

Triglyceride

$$\text{Triglyceride} + 3 [\text{H}_3\text{C}-\text{OH}] \xrightarrow{\text{Catalyst}} \text{Glycerol} + 3 \text{Methyl Ester (R)}$$

Glycerol

Methyl Ester (R)

Triglyceride

SOY

BIODIESEL

CHEMISTRY EDUCATION KIT

(VERSION 2.0)



INTRODUCTION

The development of environmentally sustainable sources of energy and chemicals is an ongoing global challenge for this century. With limited worldwide reserves of petroleum, the search for alternative renewable fuels which can supplement or replace petroleum is an important facet of this challenge. Biodiesel, a renewable fuel based upon vegetable oils like soybean oil, as well as animal fats, is one of the potential solutions.

Biodiesel provides reductions in harmful emissions, is derived from renewable sources, and can be used in every diesel vehicle without any modification. For these and dozens of other reasons, it is one of the best options for renewable fuels in this industry, and shows a promising future as a cleaner burning, renewable, environmentally friendly alternative to petroleum diesel fuel.

Fleet operators, school corporations, government agencies and private owners around the world are using biodiesel to supplement or replace petroleum diesel in their vehicles, with more and more people switching every day.

The purpose of this education kit is to engage high school students in activities which teach them basic chemistry principles and skills through the production and testing of biodiesel fuel in the classroom.

With these lessons, students will gain a better appreciation not only for principles of chemistry, but will be able to see how chemistry affects them in their daily lives, and can improve the future of the world they live in.

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CONTENTS OF KIT

PROVIDED WITH THIS KIT

The following materials and equipment are provided in this kit for use in the lesson plans. With the option of the chromatography plates, the equipment contained in this kit is reusable when properly cared for. A DVD showing the experiments being performed is also included. It is formatted to run on a standard TV DVD player system.

Consumables:

- 2 Chromatography Plates, Silica Gel Plates with plastic backing

Equipment:

- 10 Nalgene Polyethylene Reaction Bottles, 250 ml
- 20 Transfer Pipettes, 1.7ml
- 20 Micro Pipettes, 2 μ L
- 30 Centrifuge Tubes, 50 ml
- 2 Laboratory Lamps with Fiberglass Wicks
- 30 (2) Dram Glass Sample Bottles with Lids
- 1 Bottle of Urea, 100g

TO BE PROVIDED BY THE INSTRUCTOR

The following materials and equipment must be provided by the instructor in order to complete the experiments outlined in this kit.

Consumables:

- **Soybean Oil** (Vegetable Oil), food grade.
Approx. 2 quarts needed per lesson set.
Available at any supermarket or food service supplier.
- **Styrofoam Pellets** Standard packing peanuts
Approx. 50 pellets needed per lesson set.
- **Methyl Alcohol** (CH₃OH) ACS certified or better.
Approx. 500 ml needed per lesson set.
- **Potassium Hydroxide** (pellets or powder) (KOH)
ACS certified or better. Approx. 20 grams needed per lesson set.
- **Ether, Petroleum**
ACS certified or better. Approx. 100 ml needed per lesson set.
- **Ether, Ethyl** (C₄H₁₀O)
ACS certified or better. Approx. 50 ml needed per lesson set.
- **Acetic Acid** (C₂H₄O₂)
Glacial or ACS certified. Approx. 10 ml needed per lesson set.
- **Iodine** (flakes or crystals) (I₂)
ACS certified or better. Approx. 50 grams needed per lesson set.



CONTENTS OF KIT

Equipment:

This equipment list is based upon 1 set of equipment per group of 2 students. This kit contains equipment for 10 groups. Safety equipment should be provided for all students based on laboratory practices.

- 1 Graduated cylinder, 100 or 250 ml
- 1 Graduated cylinder, 25 or 50 ml
- 2 Electronic balances accurate to 0.01 gm
- 10 Glass plates (6 inches square or larger, see pg 3-3)
- 10 Rulers
- 10 Forceps
- 10 Magnetic stirring plates
- 10 Magnetic stirring bars
- 10 125 ml Erlenmeyer flasks
- 10 Plastic weighing dishes
- Nitrile Safety Gloves
- Safety Goggles

The Indiana Soybean Alliance and its affiliates (collectively, "ISA") have attempted to provide accurate information in connection with this educational science kit and all its components (the "Kit"). However, ISA shall not be held responsible for any errors, inaccuracies, omissions, or other defects in the Kit. Your use of the Kit is at your own risk. The Kit is provided AS IS AND WITHOUT ANY WARRANTIES, WHETHER EXPRESS OR IMPLIED, INCLUDING WITHOUT LIMITATION ANY IMPLIED WARRANTIES OF MERCHANTABILITY OR FITNESS FOR A PARTICULAR PURPOSE. WITHOUT LIMITING THE FOREGOING, ISA DOES NOT WARRANT ANY RESULTS THAT MAY BE OBTAINED BY USING THE KIT, NOR DOES IT WARRANT THE KIT TO BE COMPLETE OR ERROR FREE.

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TEACHERS' EVALUATION

Your input is important to us. Please mail, fax or email a completed copy of this evaluation form to:
Indiana Soybean Alliance, 8425 Keystone Crossing, Indianapolis, IN, 46240, Fax: 317-347-3626.
Email: chemistrykit@indianasoybean.com

You may also complete this evaluation online at www.indianasoybean.com/chemistrykit

Name _____

Name of School _____

School Address _____

City _____ State _____ Zip _____

1. What did you like best about the lessons in this kit? _____

2. What did you like least about the kit? _____

3. What changes should be made to improve this kit? _____

4. Were the lessons appropriate in content and complexity for high school students? _____

5. Were adequate materials supplied? _____

6. Was the Teachers' background clear and complete? _____

7. Did your students enjoy the lab activities? _____

8. Did the lab activities teach the basic concepts they were designed to teach? _____

9. Were the instructions for the activities clear? Did you obtain the anticipated results? _____

10. Would you use this kit again? Why or Why not? _____

11. What grade level/course was this kit used? _____

Additional Comments: _____

STUDENTS' EVALUATION

Fill out this evaluation after using the kit in your classroom and return to your instructor.

Indiana Soybean Alliance, 8425 Keystone Crossing, Indianapolis, IN, 46240, Fax: 317-347-3626.

Email: chemistrykit@indianasoybean.com

Student's Name _____

1. What did you like best about the lessons in this kit? _____

2. What did you like least about the kit? _____

3. Did your teacher/instructor have the appropriate material to teach this kit? _____

4. Did you enjoy the lab activities? _____

5. What lab activity was your most favorite? _____

6. Were the instructions clear and easy to understand? _____

7. What grade level are you? _____

8. How would you rate your teacher/instruction on teaching the kit? (circle one)

Fair

Good

Excellent

Additional Comments: _____

ACADEMIC STANDARDS

National Science Education Standards are available via <http://www.map.edu/> or by phone at 1-800-624-6242.

