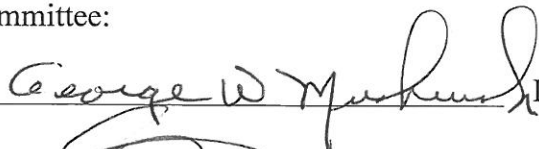
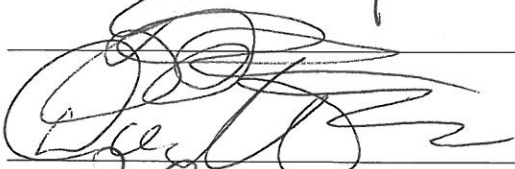

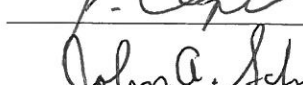
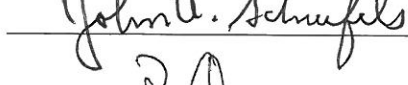
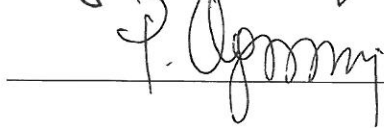
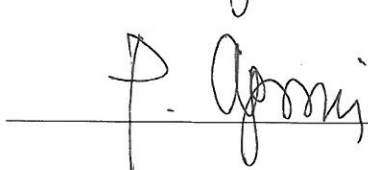


CANOLA DERIVED BIODIESEL AS BLENDING AGENT FOR PETROLEUM  
BASED FUELS

by

Phung Kim Vo  
A Thesis  
Submitted to the  
Graduate Faculty  
of  
George Mason University  
in Partial Fulfillment of  
The Requirements for the Degree  
of  
Master of Science  
Chemistry

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Fall Semester 2013  
George Mason University  
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A Thesis submitted in partial fulfillment of the requirements for the degree of Master of Science at George Mason University

by

Phung Kim Vo  
Bachelor of Science  
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Director: George W. Mushrush, Professor  
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## **DEDICATION**

This thesis is dedicated to my family, especially my parents, Vo Trung Mai and Ha Thi Minh Phi, for their unconditional love, support and belief in me. I also dedicate this thesis to my love, Phan Nguyen Lap, for always being by my side, and his words of encouragement have been a big support through my graduate school.

## **ACKNOWLEDGEMENTS**

I would like to express my deepest appreciation to my research mentor, Dr. George W. Mushrush, for his guidance, kindness and encouragement during my undergraduate and graduate school to finish this research. I would not be able to get this far without his support.

I would also like to thank Mr. Harry Castillo for giving me opportunities to assist his doctoral project, which gave me helpful knowledge to carry out my own research project. I would also like show my appreciation to Mr. Michael Girgis for his great help during my laboratory work, Ms. Jun Liu for helping me perform GC/MS and her feedback in my GC/MS analysis.

And again, I would like to send my appreciation to my parents and my brother for their financial support throughout my college life, and my boyfriend, for his support, encouragement and unwavering belief in me all these years. For all my friends who have been always by my side when I was about to give up halfway, I would like to thank you all and be grateful for all you guys have done for me.

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## LIST OF ABBREVIATIONS OR SYMBOLS

U.S. Energy Information Administration.....	EIA
Fatty Acid Methyl Ester.....	FAME
Gas Chromatography/Mass Spectrometry .....	GC/MS
DCM .....	Dichloromethane
RT .....	Retention time
ARS.....	Agriculture Research Service

## **ABSTRACT**

### **CANOLA DERIVED BIODIESEL AS BLENDING AGENT FOR PETROLEUM BASED FUELS**

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George Mason University, 2013

Thesis Director: Dr. George W. Mushrush

The fuel industry has been under significant pressures due to economic and environmental concerns. Finding cleaner and safer alternative fuel source was put in high demand. Biodiesel has been the most favorable source in the biofuel field for a while, and soybean has been the leading feedstock for making biodiesel. There are also other types of oil such as corn, palm, peanut or canola oil for making either 100% biodiesel (B100) or biodiesel blending stock for petroleum based fuels. Canola oil when crushed out will give up to 44% of oil content, the highest oil content among other types of the agricultural oil while soybean has about 20% and corn has 5% of oil content respectively. Because of its properties such as high flash point, lower cloud point and the stability in cold-weather region, and flexible crop season in fall and spring, canola oil methyl ester, derived product of canola oil esterification, was taken into research in which it was subjected to conditions that mimic the petroleum refining process (delayed coking). Four

tubes of canola oil ME went through pyrolysis process (90 psi and 400°C) in different time interval: 15, 30, 60 and 90 minutes. Result from GC/MS showed the presence of FAMEs such as palmitic, oleic, elaidic, and stearic acid methyl ester. In GC of 90-minute interval, all FAMEs except stearic acid methyl ester were replaced by hexadecane peaks ( $C_{16}H_{34}$ ). The total area percentage of all hexadecane peak in GC of 90-minute is 32.27 %.

