processes, especially physical and chemical, have been developed to enhance this adsorptive material.

Activated carbon (AC) is nowadays used as an adsorptive material for different fields, such as in water and air purification, energy storage, and nitrogen separation in industry [59]. To start with, activated carbon is defined as a carbon material with a very large inner surface area and a well-developed porous structure [60]. Activated carbon consists of carbonaceous material obtained from raw materials such as charcoal, peat, lignite, bituminous coal, coconut shells, etc. It is usually composed of 87% to 97% of carbon, but also contains other elements depending on the raw material and activation method used [61]. Activated carbon’s porous structure enables it to absorb materials in the liquid and gas phases. Activated carbon can usually be found under the form of pellets, powder, granular, or ball shapes [61].
The purification process of biodiesel using activated carbon works in a way that the impurities and contaminants are attracted and absorbed by AC, a mechanism similar to the one of ion exchange resins.
The effects of activated carbon produced from spent tea waste on biodiesel impurities and contaminants were investigated in a research study [64]. The study also compared the use of AC to water washing and ion exchange resin gel. AC showed better. In fact, the AC yield was higher than the one of the two other purification methods. Moreover, properties such as viscosity, density, Iodine value, acidity value, and have met the ASTM standards while using AC for one purification cycle [64]. In addition, AC provided an efficient adsorption of methanol, glycerol and free catalyst [65]. However, the AC would no longer be efficient once the AC particles are completely filled with impurities, which is the main reason why AC is not a predominant primary adsorbent.

3.10.2.3 Magnesol:

Magnesol, also known as synthetic magnesium silicate MgO\(_2\):6SiO\(_2\):H\(_2\)O, consists of a white odorless powder. It is considered as the very first biodiesel purification methods. The following figure depicts the schematic diagram followed for some dry washing methods, including Magnesol [66].

![Figure 23: Schematic of some biodiesel dry washing methods [66]](image-url)
A comprehensive study of literature showed that the purified biodiesel by Magnesol has met both the quality standards of both ASTM D675 and EN14214 [66]. As a matter of fact, the purification process functions in a manner that Magnesol attracts polar substances, be they impurities and free contaminants. This biodiesel purification method requires intensive mixing. A research [67] studied the properties of biodiesel after purification using Magnesol showed that methanol concentration was lowered to a 0.51% while the concentration of glycerol decreased to 0.03%. A similar study showed that Magnesol purification lowered the concentrations of methanol, soap, and glycerol to 98%, 92%, and 55% respectively [67]. As for the other properties, the same study showed that properties such as density, acidity, and iodine values met the international quality standards after using Magnesol [67]. Prior to using biodiesel as fuel for diesel engines, a filtration cycle is required. This can easily be achieved using a macroporous filter cloth [67].

3.10.2.4 Filtration:

In 2009, it has been found that biodiesel impurities could be removed through a filtration process using a membrane [67]. After recovering methanol and draining glycerol, unpurified biodiesel is transferred to a ceramic membrane for filtration. A similar study assessed the efficiency of the biodiesel filtration process used three ceramic membranes of different pores (0.6 µm, 0.2 µm, 0.1 µm) [68]. It was found that the smaller the pores the better the filtration. In fact, the ceramic membrane with 0.1 µm pores showed better filtration results [66]. The following figure shows the schematic of the biodiesel filtration process.
3.10.2.5 Distillation:

Distillation can also be used to purify biodiesel from impurities, contaminants, and volatile matter. There are various types of distillation methods which can be implemented to achieve an efficient biodiesel refinement, such as azeotropic distillation, MD distillation, and extractive distillation; not to forget the traditional distillation [69]. After separating crude biodiesel from glycerol and excess of methanol, it is passed through a distillation column while maintaining a high temperature which can sometimes reach 120°C [69]. Another way to conduct the biodiesel refinement is to implement a molecular distillation (MD) under high-vacuum by creating evaporation and condensing cycles [69]. The distillation method can reach a 98% separation yield. However, this purification method requires very high temperatures.
3.11 Glycerol as a by-product: a potential energy source

The production of biodiesel leads to generating glycerol which is a by-product. Literature have shown that 10 kg of this by-product is usually obtained for every 100 kg of produced biodiesel [70]. This by-product can be valorized by being used in making soap and pharmaceutical products. However, this process requires a very expensive purification unit. Several studies have investigated other valorization ways of this by-product. In fact, some studies have demonstrated that glycerol can be used as a carbon source for the fermentation process and generation of biogas [70]. In fact, the high Carbon content of glycerol has made this latter a great option to be integrated in anaerobic digester in order to increase methane production. Another research examined the potential of using glycerol along with biomass to make combustible pellets as a coal substitution [71].