National Science Content Standards 9-12	Lesson Plans Which Apply
A: Science As Inquiry	
Abilities Necessary to do Scientific Inquiry	1, 2, 3, 4, 5, 6
Understandings about Scientific Inquiry	1, 2, 3, 4, 5, 6
B: Physical Sciences	
Structure of Atoms	
Structure and Properties of Matter	2, 3, 4, 5, 6
Chemical Reactions	2, 3, 4, 5, 6
Motions and Forces	
Conservation of Energy and the Increase in Disorder	
Interactions of Energy and Matter	2
C: Life Sciences	
The Cell	
The Molecular Basis of Heredity	
Biological Evolution	
The Interdependence of Organisms	
Matter, Energy and Organization in Living Systems	
The Behavior of Organisms	
D: Earth and Space Science	
Energy in the Earth System	
Geochemical Cycles	
The Origin and Evolution of the Earth System	
The Origin and Evolution of the Universe	
E: Science and Technology	
Abilities of Technological Design	1, 2, 3
Understandings about Science and Technology	1, 2, 3, 4, 5, 6
F: Science in Personal and Social Perspectives	
Personal and Community Health	
Population Growth	
Natural Resources	1
Environmental Quality	1, 3
Natural and Human-Induced Hazards	1
Science and Technology in Local, National and Global Challenges	1, 3
G: History and Nature of Science	
Science as a Human Endeavor	1
Nature of Scientific Knowledge	
Historical Perspectives	1

STUDENT PRELAB QUESTIONNAIRE

Name _____ Class _____ Date _____

Please answer the following questions to the best of your ability.

Are you familiar with the diesel engine and how it works? (circle one) Yes No

Do you or someone in your family drive a diesel vehicle? (circle one) Yes No

Do you operate diesel machinery, either at home or work? (circle one) Yes No

Have you ever heard of biodiesel fuel? (circle one) Yes No

If so, where have you heard about biodiesel?

Have you ever heard of other types of alternative energy? If so, please list those you are familiar with.

In the following questions, please circle the number which corresponds to your interest in the topic listed to the left. With the number 1 corresponding to something that does not interest you at all and the number 5 corresponding to something you are very interested in.

	Not interested			Very interested	
	1	2	3	4	5
The Price of Fuel at the Pump	1	2	3	4	5
Renewable Resources	1	2	3	4	5
New Uses for Agricultural Products	1	2	3	4	5
Recycling and Environmental Stewardship	1	2	3	4	5
Applications of Chemistry in Industry	1	2	3	4	5

STUDENT POST-TEST QUESTIONNAIRE

Name _____ Class _____ Date _____

Please answer the following questions to the best of your ability.

Did you enjoy the experiments in this kit? (circle one)

Yes, very much Yes, somewhat I don't care either way No, not really

Which experiments were most interesting to you? (circle as many as you like)

Lesson 2, Transesterification of Soybean Oil to Fatty Acid Methyl Esters

Lesson 3, Physical and Chemical Property Differences of Soybean Oil and Fatty Acid Methyl Esters

Lesson 4, Thin Layer Chromatography to Examine the Biodiesel Reaction

Lesson 5, Determining Biodiesel Concentrations in Fuels

Lesson 6, Urea Inclusion Fractionation of Biodiesel. Why? _____

Have these lessons increased your awareness of important issues that affect your daily life?

What issues has it helped you with? _____

What fact about biodiesel did you find most interesting? _____

How has this kit affected your interest in Chemistry? (circle one)

Very positively Somewhat positively No change Somewhat negatively Very negatively

In the following questions, please circle the number which corresponds to your interest in the topic listed to the left. With the number 1 corresponding to something that does not interest you at all, and the number 5 corresponding to something you are very interested in.

	Not interested			Very interested	
	1	2	3	4	5
The Price of Fuel at the Pump	1	2	3	4	5
Renewable Resources	1	2	3	4	5
New Uses for Agricultural Products	1	2	3	4	5
Recycling and Environmental Stewardship	1	2	3	4	5
Applications of Chemistry in Industry	1	2	3	4	5



THE DIESEL ENGINE AND THE DEVELOPMENT OF BIODIESEL





TEACHERS' BACKGROUND MATERIAL

This lesson will introduce the students to the development of the diesel engine and provide a brief history of biodiesel fuel. This material is essential to establish the reasons behind the development of biodiesel and its growing importance in the renewable fuels industry. Please read the information on the following pages, and review the additional information in the Appendix A-1 to familiarize yourself with biodiesel and topics related to biodiesel which may be helpful in the classroom setting.

SUGGESTIONS

Depending upon the preference of the instructor, it may be desirable to reproduce and distribute this lesson as a homework reading assignment, along with the pretest in order to make maximum use of lab periods.

It may be helpful to give a short overview lecture on what the students will be doing over the course of the lessons, as well as a brief introduction to topic of biodiesel.

Introduction of the following topics for classroom discussion may help spur interest in the material:

- Current price of diesel fuel
- Production of petroleum and its impact on society and the environment
- Recent news on oil prices, pollution or global warming topics
- A survey of how many students own or use diesel vehicles or equipment
- Examples of how diesel engines are used in everyday life (school bus, delivery truck, trains, etc.)
- Examples of career opportunities in alternative fuels (chemists, engineers, farming, recycling, etc.)
- Examples of interesting feedstock (algae, used cooking oil, soybean oil)

GENERAL NOTES

The following handout is a list of frequently asked questions and answers about biodiesel. It may be distributed as an additional handout to the students or used as background material for the teacher. Additional information is available in Appendixes 1-5. It may be helpful to review the appendix material and distribute portions of the appendix pertinent to student interests.



LESSON 1

FREQUENTLY ASKED QUESTIONS ABOUT BIODIESEL

What is biodiesel?

Biodiesel is the name of an environmentally beneficial fuel produced from domestic, renewable resources whose chemical structure is a mixture of long chain fatty acid methyl esters (FAME). Biodiesel can be blended at any level with petroleum diesel to create a biodiesel blend. It can be used in compression-ignition (diesel) engines with no major modifications. Biodiesel is simple to use, biodegradable, nontoxic, and essentially free of sulfur and aromatics. (Note: The term 'biodiesel' is often confusing. Strictly speaking, biodiesel refers to the pure fatty acid methyl esters (FAME). However, the term biodiesel is sometimes used to describe blended fuel mixtures containing both FAME and petroleum diesel.)

Can I use biodiesel in my car?

Biodiesel is safe to use in any vehicle or machine that is equipped with a diesel (compression ignition) engine. Cars which use gasoline (spark ignition) cannot burn diesel fuel or biodiesel.

Is biodiesel used as a pure fuel or is it blended with petroleum diesel?