## **1.0 BACKGROUND**

### **1.1 Gasoline**

Gasoline is the main fuel source now in the automobile industry despite the fluctuation in supply and pricing. Crude oil is the raw material for gasoline which must undergoes complex refining process to crack into lighter hydrocarbon chains ( $C_4$ - $C_{12}$  hydrocarbon for gasoline). Other products from crude oil along with gasoline are naphtha, kerosene, diesel, heating oil, or lubricating oils. In the United States, gasoline is blended with approximately 15% of  $C_4$ - $C_8$  alkanes, 25-40% of  $C_4$ - $C_{10}$ , 10% cycloalkanes, and maximum of 25% aromatic compounds, and about 10% of alkenes and cyclic alkenes (Ritter 2005). Some other components sometimes found in gasoline are toluene, xylene, and even lead (which appear as a big concern for human health and environment).

Gasoline appears as colorless or pale brown; boiling points depend on percentage of distillation. Its flash point is very low compared to other fuel sources,  $-46^{\circ}\text{C}$  (ATSDR Gasoline Profile 1995). Gasoline is the main consumer fuel sources and even though diesel and biodiesel are more economical, vehicles running on gasoline engine are still cheaper than ones run on diesel and biodiesel. World consumption for crude oil in 2012 was 89.2 million barrel per day (bpd), and is expected to reach 1.05 million bpd in 2013 and 1.4 in 2014. United States produced about 6.4 million bpd of crude oil in 2012; however, this number has decreased since 2012 probably due to increases in crude oil

import from unstable oil-producing countries (IER 2012). Table 1 below lists some general properties of gasoline such as solubility, density, auto-ignition temperature and flash point.

**Table 1: Gasoline Properties**

<b>Property</b>	
<b>Color</b>	Colorless/Pale Brown
<b>Physical State</b>	Liquid
<b>Boiling Point (Depending on % of distilled)</b>	10% distilled 60°C
	50% distilled 110°C
	90% distilled 170°C
<b>Density</b>	0.7-0.8 g/cm <sup>3</sup>
<b>Solubility</b>	Insoluble in water
	Soluble in absolute alcohol, ether, chloroform, and benzene
<b>Auto-ignition temperature</b>	280-486°C
<b>Flash Point</b>	-46°C

## 1.2 Jet Fuel

Jet fuel does not have a specific formula or general properties like gasoline. It is a special type of blended fuel from fractionated distillation of crude oil (Alglas). Jet fuel is more complex than gasoline because of its working conditions like aircraft turbine engines which are often required to operate in extreme weather conditions. Kerosene, thin oil distilled from petroleum or shale oil, is the most common compounds in most of jet fuel types. There are many jet fuel types such as JET A-1, JP4, JP5, JP7, or JP8, depending on their purpose of usage. JET A-1 is most commonly used in commercial aviation; JP4, 5, 7 and are mostly used in U.S Navy and U.S Air Force. Each jet fuel type has different melting and boiling point, density, auto-ignition temperature and flash point. Jet fuel is less volatile than gasoline due to higher flash point. JP5 which is mainly served as U.S

Navy fuel has about 53% of C<sub>9</sub>-C<sub>16</sub>, 31% of cyclo-paraffin, and 16% of aromatic compounds. JP-8 is similar to Jet A-1, mainly composed of C<sub>8</sub>-C<sub>9</sub> aliphatic hydrocarbons, 9% of C<sub>10</sub>-C<sub>14</sub> and 65% of C<sub>15</sub>-C<sub>17</sub> (Henderson et al 1996). Table 2 displays general properties of JP-5 and JP-8 used in military aircrafts (ATSDR Jet Fuel Profile 1998).

**Table 2: Jet Fuel (JP-5 and JP-8) Properties**

	JP-5	JP-8
<b>Color</b>	Clear and Bright	Clear and bright
<b>Melting point</b>	-46°C	-52°C
<b>Boiling point</b>	170°C	170°C
<b>Density</b>	0.788-0.845kg/L	0.775-0.840kg/L
<b>Auto-ignition temp</b>	229°C (kerosene)	
<b>Flash Point</b>	60°C	38°C

