ÇUKUROVA UNIVERSITY
INSTITUTE OF NATURAL AND APPLIED SCIENCES

MSc THESIS

Nursima BÜYÜK

ANALYSIS OF PRODUCTION PARAMETERS OF BIODIESELS DERIVED FROM CANOLA, PALM AND COTTON OILS

DEPARTMENT OF AUTOMOTIVE ENGINEERING

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We certify that the thesis titled above was reviewed and approved for the award of degree of the Master of Science by the board of jury on 09/09/2016

……..……..	……..	…….....
Asst. Prof. Dr. Tayfun ÖZGÜR	Assoc. Prof. Dr. Mustafa ÖZCANLI	Asst. Prof. Dr. Erinç ULUDAMAR
SUPERVISOR	MEMBER	MEMBER

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Registration Number:

Prof. Dr. Mustafa GÖK
Director
Institute of Natural and Applied Sciences

ABSTRACT

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ANALYSIS OF PRODUCTION PARAMETERS OF BIODIESELS DERIVED FROM CANOLA, PALM AND COTTON OILS

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: Assoc. Prof. Dr. Mustafa ÖZCANLI
: Asst. Prof. Dr. Erinç ULUDAMAR

Biodiesel is an alternative fuel for diesel engines that is gaining attention in last years. Its primary advantages are that it is one of the renewable fuels currently available and it is also non toxic and biodegradable. It can be also used directly in most diesel engines without requiring extensive engine modifications. Due to increase, environmental pollution and suitability of fossil fuels have lead to concerns in the looking alternative fuels for diesel In this regard to replace diesel fuel with oxygenated fuel such as biodiesel. Mainly biodiesel refers to a vegetable oil or animal fat based diesel fuel consisting of long chain alkyl (methyl, ethyl or propyl) esters. Biodiesel typically made by chemically reacting lipids (eg. Vegetable oil,soybean oil, animal fat (tallow)) with an alcohol producing fatty acid esters.And various types of catalysts that have been used to effect the transesterification of vegetable, waste and animal oils in biodiesel production. Aims of this thesis, is evaluation of biodiesel production parameters on the effect of biodiesel conversion rate.

Key Words: Biodiesel, Biodiesel production, Transesterification, Catalyst

ÖZ

YÜKSEK LİSANS TEZİ

KANOLA, PALM VE PAMUK YAĞLARINDAN ÜRETİLEN BİYODİZELLERİN ÜRETİM PARAMETRELERİNİN İNCELENMESİ

Nursima BÜYÜK

ÇUKUROVA ÜNİVERSİTESİ
FEN BİLİMLERİ ENSTİTÜSÜ
OTOMOTİV MÜHENDİSLİĞİ ANABİLİM DALI

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: Doç. Dr. Mustafa ÖZCANLI
: Yrd. Doç.Dr. Erinç ULUDAMAR

Biyodizel, geçtiğimiz yıllarda ilgi çekmeye başlayan, dizel motorlar için alternatif bir yakıttır. En öncelikli avantajlarından biri, mevcut yenilenebilir yakıt kaynaklarından en yaygın ve erişilebilir olması, zehirli olmaması ve geri dönüşümlü olmasıdır. Ayrıca dizel motorlarda, motor içerisinde bir modifikasyon gerektirmeksizin direk olarak kullanılabilir. Fosil yakıtların çevre kirliliği ve doğada mevcudiyetinin azalması, dizel yakıtlara alternatif arayışına olan ilgiyi arttırmıştır. Bu bakımdan, dizel yakıtlar biyodizel gibi oksitlenmiş yakıtlarda değiştirilmeye başlanmıştır. Temel olarak biyodizeller bitkisel, ya da uzun alkil (metil, etil, propil) ester zincirli hayvansal yağlar olarak refere edilir.Biyodizel, temel olarak kimyasal olarak yağlar ile (bitkisel yağlar, soya yağı, hayvansal yağ (iç yağı) ve alkolün tepkimsei ve yağ asit esterleri oluşturması ile meydana gelir. Biyodizel oluşum tepkimesi sırasında tepkime verimini arttırmak amacı ile çeşitli katalizörler kullanılmaktadır. Bu tezin amacı biyodizel üretim parametreleri incelenerek kullanılan katalizörlerin biyodizel dönüşüm oranı üzerine etkilerinin incelenmiştir.

Anahtar Kelimeler: Biyodizel, biyodizel üretimi, transesterifikasyon, katalizör

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Finally, I would like to thank to my most valuable possession; parents and sister. They encouraged me, worried about me, gave me the necessary support throughout my life. I certainly would not have succeeded to finish my thesis without their support.

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LIST OF ABBREVIATIONS AND NOMENCLATURE

SO_2	: sulphur dioxide
NaOH	: sodium hydroxide
KOH	: potassium hydroxide
CH_3OH	: methanol
$C_3H_8O_3$	: glycerol
FFA	: free fatty acid
TLC	: Thin layer chromatography
GC	: Gas chromatography
HPLC	: high performance liquid chromatography
CO	: carbon monoxide
MO	: methyl oleate
PME	: palm methyl ester
BSEC	: brake specific energy consumption
BSFC	: brake specific fuel consumption
RBO	: rice bran oil
HC	: hydrogen clorur
PM	: particular matter
$NaOCH_3$	: Sodium methoxide
$KOCH_3$	: Potassium methoxide
FAME	: fatty acid methyl ester
EGR	: Exhaust gas recirculation
FAAEs	: Fatty Acid Alkyl Esters

1. INTRODUCTION

Biodiesel is an alternative fuel for diesel engines that is gaining attention in last years. Its primary advantages are that it is one of the renewable fuels currently available and it is also non toxic and biodegradable. It can also be used directly in most diesel engines without requiring extensive engine modifications. Gerpeny, 2004). Due to increase environmental pollution and suitability of fossil fuels have lead to concerns in the looking alternative fuels for diesel engines. (Mani, 2009). In this regard, to replace diesel fuel with oxygenated fuel such as biodiesel. (Datta, 2016). Mainly biodiesel refers to a vegetable oil or animal fat based diesel fuel consisting of long chain alkyl (methyl, ethyl or propyl) esters. Biodiesel is typically made by chemically reacting lipids (eg. Vegetable oil, soybean oil, animal fat (tallow)) with an alcohol producing fatty acid esters. (Omidvarborna, 2014)

After evaluating advantages and disadvantages of using biodiesels; the chronological development of biodiesel have to be evaluated.

Some of the advantages of using biodiesel as a replacement for diesel fuel are:

- Renewable fuel, obtained from vegetable oils or animal fats
- Low toxicity, in comparision with diesel fuel
- Degrades more rapidly than diesel fuel, minimizing the environmental cosequences of biofuel spills.
- Lower emission of contaminants; carbon monoxide, particulate matter, polycyclic aromatic hydrocarbons, aldehydes.
- Lower health risk, due to reduced emissions of carcinogenic substances.
- No sulphure dioxide (SO_2) emissions.
- Higher flash point (100°C minimum)

- May be blended with diesel fuel at any proportion; both fuels may be mixed during the fuel supply to vehicles.
- Excellent properties as a lubricant.
- It is the only alternative fuel that can be used in a conventional diesel engine, without modifications.
- Used cooking oils and fat residues from meat processing may be used as raw materials.

There are certain disadvantages of using biodiesels as a replacement for diesel fuel that must be taken into consideration:

- Slightly higher fuel consumption due to lower calorific value of biodiesel.
- Slightly higher nitrous oxide ($NO_{x)}$ emissions than diesel fuel.
- Higher freezing point than diesel fuel. This may be inconvenient in cold climates.
- It is less stable than diesel fuel, and therefore long-term storage (more than six months) of biodiesel is not recommended.
- May degrade plastic and natural rubber gaskets and hoses when used in pure form, in which case replacement with TeflonÒ components is recommended.
- It dissolves the deposits of sediments and other contaminants from diesel fuel in storage tanks prior to filling with biodiesel is recommended.

It must be noted that these disadvantages are significantly reduced when biodiesel is used in blends with diesel fuel. (Romano, 2011)

The most preferred method of of biodiesel production is transesterification chemical process. (Rasimoğlu,2014) (Attia, 2016)

Transesterification of a vegetable oil was conducted as early as 1853 by Patric Duffy, four decades before the first diesel engine became functional. Rudolf Diesel's prime model, a single 10 ft(3.0m) iron cylinder with a flywheel at its base, ran on its own power for the first time in Augsburg, Germany, on 1893 running on nothing but peanut oil. In remembrance of this event,10 August has ben declared "International Biodiesel Day." (Duffy, 1853) Even its often reporterd that Diesel designed his engine to run on peanut oil, but this is not the case.In 1900 there was shown by Otto company a small diesel engine, which, at the request of the French government ran on arachide (earth-nut or peanut) oil, and worked so smoothly. (Rob,1898).The engine was constructed for using mineral oil, and then worked on vegetable oil without any alterations being made. The French Government at the time thought of testing the applicability to power production of the Arachide, or earth-nut, which grows in considerable quantities in their African colonies, and can easily be cultivated there." Diesel himself later conducted related tests and appeared supportive of the idea.[42] In a 1912 speech Diesel said, "the use of vegetable oils for engine fuels may seem insignificant today but such oils may become, in the course of time, as important as petroleum and the coal-tar products of the present time."(The Biodiesel Handbook, ISBN 978-1-893997-79-0) Despite the widespread use of petroleum-derived diesel fuels, interest in vegetable oils as fuels for internal combustion engines was reported in several countries during the 1920s and 1930s and later during World War II. Belgium, France, Italy, the United Kingdom, Portugal, Germany, Brazil, Argentina, Japan and China were reported to have tested and used vegetable oils as diesel fuels during this time. Some operational problems were reported due to the high viscosity of vegetable oils compared to petroleum diesel fuel, which results in poor atomization of the fuel in the fuel spray and often leads to deposits and coking of the injectors, combustion chamber and valves. Attempts to overcome these problems included heating of the vegetable oil, blending it with petroleum-derived diesel fuel or ethanol, pyrolysis and cracking of the oils.

On 31 August 1937, G. Chavanne of the University of Brussels (Belgium) was granted a patent for a "Procedure for the transformation of vegetable oils for their uses as fuels" ("*Procédé de Transformation d'Huiles Végétales en Vue de Leur Utilisation comme Carburants*") Belgian Patent 422,877. This patent described the alcoholysis (often referred to as transesterification) of vegetable oils using ethanol (and mentions methanol) in order to separate the fatty acids from the glycerol by replacing the glycerol with short linear alcohols. This appears to be the first account of the production of what is known as "biodiesel" today. (Knothe, 2007)

Research into the use of transesterified sunflower oil, and refining it to diesel fuel standards, was initiated in South Africa in 1979. By 1983, the process for producing fuel-quality, engine-tested biodiesel was completed and published internationally. (SAE Technical Paper series no. 831356.,1983) An Austrian company, Gaskoks, obtained the technology from the South African Agricultural Engineers; the company erected the first biodiesel pilot plant in November 1987, and the first industrial-scale plant in April 1989 (with a capacity of 30,000 tons of rapeseed per annum).

Throughout the 1990s, plants were opened in many European countries, including the Czech Republic, Germany and Sweden. France launched local production of biodiesel fuel (referred to as *diester*) from rapeseed oil, which is mixed into regular diesel fuel at a level of 5%, and into the diesel fuel used by some captive fleets (e.g. public transportation) at a level of 30%. Renault, Peugeot and other manufacturers have certified truck engines for use with up to that level of partial biodiesel; experiments with 50% biodiesel are underway. During the same period, nations in other parts of the world also saw local production of biodiesel starting up: by 1998, the Austrian Biofuels Institute had identified 21 countries with commercial biodiesel projects. 100% biodiesel is now available at many normal service stations across Europe.

The most cursory look at the literature relating to biodiesel will soon reveal the following relationship for prediction of biodiesel from fats and oils.

100 units of oil + 10 units of methanol → 100 units of biodiesel + 10 units of glycerol This equation is a simplified form of the following transesterification reaction as demonstrated on Figure 1.1.

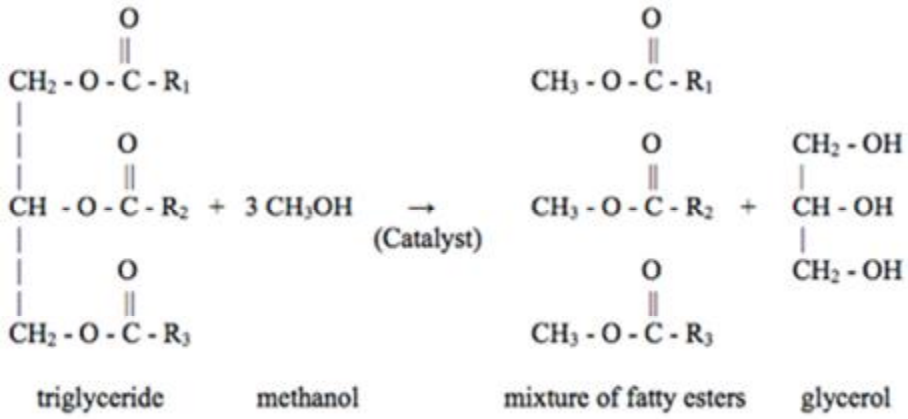

Figure 1.1. Transesterification Reaction

Where R_1, R_2 and R_3 are long chains of carbons and hydrogen atoms, sometimes called fatty acid chains.There are five types of chains that are common in soybean oil and animal fats (others are present in small amounts):

Palmitic : R = - $(CH_2)_{14}$ – CH_3 16 carbons,(including the one that R is attached to)

Stearic : R = - $(CH_2)_{14}$ – CH_3 18 carbons,0 double bonds (18 :0)

Oleic : R = - $(CH_2)_{14}$ – CH_3 18 carbons,1 double bonds (18:1)

Linoleic : R = - $(CH_2)_7$ CH=CH-CH_2-CH=CH$(CH_2)_4CH_3$

18 carbons, 2 double bonds (18:2)

Linolenic : R = - $(CH_2)_7$ CH=CH-CH_2-CH=CH-CH_2-CH=CH-CH_2-CH_3

18 carbons, 3 double bonds (18:3)

These chains are designated by two numbers seperated by a colon.The first number designates the number of carbon atoms in the chain and the second number designated the number of double bonds.The number of carbon atoms includes the carbon that is double bonded to the oxygen atom at the end of fatty acid(called carboxylic carbon).This is the end that the methanol attaches to when methyl esters are produced.Generally it means that and oil such as soybean oil to consist of pure triolein.Triolein is a triglyceride in which all three fatty acid chains are oleic acid.This is near the actual number of carbons and hydrogens and gives a molecular weight that is near the value for soybean oil. If triolein is reeacted with methanol, the reaction will be based one the fact that one molecule of triolein reacts with 3 molecules of methanol to produce 3 molecules of methyl oleate, the biodiesel product and one mole of glycerol.

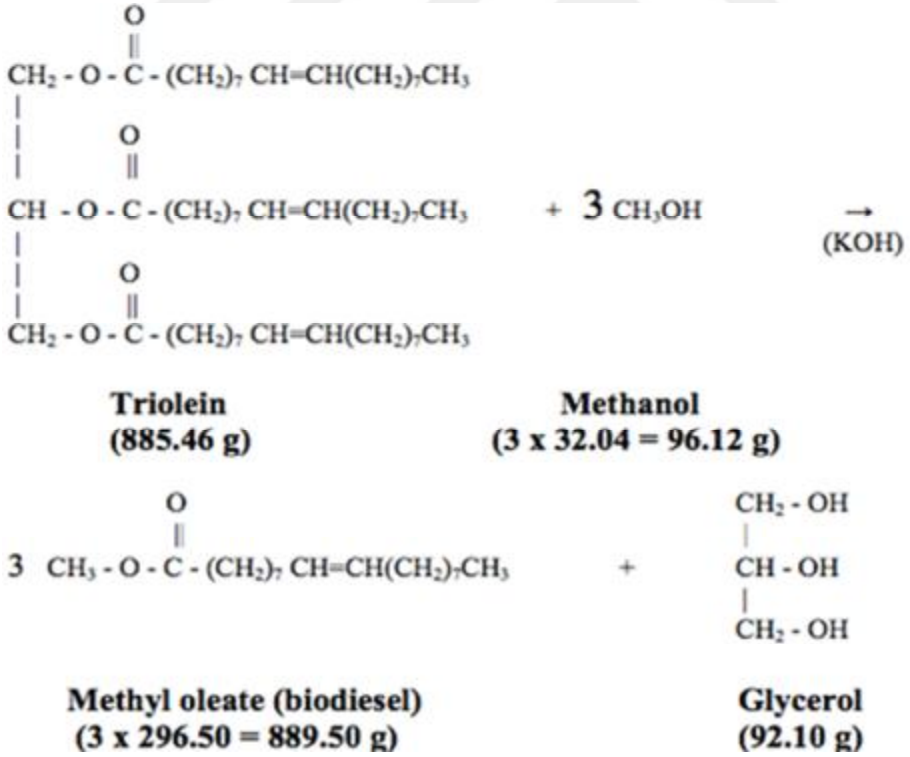

Figure 1.2. Transesterification of triolein

Therefore, the molecular weight of triolein is 885.46 and one mole of triolein weights 885,46 grams. Three moles of methanol weigh 96.12 g, 3 moles of methyl oleate weigh 889.50 g and 1 mole of glycerol weighs 92.10 g as mentioned on Figure 1.2. Although reactions written as conduction, in general, reactions can be encouraged to progress by adding an excess of one of the reactants or by removing one of the products. The reaction of triolein with 100 % excess (XS) methanol mentioned on Figure 1.3.