Biodiesel can be used as a pure fuel or blended with petroleum in any percentage. Biodiesel usually costs more than petroleum diesel (depending on the price of petroleum)! The most common blend is called B20 (a blend of 20 percent by volume biodiesel with 80 percent petroleum diesel). Blends at this level have demonstrated significant environmental benefits with a minimal increase in cost to consumers.

Is it approved for use in the US?

Biodiesel is registered as a fuel and fuel additive with the Environmental Protection Agency (EPA) and meets clean diesel standards established by the California Air Resources Board (CARB). B100 (100 percent biodiesel) has been designated as an alternative fuel by the Department of Energy (DOE) and the US Department of Transportation (DOT). Most engine manufacturers have certified their engines/fuel system for biofuels blends up to B20. While higher level biodiesel blends can be used, these may cause rubber hoses/seals to soften so manufacturers do not warrant their systems for levels higher than B20.

How do biodiesel emissions compare to petroleum diesel?

Biodiesel is the first alternative fuel to have fully completed the health effects testing requirements of the Clean Air Act. The use of biodiesel in a conventional diesel engine results in substantial reduction in many pollutants.

Some of the main pollutants typically considered are:

- Particulate Matter (Black Smoke) – Emissions of particulate matter (PM) have been linked to respiratory diseases and are generally considered to be a human health hazard.
- Carbon Monoxide (CO) – Carbon Monoxide is a poisonous gas.
- Unburned Hydrocarbons (CxHy) – Compounds which contribute to localized formation of smog.
- Nitrogen Oxides (NO_x) – Compounds which contribute to localized formation of smog.
- Sulfur Oxides – Compounds which are major contributors to forming acid rain.
- Polycyclic Aromatic Hydrocarbons (PAH and nPAH) – These compounds have been identified as carcinogenic (cancer causing) compounds.



Of the major exhaust pollutants, both unburned hydrocarbons and nitrogen oxides are ozone or smog forming precursors. The use of biodiesel results in a substantial reduction of unburned hydrocarbons and particulate matter but emissions of nitrogen oxides are either slightly reduced or increased depending on the duty cycle of the engine and testing methods used. (Note: Recent research on diesel engine operation using biofuels blends has demonstrated that any increases in NO_x formation can be eliminated by controlling diesel engine combustion operations). Based on engine testing, using the most stringent emissions testing required by EPA for certification of fuels or fuel additives in the US, the overall ozone forming potential of biodiesel emissions was nearly 50 percent less than that measured for diesel fuel. In addition, the exhaust emissions of sulfur oxides (major components of forming acid rain) from biodiesel are essentially eliminated compared to diesel.

Can biodiesel help reduce “global warming”?

A 1998 biodiesel lifecycle study, jointly sponsored by the US Department of Energy and the US Department of Agriculture, concluded biodiesel reduces net carbon dioxide emissions by about 80 percent compared to petroleum diesel.¹ This is due to biodiesel’s closed carbon cycle. The CO_2 released into the atmosphere when biodiesel is burned is used in the process of photosynthesis required for growing plants. The plants are later processed into fuel. Figure 1.1 depicts the CO_2 life-cycle which involves plant growth, followed by processing of the crop to produce food as well as fuel. The fuel is then used for transportation (and other applications) and CO_2 is released as a product of the fuel burning in engines.

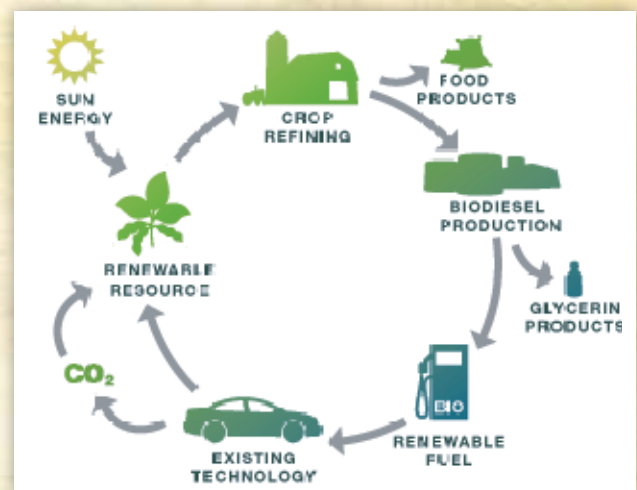


Figure 1.1 CO_2 life-cycle analysis.

Is biodiesel better for human health than petroleum diesel?

A comprehensive health effects testing program was conducted by Southwest Research Institute and Lovelace Respiratory Research Institute to ensure benefits of biodiesel to the environment and to public health. Results of the health effects testing concluded that biodiesel is non-toxic and biodegradable, posing no threat to human health. Also among the findings of biodiesel emissions compared to petroleum diesel emissions in this testing:

- The ozone forming potential of hydrocarbon exhaust emissions from biodiesel is 50 percent less.
- The exhaust emissions of carbon monoxide (a poisonous gas and a contributing factor in the localized formation of smog and ozone) from biodiesel are 50 percent lower.
- The exhaust emissions of particulate matter (recognized as a contributing factor in respiratory disease) from biodiesel are 30 percent lower.

¹ Information from <http://www.nrel.gov/docs/legosti/fy98/24089.pdf>



FREQUENTLY ASKED QUESTIONS ABOUT BIODIESEL (continued)

- The exhaust emissions of sulfur oxides (major components of forming acid rain) from biodiesel are completely eliminated.
- The exhaust emissions of hydrocarbons (a contributing factor in the localized formation of smog and ozone) are 95 percent lower.
- The exhaust emissions of aromatic compounds known as PAH and NPAH compounds (suspected of causing cancer) are substantially reduced for biodiesel compared to diesel. Most PAH compounds were reduced by 75 percent to 85 percent. All NPAH compounds were reduced by at least 90 percent.

Depending on speed and load, the nitrogen oxides (NO_x) emissions for biodiesel combustion could be higher or lower than that of diesel. However, some studies have shown increases in NO_x emission of up to 40 percent for biodiesel compared to diesel. This is undesirable since NO_x is a precursor to smog, which is a type of air pollution that could cause respiratory disorders. There are however ways to reduce the NO_x emissions produced by biodiesel combustion, which will be discussed later in this chapter.

Does biodiesel cost more than other fuels?

The cost of all fuels change every day, due to commodity market price fluctuations. The relative cost of biodiesel vs. petroleum diesel will change due to changes in the prices of petroleum and natural oils (such as soybean oil). Historically, the cost of biodiesel has been slightly higher than petroleum diesel, but this may change if petroleum prices increase. When reviewing the high costs associated with other alternative fuel systems, many large truck fleet managers have determined biodiesel is their least-cost-strategy to comply with state and federal regulations. Use of biodiesel does not require major engine modifications and provides substantial environmental benefits. That means operators keep their fleets, their spare parts inventories, their refueling stations and their skilled mechanics. The only thing that changes is improved environmental quality.

Where can I purchase biodiesel?