### 1.3 Pressure on Fuel Industry

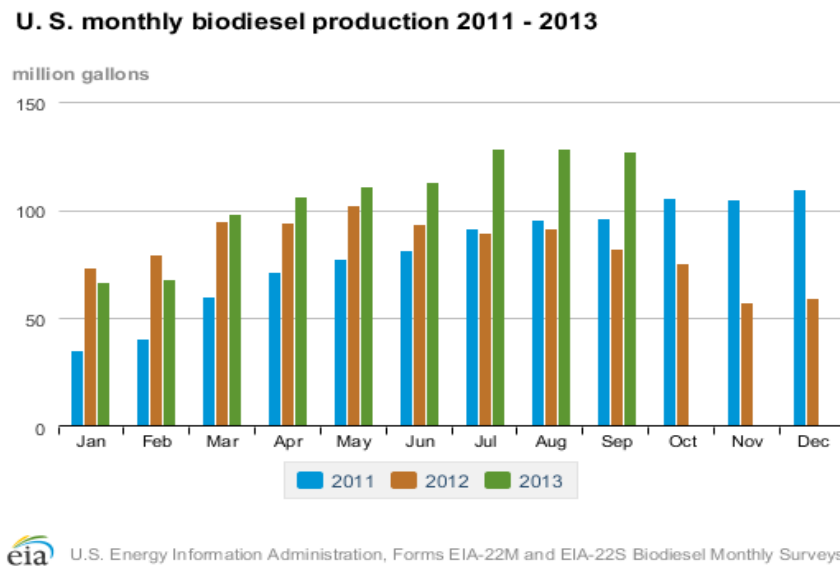
Fuel prices in the US have been increasing in the past couple years, and not showed any sign of stabilizing. Since the United States only produces about half of the amount of crude oil barrel to satisfy domestic demand, it must rely heavily on importing from other oil-producing countries. Imbalance between supply and demand has sparked many interests in alternative options to petroleum. These alternatives come in both flavors: non-renewable sources such as used automobile oil and renewable sources such as biofuels. Diesel has always been a major source of energy for heavy duty vehicles and equipment. However, it remains an unpopular choice for consumer vehicles due to the several factors such as higher cost of ownership, lack of choices, and its negative impact on public health effects due to emission exhaust. Diesel exhaust tends to have higher amount of carbon

dioxide, carbon monoxides, nitrogen oxides, sulfur oxides, or aldehyde compounds such as formaldehydes and acetaldehyde, benzene, some polycyclic aromatic hydrocarbons (PAHs) and nitro-PAHs, and other pollutants which ultimately can cause cancers or genetic mutations (UCUSA 2002).

## **1.4 Biodiesel**

Biodiesel has been promoted for a few decades as a much safer alternative to petroleum-derived fuel. With the higher density ( $0.88\text{g/cm}^3$ ) compared to fossil diesel density ( $0.83\text{g/cm}^3$ ), the energy level of biodiesel is about 13% higher than diesel (40.70% for biodiesel versus 38.20% for diesel). Studies have shown that compared to petroleum-based diesel, biodiesel does not release harmful emissions which can cause damaging risk to human health and substantial impact on environment. Biodiesel is the yellow grease part remained after filtering out all food particles from waste cooking oils. It is the product of reaction between agricultural oil and excess amount of alcohol in the presence of a catalyst (either strong acid or strong base). Canola oil and other kinds of oil such as corn oil, peanut oil, or soybean oil can become sources for biodiesel in order to solve the economic and environmental pressure on the oil and fuel industry. Using biodiesel can de-stress the industry and solve some of the environmental concerns such as global warming due to high amount of carbon dioxide; air, water and land pollution due to oil spills; emission exhaust; and acid rain. Biodiesel has become more favorable because of its bio-degradability, renewability, and at lower cost than diesel and gasoline, even with partial-biodiesel fuel. Compared to engines operating on regular fuels, biodiesel engines produce less toxic emission exhaust (carbon monoxide) or sulfur oxides. It can also help

enhancing fuel stability in both hot and cold weather regions, higher flash point, and decreasing emission exhaust.



**Figure 1: Biodiesel Production 2011-2013**

Production of biodiesel in the United States was 127 million gallons in September 2013, with approximately 64% from the Midwest region; compared to 20122, this is an increase of 31.1 million gallons. Biodiesel is both sold as B100 (100% biodiesel) or as blending stock with other fuel sources. About 976 million pounds of feedstock was used to produce biodiesel, and soybean oil is still the leading stock for biodiesel with 502 million pounds used during September 2013 (EIA Report 2013).

## 1.5 Canola Oil

Canola oil is rapeseed oil with low erucic acid concentration. Its name was changed in 1979 after Western Canadian Oilseed Crushers Association succeeded in lowering the amount of erucic acid (less than 2% now in canola oil) in rapeseed oil to decrease the risk of cardiovascular diseases. Canola has relatively high crop production for its wide



availability of seeded types in both fall and spring seasons (Oplinger et al 1989). Another advantage of growing canola is its adaptability to extreme weather conditions especially in cold regions such as North Dakota, South Dakota, Michigan or the Midwest regions of United States. According to report from U.S Canola Association, approximately 1.5 million acres of canola are grown in the U.S, mainly in North Dakota. Demand for canola oil in food industry is increasing; however, ratio of supply versus demand is 1:3, which means that crop production needs to be accelerated and expanded in order to provide sufficiency for both feedstock and fuel industry.