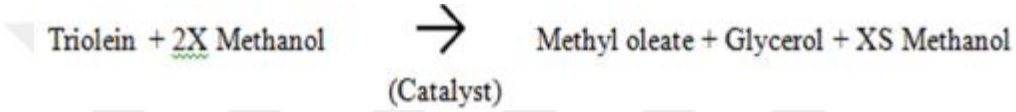

Figure 1.3. Transesterification of Triolein with 100% Excess Methanol

On the basis of 100 units of oil, the reaction mass balance with 100 % XS methanol demonstrated on Figure 1.4.

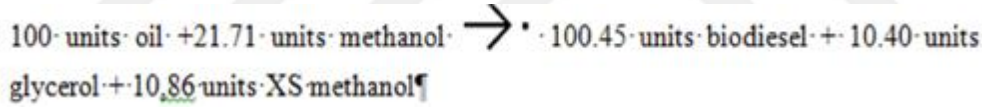

Figure 1.4. Transesterification of Triolein

The reaction also requires about 1% (based on the weight of oil) of sodium hydroxide or a similar catalyst that mostly ends up in the glycerol. These quantities can be converted to volumes by including the density of the reactants and products given in below Table 1.

Table 1.1. Density of Biodiesel Reactants (kg/liter)

The handbook of Chemistry and Physics,1970	
Triolein	0,8988
Methanol	0,7914
Methyl Oleate	0,8739
Glycerol	1,2613

On a volume basis, the reaction becomes:

100 liters of oil+ 24,65 liters' methanol → 103,3 liters' methyl oleate + 7,42 liters glycerol + 12,33 liters XS methanol

As main indicated from the beginning, biodiesel is derived from vegetable oils and the main component of vegetables are the triglicerides. Triglycerides are esters of glycerol with long chain acids, commonly called fatty acids. Besides triglycerides, mono- and diglycerides can also exist. They are formed as intermediates during the transesterification reaction. This is one of the problem when conducting chemical reaction in general, not only the transesterification reaction. It is almost always the goal of chemical reaction to obtain products that are as pure as possible. However, hardly any chemical reaction proceeds to full completion. Therefore, often intermediates can contaminate the final product. Other materials that can contaminate biodiesel are residual methanol, glycerol and catalyst.

The catalyst used for carrying out the transesterification is usually sodium hydroxide (NaOH) or potassium hydroxide (KOH). These compounds belong to a class of materials known as bases and also are inorganic compounds (inorganic compounds are often used in organic chemistry for carrying out or catalyzing reactions.) Other bases are also suitable for the transesterification reaction. The counterparts of bases are known are acids. Many acids can also be used as catalysts in the transesterification reaction. However, the base-catalyzed reaction has advantages such as a higher reaction rate.

In the soap formation, a fatty acid and based reacted to form a new compound, which was called soap and water. Compounds such as soap, in which the hydrogen (proton) of an acid has been replaced with a metal ion, are often called salts. The reason that such compounds exist is that materials such as NaOH (or KOH) can split apart (dissociate) in a fashion that gives Na^+ and OH^- (or K^+ and OH^-) in which the protons and electrons are not evenly distributed, leading to charged particles. Thus, having the same charge, Na^+ or K^+ can replace H^+ here.

Two of the most commonly used catalysts for transesterification are NaOH and KOH.These catalysts operate by reacting with alcohol according to the reaction given below (written using methanol and NaOH but other alcohols and catalysts could be substituted).

$$CH_3OH + NaOH \rightarrow CH_3O\text{-}Na + H_2O$$

Similar to H_2O "consisting" of $H^{'}$ and OH^{-},CH_3O-Na can be seen as consisting of CH_3O^{-} (alkoxide; alkylate) and Na^{+}.CH_3O^{-} is the species that attacks the ester moieties in the glycerol molecule in the following fashion :

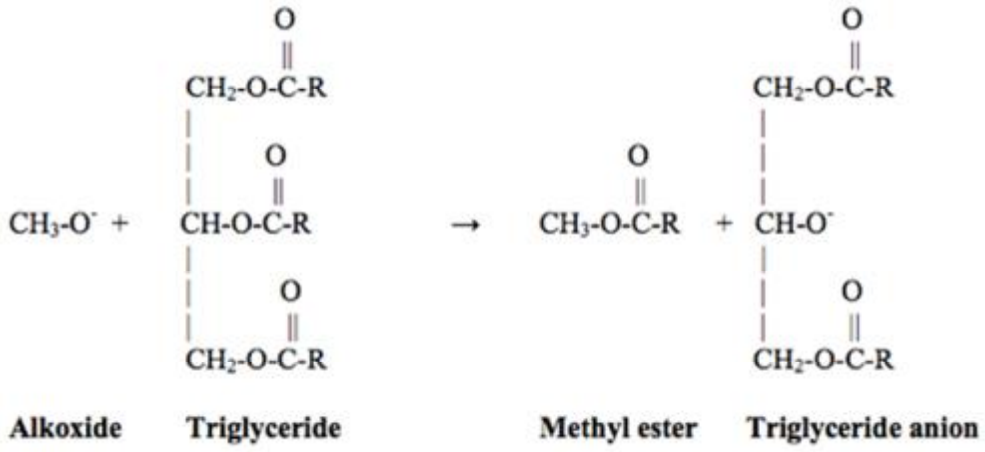

Figure 1.5. Transesterification of ester molecule

As mentioned on Figure 1.5, while ester molecule is complete after this reaction, the anion of the triglyceride needs to pick up a proton to give a stable product (a diglyceride in this case). If this proton is taken from methanol, then the alkoxide catalyst will be recovered.

Several other reactions could be written that would allow the anion of the triglyceride to pick up a proton, such as reaction with free fatty acids and water. The presence of water affects the transesterification negatively because the

triglyceride anion.

While the ester molecule is complete after reaction, anion of the triglyceride needs to pick up a proton to give a stable product (a diglyceride in this case). If this proton taken from methanol, then the alkoxide catalyst will be recovered.

Several other reactions could be written that would allow the anion of the triglyceride to pick up a proton, such as reaction with free fatty acids and water. The presence of water affects the transesterification negatively because the triglyceride anion in below equation will react with the water to form OH^- which can behave in a fashion similar to CH_3-O but a free fatty acid will result instead of a methyl ester.

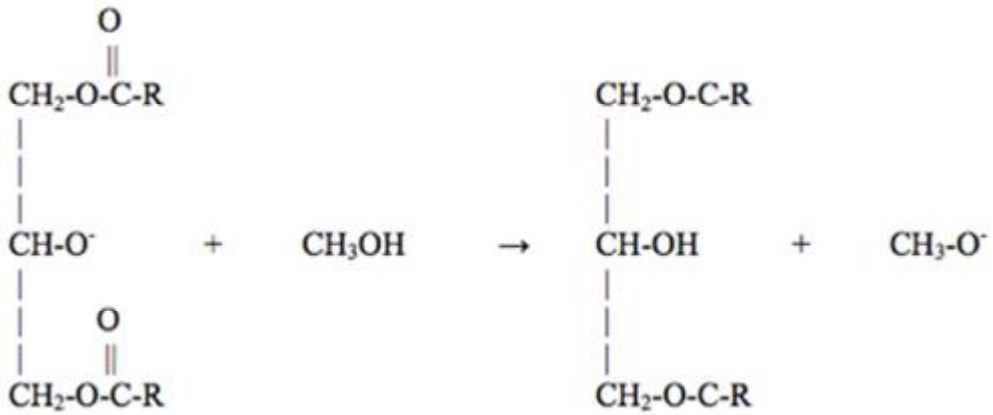

Figure 1.6. Effect of the triglyceride in transesterification

With the water to form OH^-, which can behave in a fashion similar to CH_3-O but a free fatty acid will result instead of a methyl ester as demonstrated in Figure 1.6.

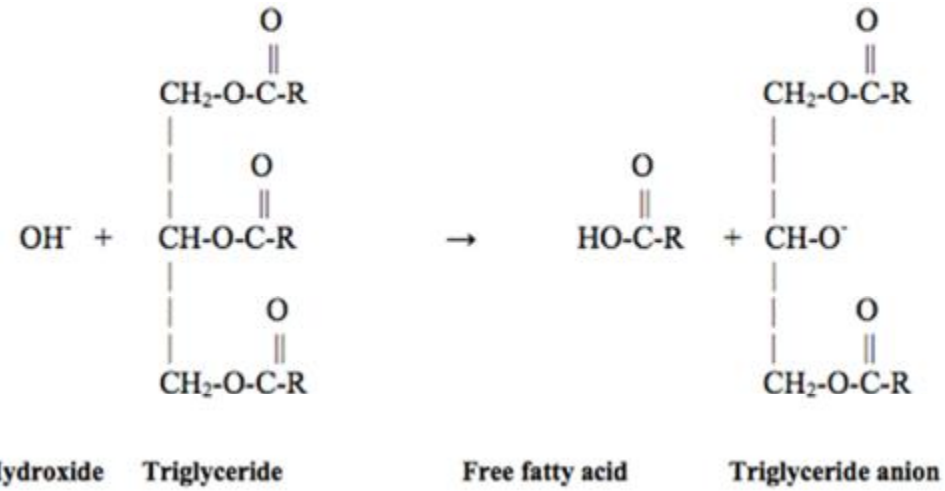

Figure 1.7. Effect of the free fatty acid in transesterification process

As mentioned in Figure 1.7 the free fatty acid can react with the Na^+ to form soap. Some water in the system can be tolerated, because $R\text{-}O^-$ is a stronger base than OH^- so the transesterification reaction occurs at a higher rate than the "saponification" of glycerol leading to free fatty acids. As given in Figure 8. the equilibrium of the following reaction (formation of free fatty acids from the ester) is far on the left side of the reaction equation mentioned on Figure 1.8.

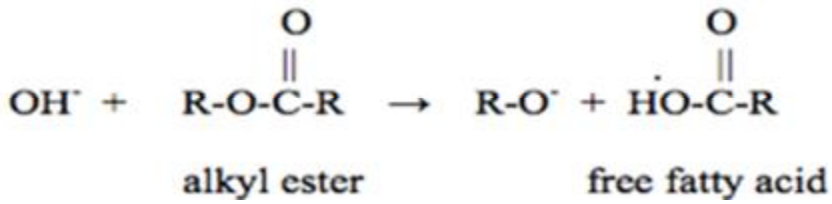

Figure 1.8. Formation of free fatty acids from the ester

As a result of the slow reaction rate, only very minor amounts of free fatty acids are formed during transesterification if the reaction is free of water at the beginning.Another aspect of the weaker basicity of OH^- vs $R\text{-}O^-$ is that the ester moieties in the triglyceride molecules will react less with OH^- than $R\text{-}O^-$.However when too much catalyst (or water) is present becomes more prevalent and causes

enhanced formation of mono- and diglyceride molecules instead of reactions with all positions in the glycerol backbone. Thus the effect of too much catalyst or too much water leads to the same result, namely, enhanced formation of undesirable mono- and diglycerides as well as free fatty acids. Too much catalyst can lead to soap formation when conditions encourage free fatty acid production.

Because of the possibility of the reactions described above leading to free fatty acids and mono and diglycerides, direct use of sodium or potassium alkylate (R-ONa or R-OK; the alkylate moiety must correspond to the R- moiety in the alcohol) as catalysts is becoming of greater interest. (Gerpeny,2004)

There are several methods available for biodiesel productions:

An alternative, catalyst- free method for transesterification uses supercritical methanol at high temperatures and pressures in a continuous process. In the supercritical state, the oil and methanol are in a single phrase, and reaction occurs spontaneously and rapidly (Unkyakiat, 2006). The process can tolerate water in the feedstock, free fatty acids are converted to methyl esters instead of soap, so a wide variety of feedstock can be used. Also the catalyst removal step is eliminated(Unkyakiat, 2006).High temperatures and pressures are required, but energy costs of production are similar or less than catalytic production routes (Kusdiana, 2007).

Ultra and high shear in line or batch reactors allow production of biodiesel continuously, semi continuously and in batch mode. This drastically reduces production time and increases production volume.

The reaction takes place in the high energetic shear zone of the Ultra and High shear mixer by reducing the droplet size of the immiscible liquids such as oil or fats and methanol. Therefore the smaller the droplet size the larger the surface area the faster the catalyst can react.

In the ultrasonic reactor method, the ultrasonic waves cause the reaction mixture to produce and collapse bubbles constantly. This cavitation simultaneously provides the mixing and heating required to carry out the transesterification

process. Thus using and ultrasonic reactor for biodiesel production drastically reduces the reaction time, reaction temperatures, and energy input.Hence the process of transesterification can run inline rather than using the time consuming batch processing.Industrial scale ultrasonic devices allow fort he industrial scale processing of several thousand barrels per day (Hielscher, 2016).

Large amounts of research have focused recently on the use of enzymes as catalyst for the transesterification. Researchers have found that very good yields could be obtained from crude and used oils using lipases. The use of lipases makes the reaction less sensitive to high free fatty acid content, which is a problem with the standard biodiesel process. One problem with the lipase reaction is that methanol can not be used because it inactivates the lipase catalyst after one batch. However, if methyl acetate is used instead of methanol, the lipase is not in activated and can be used for several batches, making the lipase system much more cost effective (Wei, 2004).

Lipids have been drawing considerable attention as a substrate for biodiesel production owing to its sustainability, non toxicity and energy efficient properties. However, due to cost reasons attention must be focused on the non edible sources of lipids, in particular oleaginous microorganisms. Such microbes have the ability to assimilate the carbon sources from a medium and convert the carbon into lipid storage materials. The lipids accumulated by these oleaginous cells can then be transesterified to form biodiesel (Wei, 2004).

The aim of this thesis was to analyse the production parameters of biodiesels derived from canola, palm and cotton oils.

2. PRELIMINARY WORK

Biodiesel productions and its process parameters has been attracting topic for the researcher from past to present. It seem that biodiesel and its process parameters will be continue to be attractive for many years because of lack of Sulphur dioxide emissions, renewability of product, lower emission residues and toxicity and lower healt risky properties and so the sustainability trend of the universe. Therefore, this section represents detailed literature review of biodiesel production properties. Studies were categorized according to chronology.

Patil et al. (2009), stated the non edible vegetable oils such as Jatopha curcas and Pongamia glabra (karanja) and edible oils such as corn and canola were found to be good viable sources for producing biodiesel. Biodiesel production from different edible and non edible vegetable oils was compared in order to optimize the biodiesel production of non edible and edible vegetable oils were investigated.A two step and single step transesterification process was used to produce biodiesel from high free fatty acid (FFA) non edible oils and edible vegetable oils, respectively.This process gives yields of about 90-95 % for J.curcas, 80-85% for P.glabra, 80-95% for canola, and 85-96% for corn using potassium hydroxide (KOH) as a catalyst.The fuel properties of biodiesel produced were compared with ASTM standarts for biodiesel.

Sahoo et al. (2009), stated that petroleum sourced fuels is now widely known as non renewable due to fossil fuel depletion and environmental degradation. Renewable, carbon neutral, transport fuels are necessary for environmental and economic sustainability. Biodiesel derived from oil crops is a potential renewable and carbon netural alternative to petroleum fuels. Chemically, biodiesel is monoalkyl esters of long chain fatty acids derived from renewable feed stock like vegetable oils and animal fats.It is produced by transesterification in which, oil or fat is reacted with monohyric alcohol in presence of a catalyst.The process of transesterification is affected by the mode of reaction condition, molar

ratio of alcohol to oil, type of alcohol, type and amount of catalystrs, reaction time and tempereature and purity of reactants.In the present paper various methods of preparation of biodiesel from non edible filtered Jatropha (Jatropha curcas), Karanja(pongamia pinnata) and Polanga(Calophyllum inophyllum) oil have been described.Mono esters (biodiesel) produced and blended with diesel were evaluated. The technical tools and processes for monitoring the transesterification reactions like TLC, GC and HPLC have also been used.

Tuccar et al. (2014), stated that citrus sinensis biodiesel cause a slight reduc- tion in torque and brake power values. Regarding to experimental design, citrus sinensis biodiesel was blended with conventional diesel fuel with volu- metric ratios of 5%, 10% and 20%. Fuel properties of blends and pure citrus sinensis biodiesel were found out and performance characteristics and exhaust emissions of the engine fueled with blends were analyzed. The engine performance experiments indicated that citrus sinensis biodiesel cause a slight reduc- tion in torque and brake power values. Exhaust emission tests revealed that while CO emission values decrease, NO_X emission values increase with citrus sinensis biodiesel usage. To conclude, citrus sinensis biodiesel can be used as a very promising additive to improve CO emissions of conventional diesel fueled engine.

Altaie et al.(2015), stated that increasing the MO proportion of palm oil methyl ester biodiesel until 50% (vol%, vol%) led to maximum improvements in cloud point and cold filter plugging point, which were reduced to 70.38% and 91.69%, respectively. Regarding to the investigation, the physicochemical properties of specified blends of technical-grade methyl oleate (MO) and PME, namely, PME80/MO20, PME70/MO30, PME60/MO40, and PME50/MO50 (vol/vol%) were studied. The aim was to determine the optimum blend and achieve better cold-flow properties than neat PME. Differential scanning calorimetry analysis showed that increasing the MO proportion until 50% (vol%, vol%) led to maximum improvements in cloud point and cold filter plugging point, which were reduced to 70.38% and 91.69%, respectively. Important fuel properties (i.e., cetane

number (CN), kinematic viscosity, density, gross heating value, net heating value, flash point, oxidation stability, and acid value) were also examined. All fuel properties of PME–MO blends were observed within the specified permissible limits of biodiesel standard (ASTM D 6751).