Biodiesel is available anywhere in the US. The National Biodiesel Board (NBB) maintains a list of registered fuel suppliers. A current list is available on the biodiesel web site at www.biodiesel.org or by calling the NBB's toll free number at (800) 841-5849.

Who can answer my questions about biodiesel?

NBB maintains the largest library of biodiesel information in the US. Information can be requested by visiting the biodiesel web site at www.biodiesel.org, by emailing the NBB at info@biodiesel.org, or by calling the NBB at (800) 841-5849.

THE DIESEL ENGINE AND THE DEVELOPMENT OF BIODIESEL

Name _____ Class _____ Date _____

THE HISTORY OF THE DIESEL ENGINE

In the early 1890s, a German inventor named Rudolph Diesel invented and patented a new design of what he called an “improved heat engine.” The diesel engine is an internal combustion engine which operates on a four stroke cycle. The four strokes are intake, compression, power and exhaust.

1) Intake stroke

In the intake stroke the intake valve opens and fresh air (containing no fuel) is drawn into the cylinder as the piston moves down.

2) Compression stroke

The intake valve closes and the piston rises during the compression stroke. The air in the engine is compressed, causing its temperature to rise.

3) Power stroke

At the end of the compression stroke, the air is hot enough to ignite fuel. When the piston is near the top of the cylinder, the fuel injector drives fuel into the cylinder. The fuel immediately ignites upon contact with the hot compressed air. As the fuel burns, the gas in the cylinder heats and expands, driving the piston downwards during the power stroke.

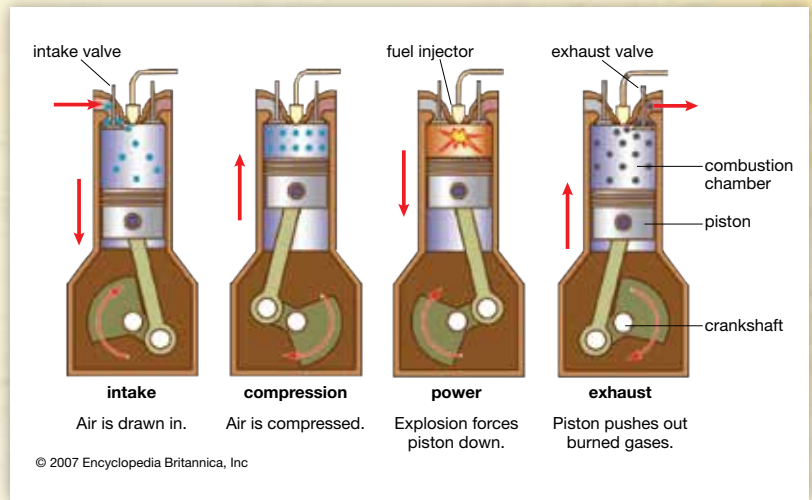


Figure 1.2 Four stroke cycle of a diesel engine.

4) Exhaust stroke

In the last stroke, the exhaust valve opens, and the exhaust is driven out of the cylinder.

Each stroke corresponds to the piston moving up or down once. The linear (up and down) motion of the piston causes the crankshaft to rotate, which leads to rotation of the wheels of the vehicle.

Diesel designed his engine to run on a very wide variety of fuels, including vegetable oils. In fact, when he demonstrated his engine at an exhibition fair in Paris in 1903, he ran it on peanut oil. Diesel envisioned that his engine would someday replace the large and inefficient steam engines of the day and utilize low cost and locally produced fuels, such as vegetable oils. The Diesel engine did go on to replace steam engines in many applications, and by the 1950s had nearly completely replaced the steam engine in railway locomotives, power generation, construction machinery and agricultural equipment. However, the engine being used in the 1950s bore little resemblance to the one Diesel invented in 1893.



By the time of Diesel's death in 1913, the fledgling petroleum industry had begun to produce what was referred to as middle distillate fuel, which was a by-product of the production of gasoline from crude oil. Engineers found that this fuel, while unsuitable for gasoline engines, burned well in the compression ignition design of Diesel's engine. Eventually, the Diesel engine was redesigned to run on this inexpensive fuel, which was then named Diesel fuel. Running on low cost petroleum, the efficient and rugged Diesel engines began to make inroads into the automobile and truck industries in the 1930s and 1940s. After World War II, the Diesel engine began to replace gasoline engines in many applications that required a long life and efficient power. The diesel engine continued to proliferate and underwent many redesigns to make it lighter, more efficient, and less expensive. Today's modern diesels are more efficient and last longer than comparable gasoline engines, and are used in everything from light commuter cars to the world's largest cargo ships and mining equipment.

The Oil Crisis and Pollution Concerns

As oil fields in North America and Europe dried up in the 1950s, the world's oil supply shifted to the Middle East and offshore drilling to supply the large amounts of crude oil needed to supply the gasoline and diesel fuel that ran the world's economy.

In 1961 the largest oil producing nations in the Middle East banded together to form the Organization of Petroleum Exporting Countries, or OPEC. This new organization allowed the countries to control production and consequently the price of crude oil. In 1973 and again in 1978, OPEC drastically cut production and the price of oil sky rocketed, sending world economies into a crisis. This created grave concerns over the stability of foreign oil supplies.

In addition to problems with supply, many scientists were becoming concerned with the emissions from cars running on petroleum fuel, which lead to the accumulation of so-called "greenhouse gases." These emissions trap solar energy in the earth's atmosphere and are thought to lead to global climate change. In addition, it was found that many emissions from petroleum-fueled engines had toxic effects on animal and plant life.

The Rediscovery of Soybean Oil-based Fuels

Realizing the problem of dependency on foreign oil and the growing environmental problems from the combustion of petroleum, engineers and scientists worked to develop alternatives to using petroleum in transportation fuels. Soybean oil presented an immediate draw in the US since it is the most widely produced vegetable oil. Early work showed that vegetable oils provided nearly the same amount of power as diesel fuel, and actually reduced several harmful emissions. However, long term testing resulted in the engines developing carbon and "gum" deposits. The problem was that the vegetable oil was much more viscous than petroleum diesel fuel for which modern diesel engines were designed. Viscosity is a measure of the resistance to flow of liquids. Think of pouring maple syrup and water from the same container. The maple syrup is much more viscous, more resistant to flow, than the

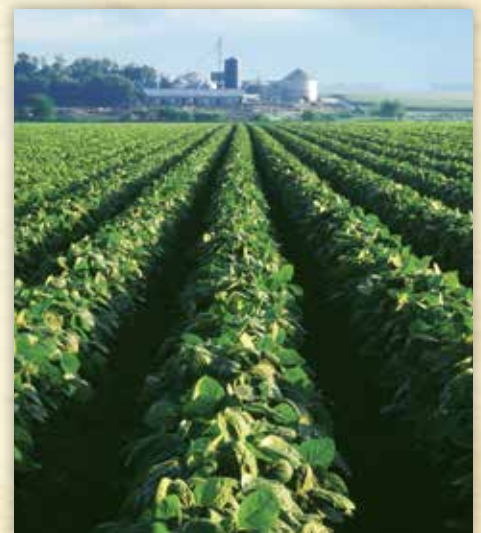


Figure 1.3 Soybean field

water. In this same manner, vegetable oils are much more viscous than petroleum diesel. It was found that the diesel engine's injection system, which is responsible for spraying very small droplets of fuel into the combustion chamber, could not properly spray the viscous vegetable oils. The larger vegetable oil droplets would not burn completely, and as a result would deposit carbon on the injectors and valves of the engine, eventually leading to engine failure. It was determined that it was necessary to reduce the viscosity of vegetable oils if they would be successful as diesel fuel replacements.