III. STEEPLE Analysis:

Successful projects are the ones that are planned both effectively and efficiently. Strategic planning is a must phase before realizing a project. In fact, strategic planning is highly crucial in determining the path and current situation of the project as well as the possibility of either its success or failure. Thus, before the implementation of a project, it is mandatory to conduct many analyses on the external and internal factors and driving forces related to the project. There are numerous strategic analysis methods and tools which can offer a deep analysis and understanding of the aspects affecting a project. STEEPLE, SWOT, PEST, PESTLE are some examples of the most used tools at the professional level [70].

In this capstone project, we will tackle STEEPLE as an analysis tool along with its features and characteristics, as it is a necessary phase in carrying out engineering capstone projects at AUI.
As a matter of fact, STEEPLE Analysis allows us to foresee emerging trends by considering external factors which may affect the project in the future. The STEEPLE Analysis has to do with 7 different fields as its name implies:

- S ---- Social
- T ---- Technological
- E ---- Environmental
- E ---- Economic
- P ---- Political
- L ---- Legal
- E ---- Ethical

The following figure tackles the STEEPLE Analysis conducted for our project.
<table>
<thead>
<tr>
<th>Political &amp; Legal:</th>
<th>Technological:</th>
<th>Social:</th>
</tr>
</thead>
<tbody>
<tr>
<td>• There are no regulations or legislation when it comes to biodiesel in Morocco.</td>
<td>• Working with engineering tools in upgrading the design of the pilot unit as well as automating the plant.</td>
<td>• Promote the idea of social responsibility towards the environment as a commodity</td>
</tr>
<tr>
<td>• Lack of governance</td>
<td>• Up scaling the production from 40L to 200L of waste cooking oil</td>
<td>• Pushing the community of Ifrane to realize the importance of renewable energies.</td>
</tr>
</tbody>
</table>

**Ethical:**

• Raising awareness on sustainable development and circular economy
• Promoting the recycling process in the region

**Environmental:**

• Biodiesel is biodegradable, non-toxic, and renewable.
• Clean energy source.
• Preserved nature and ecosystem.
• Reduced pollution as a result of less GHGs emissions

**Economic:**

• Producing biodiesel can become competitive to diesel fuel.
• Optimization of the resources at hand.
IV. Design of the biodiesel production pilot unit

1. Process selection and design

As it is mentioned before, our project deals at first hand with recycling waste cooking oil by using it as a raw material in producing biodiesel. Thus, in this section, we will discuss the biodiesel production process followed in our pilot unit. Our process is pretty simple and straightforward. As a matter of clarification and based on the analysis of the collected WCO properties, we found that the FFAs content is not high (< 2%). That being said, the acid esterification is not required, and a transesterification reaction will be directly proceeded to produce biodiesel. Figure depicts the different stages that would be proceeded to produce biodiesel from WCO. We divided the process design into four main steps: pre-treating WCO, transesterification reaction methanol recovery, and last but not least, a purification stage.

1.1 WCO pre-treatment stage

Before proceeding to the biodiesel production reaction, WCO should be pre-treated. We decided to filter the oil to get rid of any food debris and pre-heat it to remove water and moisture. For filtration, we choose to opt for the traditional method, fabric. A macroporous cloth filter is all what it takes to filter WCO from any food chunks or debris. This method is simple and cost effective. Then, WCO will be heated to 50°C for about an hour to make sure all the moisture is removed to prevent the hydrolysis reaction to happen.

1.2 Processing phase

After pre-treating the raw material, WCO is pumped to the reactor tank where it will be mixed with methanol in the presence of a catalyst, which is chosen to be Potassium Hydroxide
KOH. The transesterification will require a temperature of 55°C and a good agitation for two hours long. This reaction results in methyl ester (crude biodiesel) and a by-product, glycerol.

1.3 Methanol Recovery

Methanol is a very expensive and its price is tied up to oil prices fluctuation. As it was stated before, our goal is to design a sustainable yet cost effective pilot unit, that is why we added a methanol recovery phase in our pilot unit. The recovery process is very simple. After the end of the transesterification reaction, methanol is recovered by heating up the reactor tank at 65°C to make sure all the excess of methanol boils. Then, the evaporated methanol goes through a condenser to finally be recovered and used in the next batches.

1.4 Separation phase

After the end of both the transesterification reaction and methanol recovery phases, the mixture is let to settle down for about 24 hours. The mixture is then separated to two phases due to gravity and change of density. Glycerol is placed at the bottom while crude methyl ester is at the top. Glycerol can be then drained out easily from the reaction tank.