Some advantages of canola oil are stability in cold tests, high oil content, high flash point, and low cloud point and pour point. High flash point of canola oil compared to gasoline and jet fuel can lower the risk of flammation, cloud point of  $-3^{\circ}\text{C}$  can enhance durability and safety of engines in cold weather, and low pour point can contribute to maximizing fuel usage.

**Table 3: Canola Oil Properties (Gunstone 2009)**

Property	
Density	0.914-0.917 g/cm <sup>3</sup>
Smoke Point	220-230°C
Flash Point (open cup)	275-290°C
Specific Heat (J/g)	1.91-1.916
Cloud Point	-3°C
Pour Point	-9°C
Oil Content	44%

**Table 4: Fatty Acid Composition**

<b>Fatty Acid Composition (Weight Percent %)</b>				
	<b>Canola</b>	<b>Corn</b>	<b>Peanut</b>	<b>Soybean</b>
<b>C 16:0 Palmitic</b>	4 - 5	7 - 13	6 -12.5	2.3 - 11
<b>C 18:0 Stearic</b>	1 - 2	2.5 - 3	2.5 - 6	2.4 - 6
<b>C 18:1 Oleic</b>	55 - 63	30.5 - 43	37 - 61	22 – 30.8
<b>C 18:2 Linoleic</b>	20 - 31	39 - 52	13 - 41	49 - 53
<b>C 18:3 Linolenic</b>	9 - 10	1	1	2 -10.5

Table 4 demonstrates the percentage of fatty acid composition in canola oil with highest content of oleic acid, linoleic, linolenic, palmitic and stearic acid. Those acids are expected to show in gas chromatogram of each run, and as time expands longer, they can break down to alkenes or alkanes.

## **2.0 THESIS**

As price of fuel continues to escalate in the present and more concerns arise for both environment and human health, the needs for more biodiesel increase. Greenhouse gas emission has been reduced at least 57% and up to 86% when substituting petroleum diesel with biodiesel. However, the main source for making biodiesel is soybean which is also necessary source for feedstock; limited supply of soybean together with high demand from both industries can cause shortage at some. As a result, canola was brought into research for its high oil content when crushed out compared to soybean and corn oil.

The objective of this research is examining the product of canola-derived diesel to determine its ability as blending agents for petroleum-based fuels. The method applied in this research is based on the procedure of refining petroleum in fuel industry, called delayed coking. Delayed coking is heat-required thermal cracking in completing the coking reactions with the help of a furnace, which helps to shorten the time of coking (Fahim et al 2009). Pyrolysis of agricultural oil has not been studied before so our prediction is that FAMEs will probably undergo a free radical attack at the double bond of the fatty acids in canola oil methyl ester and even break down to alkanes in either gasoline or jet fuel range. Expected results are based on those of previous research of soybean-derived biodiesel in which canola oil methyl-ester should enhance fuel stability.

### 3.0 EXPERIMENTAL

#### 3.1 Reagents

The canola methyl-ester sample was made by one graduate student at George Mason University. The general procedure was canola oil sample heated to 55°C after having water and any particulates removed, and then taken into reacting with methoxide (mixing 99% pure methanol and 85% pure potassium hydroxide). Fatty acid methyl-ester (FAMEs), or biodiesel, and glycerol by-product were formed in the ratio of 10:1. The products were separated then washed with water, and its purity was confirmed by dissolution in methanol until the clear solution formed without any deposit appearance. Figure 2 below illustrated reaction of fatty acid converted to fatty acid methyl esters.