The Chemistry of Biodiesel

Biodiesel (fatty acid methyl esters, FAME) can be created from soybean and other vegetable/animal fats/oils. [Note: Fats and oils have the same basic chemical structure. The difference in the nomenclature is that fats are generally considered solids at room temperature whereas oils are liquids at room temperature.]

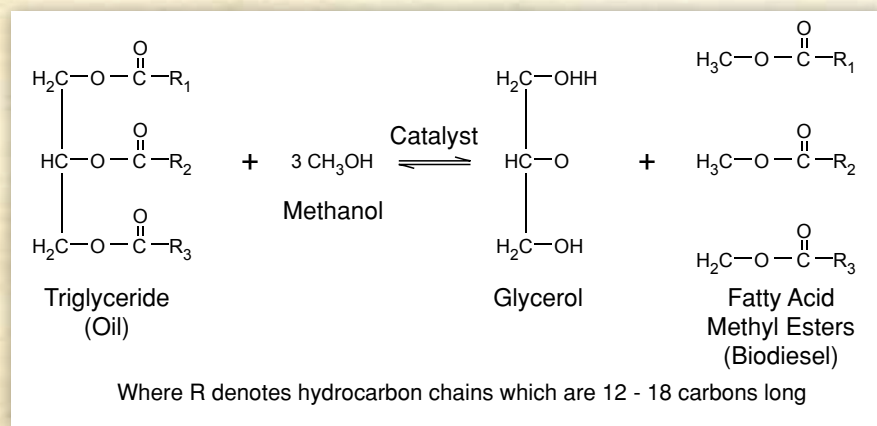
Fats/oils are called triacylglycerides or for short, triglycerides. A triglyceride is composed of 3 long chain fatty acid molecules attached to a glycerol (or glycerin) molecule (see Fig. 1.4 below). The long chain fatty acid molecule is the $-OOC-R$ structure, where R = an alkane/alkene hydrocarbon chain (C10-20). Since there are 3 fatty acids per triglyceride, the R groups may be different for each fatty acid.

Most soybean and plant oils (approx. C50-60) are much larger molecules than gasoline or diesel fuel (C6-24). In an attempt to make vegetable oil more like petroleum diesel, scientists and engineers began to examine a process commercialized in the 1940s known as transesterification, which used alcohol and a catalyst to break down triglycerides into smaller molecules known as fatty acid methyl esters (FAME).

The transesterification reaction is represented in Figure 1.4. FAMES made from soybean and other domestic oils consist of carbon chains of lengths in the range of 12-20 and are commonly called biodiesel.

[Note: The term 'biodiesel' is often confusing. Strictly speaking, biodiesel refers to the pure fatty acid methyl esters.] However, the term biodiesel is sometimes used to describe blended fuel mixtures containing both FAME and petroleum diesel. Due to similar molecular size and structure, the fatty acid methyl esters have chemical and physical properties similar to petroleum diesel fuels.

Transesterification is the process of transferring one part of an ester molecule to another molecule, usually an alcohol. (An ester is a molecule that contains a carboxylate structure, $-OOC-$.) In the case of the biodiesel



reaction, the fatty acid groups (which contain the ester structure) are transferred to methanol, a one carbon alcohol, CH_3OH . This results in the formation of 3 long chain fatty acid methyl esters (FAME) and 1 molecule of glycerol for each fat/oil molecule reacted (see Fig. 1.4). The transesterification reaction is represented in Figure 1.4.

Figure 1.4 Chemical symbol representation of the transesterification reaction



The Benefits of Biodiesel Fuel

It was quickly found that biodiesel produced from soybean and other oils combust very well in diesel engines. Biodiesel also reduces emissions of several harmful compounds. Compared to petroleum diesel, biodiesel reduces emissions of carbon monoxide, unburned hydrocarbons, and particulate emissions. In addition, since biodiesel is made from soybeans and other crops which use carbon dioxide in their growth cycle, biodiesel reduces the life cycle emissions of carbon dioxide when compared with petroleum diesel. This can decrease the concentration of carbon dioxide ('greenhouse gases') in the atmosphere.

Besides reducing many harmful emissions, biodiesel is much more environmentally benign than petroleum diesel fuel. It is biodegradable and far less toxic to humans, animals and aquatic life than petroleum diesel fuel. Due to these properties, biodiesel is ideal for use in agricultural, logging and construction equipment, and marine vessels, where fuel spills present hazards to animals and the environment.

Another desirable property of biodiesel is its very high flash point. Flash point is the temperature at which a fuel will ignite when exposed to spark or flame. Biodiesel has a much higher flash point than petroleum diesel, making it safer to transport, handle, store and use than conventional petroleum diesel fuel.

Biodiesel Feedstocks

Biodiesel is produced throughout the world from a wide variety of starting materials. In the U.S. soybean oil is the major plant oil, hence most of the biodiesel made in the U.S. comes from soybeans. With the proper production technology, almost any fat or oil can be used to produce biodiesel certified to industry required specifications. Other examples of feedstock used include:

- Oils such as canola, peanut, cottonseed, camelina, corn and palm
- Animal fats such as beef tallow, poultry fat, pork lard, and fish oil
- Used greases such as used cooking oil, waste bakery fat, trap grease, and sewer grease

The main difference in these fuels has to do with their solid/liquid characteristics. Biodiesel fuels that are made from liquid oils (e.g. soybean, canola) will have a lower freezing point than biodiesel fuels made from solid oils/fats (tallow, pork, palm). In addition to these feedstocks, there is a significant amount of research being done by industry and government on creating fuels from fast growing algae, which utilize sunlight and carbon dioxide to create oils. All of these feedstocks are renewable, meaning they utilize energy from the sun, nutrients/carbon dioxide, and water to produce the oils or fats. This allows them to be more environmentally beneficial vs. non-renewable petroleum usage.