Attia et al.(2016), stated that the the effect of WCOME blending ratio on viscosity and sooting propensity of the biodiesel–diesel fuel mixture. The engine performance has been expressed in terms of the in-cylinder pressure data as well as the engine mechanical and environmental aspects measured at engine rated speed (1500 rpm) and different engine loads. The main results of the related work showed that, the location and value of the in-cylinder peak pressure depends mainly on the engine load and the biodiesel blending ratio. The best value of Brake Specific Energy Consumption (BSEC) is attained at blended fuel containing 20% WCOME (B20) where the maximum brake thermal efficiency is also observed. While there was a range of blending ratio from B20 to B50 throughout the best engine environmental behavior is attained. Results indicated that, the use of neat biodiesel fuel (B100) at different engine loads leads to an increase of BSEC by about 8%, an increase of engine smoke opacity by about 15%, a decrease of NO_X emissions by about 10% with slight decrease of CO emissions and a decrease of the unburned hydrocarbons by about 15%. The most recommended WCOME biodiesel blending ratios vary from 30% to 50% for better engine performance and emission characteristics. In relating recommended blending ratios, the engine performance provides the following results in comparison with the corresponding values for neat fuel around 10% higher BSFC, insignificant change in g_{bth}, around 3% higher BSEC, and 2% lower T_{Exh}, while the correspond- ing engine emissions include 25% lower CO, 20% lower UHC, 6% lower NO_X, and 20% higher smoke opacity.

Nalgundwar et al (2016), stated that a dual biodiesel blend, mixture of two different kinds of biodiesel namely palm (Elaeis guineensis) and jatropha (Jatropha curcas) in diesel was considered for evaluation in a single cylinder DI diesel engine

with varying loads after going through physical prop- erties analysis. The results for lower blend of biodiesel D90PB5JB5 (i.e. 90% diesel & 10% biodiesel) with diesel showed 4.65% average increase in brake power than diesel. There was slight reduction seen in BSFC for lower blends. Higher biodiesel blend D20JB40PB40 (i.e. 20% diesel & 80% biodiesel) have showed up to 15% average increase in brake thermal efficiency. A notable decrease in exhaust gas temperature for most of biodiesel blends was observed. There were 7.1%, 17.7% and 14.5% average reductions in CO emissions with samples D90JB5PB5, D80JB10PB10 and D70JB15PB15 (biodiesel blends containing 10%, 20% & 30% biodiesel) respectively, when compared to diesel. Lower blends of biodiesel samples D90JB5PB5 and D80JB10PB10 showed 5.3% and 9.2% average increase in NOx emissions respectively, than diesel.

Rashedul et al (2014), stated that the use of additive in biodiesel fuel is inalienable both for improving properties and for better engine performance and emission control. Due to several factors like decreasing the dependence on imported petroleum, reducing global warming, increasing lubricity, and reducing substantially the exhaust emissions from diesel engine. However, there is a major disadvantage in the use of biodiesel as it has lower heating value, higher density and higher vis- cosity, higher fuel consumption and higher NO_X emission, which limits its application. Here fuel additives become essential and indispensable tools not only to minimize these drawbacks but also generate spec- ified products to meet the regional and international standards. Fuel additives can contribute towards fuel economy and emission reduction either directly or indirectly. Their use enable vehicle performance to be maintained at, or near, optimum over the lifetime of the vehicle. A variety of additives are used in automotive biodiesel fuel to meet specification limits and to enhance quality. For example, metal based additives, oxygenated additives, antioxidants, cetane number improvers, lubricity improvers and cold flow improvers are used to meet specifications and quality. This article is a literature review of the effect of various additives on biodiesel properties, engine

performance and exhaust emission characteristics and the corresponding effect factors were surveyed and analyzed in detail. The review concludes that the use of additive in biodiesel fuel is inalienable both for improving properties and for better engine performance and emission control. Therefore, in order to find the appropriate fuel additives in the combustion applications, more experiments are needed to explore the different related mechanisms.

Chuah et al (2015), stated a new route for intensification of methyl ester synthesis in Malaysia via alkali- catalysed transesterification of waste cooking oil derived from palm olein using a hydrodynamic cavitation reactor. The effects of the oil to methanol molar ratio (1:4–1:7), catalyst loading concentration (0.5–1.25wt%) and reaction temperature (50–65–C) have been investigated using an optimised plate with 21 holes of 1 mm diameter and an inlet pressure of 2 bar in a 50 L of hydrodynamic cavitation reactor assisted by a double diaphragm pump. Optimal conversion of 98.1% was achieved in 15min in a hydrodynamic cavitation reactor with 1:6 molar ratio of oil to methanol, 1 wt% of catalyst and 60 $^{\circ}$C of reaction temperature. It has been observed that a significant reduction in the optimum reaction time (about 6 fold) for transesterification from 90 min for mechanical stirring approach to 15 min for the hydrodynamic cavitation approach. Optimal yield efficiency of 12.50 10^{-4} g/J was found using hydrodynamic cavitation and it was 8 fold higher than 1.5 10^{-4} g/J when mechanical stirring was used.

Lin et al (2009), stated the report on the successfully production of biodiesel by transesterification of crude rice bran oil (RBO). The process included three-steps. Firstly, the acid value of RBO was reduced to below 1 mg KOH/g by two-steps pretreatment process in the presence of sulfuric acid catalyst. Secondly, the product prepared from the first process was carried out esterification with an alkaline catalyst. The influence of four variables on conversion efficiency to methyl ester, i.e., methanol/RBO molar ratio, catalyst amount, reaction temperature

and reaction time, was studied at this stage. The content of methyl ester was analyzed by chromatographic analysis. Through orthogonal analysis of parameters in a four-factor and three-level test, the optimum reaction conditions for the transesterification were obtained: methanol/RBO molar ratio 6:1, usage amount of KOH 0.9% w/w, reaction temperature 60 °C and reaction time 60 min. In the third step, methyl ester prepared from the second processing step was refined to become biodiesel. Fuel properties of RBO biodiesel were studied and compared according to ASTM D6751-02 and DIN V51606 standards for biodiesel. Most fuel properties complied with the limits prescribed in the aforementioned standards. The consequent engine test showed a similar power output compared with regular diesel but consumption rate was slightly higher. Emission tests showed a marked decrease in CO, HC and PM, however, with a slight increase in NO_X.

Ramezani et al.(2010), stated the parameters affecting castor oil transesterification reaction were investigated. Applying four basic catalysts including $NaOCH_3$, NaOH, $KOCH_3$ and KOH the best one with maximum biodiesel yield was identified. Using Taguchi method consisting four parameters and three levels, the best experimental conditions were determined. Reaction temperature (25, 65 and 80 $^{\circ}C$), mixing intensity (250, 400 and 600 rpm), alcohol/oil ratio (4:1, 6:1 and 8:1) and catalyst concentration (0.25, 0.35 and 0.5%) were selected as experimental parameters. It was concluded that reaction temperature and mixing intensity can be optimized. Using the optimum results, it proposed a kinetic model which resulted in establishing an equation for the beginning rate of transesterification reaction. Furthermore, applying ASTM D 976 correlation, minimum cetane number of produced biodiesel was determined as 37.1

Dias et al. (2013), stated that the best temperature and reaction time to produce biodiesel from raw castor oil were 65 °C and 8 h, where models predict a product yield of 73.62% (w/w) and a purity of 83.41% (w/w). Regarding to related study, mechanical and chemical oil extraction procedures were evaluated.

Biodiesel was produced by homogenous alkaline trans- esterification and experimental planning was conducted to evaluate the influence of temperature and reaction time in product yield and quality; 20 experiments were performed. A 54.1% (w/w) oil yield was obtained by Soxhlet extraction with methanol after grinding the seeds. Product yield ranged from 43.3 to 74.1% (w/w), biodiesel quality varied and the conditions that lead to the best product were established. Results indicate that, to achieve higher product yields and quality using raw oil, longer reaction times are required compared to what is generally reported for refined oil. Statistically significant predictive models were obtained to estimate product yield and quality as function of the studied reaction variables.

Rasimoglu et al. (2014), stated the effects of parameters of transesterification on the cold flow properties of corn oil based biodiesel such as cloud point, pour point and cold filter plugging point. Reaction parameters examined were the transesterification temperature (in the range of 20e60 –C), reaction time (10e60 min), alcohol-to-oil ratio (3.15:1e12.85:1 in moles), amount of catalyst (0.25 e2 $g_{catalyst}$/100 mL corn oil) and stirring speed (300e800 rpm). As a result, it has been observed that when the transesterification reaction period is kept longer than 10 min, there were no changes in cold flow properties of the biodiesel obtained. In addition, better cold flow properties were monitored when alcohol-to-oil ratio was kept between 3.15:1 and 4.15:1. While no effect of reaction temperature on cold flow properties was observed above 20 –C, amount of basic catalyst used in the experiments gave the lowest cold flow properties at the percent of 0.75. Stirring speed has been ineffective in terms of cold flow properties in the transesterification process.

Kılıc, Uzun and Pütün (2013), stated the optimization of biodiesel production from castor oil using full factorial design was investigated. The biodiesel production was carried out in a batch laboratory scale reactor by alkaline-catalyzed transesterification process. Experimental design was used in the

evaluation of process variables. Effects of temperature, methanol/oil molar ratio and catalyst concentration were optimized according to the 2^3 full factorial central composite design (CCD). For determining the influence of purification methods on biodiesel yield, different purification methods were applied to the product after transesterification reaction. Second- order model was obtained to predict biodiesel yield as a function of these variables. According to the experimental results this process gave an average yield of biodiesel more than 90%.

Pullen and Saeed (2015), stated the effects of catalyst type, number of reaction stages, the free fatty acid (FFA) and water content of the reactants on the progress of rapeseed base-catalyzed transesterification under the optimized reaction conditions used in scale-up production. Firstly, the efficacy of different alkaline-base catalysts: sodium hydroxide (NaOH), potassium hydroxide (KOH), and sodium methoxide (CH_3ONa) were investigated and compared. The order of catalyst efficacy was found to be NaOH N CH_3ONa N KOH at 1% m/m concentration: NaOH was most potent achieving the highest conversion to FAME in the shortest time. The effect of performing a 2 stage base-catalyzed reaction, where glycerol was removed prior to a second reaction stage, was investigated to determine any increase in the overall conversion to FAME relative to the single stage process. The effects of increasing the reactant FFA and water content on completeness of transesterification using 1% m/m NaOH were also studied. End-product FAME content was sig- nificantly reduced at N5% m/m acid content (acid value N 10 mg KOH/g). Above ~7% m/m acid (~14 mg KOH/g), the reaction was stopped due to excessive soap/gel formation. The FAME content was not especially sensitive to reactant water contamination. Only at a water level of N 6000 ppm was the FAME content.

Swaminathan and Sarangan (2012), stated that adding Bio-Fish Oil to the list of biodiesel, experimental studies were made using this bio oil in a single cylinder diesel engine and generator set as substitute for diesel fuel and the results

are discussed. It is also blended with oxygenate and EGR technique also used to improve the performance and reduce the emission of the engine. Encouraging results were obtained from this investigation. The percentage reduction is CO e 91%, CO_2 e 62%, NO_X e 92% and C_XH_Y e 90% were attained when the engine was run at maximum load using BFO with 2% additive with EGR and there was reduction in all the percentages when the engine was run in other loads also. In the case of NO_X, there was an increase of this emission by about 48% in the maximum load with BFO when compared with diesel, for obvious reasons and that was also reduced because of the addition of oxygenates and EGR technique. The optimum values were obtained with 2% of additive. When this percentage is increased or decreased the emission were increased.

Lavanya et al. (2012), stated that high yielding castor genotypes ideal for biodiesel production. The material evaluated included seed of 15 castor genotypes grown in rainfed conditions of Alfisols at Hyderabad, India. Variability for palmitic, stearic, oleic, linoleic fatty acids was recorded. Ricinoleic acid, the predominant mono unsaturated fatty acid varied among castor genotypes from 86.7 to 92.1%. Correlation coeffecients between fatty acid profile and biodiesel traits were computed. Genotypes 48-1 and DCH-200 exhibited high O/L ratio, low Iodine value (IV) and high cetane number (CN) which indicates higher stability, longer shelf life, quick ignition and greater combustion quality. Genotype DPC-9 exhibited potential as a female parent for development of biodiesel suitable hybrid.

Ayetor et al. (2015), conducted a study about the effect of H_2SO_4 on viscosity of methyl esters from Jatropha oil (JCME), palm kernel oil (PKOME) from Elaeis guineensis species, and coconut oil (COME) and effect of methanol to oil molar mass ratio on yield of the three feedstocks. Decreased the Methyl ester yield by esterification process using sulphuric acid anhydrous (H_2SO_4). Jatropha methyl ester experienced a viscosity reduction of 24% (4.1–3.1 mm^2/s) with the addition of 1% sulphuric acid. Regarding to their study, palm kernel oil (PKOME),

coconut oil (COME) and Jatropha oil (JCME) were used as feedstocks for the production of biodiesel to investigate optimum parameters to obtain high yield. For each of the feedstock, the biodiesel yield increased with increase in NaOH concentration. The highest yield was obtained with 1% NaOH concentration for all. The effect of methanol in the range of 4:1–8:1 (molar ratio) was investigated, keeping other process parameters fixed. Optimum ratios of palm kernel oil and coconut oil biodiesels yielded 98% each at methanol: oil molar ratio of 8:1. The physiochemical properties obtained for each methyl showed superior properties compared with those reported in published data.

Brennan and Owende (2010), reviewed the technologies underpinning microalgae-to-biofuels systems, focusing on the biomass production, harvesting, conversion technologies, and the extraction of useful co-products. It also reviewed the synergistic coupling of microalgae propagation with carbon sequestration and wastewater treatment potential for mitigation of environmental impacts associated with energy conversion and utilisation. It was found that, whereas there are outstanding issues related to photosynthetic efficiencies and biomass output, microalgae-derived biofuels could progressively substitute a significant proportion of the fossil fuels required to meet the growing energy demand.

Bankovic-Ilıc et al. (2012) , reviewed various methods for biodiesel production from common non-edible oils employing alcoholysis reactions. The aim of this paper is to present the possibilities of the use of non-edible oils into biodiesel production, to consider the various methods for treatment of non-edible oils and to emphasize the influence of the operating and reaction conditions on the process rate and the ester yield. The special attention is paid to the possibilities of optimization, kinetics and improvement of biodiesel production from non-edible oils.

Yang et al. (2014), stated that use of non-edible oils can be guaranteed as sustainable feedstock for biodiesel since most of the non-edible plants can be grown on wastelands to reclaim them, not compete with food crops for limited

lands, are relatively cheap, available and offer similar or even higher yields of biodiesel and fuel properties as the edibles. Developing biodiesel industry in Africa can help curb the high rate of unemployment through job creation as well as increase in income level of the rural populace. Weaning African economies from oil import dependencies could also be an economic achievement. It can be deduced from the study that there are promising non-edible oil resources in the system for biodiesel industrialization in Africa.

Takase et al. (2015), studied the corelation between the limited reserve of the fossil fuels and among the highlights of this expatiate review include oil as feedstock for biodiesel, the need for non-edible feedstocks, neem, karanja, rubber, jatropha and their value chains, methods of modifying oil to biodiesel, factors affecting biodiesel production, application of the selected non- edible seed biodiesels to engines for performance and emission characteristics and the outlook. The demand for petroleum has risen rapidly due to increasing industrialization and modernization of the World.The limited reserve of the fossil fuels is also dwindling alongside escalation in the prices.The threats from these and food insecurity are, however, drawing the attention of researchers for alternative fuel which can be produced from renewable feedstocks.Biodiesel as the most promising alternate is currently produced from conventionally grown edible plant oils such as rapeseed, soybean, sunflower and palm. The use of the edible oils is worsening the current competition of oil for food and for fuel. Focus on the use of non-edible resources is presently directed to jatropha, mahua, pongamia, calophyllum tobacco, cotton oil, etc. Discrepancies between the expectation and realities regarding these non-edible oils are necessitating efforts for diversification of the feedstocks to resources that could guarantee energy production without affecting food security. Neem, karanja, rubber and jatropha are evergreen multipurpose non-edible plants that are widely available and can be grown in diverse socio-economic and environmental conditions. These plants are described as golden trees that have multiple uses such as for fuels, medicines, dyes, ornamentals, feeds, soil enrichment, afforestation.

Bhuiya et al. (2016), rewieved the several aspects of non-edible oils which are termed as 2nd generation biodiesel, such as the biodiesel's physico-chemical properties, and its effect on engine performances and emissions. In addition, the effect of biodiesel on engine power, fuel economy and emissions including regulated and non-regulated, and the corresponding effect factors are surveyed and analysed in detail. It is found from the review that the biodiesel fuel properties vary depending on the sources of feedstocks which have considerable impact on engine performances and emissions characteristics. The use of biodiesel leads to the substantial reduction in key pollutants namely particulate matter (PM), hydrocarbon (HC), and carbon monoxide (CO) emissions accompanying with the imperceptible power loss, slight increase in fuel consumption and slight increase in NOx emission on conventional diesel engines with no or fewer modification. By the related review introduces a potential guideline on further research for improving engine performance and emission characteristics using different 2nd generation biodiesels and their blends. Regarding to his study provided a comparative baseline to make an easy comparison among the biodiesels in respect of fuel properties, engine performance and emission features.

Datta and Mandal (2016), reviewed the production, performance and emissions from a compression ignition engine using biodiesel as alternate to fossil based diesel fuel. The properties of biodisel produced from different sources and their fatty acid composition have also been described.The experimental set up used by different researchers for the investigations and their findings regarding performance and emissions with respect to mineral diesel have been presented in short for a large number of studies.For better illustration of the facts, results of a few experimental studies available in the literature have been presented in the form of different graphs for selective important performance and emission parameters as case studies.The overall impression is that the performance of the engine slightly deteriorates with the use of biodiesel partially or fully instead of diesel, but the environmental aspects are significantly improved.

Suh and Lee (2016), reviewed the spray atomization characteristics, combustion performance and emissions reduction characteristics of biodiesel-fuleled compression igniticion (CI) engine, including physical fuel properties such as the oxygen content, density, viscosity and cloud point.