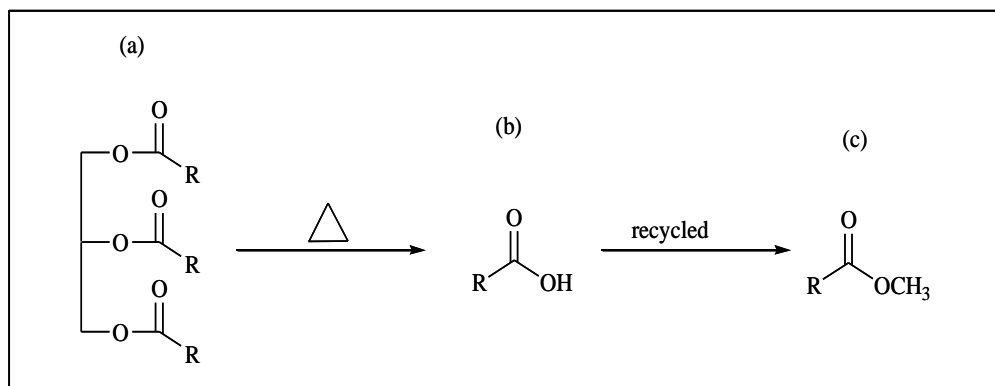


Figure 2: Conversion Reaction of Fatty Acid to FAME

### 3.2 Method

Set-up apparatus for this research included a vacuum machine, hot water container and isopropyl container to keep dry-ice for quenching process (showed in figure 6). Four 15 cm long, 6.4 mm-o.d.(1/4-inch-o.d.) 316 stainless steel tubes with one end closed and the other fitted with a stainless steel valve via a Swagelok fitting with 0.1 g of canola oil methyl ester in each tube were the significant parts of the experiment because it ensured that no significant of residues forming on the tube wall. Each tube was attached to the vacuum system, cooled to 195K then subjected to three freeze-pump-thaw cycles (Mushrush et al 1984). The first stainless tube to the valve with vacuum valve was opened for 3 minutes then closed; the tube was into a silver dry ice container for another 3 minutes (freeze), opened the valve gently (pump), put the tube into hot water container for 3 minutes (thaw), then closed the valve, repeated this process for another two times before closing both valves and closed the tube tightly. The same procedure was applied for another three tubes (30, 60 and 90 minutes). Four tubes were left to room temperature and pyrolyzed by being inserted the 90-minute tube first into 7-mm holes in a 15-cm diameter aluminum block fitted with heaters and temperature controller (Furnace 47900), followed by 15, 60 and 30-minuted tubes. Pyrolysis temperature was 400°C with pressure of 90 psi (620 kPa). Stainless steel tube 316 would not show any catalytic behavior below 823°C, as seen in the study of thermal cracking of n-octane by Marschner group. After the pyrolysis period, all tubes were cooled down to room temperature (22°C), and tightened valves were removed. About 100  $\mu$ L of toluene was first added to each tube, followed by 0.5 cc of DCM (about 3 times for each tube) until liquid poured into vial

appeared clear, then 0.1cc of toluene was added last before closing the Teflon cap liner vial. Same procedure was also applied for other 3 tubes, and all vials were left at 0°C in the fridge for 24 hours before diluting them to 1/100 and 1/1000 with DCM to run for GC/MS.



**Figure 3: Stainless Steel Tube 316**



**Figure 4: Furnace 47900**



**Figure 5: Experimental Set-up**

### **3.2.1 Gas Chromatography/Mass Spectrometry**

The result from the experiment was analyzed by combined capillary GC/MS in Prince William Campus of George Mason University. The GC/MS system was Agilent Technologies Model 6890, a gas chromatography configured for splitless injection (1  $\mu$ L), with a Finnegan INCOS 50B mass spectrometer. The GC part was equipped with all glass inlet system which has 0.32 mm x 30m fused silica capillary column (column flow 1mL/min, cross-linked 5% phenyl methyl silicone supplied by Agilent Technologies. The set up injector temperature was 250°C and the detector temperature was 320°C. Initial temperature was set at 60°C for 5 minute, a ramp of 5°C/min, to a final temperature of 260°C. The Finnegan INCOS 50B mass spectrometer was operated in the electron impact ionization mode (70 eV) with continuous scan acquisition from 50 to 300 amu at a cycling rate of 1 scan/sec. The electron multiplier was 1050 V; source temperature was 200°C; and the transfer line temperature was 290°C (Mushrush et al 2012).

## 4.0 RESULT AND ANALYSIS

### 4.1 Result Data from GC/MS

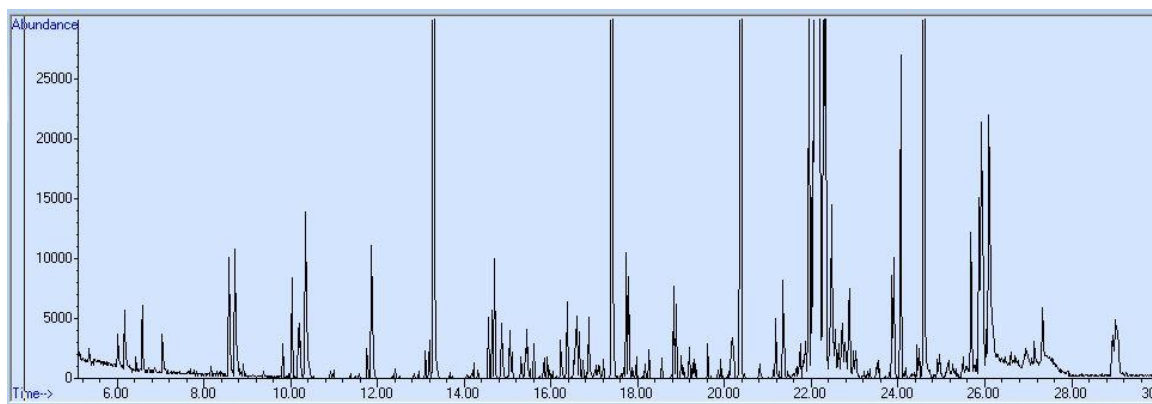


Figure 6: GC 15-minute Interval

Table 5: MS 15-minute Area % Report

# peak	R.T min	% of total	Result
4	20.369	10.13	Palmitic acid ME
5	22.091	37.62	Oleic acid ME
6	22.139	30.94	Elaidic acid ME
7	22.296	7.26	Stearic acid ME