Figure 1.5 Integrity Biofuels – Central Indiana's first soy biodiesel fuel production facility



The Biodiesel Industry

The production of biodiesel has become an established and growing alternative fuel industry worldwide. In the United States, the biodiesel industry has grown to an industry with over 130 production plants in 39 states, with an annual capacity of over 1.5 billion gallons. This industry is estimated to employ over 50,000 people in a wide range of skilled, high paying jobs. Examples of biodiesel careers include:

- Chemical and Mechanical Engineers (Biodiesel plant design, construction and operation)
- Operators and Technicians (Biodiesel plant operation and maintenance)
- Chemists and Scientists (Fuel quality testing and new feedstock research)
- Fabricators and Contractors (New biodiesel plant construction and expansion)
- Truck drivers (transport of feedstock, biodiesel, byproducts, chemicals)
- Farm related employment (production, harvesting and processing of oilseeds)



Figure 1.5a Sequential Pacific Biodiesel in Salem, OR is an example of a diversified biodiesel plant which utilizes soybean oil, canola oil, and waste cooking oil to produce over 5 million gallons of ASTM specification biodiesel each year. This fuel is blended with petroleum and sold in Oregon, Washington, Idaho, and California.



Figure 1.5b Louis Dreyfus Biodiesel plant, located in Claypool, Indiana, is the largest integrated soybean/biodiesel processing plant in North America. The annual capacity for biodiesel production is 88 million gallons/year. This is a BQ9000 certified plant, which ensures high quality product/processing at each step in the manufacturing process.



The Challenges of Biodiesel Fuel

While biodiesel does have many advantages, there are several challenges that must be overcome when running an engine on biodiesel. First, biodiesel has a slightly lower energy content than petroleum diesel. This means that in order to get the same amount of power (or torque) from a diesel engine, more fuel will be required when using biodiesel.

In addition, emissions of nitrogen oxides or NO_x can increase when using biodiesel. Nitrogen oxides are smog producing emissions and are strictly regulated by the Environmental Protection Agency (EPA). Figure 1.6 shows how most emissions of carbon monoxide (CO), particulate matter (PM), and hydrocarbons (C_xH_y) decrease as the percentage of biodiesel increases but the NO_x level increases. In fact, more recent studies on modern engines have reported even higher NO_x increases of over 40 percent when using 100 percent biodiesel. (Note: Recent research on diesel engine operation using biofuels blends has demonstrated that any increases in NO_x formation can be eliminated by controlling diesel engine combustion operations)

Biodiesel also has a higher cloud point, which means that the fuel will begin to gel when it gets too cold. In order for biodiesel to be used in cold weather, additional processing must be done to the fuel or additives must be used. Currently, biodiesel is commonly blended with diesel fuel in blends containing up to 20 percent biodiesel. With these lower biodiesel blends, gelling and emissions problems are less severe.

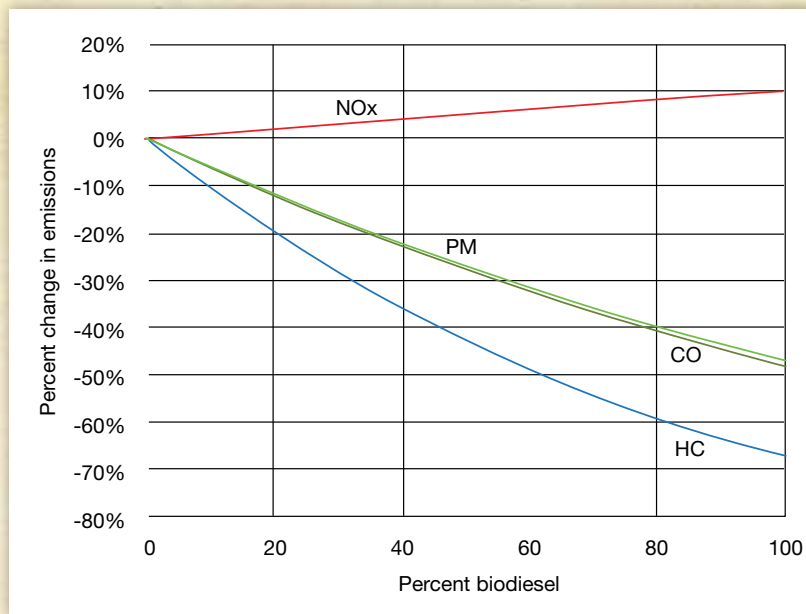


Figure 1.6 Average impact of biodiesel blends on emissions from pre-1998 heavy-duty n-highway engines in 2002 EPA study.



TRANSESTERIFICATION OF SOYBEAN OIL TO FATTY ACID METHYL ESTERS





TEACHERS' BACKGROUND MATERIAL

In this lesson, the students will convert soybean oil into Fatty Acid Methyl Esters (FAME) using methanol and potassium hydroxide as a catalyst. This lab is designed to be performed over two partial class periods. In the first class period, the students will measure out the reagents, prepare the catalyst alcohol mixture and begin the reaction on magnetic stirring plates. After a minimum 4-hour reaction period and an 8-hour settling period, the students will decant the glycerin by-product and perform mass balance and molar calculations on the reaction.

BACKGROUND OF THE REACTION

In this reaction, triglycerides composed of a glycerol molecule and 3 fatty acid molecules are converted to mono esters of a lower alcohol, in this case methanol. The reaction is reversible, and is most easily catalyzed with strong base catalysts such as potassium hydroxide. Acid catalysts have also been used, however the reaction rates are significantly slower and a high conversion is difficult without elevated temperatures and pressures. A schematic representation of the reaction is shown in Figure 2.1.

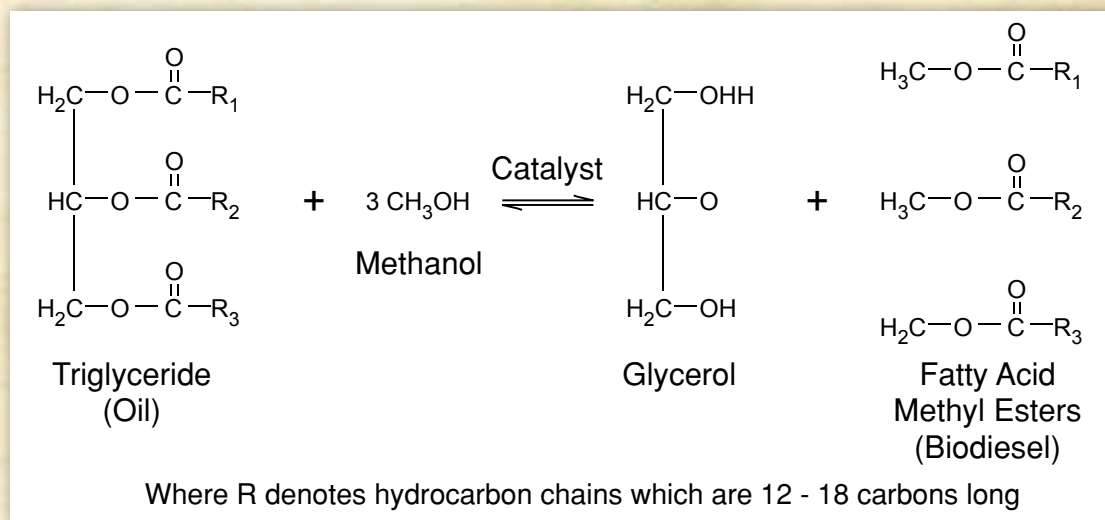


Figure 2.1 Transesterification reaction

In practice, the transesterification is driven to the right by using excess methanol, typically 6:1 molar ratio methanol to oil, representing twice the required molar ratio. The catalyst and alcohol are premixed to avoid the formation of soaps which can occur when adding the base catalyst directly to the neutral oil. The reaction can be carried out at room temperature or at elevated temperatures, with faster conversion occurring at reaction temperatures close to the boiling point of methanol. However, in this experiment, the reaction will be carried out at room temperature to increase safety and avoid the need for fume containment or recovery equipment.