Different fuel properties of biodiesel such as higher oxygen content and cetane number induce shorter ignition delay, improved combustion efficiency, and reduction of emissions. However, higher viscosity causes coarse atomization, and a higher cloud point indicates that the fuel injection system needs to be modified.

A slightly shorter injection delay was observed due to the low compressibility of biodiesel, and the production of biodiesel spray is similar to that of diesel spray despite having different fuel properties. The higher surface tension of biodiesel results in a higher SMD distribution, and thus the droplet momentum of biodiesel, which is related to the spray velocity, is greater than that of diesel fuel. A lower maximum combustion pressure and indicated mean effective pressure (IMEP) were observed for biodiesel fuel due to its low heating value. Biodiesel combustion with exhaust gas recirculation (EGR) shows a higher combustion pressure and rate of heat release than those of diesel fuel due to its oxygen content. This oxygen content induces reduced HC, CO, and PM emissions, but there is an increase of NO_X emission. However, a high EGR strategy can reduce the NO_X emission in biodiesel-fueled CI engines (Suh, 2016).

Dai et al. (2015), stated that the catalyst of 4(mol (Li_2CO_3)/mol (Al_2O_3)) being calcined at 900°C showed the optimum activity. For this statement the AlO_2 catalyst was prepared by solid-state reaction and then applied to the biodiesel production by transesterification reaction between methanol and soybean oil. This was the first attempt to use $LiAlO_2$ as a catalyst for biodiesel production. XRD, BET and FE-SEM demonstrated that the Li compound was incorporated into Al_2O_3form $LiAlO_2$ with an enhanced basicity. The maximum conversion achieved 97.5% with 2 h reaction time at 65 °C, 24:1 M ratio of methanol to oil and 8 wt.%

of catalyst. $LiAlO_2$ could be easily recovered and reused for six cycles without significant deactivation.

Bilgin et al. (2015), conduct a study about transesterification reaction parameters to produce the lowest kinematic viscosity waste cooking oil biodiesel by using sodium hydroxide (NaOH) as catalyst and ethanol (C_2H_5OH) as alcohol. For this purpose, the individual effectts of main reaction parameters such as catalyst concentration (0,50-1,75%), reaction temperature (60-90°C), reaction time (60-150 min) and alcohol/oil molar ratio (6:1-15:1) on the kinematic viscositics of produced biodiesels were investigated, respectively. According to results, reaction parameters giving the lowest kinematic viscosity of 4.387 cSt were determined as 1.25% catalyst concentration,70°C reaction temperature, 120 minutes reaction time and 12:1 alcohol/oil molar ratio.

Boz and Sunal (2009), stated that Zinc oxide supported catalysts were prepatated by impregnation method with aqueous solutions of KOH and K_2CO_3 and tested for the transesterification of canola oil with methanol resulting in Fatty acid Methyl ester FAME) at the temperature range between 298-333 K and methanol/oil ratio of 6/1 -15/1.Both catalyst,KOH and K_2CO_3 impregnated into ZnO, were found to be the active catalysts resulting in very high conversion of triglyceride (94% and 96% respectively) for transesterification of canola oil at 333 K with methanol/oil ratio of 12/1.Methanol was found to be much more reactive than ethanol in the transesterification reaction.

Mani and Nagarajan (2009), stated that the influence of injection timing on the performance, emission and combustion characteristics of a single cylinder, four stroke, direct injection diesel engine has been experimentally investigated using waste plastic oil as a fuel. Tests were performed at four injection timings (23°,20°,17° and 14° bTDC). When compared to the standard injection timing of 23° bTDC the retarded injection timing of 14°bTDC resulted in decreased oxides of nitrogen, carbon monoxide and unburned hydrocarbon while the brake thermal

efficiency, carbon dioxide and smoke increased under all the test conditions.

Wen et al. (2006), stated the way of production of biodiesel from agricultural products grown within the Commonwealth and can be used by Virginia farmers. It is the most commonly made from oil extracted from soybeans, one of the top agricultural products in Virginia and there is a lot of interest in biodiesel production across Virginia. In general, biodiesel can be produced from any of the following: pure vegetable oil (soybean, canola, sunflowers); rendered animal fats; or waste cooking oil. The oil is converted into biodiesel through a chemical process called transesterification. Glycerin is removed as a byproduct of the reaction, and the resulting fuel can be blended with petroleum diesel, or used directly as a neat fuel. Biodiesel should be evaluated according to the protocols outlined in the Biodiesel Standard ASTM (American Society for Testing and Materials) D6751 before use. In Virginia Cooperative Extension (VCE) publication 442-880 (the basics of biodiesel fuel are discussed as well as the myths and questions about biodiesel usage. This new publication presents the procedures for producing biodiesel, with particular emphasis on small-scale production.

Hillon et al. (2003), stated that increasing biodiesel consumption requires optimized production processes that are compatible with high production capacities and that feature simplified operations, high yields, and the absence of special chemical requirements and waste streams. The high quality of the glycerol by-product obtained is also a very important economic parameter. A heterogeneous catalyzed continuous process allows all these objectives to be attained.

Ejikeme et al. (2010), states the various types of catalysts that have been used to effect the transesterification of vegetable, waste and animal oils in biodiesel production. They evaluate the conversion of component triglycerides in oils to simple alkyl esters with short chain alcohols like methanol and ethanol amongst others is achieved mainly by transesterification. The transesterification reaction, a reversable process proceeds appreciably by the addition of catalysts, which can be acidic,basic or organic in nature, usually in molar excess of alcohol. The economy

of the process depends on the type and quantity of catalyst used among other factors. The catalyst can be homogeneous or heterogeneous depending on whether it is in the same or different phase with the reactants; oils and alcohols.

Anastopoulos et al. (2009), reviewed that the transesterification reaction of four different vegetable oils (sunflower, rapeseed, olive oil and used frying oil) with ethanol, using sodium hydroxide as catalyst, were studied. The ester preparation involved a two-step transesterification reaction, followed by purification. The effects of the mass ratio of catalyst to oil (0.25 – 1.5%), the molar ratio of ethanol to oil (6:1 – 12:1), and the reaction temperature (35 – 90 °C) were studied for the conversion of sunflower oil to optimize the reaction conditions in both stages. The rest of the vegetable oils were converted to ethyl esters under optimum reaction parameters. The optimal conditions for first stage transesterification were an ethanol/oil molar ratio of 12:1, NaOH amount (1% wt/wt), and 80 °C temperature, whereas the maximum yield of ethyl esters reached 81.4% wt/wt. In the second stage, the yield of ethyl esters was improved by 16% in relation with the one-stage transesterification, which was obtained under the following optimal conditions: catalyst concentration 0.75% and ethanol/oil molar ratio 6:1. The fuel properties of the esters were measured according to EN test methods. Based on the experimental results one can see that the ethyl esters do not differ significantly from methyl esters. Moreover, the results showed that the values of density, viscosity, and higher heating value of ethyl esters were similar to those of automotive and heavy duty engine diesel fuel. However, the CFPP values were higher, which may contribute to potential difficulties in cold starts. On the other hand, the flash points, which were higher than those of diesel fuel constituted a safety guarantee from the point of view of handling and storage.

Ghaly et al. (2010), stated that the most popular microbes used for their lipases have been filamentous fungi and recombinant bacteria. A summary of lipases used in transesterification and their optimum operating conditions is provided. In addition to the choice of lipase employed, factors which make the

transesterification process feasible and ready for commercialization are: enzyme modification, the selection of feedstock and alcohol, use of common solvents, pretreatment of the lipase, alcohol to oil molar ratio, water activity/content and reaction temperature. Optimization of these parameters is necessary in order to reduce the cost of biodiesel production. Use of no/low cost waste materials as feedstocks will have double environmental benefits by reducing the environmental pollution potential of the wastes and producing an environmentally friendly fuel.

3. MATERIAL AND METHOD

The objective of this thesis was to analysis of production parameters of biodiesels derived from canola, palm and cotton oils

Because of its high viscosity and low volatility, the direct use of feedstock in diesel engines can cause problems including: high carbon deposits, scuffing of engine liner, injection nozzle failure, gum formation, lubricating oil thickening and high cloud and pour points. In order to avoid these problems, the feedstock is chemically modified to its derivatives which have properties more similar to conventional diesel fuels. The free fatty acids and triglycerides contained in the oil are reduced to Fatty Acid Alkyl Esters (FAAEs) .The three most recognized methods of biodiesel production are: Pyrolysis, microemulsification and transesterification.

In this thesis biodiesel productions are provided by transesterification methodology from canola oil, cotton oil and palm oil. In order to produce biodiesel from related feedstock oils (canola oil, palm oil and cotton oil) the biodiesel equipments at the University of Cukurova Automotive Engineering Department Automotive Engineering Laboratories was used.

3.1. Biodiesel Production from Feedstocks

In this study, canola oil, palm oil and cotton oil were bought from market. In order to produce biodiesel from feedstocks the biodiesel production equipments at the University of Cukurova Automotive Engineering Department Automotive Engineering Laboratories was used.

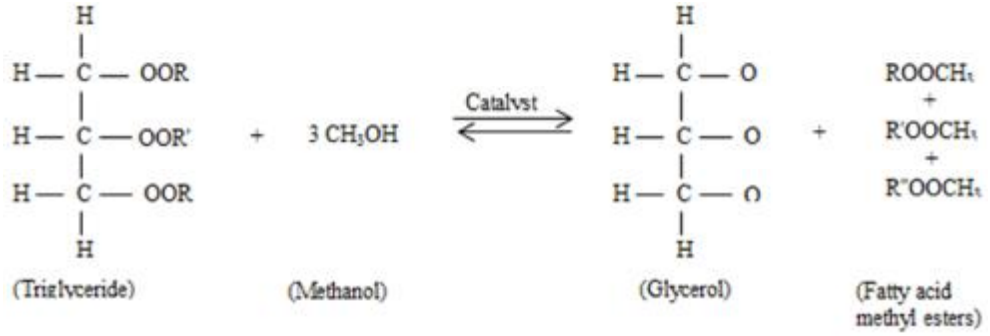

Figure 3.1. General equation for transesterification of triglycerides with methanol

Transesterification reaction (Figure 3.1) was carried out in a spherical glass reactor equipped with reflux condenser, stirrer and thermometer. Sodium hydroxide (NaOH) and potassium hydroxide (KOH) used as catalyst and methanol (CH_3OH) was chosen as alcohol. Methanol was preffered as alcohol because it is the cheapest one. At the transesterification reaction, mol ratio of alcohol and oil was altered from 4:1 to 10:1 During the transesterification process mass ratio of catalyst and oil altered as 0.3, 0.5, 0.7 and 0.9%. Senstive scales used to measure the oil, catalyst and alcohol amounts. In order to obtain sodium methoxide, methanol and NaOH were mixed and KOH and methanol were mixed to obtain potassium methoxide by the same way. Then sodium methoxide (or potassium methoxide) and canola oil were mixed in the small scale reactor. Detailed view of small scale reactor can be seen in Figure 3.2. The mixture was heated up to 60°C and kept at this temperature for one hour; during this period, mixture was stirred continuously at the rate of 800 rpm. Temperature kept at 60 °C constantly during process time.At the end of the reaction period, the crude methyl ester was waited at separating funnel for 8 hours. And then, deep phase containing crude glycerin was separated from crude methyl esters. Then the crude methyl ester was washed three times with warm water. Water and all the other substances precipitated at the bottom.After washing process crude methyl ester was dried at 105°C for one hour. After the drying process, canola/palm/cotton methyl ester let to be cool down. In

order to neglect the possibility of impurities the canola/palm/cotton methyl ester drained from paper filter. This production process was repeated for 2 times and eacth party collection in the same vessel to get a homogenous biodiesel as demonstrated in Figure 3.3

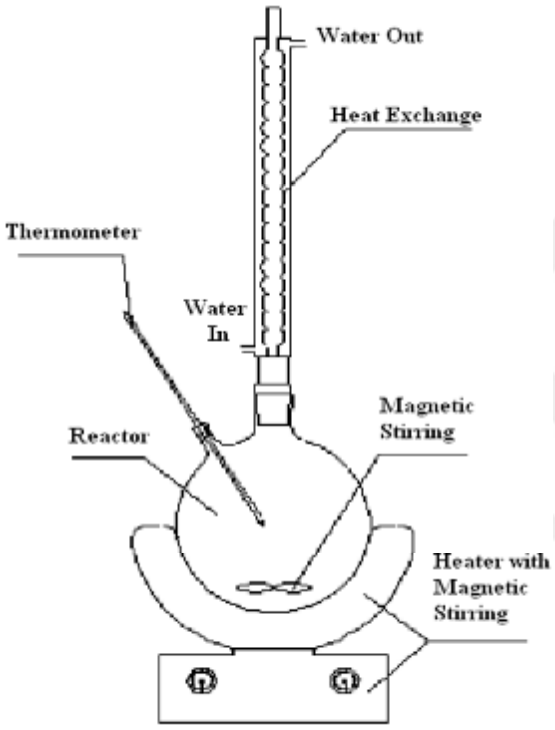

Figure 3.2. Small scale reactor

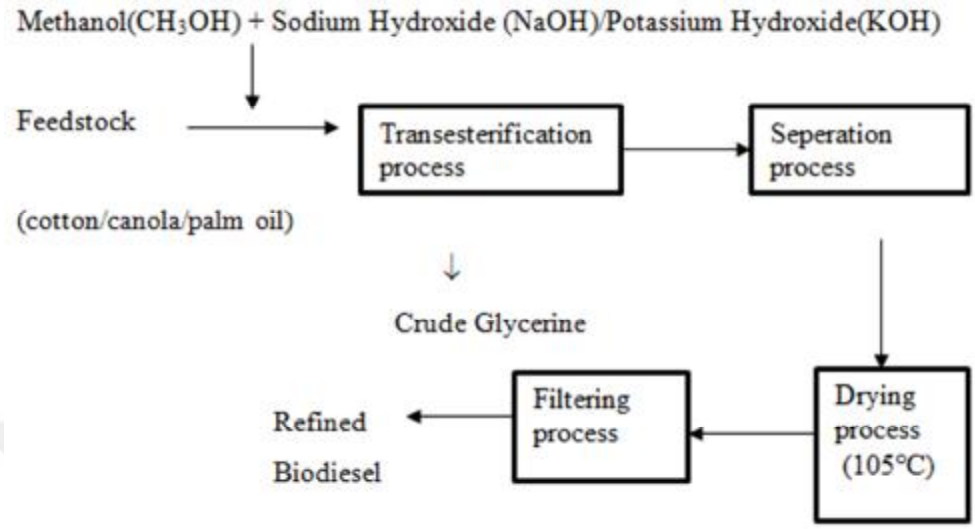

Figure 3.3. Flow diagram of biodiesel production from feedstocks

3.2. Fuel Properties Measurement Methods

The fuel quality measurements of the blends were done accordingly to EN 14214 and EN 590. The testing equipments and their measuring methods were briefly explained below.

Fuel analysis measurements were performed at the Cukurova University Automotive Engineering Laboratories. The fuel properties were measured according to the standard test method. Obtained biodiesel conversion rate is measured via the below equation:

$$\% \text{ conversion ratio} = \frac{produced\ methyl\ ester\ amount\ (gr)}{spent\ oil\ amount\ in\ process\ (gr)} \text{ x } 100 \text{[Altaie et. al, 2015]} \quad (3.1)$$

The measured fuel properties are density, kinematic viscosity, pour point, cloud point and cold filter plugging point. Table 3.1 shows the spesifications of fuel measurements devices.

Table 3.1. The spesifications of Fuel Properties Measurement Device

Property	**Device**	**Accuracy**
Density (g/cm^3)	Kyoto Electronics DA-130	±0.001 g/cm^3
Kinematic viscosity (mm^2/s)	Tanaka AKV-202	±0.01 mm^2/s
Pour Point (ºC)	Tanaka MPC 102L	±1 ºC
Cloud Point (ºC)	Tanaka MPC 102L	±1 ºC
Cold Filter Plugging Point (ºC)	Tanaka AFP-102	±1 ºC

3.2.1. Density Measurement:

The density measurement was done using Kyoto Electronics DA-130 type densimeter (Figure 3.4). This densimeter uses the resonant frequency method to measure the densities. With the densimeter we can also measure the specific weight, API gravity, % Brix, volume and mass alcohol rates. The measurement interval of the device is 0 to 2 g/cm^3 and 0 to 40 ºC. The device has a sensitivity of ±0.001 g/cm^3, and a stability of 0.0001 g/cm^3. The device is measuring according to the standards of TS 6311, ASTM D 4052-96.

Figure 3.4. Kyoto Electronics DA-130 portable densimeter

3.2.2. Viscosity Measurement:

To define kinematic viscosity, it is useful to begin with the definition of viscosity. Simply stated, viscosity, which is also called dynamic viscosity (η), is the ease with which a fluid will flow. Technically it is the ratio of the shear stress to the shear rate for a fluid. In contrast, the kinematic viscosity (ν) is the resistance to flow of a fluid under gravity (GERPEN, 2004).

The viscosities of the fuels were measured with Saybolt Universal Viscosimeter (Figure 3.5) produced from Ubbelohde tube with ASTM D88 standards. The meaurement results were recorded in seconds. Then using a conversion table, the results were converted from SSU (saybolt universal second) to centistrokes (cSt) unit. The measurements were done at 40°C according to the TS EN 14214 standard.

Figure 3.5. Saybolt Universal Viscosimeter

3.2.3. Cetane Number Measurement:

Perhaps the most important measure of ignition characteristic of diesel and/or biodiesel fuels in the cetane number, since it directly pertains to ignition

within compression ignition engines. The cetane number is the primary specification measurement used to match fuels and engines. It is commonly used by refiners, marketers and engine manufacturers to describe diesel fuels. (GERPEN, 2004)

The cetane number and indexes were measured by Zeltex ZX440 (Figure 3.6) type device, which works under the close infrared spectrometer (NIR) principal. With the help of this principal the Cetane number measurement experiment became very fast and cheap with only 3% error compared to the time consuming expensive motor tests.