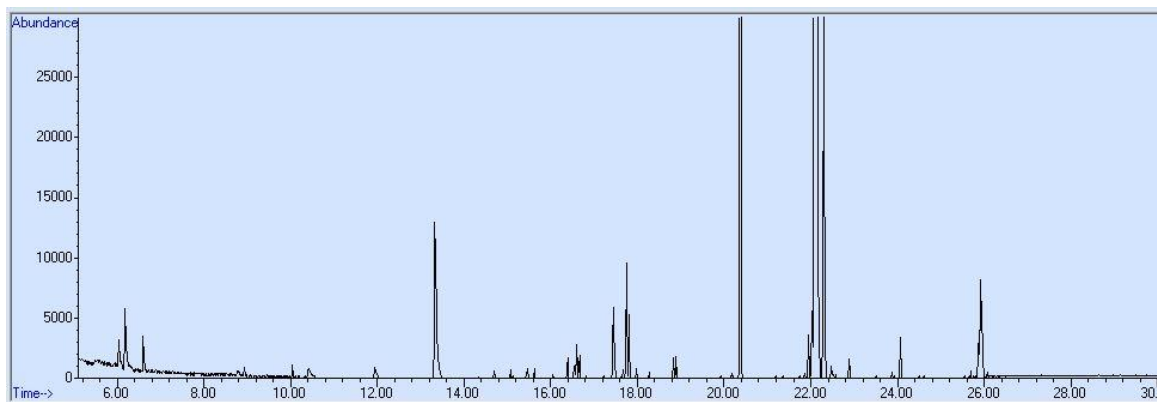


Figure 7: GC 30-minute Interval

Table 6: MS 30-minute Area % Report

# peak	R.T min	% of total	Result
1	20.372	10.91	Palmitic acid ME
2	22.084	60.98	Oleic acid ME
3	22.125	28.11	Elaidic acid ME
4	22.293	####	Stearic acid ME