1.5 Purification phase: water washing

As stated previously, there exist many techniques and methods to purify crude biodiesel. As a purification method, we chose the water washing, also known as wet washing. This technique will in fact remove all the impurities, contaminants, and volatile matter by increasing the interaction between crude biodiesel and water while reducing the risk of emulsification. As a matter of clarification, this purification stage is optional in our pilot unit. It can be eliminated in the case of producing crude biodiesel which can be used for heating purposes, while it will be proceeded in
the case of producing pure biodiesel that has to meet international quality standards, ASTM and EN standards in order to be used as a substitute of conventional diesel or in fuel blends. For each batch, a water amount of twice the WCO volume will be added to the reactor tank. After an agitation of one hour, the aqueous phase will be drained out after 24 hours of settling down the mixture.

The following diagram depicts our designed biodiesel production process in detail.

- Filtering WCO.
- Heating WCO at $T = 50^\circ C$ for 30min.
- Preparing the potassium methoxide solution: Methanol + Catalyst (KOH)

- Pumping the WCO to the reaction tank.
- Pumping the potassium methoxide solution to the reaction tank
- Reaction + agitation at a temperature of $T = 55^\circ C$ for 2hours.
- Methanol recovery at $T = 65^\circ C$.
- Setting the mixture for 24h.
- Draining glycerol.

- 2 water washing cycles to purify crude biodiesel.
- Drying the washed biodiesel.
- Filtering biodiesel using two different filters.
2. Process Modules

In this section we will discuss the different modules which will be implemented in our biodiesel production pilot unit plant. As a matter of fact, the less the number of process modules, the better is the design of the pilot unit plant. Moreover, this will allow us to build an economically feasible plant and save the space. We started by designing six different modules: Heating module, Reception module, Reaction module, Separation module, Purification module, and a Clean-In Place module. Then, we decided to lower the number to four modules in a way that the reaction, separation and purification modules would be grouped under one module.

- Q --- Heating module
- R --- Reception module
- RSP --- Reaction/Separation/Purification module
- CIP --- Clean in place module

The Clean in place module can be purchased and easily be integrated to our biodiesel pilot unit. The following figure depicts the relation between the stated modules in our biodiesel production pilot unit.
3. Process Flow Diagram (PFD)

In this section, we will shed light on the proposed process flow diagram for our biodiesel production pilot unit. The design is created using the software Edraw.
<table>
<thead>
<tr>
<th>Element(s)</th>
<th>Name</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Methanol inlet</td>
</tr>
<tr>
<td>2</td>
<td>Catalyst (KOH) inlet</td>
</tr>
<tr>
<td>3</td>
<td>200L of WCO reception</td>
</tr>
<tr>
<td>4</td>
<td>400 L of hot water inlet</td>
</tr>
<tr>
<td>5, 18</td>
<td>Agitators</td>
</tr>
<tr>
<td>6, 19</td>
<td>Heating elements</td>
</tr>
<tr>
<td>7</td>
<td>Potassium methoxide tank</td>
</tr>
<tr>
<td>8</td>
<td>WCO reception Tank</td>
</tr>
<tr>
<td>9, 10, 21, 25</td>
<td>Control valve</td>
</tr>
<tr>
<td>11, 13, 15, 16, 26, 27</td>
<td>3-way valves</td>
</tr>
<tr>
<td>12</td>
<td>Peristaltic pump</td>
</tr>
<tr>
<td>14</td>
<td>Centrifugal pump</td>
</tr>
<tr>
<td>17</td>
<td>Shower element</td>
</tr>
<tr>
<td>20</td>
<td>Reaction tank</td>
</tr>
<tr>
<td>22</td>
<td>Condenser</td>
</tr>
<tr>
<td>28, 29</td>
<td>Filters</td>
</tr>
<tr>
<td>30</td>
<td>Recovered methanol outlet</td>
</tr>
<tr>
<td>31</td>
<td>Glycerol outlet</td>
</tr>
<tr>
<td>32</td>
<td>Crude biodiesel outlet</td>
</tr>
<tr>
<td>33</td>
<td>Pure biodiesel outlet</td>
</tr>
</tbody>
</table>
4. Process Explanation

In this section, we will describe the biodiesel production process occurring in our designed biodiesel production pilot unit.

Pretreatment:

- 1.73Kg of KOH (catalyst) and 40L of Methanol are introduced to tank 7.
- Agitator 5 is turned on to mix the KOH with methanol at a temperature of $T = 50^\circ$C. After $t = 30$ min, it is turned off.
- After filtering WCO to get rid of any food chunks, it is heated at a temperature of $T = 50^\circ$C for $t = 1h$.