Figure 3.6. Zeltex ZX440

3.2.4. Cold Flow Measurement

A fuel property that is particularly important for the low temperature operability of diesel fuel is the cloud point. The cloud point is the temperature at which a cloud of wax crystals first appears in a liquid upon cooling. Therefore, it is an index of the lowest temperature of the fuel's utility under certain applications. Operating at temperatures below the cloud point for a diesel fuel can result in fuel

filter clogging due to the wax crystals. As described in ASTM D 2500, the cloud point is determined by visually inspecting for a haze in the normally clear fuel, while the fuel is cooler under carefully controlled conditions.

A second measure of the low temperature performance of diesel/biodiesel fuels is the pour point. The pour point is the lowest temperature at which a fuel sample will flow. Therefore, the pour point provides an index of the lowest temperature of the fuel's utility for certain applications. The pour point also has implications for the handling of fuels during cold temperatures. The standard procedure for measuring the pour point of fuels is ASTM D 97.

Tanaka MPC-102 (Figure 3.7) was used to determine pour point of fuels. Pour Point measurement is made utilizing a new ASTM test method, namely "Air Pressure Method" (ASTM D6749 on "Standard Test Method for Pour Point of Petroleum Products (Automatic Air Pressure Method 1)), which yields eventually no bias against the conventional test method, repeatability/reproducibility of $1^{o}C$ /$2^{o}C$ and 2-3 times faster determinations. The device can measure according to the ASTM D6749/D97, ISO 3016 (PP), ASTM D2500, ISO 3015 (CP). Measuring range is +51 ^{o}C to – 40 ^{o}C with tap water of 20 ^{o}C and +51 ^{o}C to – 65 ^{o}C with cooling liquid of -35 ^{o}C. Amount of applied air pressure for pour point detection, to accommodate different sample types: L (low) for diesel fuels, H (high) for lube oils, VH (very high) and UH (ultra high) for residual fuels and similar samples. Testing intervals: 1.0 ^{o}C, 2.5 ^{o}C, 3.0 ^{o}C.

Figure 3.7. Tanaka MPC-102L

3.2.5. Cold Filter Plugging Point Device

The most important practical disadvantage of biodiesel is that it has a higher pour cold filter plugging point than mineral diesel oil. The cold filter plugging point measures the temperature at which paraffin or, in the case of biodiesel, ester structures, crystallize before injection, thereby blocking the filter. The ensuing lack of fuel can result in engine failure, mainly in cool to cold temperatures. The raw materials used in the manufacture of biodiesel influence the cold filter plugging point. In this study Tanaka AFP-102 cold filter plugging point device was used. The model AFP-102 automatically executes the Cold Filter Plugging Point (CFPP) test method.

Figure 3.8. Tanaka AFP-102

4. RESULTS AND DISCUSSION

All kinds of plant oils, animal fats, greases and waste materials such as animal rendering, fish processing and cooking oil wastes could be espoused as the feedstocks for biodiesel production through transesterification. In this thesis palm oil, cotton oil and canola oil are studied. Chemical transesterification using alkalys and acids has tranditionally been used. However, transesterification in presence of alkaline catalysts has been the method of choice for biodiesel production and is 100% commercialized. The multi-step purification of end products, separation of glycerol, waste water treatment and the intensive energy use of the chemical transesterification have given chance to the less energy intensive, robus and highly active enzymatic transesterification**.** (Ghaly et al, 2010)

4.1. Effects of Catalys Concentration for Canola Oil

In this thesis, canola biodiesel is produced via the transesterification method with different amounts of catalyst and alcohol/molar ratio by using sodium hydroxide (NaOH) and potassium hydroxide (KOH) as catalyst. The converison ratio, viscosity, density and cold flow properties of produced biodiesel samples were measured and compared.

In the first reaction section, alcohol/oil ratio is fixed as 6:1 and methanol/oil ratio was altered as 0.3, 0.50, 0.7 and 0.9%. During the reaction temperature kept as constant at 60 °C and reaction time is kept as 60 min. as well

Table 4.1. Effect of Catalyst Ratio on Conversion Rate %

NaOH (6:1) amount	0.3 %catalyst (w/w%)	0.5% catalyst (w/w%)	0.7 catalyst (w/w%)	0.9 % catalyst (w/w%)	EN 590	Diesel
Conversion rate (%)	80	89	90	86	-	-
Glyserin Amount (g)	58	72.7	80	92		
Density(g/cm^3)	0.888	0.886	0.883	0.884	820-845	833
Viscosity (cSt)	4.93	4.60	4.32	4.44	2.0-4.5	2.85
Pour Point (°C)	-3	-6	-10	-9		-10
Cloud Point (°C)	+2	+1	-2	-1	-10	
Cold Filter Plugging Point (°C)	-5	-7	-14	-10	-20	

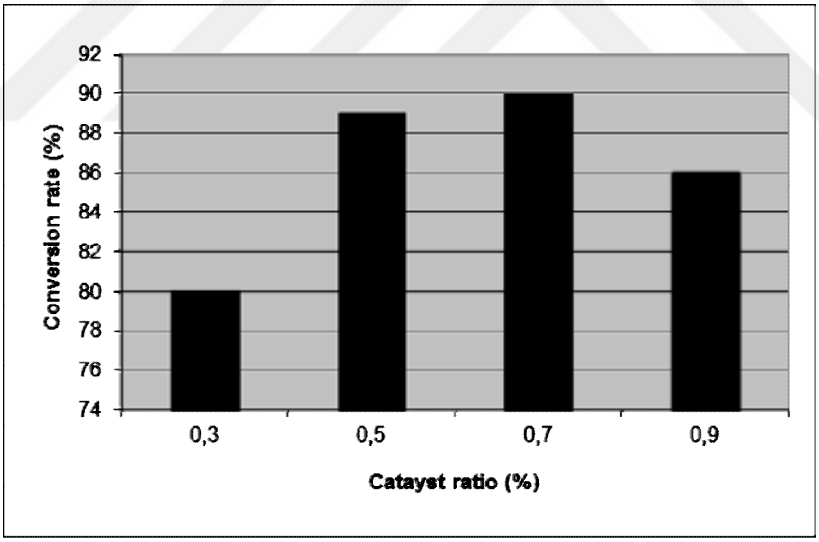

Figure 4.1. Effect of catalyst ratio on conversion rate

As demonstrated on Figure 4.1 and Table 4.1 in the reaction of transesterification the maximum conversion rate is obtained at 0.7% catalyst concentration. At constant temperature (60°C), constant reaction time (60 min)

and constant methanol ratio (6:1) ; maximum conversion rate (%90) obtained at 0,7% catalyst ratio.After 0,7% catalyst ratio, conversion rate started to decrease again, due to saponification seen at excess catalyst amounts.

Table 4.2. Effect of Alcohol ratio on Conversion Rate %

NaOH (%0.7) amount	4:1 Alcohol	6:1 Alcohol	8:1 Alcohol	10:1 Alcohol
Conversion Rate (%)	85	90	88	84
Gyliserin Amount (g)	90	80	90	97
Density (g/cm^3)	0.884	0.883	0.887	0.889
Viscosity (cSt)	4.41	4.32	4.50	4.95
Pour Point (^0C)	-8	-10	-7	-5
Cloud Point (^0C)	-2	-2	+1	+2
Cold Filter Plugging Point (^0C)	-10	-14	-8	-5

After the previous transesterification period. NaOH catalyst amount kept as constant at 0,7% as well as temperature (60°C) and reaction time (60 min); and alcohol amount was altered as 4:1 ,8:1 and 10:1

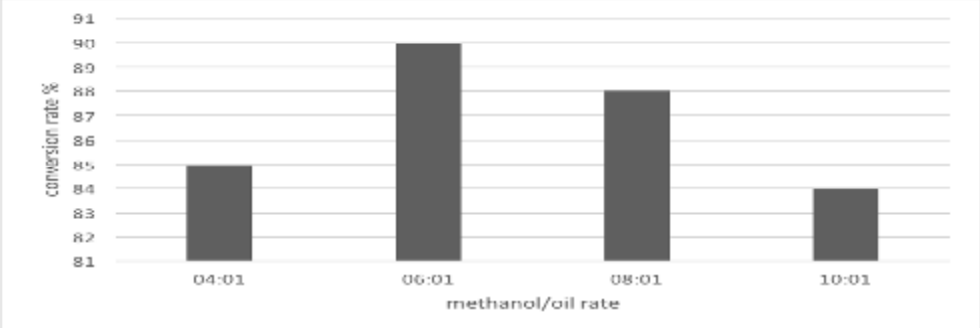

Figure 4.2. Effect of methanol:oil rate on conversion rate

As demonstrated on Figure 4.2 and Table 4.2, in the reaction of transesterification the maximum conversion rate is obtained at 4:1 alcohol rate. At constant temperature (60°C), constant reaction time (60 min) and constant catalyst ratio at 0.7% ; maximum conversion rate (%90) obtained at 6:1 alcohol rate.

Regarding to previous transesterification processes; maximum conversion rate parameters are defined for NaOH catalyst. After these processes, catalyst is altered as KOH and all processes are applied for alternative catalyst as well.

Table 4.3. Effect of Catalyst ratio on Conversion rate

KOH (6:1) amount	% 0.3 catalyst	% 0.5 catalyst	% 0.7 catalyst	% 0.9 catalyst	EN 590	Diesel
Conversion Rate (%)	74.3	78.3	79.1	80.5	-	-
Gyliserin Amount (g)	40	47	54	42		
Density (g/cm^3)	0.894	0.892	0.890	0.887	820-845	833
Viscosity (cSt)	5.6	5.3	4.9	4.7	2.0-4.5	2,85
Pour Point (^0C)	-6	-7	-5	-6		-10
Cloud Point (^0C)	+6	+4	+2	-3	-10	
Cold Filter Plugging Point (^0C)	-5	-11	-6	-20	-20	

In the first reaction section, KOH amount fixed as 6:1 and catalyst /oil ratio was altered as 0.3, 0.5, 0.7 and 0.9 % .During the reaction temperature kept as constant at 60 °C and reaction time is kept as 60 min. as well.

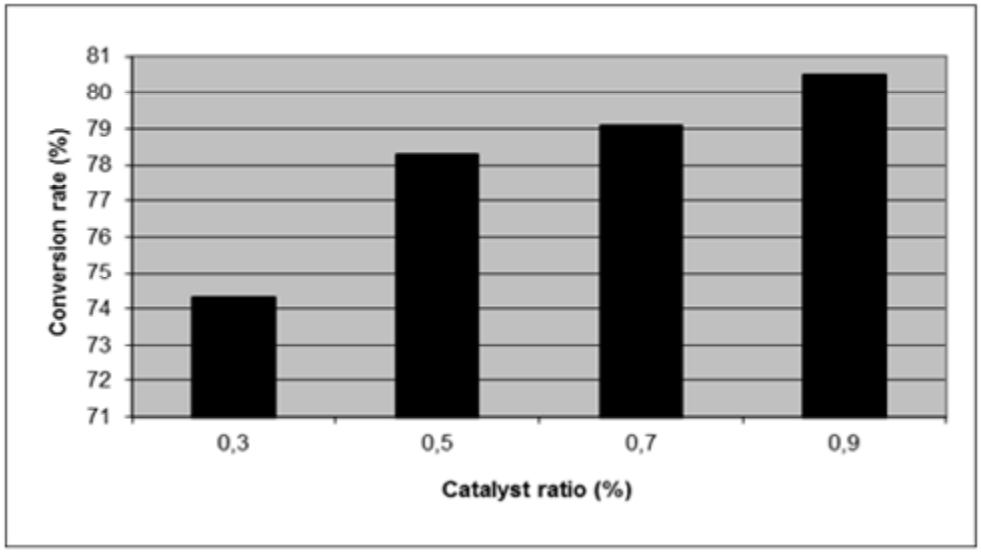

Figure 4.3. Effect of catalyst/oil ratio on conversion rate

As demonstrated on Figure 4.3 and Table 4.3 in the reaction of transesterification the maximum conversion rate is obtained at 0.9% catalyst concentration. At constant temperature (60°C), constant reaction time (60 min) and constant potassium hydroxide (KOH) ratio (6:1) maximum conversion (%) 80.5 obtained at 0.9% catalyst ratio.

Table 4.4. Effect of Alcohol rate on Conversion Rate

KOH (%0.9) amount	4:1 Alcohol	6:1 Alcohol	8:1 Alcohol	10:1 Alcohol
Conversion Rate (%)	77	80.5	78	65
Gyliserin Amount (g)	75	42	70	90
Density (g/cm^3)	0.889	0.887	0.890	0.895
Viscosity (cSt)	4.9	4.7	5.1	5.5
Pour Point (^0C)	-5	-6	-5	-5
Cloud Point (^0C)	-2	-3	+1	+2
Cold Filter Plugging Point (^0C)	-13	-20	-12	-10

After the previous transesterification process, KOH catalyst amount kept as constant at 0.9 % as well as temperature (60°C) and reaction time (60 min); and alcohol arate was altered as 4:1 ,8:1 and 10:1

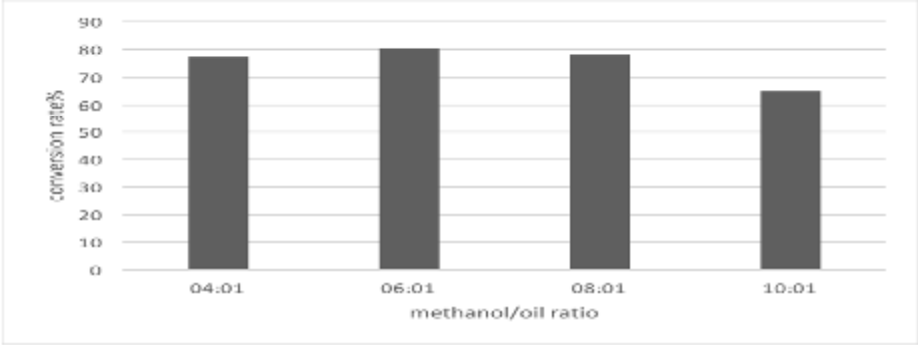

Figure 4.4. Effect of alcohol rate on conversion rate

As demonstrated on Figure 4.4 and Table 4.4 during the reaction of transesterification the maximum conversion rate is obtained at 4:1 alcohol rate. At constant temperature (60°C), constant reaction time (60 min) and constant catalyst ratio 0.9% ; maximum conversion rate (%80,5) obtained at 6:1 alcohol rate.

After evaluation of the each catalyst reaction parameters for maximum conversion rate; both catalysts are compared among the altered 0.3, 0.50, 0.7 and 0.9 % catalyst/oil ratio; at constant alcohol /oil ratio as 6:1, constant reaction temperature as 60 °C, constant reaction temperature as 60 minutes. On comparision of catalysts (KOH and NaOH), regarding to the evaluation results, as given on the below diagram for NaOH catalyst maximum conversion rate obtained as %93 and %91 for KOH catalyst. Its reported that when catalyst amount increased from %0.3 to 0.7%, biodiesel conversion rate is increased as well. And this is a strong evidence that presence of the catalyst in the reaction, increasing the reaction rate. But at the mean time, after optimum catalyst addition (as 0,7% catalyst ratio for NaOH and 0,9% catalyst ratio for KOH), saponification begins,

and saponification increases the viscosity and the increasing the viscosity, the decreases the conversion rate.

Comparision of viscosity amounts of KOH and NaOH catalyst added processes; its reported that minimum viscosity amounts obtained at 0.9% amount of KOH and 0,7% NaOH additions. When preliminary studies were examined, it was observed that the increase the biodiesel yield, the decrease the viscosity. After hydrolysis of the optimum amount of catalyst to occur leads to increase the viscosity because it reduces the biodiesel yield ; due to saponification of hydrolised triglycerides in feedstock oils.

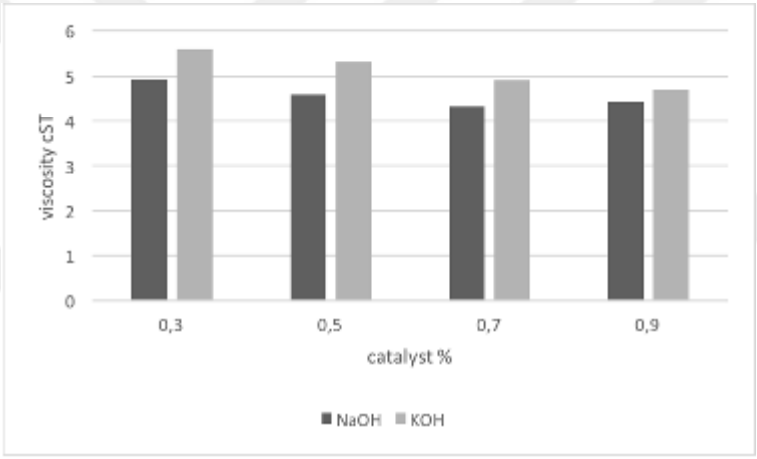

Figure 4.5. Effect of the Kinematic Viscosity (cSt) on Catalyst (%)

As demonstrated on Figure 4.5, even the lower concentrations, the lower catalyst rate was determined; the increase the viscosity, the higher the rate of catalyst. The lowest amount of viscosity is obtained at 0.9% KOH catalyst usage rate of 4.7 cSt and 0.7% NaOH catalyst usage rate of 4.32 cSt .