Significant conversion (approx. 50 percent) occurs within the first ten minutes of agitation of the methanol/catalyst and triglycerides phases, although higher conversion takes significantly longer (4+ hours). When agitation is ceased, the insoluble glycerol by-product will begin to settle to the bottom of the reaction vessel. If the reaction proceeded properly, this settling should be complete after 8 hours, giving a clear phase separation.



LESSON 2

The following pages provide a more substantial background on the organic chemistry behind vegetable oils, their structure and conversion to biodiesel. It is designed as a reference for the instructor to create lecture material around the lab experiment.

THE CHEMISTRY OF VEGETABLE OILS AND BIODIESEL

Plants absorb energy from the sun using it to drive biochemical reactions that synthesize fats/oils. Fats/oils are energy dense forms of chemical energy which are used by plants during seed germination and by mammals as a metabolic energy fuel. This kind of renewable resource production is a critical source of energy and raw materials for the future production of fuels and materials to replace current petroleum-based products.

The terms fat and oil refer to triglycerides, which are 3 fatty acids linked to a glycerine/glycerol molecule.

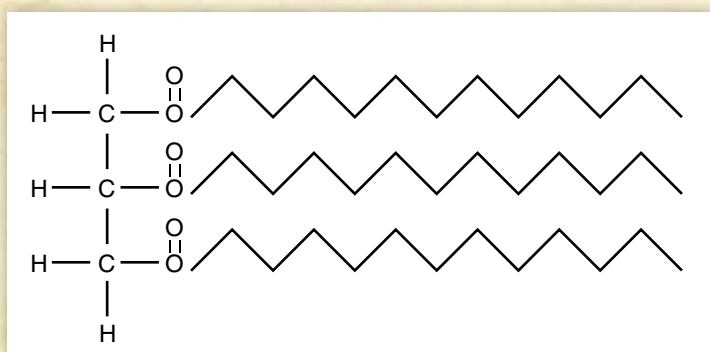


Figure 2.2 Chemical structure of triglyceride

The 3 carbon chain above is called a glyceride, based on its original tri-alcohol structure called glycerine or glycerol (see below).

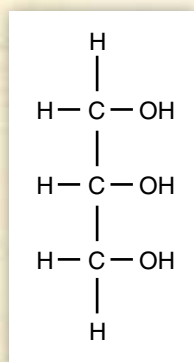


Figure 2.3 Chemical structure of glycerol

Other synonyms of glycerin are glycerine; 1,2,3-propanetriol; glycerol; 1,2,3-trihydroxypropane; glycol alcohol; trihydroxypropane; and propanetriol.



Fatty esters can be graphically represented in several ways. The angled lines refer to a hydrocarbon structure composed of carbon bonds with attached hydrogen atoms.

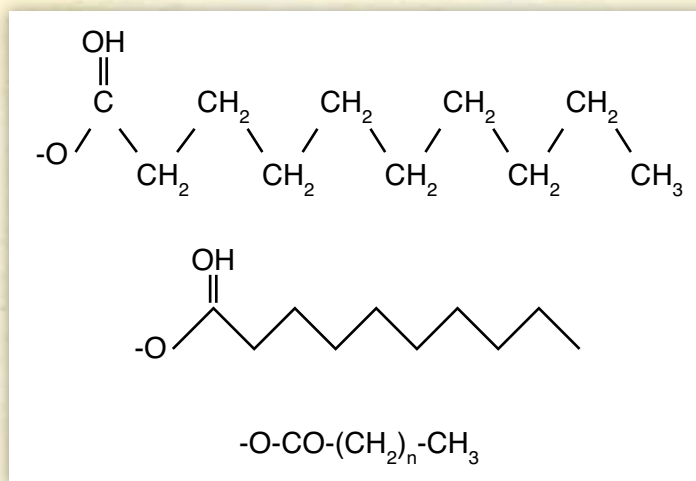


Figure 2.4 Chemical structures of fatty acid ester

The chemical bond between the hydrocarbon chain and the glycerol molecule is called an ester linkage and has the chemical structure below, where R1 and R2 refer to any attached chemical structures.

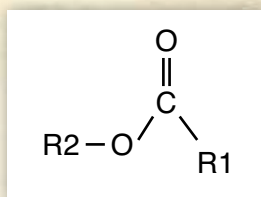


Figure 2.5 Chemical structure of an ester

The ester group is also called a carboxylate. If R2 is a hydrogen atom, this is called a carboxylic acid since the hydrogen can be dissociated as an acidic proton. Reactions where the R2 group is exchanged for another group (such as methanol replacing glycerol), the reaction is called a transesterification or a transfer of the ester group from the glycerol to the methanol.

Nomenclature of esters is based on the 2 groups attached to the ester carboxylate structure. For example, H-O-CO-CH_3 is called acetic acid and is a component of vinegar. If the hydrogen atom (R2) is exchanged with methanol, $\text{CH}_3\text{-O-CO-CH}_3$, this is called methyl acetate. The R1 group determines the carboxylate structure name. For example if the R1 group is a 16 chain hydrocarbon, this becomes a methyl palmitate.



LESSON 2

Vegetable Oil Background

The most common esters found in nature are fats and vegetable oils, which are esters of glycerol and fatty acids. Traditional domesticated plants producing high levels of oils are soybean, sunflower, palm and coconut. Due to the similarity of chemical structure to petroleum, both are called 'oil' and have similar physical properties, such as viscosities, lubricity, density and energy content. Vegetable oils were the first fuels used in the development of compression ignition engines by Rudolph Diesel in the 1890s, initially designed to run on peanut oil. The subsequent discovery of abundant supplies of petroleum fossil fuels along with global demand for cheap fluid energy supplies led to the growth of the petrochemical industry in the mid-1900s. Interestingly, in a 1912 speech, Rudolf Diesel is quoted as having 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." Nearly a century later, his vision is becoming reality.