Treatment:

- 200L of WCO is pumped to tank 20. Valve 11, 15, and 16 are opened while pump 14 is turned on.
- Methoxide solution is pumped as well to the reaction tank 20. Temperature is maintained at $T = 55^\circ$C. Valve 10, and 16 are opened while pump 12 is turned on.
- Agitator 18 is turned on to mix the mixture. After $t = 2h$, agitator 18 is turned off.
- Temperature is elevated to $T = 65^\circ$C. The circuit \{13,14,15,17\} is used to recover methanol, the valve 21 is open. Methanol is condensed through element 22.
- After reaching a conductivity (To be determined experimentally), the valve 24 is closed and temperature is let to decrease to $T = 40^\circ$C.
- The mixture is let to settle down for 24h. The mixture is split into two phases. Valve 26 is opened. The glycerol is then drained out and stored as byproduct. A conductivity sensor is placed in the reaction tank to detect conductivity. After a change of conductivity, valve 26 is closed.

Posttreatment:

- Two water washing cycles:
  - After draining glycerol, 400L of hot water is added to the reaction tank 20. Agitator 18 is turned on to mix the solution for $t = 1h$.
  - The mixture is afterwards let to settle down for 24h. The mixture is divided then to two phases (aqueous and organic)
  - The aqueous phase (waste water) is drained out. Valve 25 opened.
- Biodiesel drained out:
  - After $t = 1h$, biodiesel is filtered through two filters of different pores valve 26 and 27 opened.
  - ⇒ Pure Biodiesel.
V. Biodiesel Production Cost

The components needed in our biodiesel pilot unit are as follows:

- Waste Cooking Oil WCO
- Methanol
- KOH

Collecting waste cooking oil is free of charges. However, methanol is the most expensive reactant in our production process. Based on a financial analysis previously done and leaded by Dr. Abdelghani El Asli, we could generate the following table that sums up all the charges in detail.

Table 5: Cost estimation of the biodiesel production

<table>
<thead>
<tr>
<th>Reactants</th>
<th>Qt. Needed</th>
<th>Optimistic Price (MAD)</th>
<th>Realistic Price (MAD)</th>
</tr>
</thead>
<tbody>
<tr>
<td>WCO</td>
<td>200L</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>Methanol</td>
<td>40L</td>
<td>0.16</td>
<td>952</td>
</tr>
<tr>
<td>KOH (Catalyst)</td>
<td>1.74 Kg</td>
<td>34.8</td>
<td>243.6</td>
</tr>
<tr>
<td><strong>Total Cost</strong></td>
<td></td>
<td><strong>34.96</strong></td>
<td><strong>1195.6</strong></td>
</tr>
</tbody>
</table>

VI. Pilot Unit Automation

To automatically control an industrial process is to make it work with the strict minimum of intervention manual to better control the process by the use of automatic devices such as sensors, automatic valves, PLC, etc. The following figure depicts the process of an automated system/design. PLC, which stands for Programmable logic controller is “an industrial solid-state computer that monitors inputs and outputs and makes logic-based decisions for automated processes or machines” [23].

To lower the human force intervention throughout the different stages and phases in our biodiesel production pilot unit, we chose to use a PLC system as it is a simple, straightforward one
and can be directly linked to various setups. After building our pilot unit prototype, sensors detecting conductivity, liquid level, and heat would be purchased and easily integrated in our pilot unit plant. Throughout these sensors, PLC system would detect signals of inputs to reach the desired output. On the other hand, the outputs in our case are resumed in pumps, resistors, agitators, and valves. The following figure depicts how a PLC system unit communicates.

![PLC System Diagram](image)

*Figure 26: Automation systems using PLC [72]*

1. I/O Table

Before proceeding into running the software AUTOMGEN, we have first to identify the Input/Output units.
Table 6: I/O Table of the biodiesel pilot unit production