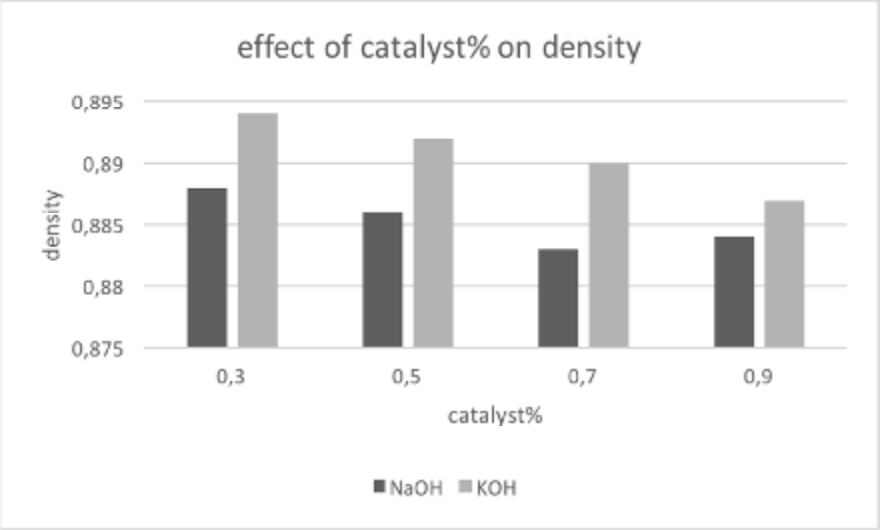

Figure 4.6. Effect of catalyst rate on density

As demonstrated on Figure 4.6 even the lower density results are obtained on the lower catalyst rates; the increasing the catalyst rate, increases the density. The lowest results obtained during usage of KOH as catalyst as 0.9% rate and density of 0,887 g/cm3, during the usage of NaOH as catalyst as %0,7 rate and density of 0,883 (g/cm^3).

After evaluation the catalyst amount on density property, effect of catalyst amounts on cold flow properties are studied. Before exhibit the cold flow property evaluations, related properties are defined. The key flow properties of winter fuel can be specified by the following temperature measures: cloud point (CP), pour point (PP), and cold filter plugging point (CFPP). The lowest temperature at which crystals start to become visible (diameter exceeds 0,5 ㎜), forming a hazy or cloudy suspension, is defined as the CP. The lowest temperature at which fuel lose its defined as the CP. The lowest temperature at which fuel lose its fluidity and becomes semi-solid(crystal agglomeration is sufficiently extensive to prevent free pour) is defined as PP. CFPP can be described as the lowest temperature, where 20

Ml of fuel still flows through a 45 mm wire mesh filter under 0.02 atm vacuum within 60 s. (Mohamad et al. 2015).

Figure 4.7, 4.8, 4.9 show the impacts of catalyst ration on cold flow properties.

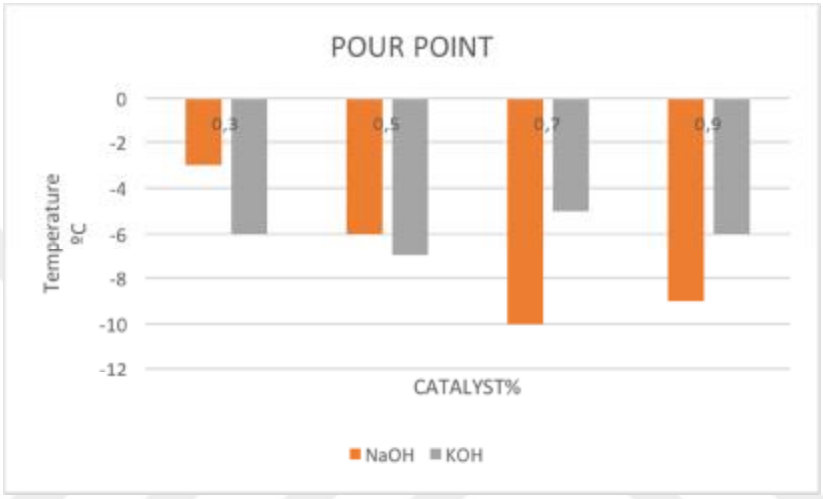

Figure 4.7. Effect of catalyst amount on Pour point °C

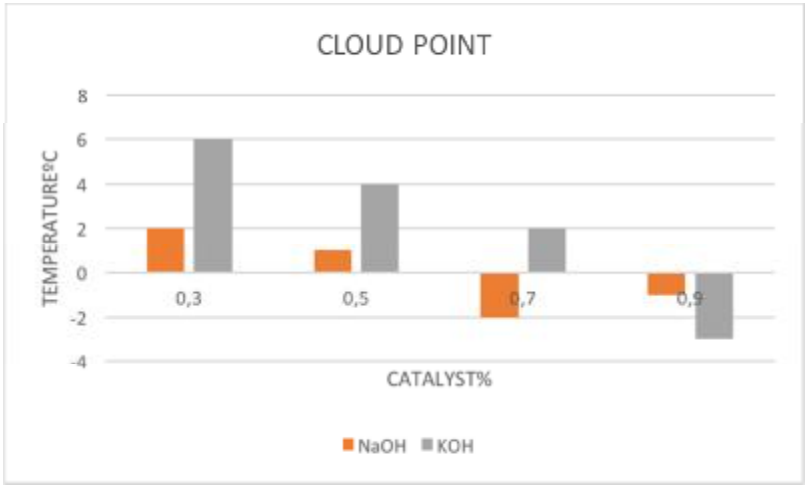

Figure 4.8. Effect of Catalyst amount on Cloud point

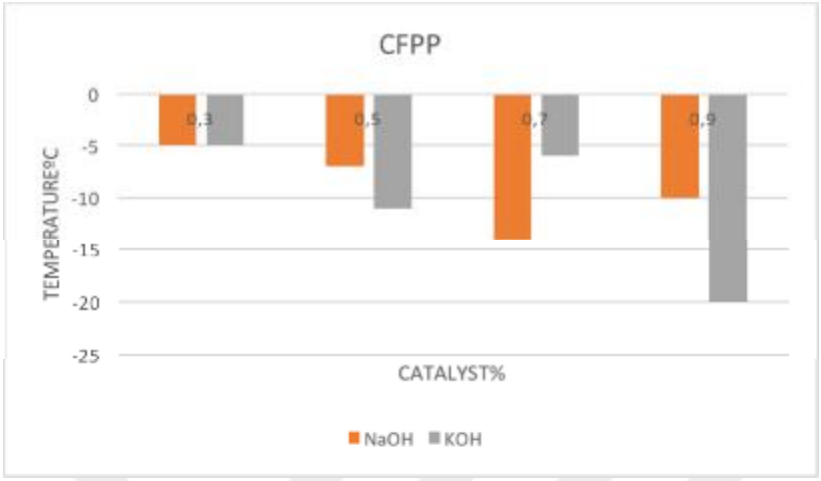

Figure 4.9. Effect of catalyst amount on cold filter plogging point

After evaluating the cold flow properties; effect of alcohol/oil rate on transesterification is evaluated.

One of the most important factor on methyl ester transformation is alcohol/oil mol ratio. In this thesis alcohol/oil ratio alters as 4:1, 6:1, 8:1 and 10:1 and catalyst amounts kept as 0,7% rate for NaOH catalyst and 0.9% rate for KOH catalyst and reaction temperature 60 °C and reaction duration as 60 minutes.

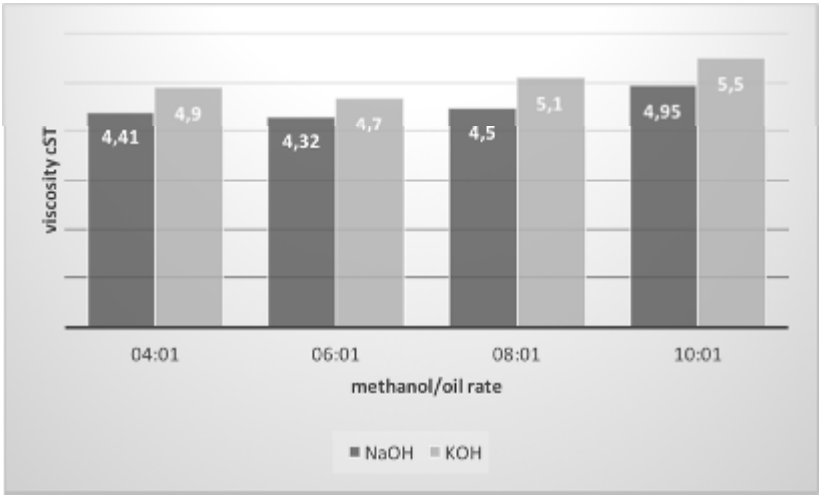

Figure 4.10. Effect of Alcohol/oil rate on kinematic viscosity

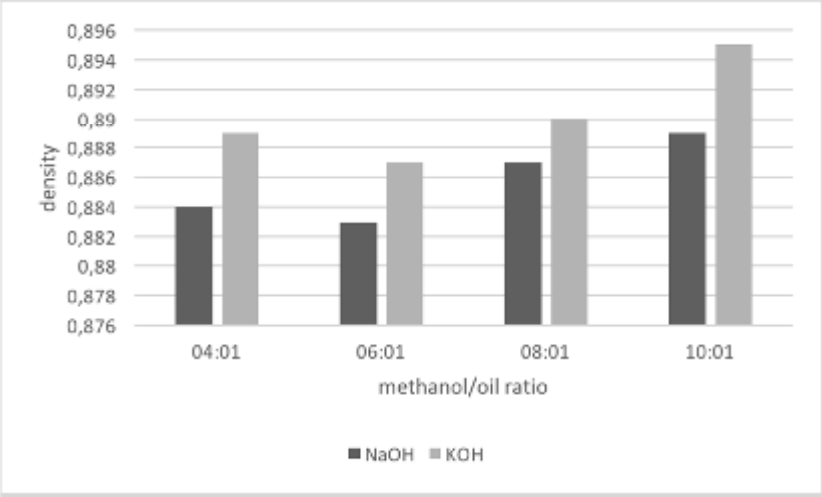

Figure 4.11. Effect of Alcohol/oil rate on density (g/cm$^{3)}$

On Figure 4.10 and 4.11, the effect of alcohol/oil rate of NaOH and KOH catalysts on viscosity and density are demonstrated. When obtained results are studied, kinematic viscosity is decreased as near the optimum alcohol/oil rate; above the optimum alcohol/oil rate kinematic viscosity increases. For NaOH catalyst 4.32 cSt is obtained at 6:1 mol rate; and for KOH catalyst 4.7 cSt obtained at 6:1 mol rate.

The increase of alcohol/oil rate up to the optimum level, the increases the conversion rate increases; due to transesterification process direction arrives to product side; and kinematic viscosity decreases. But when alcohol/oil rate exceeds the optimum level; due to increasing the solubility of glycerol in the methyl ester phase; difficulty in phase separation of the glycerin phase and ester phase, presence of dissolved glycerine during the reaction, turns the reaction side to entrant side and all these results cause decrease of transesterification efficiency and so increase the biodiesel viscosity.

And effect of alcohol/oil rate on cold flow properties are demonstrated on Figure 4.12, 4.13 and 4.14

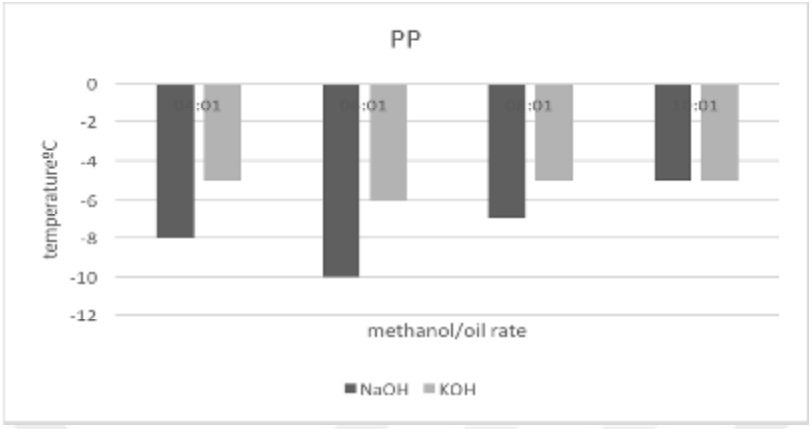

Figure 4.12. Effect of alcohol/oil on pour point

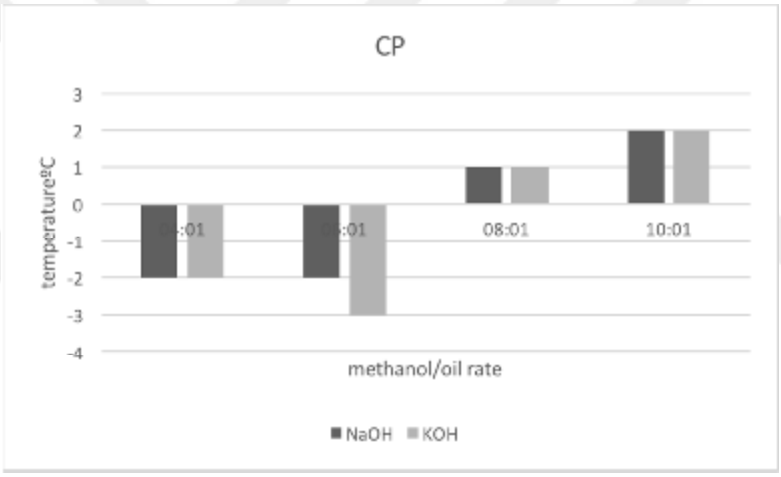

Figure 4.13. Effect of alcohol/ oil rate on cloud point

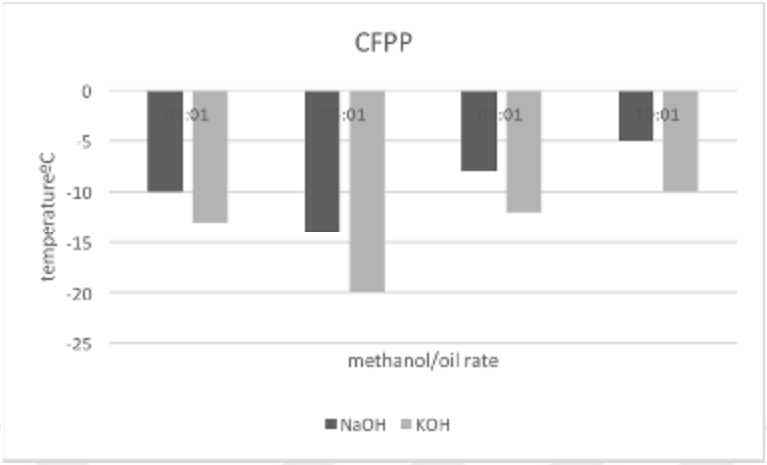

Figure 4.14. Effect of alcohol/oil rate on cold filter plogging point.

Regarding to the experimental studies based on different catalysts; during the canola biodiesel production, effect of catalyst amount and alcohol/oil molar ratio on methyl ester conversion ratio and fuel properties are studied.

The maximum methyl ester converion ratio obtained by NaOH catalyst based biodiesel is %0,7 catalyst ratio and 61 alcohol/oil mol ratio is %90; and by KOH catalyst based biodiesel is 0.9% catalyst ratio and 6:1 alcohol/oil mol ratio is %80,5.

In transesterification process for NaOH catalyst based biodiesel, the minimum viscosity value obtained at 0.7% catalyst rate and 6:1 alcohol/oil mol ratio; and for KOH catalyst based biodiesel the minimum viscosity value obtained at 0.9% catalyst rate and 6:1 alcohol/oil mol ratio.

Due to maximum conversion rates obtained during NaOH catalyst based transesterification processes, only NaOH is used as catalyst for palm oil and cotton oil transesterification experiments.

4.2. Effects of Catalyst Concentration for Palm Oil

As mentioned on the previous section only NaOH is used as catalyst during all palm oil transesterification processes.

In the first reaction section, the methanol/oil molar ratio:6:1, reaction temperature: 60°C, reaction time: 60 minutes were kept constant and catalyst concentration was altered 0.3, 0.5, 0.7 and 0.9%.Values are determined to include the parameter ranges in literature.In the reaction of transesterification, the maximum conversion rate is obtained at 0.7% catalyst concentration as demonstrated on Figure 4.15 and Table 4.5. At constant temperature (60°C), constant reaction time (60 min) and constant methanol ratio (6:1) maximum conversion (%) 92,5 obtained at % 0,7 catalyst ratio.

Table 4.5. Effect of Catalyst ratio on Conversion Rate (%)

Catalyst Ratio (%)	Methanol Ratio (%)	Reaction Time (min)	Reaction Temperature (°C)	Conversion (%)
0,3%	6:1	60	60	83,25%
0,5%	6:1	60	60	90,0%
0,7%	6:1	60	60	92,5%
0,9%	6:1	60	60	85,75%

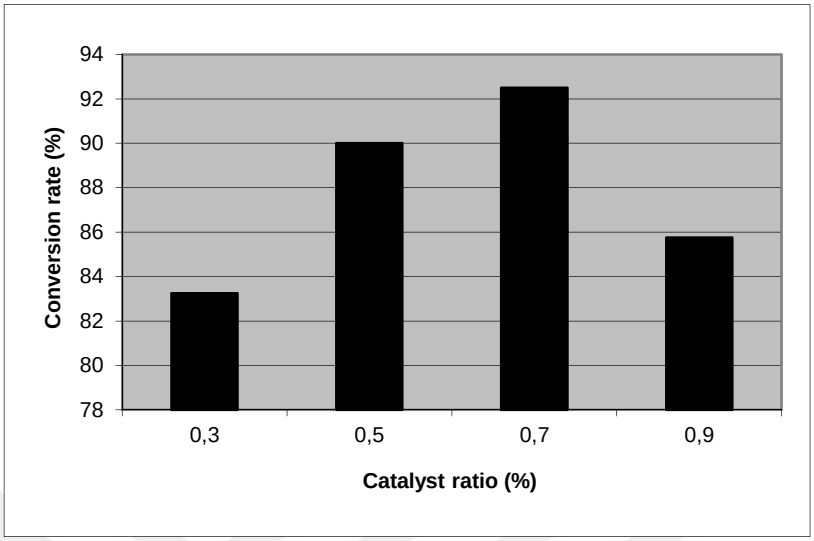

Figure 4.15. Effect of catalyst ratio on conversion rate

Then by fixing the catalyst ratio as 0,7%; at constant reaction time 60 min, constant reaction temperature (60°C); maximum conversion is obtained at 4:1 methanol ratio as mentioned on Table 4.6 and Figure 4.16 .