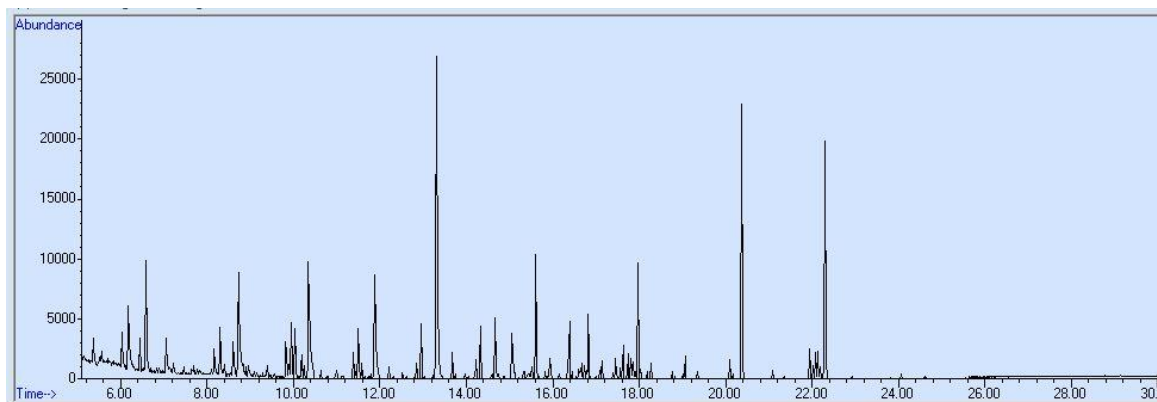


Figure 8: GC 60-min Interval

Table 7: MS 60-minute Area % Report

# peak	R.T min	% of total	Result
18	17.973	4.09	C-16
19	20.376	9.94	Palmitic acid ME
20	22.303	9.25	Stearic acid ME

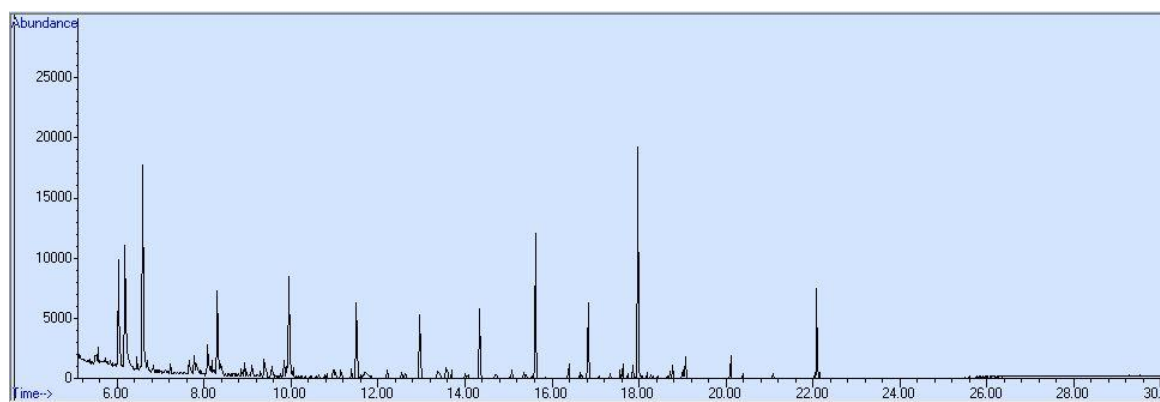


Figure 9: GC 90-min Interval

Table 8: MS 90-minute Area % Report

# peak	R.T min	% of total	Result
13	14.334	4.49	C-17
14	15.617	9.15	C-17
15	16.832	4.5	C-17
16	17.978	14.13	C-17
17	22.093	5.24	Stearic acid ME

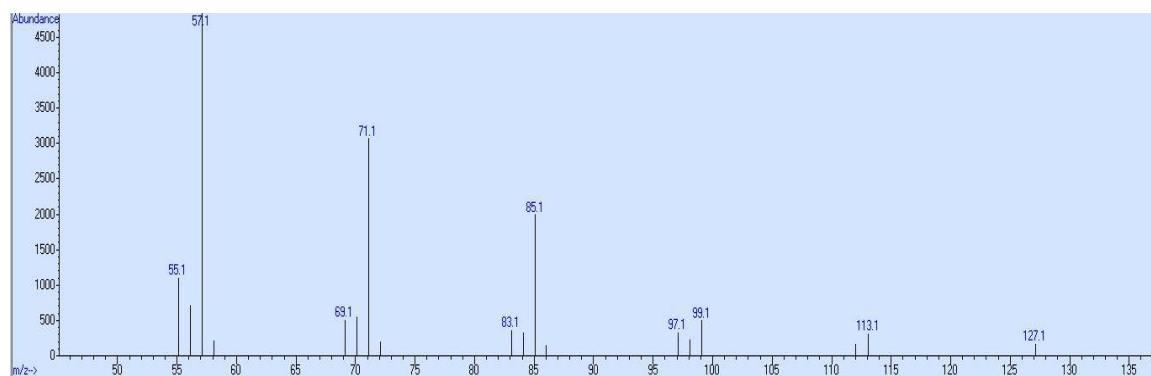


Figure 10: MS of C-17

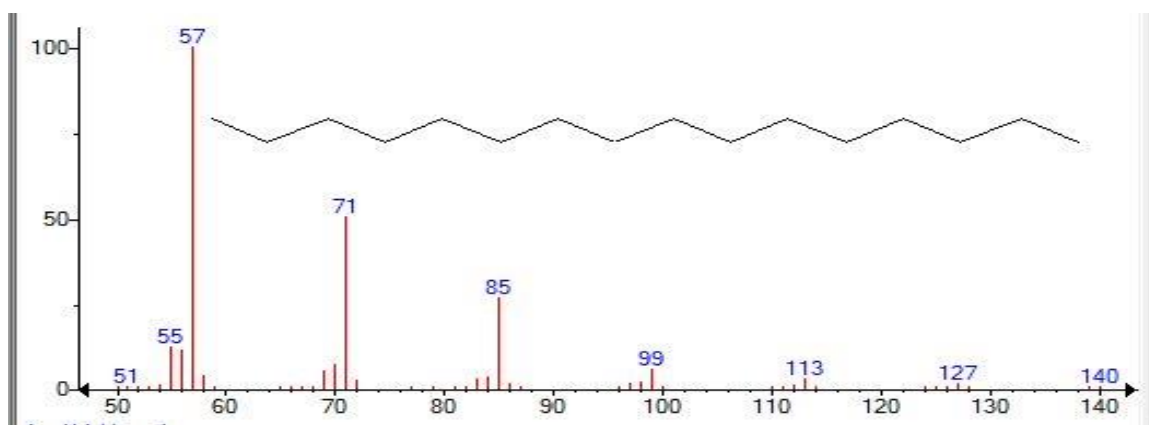
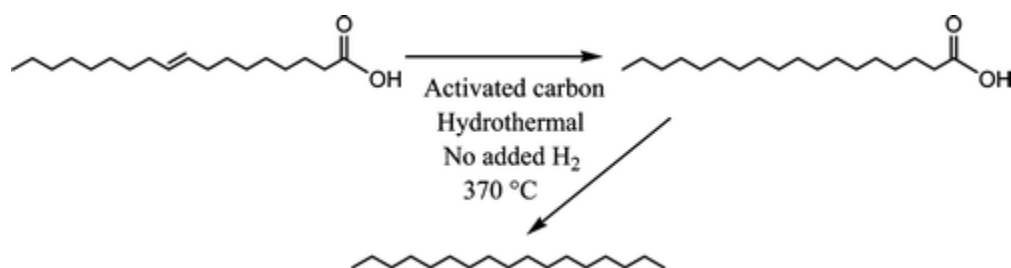