<table>
<thead>
<tr>
<th>INPUT</th>
<th>TYPE</th>
<th>OUTPUT</th>
<th>TYPE</th>
</tr>
</thead>
<tbody>
<tr>
<td>n₁: 150L of WCO.</td>
<td>Binary</td>
<td>R₆: activate Resistance 6</td>
<td>Digital</td>
</tr>
<tr>
<td>n₂: level of methoxide mixture (36.637L + 1.34Kg)</td>
<td>Binary</td>
<td>R₁₉: activate Resistance 19</td>
<td>Digital</td>
</tr>
<tr>
<td>n₃: level of added water (300L)</td>
<td>Binary</td>
<td>Ag₅: activate agitator 5 in tank 7</td>
<td>Digital</td>
</tr>
<tr>
<td>T₁: 50°C to heat WCO.</td>
<td>Analog</td>
<td>Ag₁₈: activate agitator 18 in the reaction tank 20</td>
<td>Digital</td>
</tr>
<tr>
<td>T₂: 55°C to heat the reaction tank.</td>
<td>Analog</td>
<td>v₉: open valve 9</td>
<td>Binary</td>
</tr>
<tr>
<td>T₃: 70°C to heat the reaction tank.</td>
<td>Analog</td>
<td>v₁₀: open valve 10</td>
<td>Binary</td>
</tr>
<tr>
<td>T₄: 40°C to heat the reaction tank.</td>
<td>Analog</td>
<td>v₁₃: open valve 13</td>
<td>Binary</td>
</tr>
<tr>
<td>C₁: 1050 µmhos conductivity of glycerol</td>
<td>Binary</td>
<td>v₁₅: open valve 15</td>
<td>Binary</td>
</tr>
<tr>
<td>C₂: conductivity different than the one of biodiesel (c = 0)</td>
<td>Binary</td>
<td>v₁₆: open valve 16</td>
<td>Binary</td>
</tr>
<tr>
<td>t₁: 1h to preheat the WCO</td>
<td>Binary</td>
<td>v₂₁: open valve 21</td>
<td>Binary</td>
</tr>
<tr>
<td>t₂: 30min to mix the methoxide solution</td>
<td>Binary</td>
<td>v₂₅: open valve 25</td>
<td>Binary</td>
</tr>
<tr>
<td>t₃: 2h for the transesterification reaction</td>
<td>Binary</td>
<td>v₂₆: open valve 26</td>
<td>Binary</td>
</tr>
<tr>
<td>t₄: 1h for methanol recovery</td>
<td>Binary</td>
<td>v₂₇: open valve 27</td>
<td>Binary</td>
</tr>
<tr>
<td>t₅: 24h to settle the mixture (biodiesel + glycerol)</td>
<td>Binary</td>
<td>P₁₄: Pump 14 on</td>
<td>Binary</td>
</tr>
<tr>
<td>t₆: 1h to mix water with biodiesel</td>
<td>Binary</td>
<td>P₁₂: Pump 12 on</td>
<td>Binary</td>
</tr>
<tr>
<td>t₇: 24h to settle the mix of water and biodiesel</td>
<td>Binary</td>
<td></td>
<td></td>
</tr>
<tr>
<td>t₈: 1h to mix the water again with biodiesel.</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

2. GRAFCET

This section will deal with the function block diagram language used to automate our pilot unit system. For that, we need an elaborated code that takes into consideration the different stations and
all the different valves. Thus, we chose to opt for GRAFCET as a language to represent operations and actions in our automated system.

To start with, GRAFCET, which basically stands for Gra\(p\)he Fonctionnel de Commande des \(\text{\'{E}}\)tapes et Transitions in French, “is a mode of representation and analysis of an automatism, particularly well adapted to systems with sequential evolution, that is to say can be broken down into steps. It is derived from the mathematical model of Petri nets. The GRAFCET is thus a graphic language representing the operation of an automatism by a set:

- Steps with which actions are associated.
- Transitions between steps that are associated with transition conditions (receptivity).
- Oriented links between steps and transitions.” [73]

![Figure 27: General structure of a GRAFCET](image)

Appendix B represents the GRAFCET of our system.
VII. Conclusion and Future Work

Biodiesel has indeed a promising future in the Moroccan market and fits properly its ambitious renewable energy goals. The pilot design has been successfully made as well as the attempted automation of its flow. The feasibility study of our project have made us strongly believe in its success. Future work would mainly consist of designing a glycerol valorization plant to produce combustible pellets that can substitute coal. Moreover, rather than water washing, other biodiesel purification methods could be implemented in the future. In addition, an acid esterification prior to transesterification can be integrated in our design as well, in case of using WCO with a higher FFAs content (>2%).
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Appendix A: PFD

- Preparing the reactants:

- Pumping the reactants to the reaction tank:

- Transesterification reaction:
• Methanol Recovery:

• Separation Phase:
- Separation: Draining:

- Crude Biodiesel:
• Purification: Adding Water

• Separation: Draining Water
**Pure Biodiesel:**

[Diagram of biodiesel production process with labeled components and flow paths]
Appendix B: Automgen Simulation

- GRAFCET