Table 4.6. Effect of Methanol Ratio (%) on Conversion Rate (%)

Catalyst Ratio (%)	Methanol Ratio (%)	Reaction Time (min)	Reaction Temperature (°C)	Conversion (%)
0,7%	4:1	60	60	95,70%
0,7%	5:1	60	60	94,35%
0.7%	6:1	60	60	92.5%
0,7%	7:1	60	60	85,74%

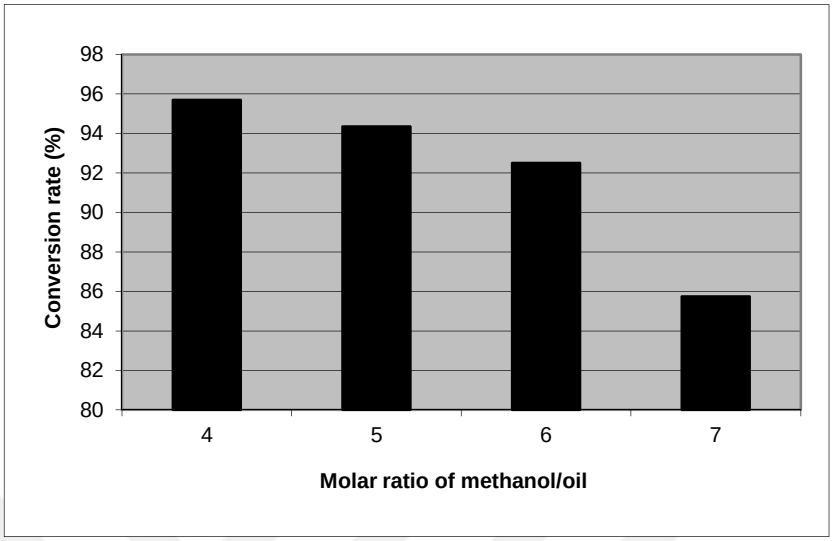

Figure 4.16. Effect of molar ratio of methanol/oil on conversion rate

Regarding to previous reaction results obtained at methanol ratio at 4:1 and on final reactions applied at constant catalyst ratio 0,7% ; constant reaction time 60 min, 60 °C maximum conversion rate is 95.7%.

Reaction temperature altered that 55°C, 60°C, 65°C, maximum conversion 88,92% is obtained on 65 °C as demonstrated on Figure 4.17 and Table 4.7.

Table 4.7. Effect of Reaction Temperature on Conversion Rate (%)

Catalyst Ratio (%)	Methanol Ratio (%)	Reaction Time (min)	Reaction Temperature (°C)	Conversion (%)
0,7%	4:1	60	50	80,10%
0,7%	4:1	60	55	83,60%
0,7%	4:1	60	60	96%
0,7%	4:1	60	65	88,92%

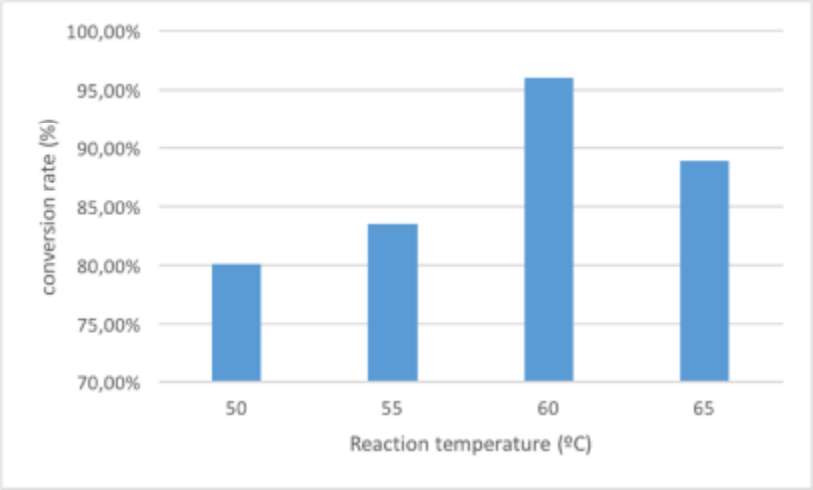

Figure 4.17. Effect of reaction temperature on conversion rate

Reaction time altered that 45, 60, 75, 120 min maximum conversion 95,7% is obtained on 75 minutes reaction temperature as seen on Figure 4.18 and Table 4.8.

Table 4.8. Effect of Reaction Time on Conversion Rate (%)

Catalyst Ratio (%)	Methanol Ratio (%)	Reaction Time (min)	Reaction Temperature (°C)	Conversion (%)
0,7%	4:1	45	60	90%
0,7%	4:1	60	60	87%
0,7%	4:1	75	60	95,7%
0,7%	4:1	120	60	85%

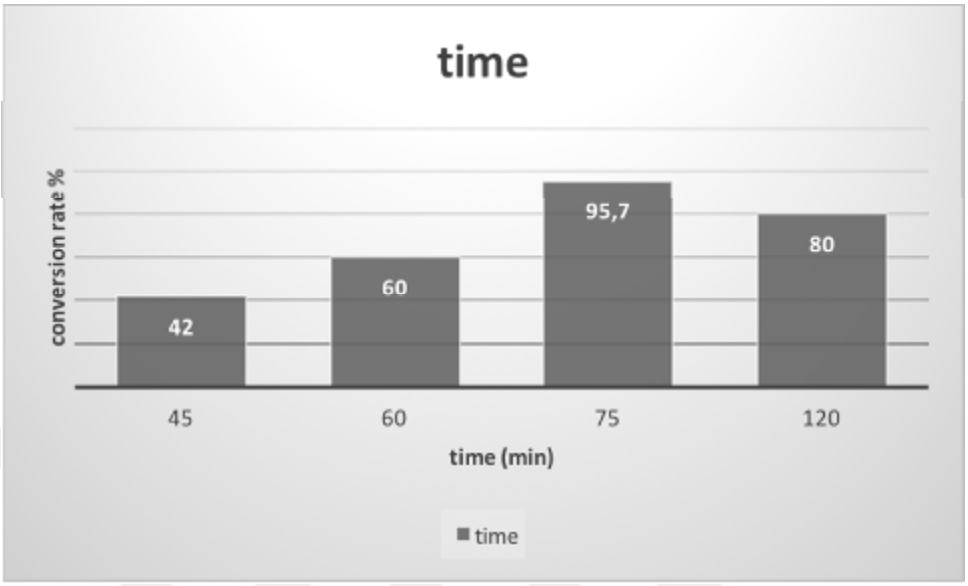

Figure 4.18. Effect of reaction time on conversion rate

Table 4.9. Fuel Properties of Diesel (Özgür ,2011)

Property	**Units**	**EN 590**	**Diesel**
Density	kg/m³	820-845	833
Viscosity	mm²/s	2.0-4.5	2.85
Flash point	°C	Min 55	58.5
Cetane number	-	Min 51	56
Pour Point	°C	-	-10

After conversion rate properties; density and viscosity properties are tested. In the first reaction section, at constant methanol rate (%) as 6:1, at constant reaction time as 60 min, at constant reaction temperature as 60°C and variation of 0,3%, 0,5%, 0,7% and 0,9% most convenient density as 0,882 and the most convenient viscosity as 4,229 obtained at catalyst ratio as 0,7% as demonstrated on Figure 4.19 and Figure 4.20 by comparing to Diesel properties as shown on Table 4.9.

Table 4.10. Effect of Catalyst Ratio on Density and Viscosity Properties

Catalyst Ratio (%)	Methanol Ratio (%)	Reaction Time (min)	Reaction Temperature(°C)	Density (g/cm^3)	Viscosity cST
0,3%	6:1	60	60	0,884	4,869
0,5%	6:1	60	60	0,882	4,089
0,7%	6:1	60	60	0,881	4,087
0,9%	6:1	60	60	0,882	4,089

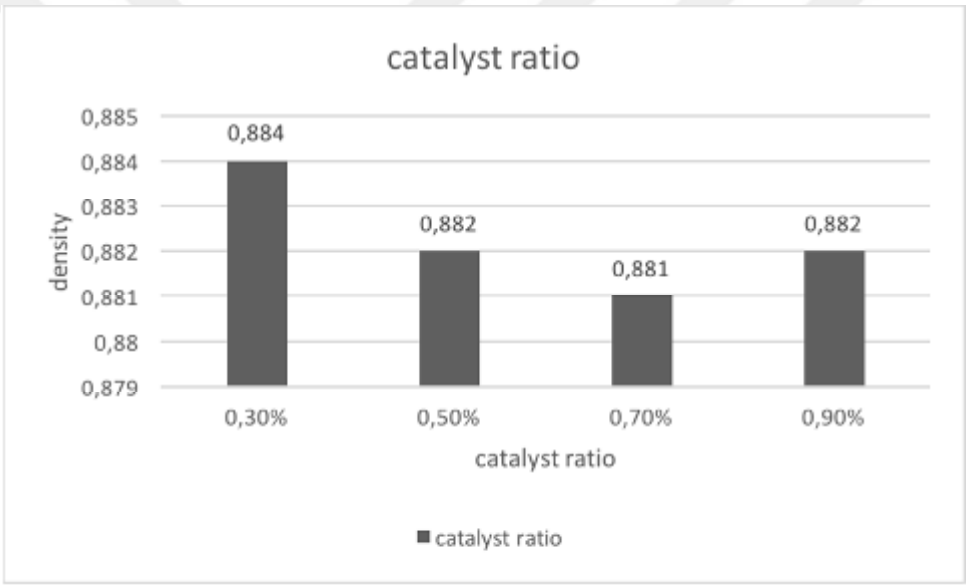

Figure 4.19. Effect of catalyst ratio on density

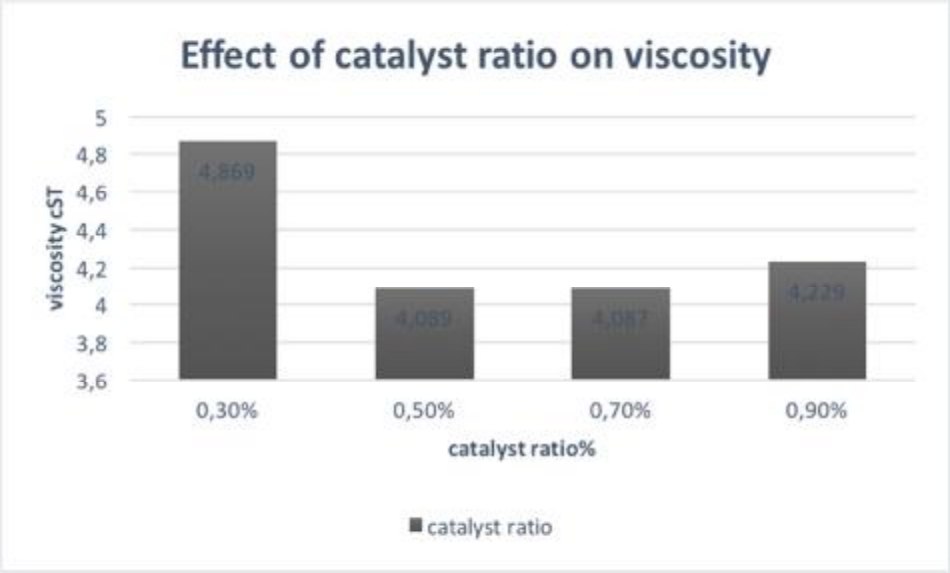

Figure 4.20. Effect of catalyst ratio on viscosity

After fixing the most convenient methanol/oil rate to reference amount as 0.7%; at constant reaction time as 60 min, at constant reaction temperature as 60 °C and methanol ratio is altered as 4:1, 5:1, 7:1. As demonstrated on Table 4.11 and Figure 4.21 and Figure 4.22 in the reaction of transesterification the most convenient density as 0,881 g/cm^3 and the most convenient viscosity as 4,087 cST is obtained at 4:1 methanol rate. At constant temperature 60 °C, constant reaction time (60 min), and constant catalyst ratio (0,7%) the most convenient density (0,881) and the most convenient viscosity (4,087).

Table 4.11. Effect of methanol ratio on density and viscosity

Catalyst Ratio (%)	Methanol Ratio (%)	Reaction Time (min)	Reaction Temperature (°C)	Density g/cm^3	Viscosity cST
0,7%	4:1	60	60	0,879	4,088
0,7%	5:1	60	60	0,878	4,189
0,7%	6:1	60	60	0,881	4,087
0,7%	7:1	60	60	0,881	4,219

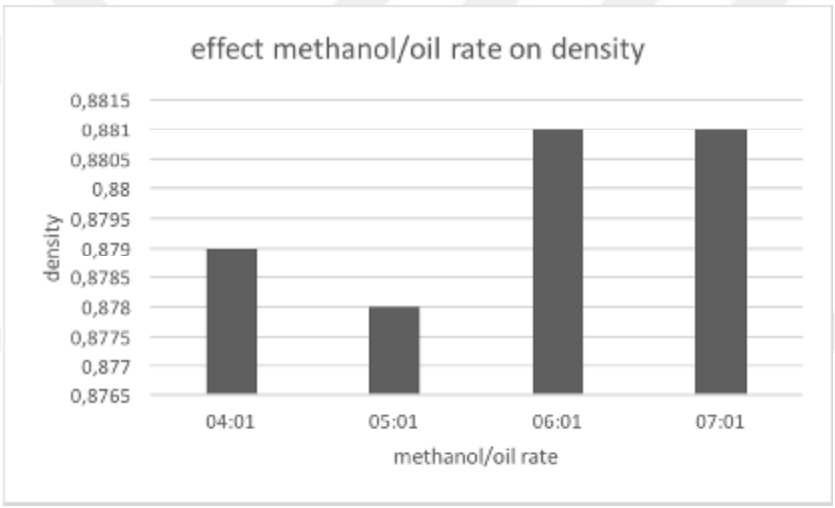

Figure 4.21. Effect of methanol/oil rate on density

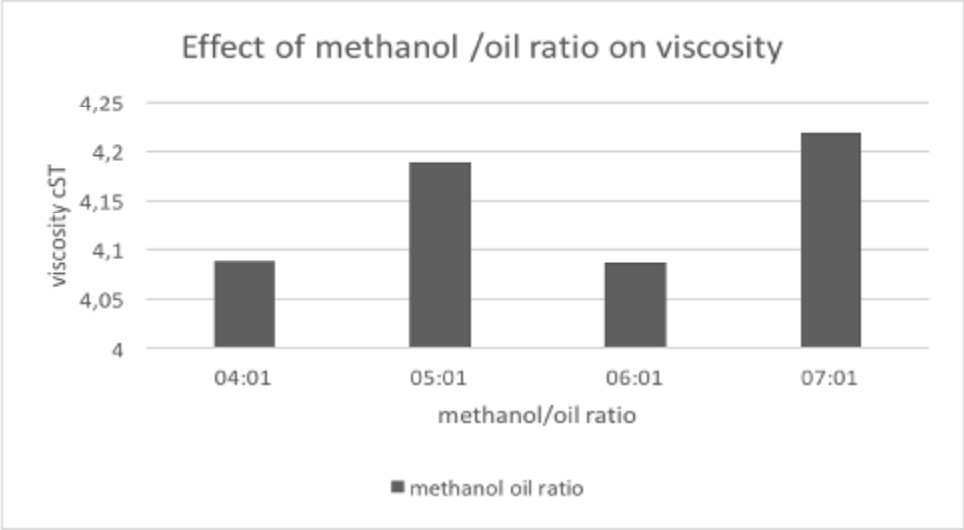

Figure 4.22. Effect of methanol/oil rate on viscosity

After evaluating the density and viscosity properties; cold flow properties are examined.

As demonstrated in Figure 4.23 and Table 4.12, in first transesterification processes on cold flow properties evaluation, methanol ratio 6:1 reaction temperature (60C) reaction time (60 min) are kept constant and catalyst ratio 0.3, 0.5, 0.7 and 0.9% And as a result of final transesterification process, the minimum PP (+4°C), CP(+6 °C) and CFPP (+2°C) are obtained at the catalyst ratio (%0,7).