Figure 11: Library MS of Hexadecane

## 4.2 Discussion

Gas chromatogram of the 15-minute and 30-minute interval showed four peaks of four main fatty acids in canola oil such as palmitic acid, stearic acid, oleic acid, and elaidic acid. Oleic acid and elaidic acid methyl esters have the highest percentage in the compounds. Only stearic acid methyl ester in GC of 30-minute didn't have any percentage in the compounds despite its peak presence in the GC. It could be the error or not having enough concentration to be detected in the GC. In 60-minute, hexadecane peak started to show at 17.973 RT with percentage of 4.09, and oleic acid and elaidic acid have disappeared, which indicated that those two FAMES may be broken down and yield the hexadecane product. All the FAMES have gone in GC of 90-minute except for stearic acid ME and yield the same product which was hexadecane  $C_{16}H_{34}$ . The MS of experimental compound for hexadecane looked almost identical to the library result, which supported that most of the FAMES except for stearic acid ME were mostly broken down. The total area percentage of all hexadecane peaks in GC of 90-minute is 32.27. Because there is no clear evidence which FAME was broken down first and which yield the most hexadecane, no detail mechanism can be given, however, palmitic and oleic

acids were studied to determine the range of hydrocarbons they converted by using activated carbons in near and supercritical water with no hydrogen added. It showed that palmitic gave products in C<sub>8</sub>-C<sub>15</sub> range, and oleic acid yield about C<sub>12</sub>-C<sub>17</sub> alkanes and some C<sub>17</sub> olefins (Savage et al 2011). The proposed mechanism is the double bond in FAMES will undergo free radical attack first, followed by leaving the carboxylic group which eventually produces long chain hydrocarbons. The figure below was result from Savage research group proposing the possible mechanism, however, our research did not use activated carbon and our temperature was higher:



**Figure 12: Possible Mechanism of Conversion Fatty Acid to Long Chain Hydrocarbon**

## **5.0 CONCLUSION**

Using waste vegetable oil as the primary reagent for biodiesel products can help to reduce about 70-95% for the petroleum cost and solve some of the environmental problems. The appearance of hexadecane after pyrolysis process is supporting evidence that canola derived biodiesel can be a suitable blending agent for petroleum based fuel. Further research can continue on examining the stability of petroleum fuel when adding canola derived biodiesel. Blending any agent into petroleum based fuels has to be done with significant consideration, for example, flash point consideration, engine types, or solubility of both petroleum fuel and blending stock. The blending agent cannot affect the stability of the original fuel. Overall, this research provides more opportunities for alternative fuels in order to reduce pressure on fuel industry, and also gives encouragement for farmers to boost the crop production to decrease competing with food industry.

## **6.0 FUTURE DIRECTIONS AND EXPECTATION**

The U.S airline industry has consumed about 19 billion gallons of fuel, and the amount will increase significantly in the near future based on its current growing rate. The result of this thesis is promising for another future of aviation bio jet-fuel; however, it has to be further examined for the stability when blending in jet fuel with different concentration. In addition, the supply of agricultural oil in food industry is not equivalent to market demand, which makes mass production of fuel from vegetable oil is still a concern. The quality of oil can also a factor in determining the quality of biofuels, therefore, some on-going research projects are looking for high quality ideal plant oil to make renewable jet fuel. Scientists have been working with farmers to improve crop production or increase harvesting process. Another possible approach is rotating the different type of oilseed, for example, ARS chemists have been working on identifying rapeseed and wheat rotation systems to determine if it can help in reducing erosion, improving the conditions of the field such as water holding capacities or herbicide needs. The main purpose of this project is to test if the rate of conversion agricultural oil to biofuels, especially jet-fuel, will increase with high-quality oil (ARS 2012).

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### **Work experience:**

#### **2010-present: Department of Chemistry, George Mason University**

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- Assisted in quality control of fertilizer, import food product (coffee, tea), or determination of chemical compounds in agricultural products.

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