Table 4.12. .Effect of Catalyst Ratio on PP, CP and CFPP

Catalyst Ratio (%)	Methanol Ratio (%)	Reaction Time (min)	Reaction Temperature (°C)	PP (°C)	CP (°C)	CFPP (°C)
0,3%	6:1	60	60	+5,0	+6,0	+3,0
0,5%	6:1	60	60	+5,0	+7,0	+3,0
0,7%	6:1	60	60	+4,0	+6,0	+2,0
0,9%	6:1	60	60	+5,0	+6,0	+3,0

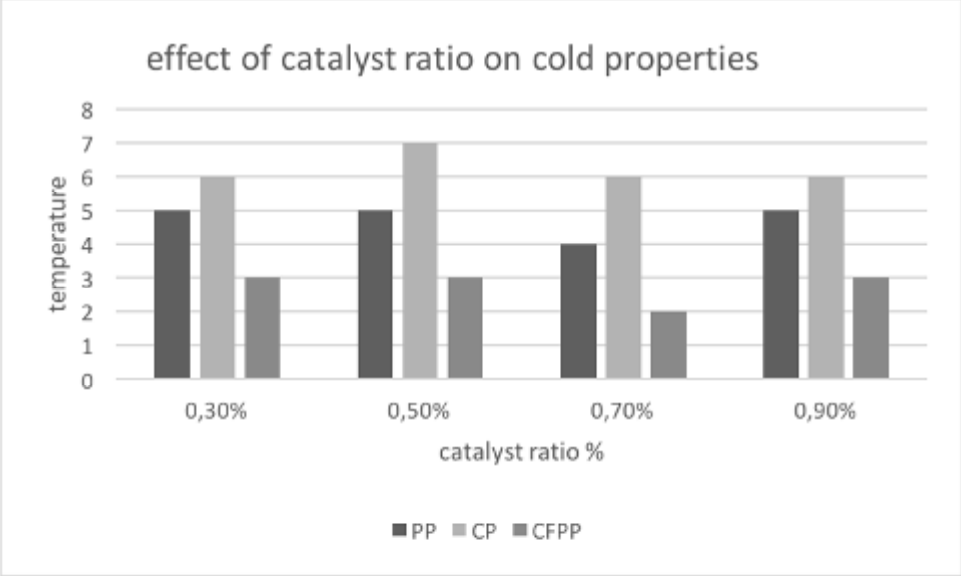

Figure 4.23. Effect of catalyst ratio on CP, PP and CFPP

Table 4.13. .Effect of Methanol/oil rate on PP, CP and CFPP

Catalyst Ratio (%)	Methanol Ratio (%)	Reaction Time (min)	Reaction Temperature (°C)	PP (°C)	CP (°C)	CFPP (°C)
0,7%	4:1	60	60	+5,0	+7,0	+3,0
0,7%	5:1	60	60	+6,0	+11,0	+4,0
0,7%	6:1	60	60	+4,0	+6,0	+2,0
0,7%	7:1	60	60	+6,0	+7,0	+4,0

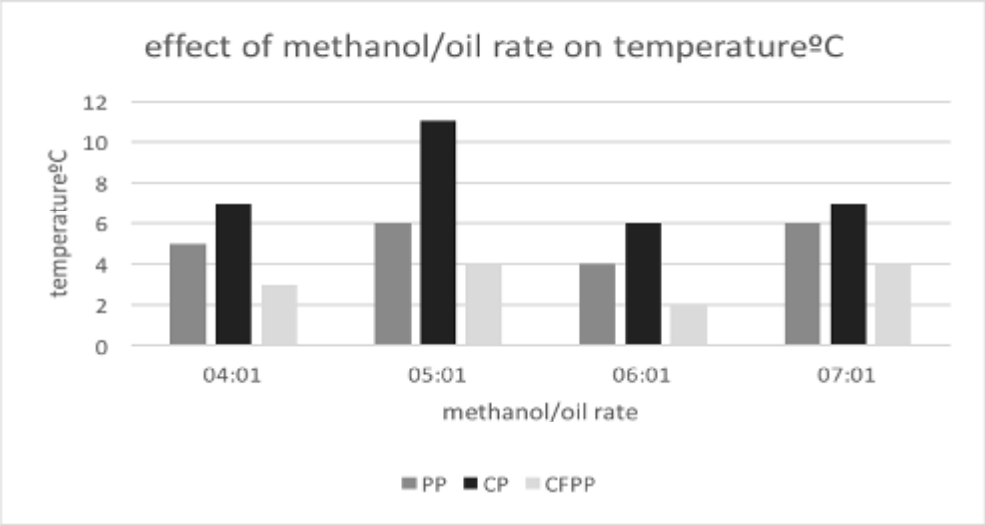

Figure 4.24. Effect of methanol/oil rate on CP, PP and CFPP

As demonstrated in Figure 4.24 and Table 4.13, minimum cold properties PP (+4°C), CP (+6°C) and CFPP (+2°C) obtained at 60 min reaction time ,60 °C reaction temperature, 6: 1% methanol ratio and %0,7 catalyst ratio.

Regarding to evaluations of palm oil as biodiesel fuel; its demonstrated that most feasible properties obtained at %0,7 NaOH catalyst ratio, 4:1 % methanol ratio, 60 minutes reaction time, 60 °C reaction temperature for 95,7% conversion rate.

And during evaluating the cold properties its demonstrated that palm oil biodiesel fuels do not have proper cold properties. To improve the cold properties for palm oil biodiesel fuels, its necessary to mix the palm oil with other biodiesels or increase the fuel additives to obtain suitable cold properties.

4.3. Effects of Catalyst Concentration for Cotton Oil

In the first reaction section, the methanol/oil molar ratio: 6:1, reaction temperature: 60°C, reaction time: 60 minutes were kept constant and catalyst concentration was altered 0.3, 0.5, 0.7, 0.9, 1.1 and 1.3%. Effects of catalyst

concentration on conversion is demonstrated in Figure 4.26 In the reaction of transesterification the maximum conversion rate is obtained at 0.3% catalyst concentration. At 0.3 % catalyst concentration a yield of 92.5% was achieved for cotton oil methyl ester. As demonstrated in Figure 4.26 that conversion rate decreases with the addition of NaOH catalyst concentration.

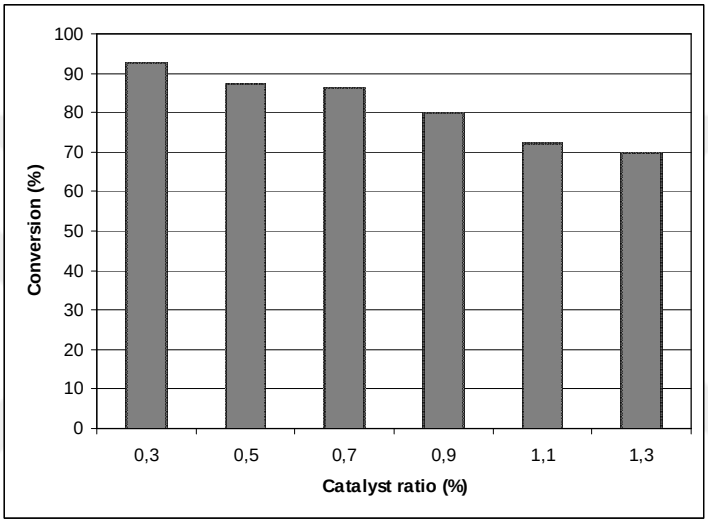

Figure 4.25. Effects of catalyst ratio on conversion

Molar ratio of alcohol to triglyceride is one of the most important parameter influencing the conversion rate of ester. In the transesterification reaction, the stoichiometric ratio needs 3:1 molar ratio alcohol/triglyceride to yield 3 mol of fatty acid alkyl esters and 1 mol of glycerol but a large amount of alcohol is needed to drive the reaction to the product side. Figure 4.26 illustrates the effects of molar ratio of methanol/oil on conversion. In transesterification reaction the maximum conversion rate was acquired at the methanol to oil molar ratio of 4:1 and conversion rate decreases at higher molar ratio of methanol to oil. As a result, 4:1 molar ratio was identified as the optimum molar ratio of methanol to oil for effective transesterification reaction.

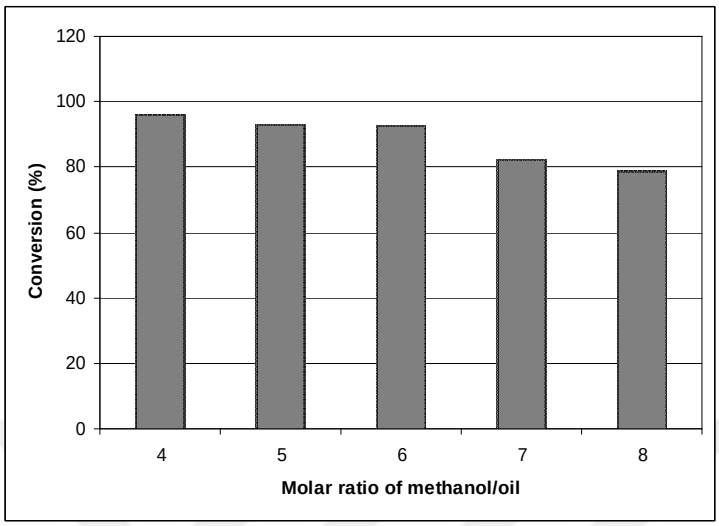

Figure 4.26. Effects of molar ratio of methanol/oil on conversion

The effects of reaction time on biodiesel conversion is demonstrated in Figure 4.28. The catalyst concentration and molar ratio of methanol to oil were set 0.3% and 4:1 respectively. During the reaction, temperature was kept at 60 ºC. It was observed that when reaction time reaches 75 min, the maximum conversion rate is obtained. After obtaining the maximum production, when the time is increasing the production decreases. The maximum conversion rate was found 96.75% for 75 min.

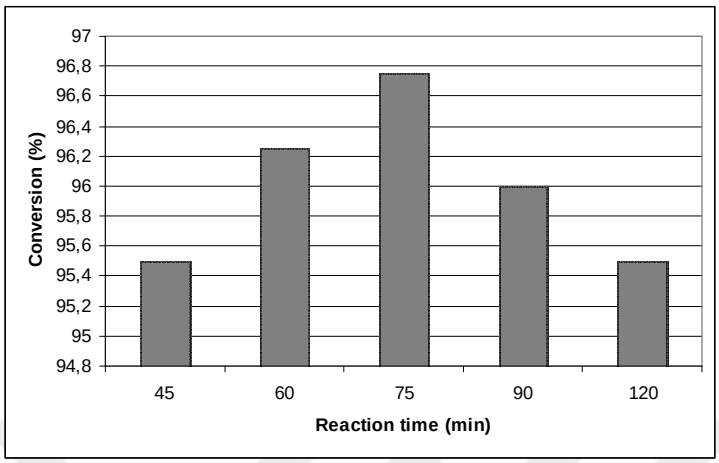

Figure 4.27. Effects of reaction time on conversion

Reaction temperature is one of the important factors affecting the progress of the transesterification reaction**.** In the experiments the reaction time was changed between 50 ºC and 70 ºC for observing maximum conversion rate. The effects of the reaction temperature on the conversion is given in Figure 4.27 As you can see in the Figure 4.29, the highest conversion was achieved at 60 ºC reaction temperature.

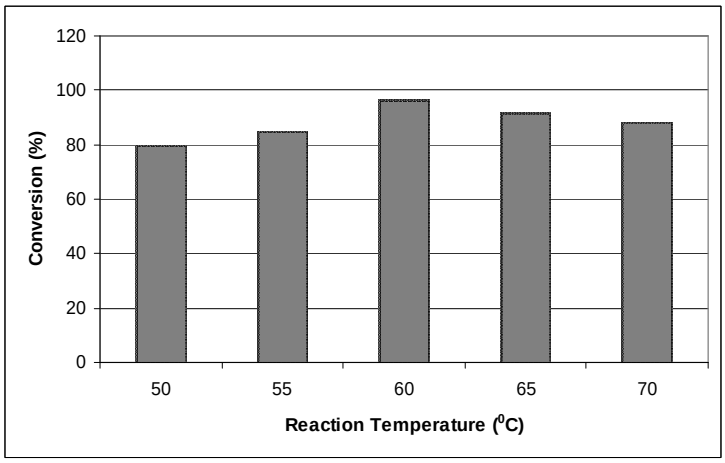

Figure 4.28. Effects of reaction temperature on conversion

The effects of catalyst concentration and molar ratio of methanol to oil on fuel properties are given in Table 4.14and Table 4.15 respectively. As mentioned in Table 4.14 with the addition of catalyst concentration density and kinematic viscosity values decrease however cold flow properties increased. Table 4.15shows that the lowest cold flow properties obtained at 8:1 molar ratio.

Table 4.14. The effects of catalyst concentration on fuel properties

Catalyst ratio (%w/w)	**Density (g/cm^3)**	**Kinematic viscosity (cST)**	**Pour Point(ºC)**	**Cloud Point (ºC)**	**Cold Filter Plugging Point(ºC)**
0.3	0.890	4.82	+5	+8	+3
0.5	0.888	4.6	+5	+8	+3
0.7	0.887	4.52	+6	+8	+4
0.9	0.886	4.38	+6	+9	+4

The effects of catalyst concentration and molar ratio of methanol to oil on fuel properties are given in Table 4.14 and Table 4.15 respectively. As you can see in Table 4.14 with the addition of catalyst concentration density and kinematic viscosity values decrease however cold flow properties increased. Table 4.15shows that the lowest cold flow properties obtained at 7:1 molar ratio.

Table 4.15. The effects of molar ratio of methanol to oil on fuel properties

Molar ratio of methanol to oil (%w/w)	**Density (g/cm^3)**	**Kinematic viscosity (cST)**	**Pour Point (ºC)**	**Cloud Point (ºC)**	**Cold Filter Plugging Point(ºC)**
4:1	0.888	4.65	+6	+9	+5
5:1	0.889	4.73	+6	+9	+4
6:1	0.890	4.82	+5	+8	+3
7:1	0.892	5.0	+4	+7	+2

Regarding to cotton oil biodiesel transesterification processes the highest biodiesel yield was achieved 96.75% with 0.3% NaOH concentration, 4:1 molar ratio of methyl alcohol to oil, 75 min of reaction time and 60ºC of reaction temperature. Biodiesel yield was reduced with the addition of catalyst. The lowest kinematic viscosity is measured 4.21 cST with 1.3% (w/w) catalyst concentration. Density and kinematic viscosity reduce with the addition of catalyst. The lowest cold flow properties obtained at 8:1 molar ratio of methanol to oil.

5. CONCLUSIONS

The aim of this experimental work on transesterification processes was investigate the effect of the effects of the biodiesel production parameters on conversion rate.

Because of its high viscosity and low volatility, the direct use of feedstock in diesel engines can cause problems including: high carbon deposits, scuffing of engine liner, injection nozzle failure, gum formation, lubricating oil thickening and high cloud and pour points. In order to avoid these problems, the feedstock is chemically modified to its derivatives which have properties more similar to conventional diesel The free fatty acids and triglycerides contained in the oil are reduced to Fatty Acid Alkyl Esters.

Experimental studies demonstrated that for Canola oil the maximum methyl ester converion ratio (%93) obtained by 0,7% NaOH catalyst ratio and 6:1 alcohol/oil mol ratio; and by %91 conversion rate obtained by0.9% KOH catalyst ratio and 6:1 alcohol/oil mol ratio. In transesterification process for NaOH catalyst based biodiesel, the minimum viscosity value obtained at 0,7% catalyst rate and 4:1 alcohol/oil mol ratio; and for KOH catalyst based biodiesel the minimum viscosity value obtained at 0.9% catalyst rate and 6:1 alcohol/oil mol ratio.

Regarding to comparision of experimental results and cost analysis results its demonstrated that NaOH catalyst is more feasible than KOH catalyst for transesterification processes.

Concerning to Canola oil for NaOH catalyst and alcohol amounts that given the minimum cold flow properties; pour point is -9°C, cloud point is -4°C and cold filter plugging point is -10°C, and for KOH catalyst and alcohol amounts, pour point is -10°C cloud point -7°C and cold filter plugging point is -23°C.

And regarding to these cold flow properties all PP, CP and CFPP values of canola oil are found suitable for usage.

Regarding to evaluations of palm oil as biodiesel fuel; its demonstrated that most feasible properties obtained at %0,7 NaOH catalyst ratio, 4:1 % methanol ratio, 60 minutes reaction time, 60 °C reaction temperature for 95,7% conversion rate.

Concerning to Palm oil for NaOH catalyst and alcohol amounts, that given the minimum cold flow properties; yield point is +5°C cloud point is +7°C and cold filter plugging point is +3°C. So regarding to this cold flow properties its demonstrated that palm oil biodiesel fuels do not have proper cold flor properties for using alone. To improve the cold flow properties for palm oil biodiesel fules, its necessary to mix the palm oil with diesel fuel or increase the fuel additives to improve the cold flow properties.

Regarding to cotton oil biodiesel transesterification processes the highest biodiesel yield was achieved 96.75% with 0.3% NaOH concentration, 4:1 molar ratio of methyl alcohol to oil, 75 min of reaction time and 60ºC of reaction temperature. Biodiesel yield was reduced with the addition of catalyst. The lowest kinematic viscosity is measured 4.21 mm^2/s with 1.3% (w/w) catalyst concentration. Density and kinemtaic viscosity reduce with the addition of catalyst. The lowest cold flow properties obtained at 7:1 molar ratio of methanol to oil.

And concerning to cold flow properties its demonstrated that cotton oil is not suitable to using alone as well as palm oil. To improve the cold flow properties its necessary to mix the cotton oil with diesel fuel or increase the fuel additives to improve the cold flow properties.

As a conclusion,regarding to comparision of all results with diesel and EN 590 fuel (as referance); its demonstrated that NaOH is more suitable to use as a catalyst during transesterification process, regarding to experimental and cost analysis. When palm oil, cotton oil and canola oil compared, the most suitable and feasible results concering fuel properties and cold properties obtained via canola oil. Also, canola oil is suitable to use alone instead of mixture with diesel fuel.

Its demonstrated that the maximum conversion ratio of canola oil, obtained at 6:1 alcohol/oil ratio,.As a result of this study, regarding to experimental results and preliminary studies %0,7 NaOH catalyst ratio, 6:1 alcohol/oil rate is the most feasible conditions to produce pure biodiesel.

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CURRICULUM VITAE

Nursima BUYUK was born on January 01,1986 in Kırıkkale After completing his education in MKE Tophane İlkokulu in Cankırı and Adnan Menederes Anatolia High School in Istanbul , she enrolled in Çukurova University Textile Engineering Department in 2004 and Mechanical Engineering Department in 2005 by double major programme.She had graduated from the Mechanical and Textile Engineering Department of Çukurova University with a Bachelor of Science degree in 2008. She had started to Master of Science education Automotive Engineering Department in 2013

After January 2010, Nursima BÜYÜK took charge in various private sector companies. She started her career in Bossa TAS as production development engineer in Denim and Sportwear Company. She continued her job through the beginning of 2014 year. Between the beginning of 2014 and end of june 2016 she worked as sales executive of YUNSA Premium Collection in Sabancı Holding.