In the latter half of the last century, the environmental effects of the combustion of fossil fuels were realized along with the recognition of their limitations as a non-renewable resource. On a global basis, billions of pounds of carbon are added to the atmosphere annually, which are believed to be possibly responsible for changes in the environment. Additionally, there are many current political and economic issues surrounding the use of petroleum, based on foreign suppliers and decreasing availability/increasing costs. For these reasons, there is a significant amount of attention toward developing alternative energy resources. Of particular interest are plant oils, since they can be used in existing mobile liquid fuel applications with minimal or no changes in fuel delivery infrastructure and engine design, in addition to being completely renewable.

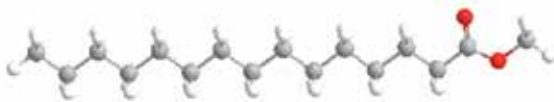
Note that triglycerides can also be obtained from animal sources (fats) such as tallow (beef fat), ghee (butter fat), lard (pork fat), chicken fat, blubber (whale/fish fat) and cod liver oil. The main differences in these oils/fats are the types of fatty acid chains. Triglycerides contain 3 fatty acids attached to a glycerol molecule via ester linkages. There are generally different fatty acids on each position, producing a heterogeneous triglyceride. Common types of fatty acids found in domestic plant and animal triglycerides are:

palmitic acid	$C_{16}H_{32}O_2$ (saturated 16:0)
stearic acid	$C_{18}H_{36}O_2$ (saturated 18:0)
oleic acid	$C_{18}H_{34}O_2$ (1 double bond 18:1)
linoleic acid	$C_{18}H_{32}O_2$ (2 double bonds 18:2)
linolenic acid	$C_{18}H_{30}O_2$ (3 double bonds 18:3)
erucic acid	$C_{22}H_{42}O_2$ (saturated 22:1)

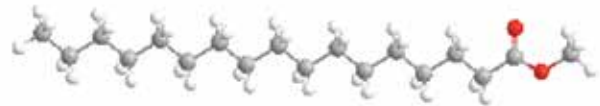


The term saturated means that there are no carbon-carbon double bonds, so all the carbons are “saturated” with hydrogen atoms (alkane structures). Myristic (14:0), palmitic (16:0), and stearic (18:0) are all saturated fatty acids. Conversely, unsaturated means that there are one or more carbon-carbon double bonds present (alkene structures). Oleic acid (18:1) is a monounsaturate, and linoleic (18:2) and linolenic (18:3) are polyunsaturates (di- and triunsaturates).

The physical molecular shape of the different fatty acid methyl esters changes with the presence of carbon double bonds. The normal single carbon bond angle is 109.5° . For saturated fatty acid esters, there are no carbon double bonds.

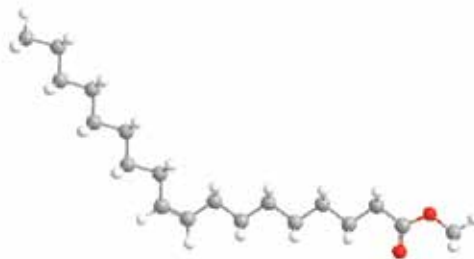


Methyl palmitate (16:0)

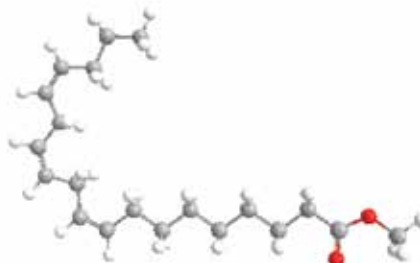


Methyl stearate (18:0)

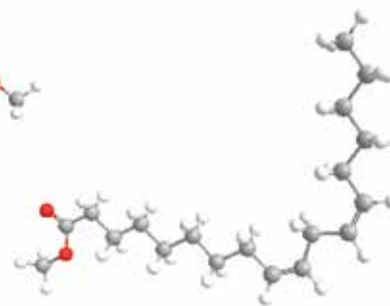
For unsaturated fatty acid methyl esters, the molecular structure contains carbon double bonds, which have slightly larger bond angles.



Methyl oleate (C18:1)



Methyl linoleate (18:3)



Methyl linoleate (18:2)



LESSON 2

The structure/composition of the fatty acid methyl esters strongly affects their physical properties. Longer carbon chains generally tend to crystallize at lower temperatures. Linear saturated (alkane type) fatty acid esters tend to crystallize much more easily than non-linear unsaturated (alkene type) esters. So the saturated fatty acid esters tend to crystallize/solidify at much higher temperatures than unsaturated fatty acid esters. See table below where x:n refers to the carbon chain length (x) and number of carbon double bonds (n). You will probably recognize that oils from beef, palm, and butter (which contain more saturated fatty acids) are solids at room temperature, whereas sources such as soybean, olive, and canola (which contain more unsaturated fatty acids) are liquids at room temperature.

SOURCE	SATURATED					UNSATURATED			
	10:0	12:0	14:0	16:0	18:0	18:1	18:2	18:3	20:n
Coconut	7	48	17	8	3	6	2		
Palm			1	46	5	39	9	1	
Soybean				11	4	22	54	8	
Olive			1	15	3	67	13	1	
Canola				3	5	45	27	9	
Beef			3	16	22	42	2	0.2	
Butter	6	6	11	26	10	28	2	1	
Jatropha				16	7	42	33	1	
Fish	6		16		3	5	2	1	14
Melting Temp. (°C)		+6	+18	+31	+39	-19	-35	-57	

Figure 2.6 Fatty Acid composition of Different Fat sources and methyl ester melting points (from Bailey's Industrial Oil and Fat Products, 5th ed.) Values shaded in pink are saturated fatty acids. Values shaded in green are unsaturated fatty acids.

Soybean Methyl Ester Formula and Molecular Weight

The average molecular weight of soybean oil methyl esters is 292.2. This was calculated using the average fatty acid distribution for soybean methyl esters below. Also below are the molecular weight and chemical formula for each of the component esters. The scientific names for the methyl esters are based on the fatty acid carboxylate structure. For example the formal name of the saturated 16 carbon chain methyl ester ($C_{15}H_{31}CO_2CH_3$) is hexadecanoic acid methyl ester. Common names have been given to these fatty acid methyl esters as listed below.

Fatty Acid	Weight %	Molecular weight	Chemical formula
Palmitic	11	270.46	$C_{15}H_{31}COO_2CH_3$
Stearic	4	298.52	$C_{17}H_{35}COO_2CH_3$
Oleic	22	296.50	$C_{17}H_{33}COO_2CH_3$
Linoleic	54	294.48	$CH_3(CH_2)_4CH=CHCH_2CH=CH(CH_2)_7COO_2CH_3$
Linolenic	8	292.46	$CO_2CH_3CH_3(CH_2CH=CH)_3(CH_2)_7COO_2CH_3$

Figure 2.7 Typical Soybean Oil Methyl Ester Fatty acid profile.



Biodiesel Fuel

Vegetable oil is converted into biodiesel fuel (fatty acid methyl esters or FAME) by transesterification. The three ester bonds within a triglyceride are transesterified to transfer the fatty acids from the glycerol molecule to an alcohol (usually methanol or ethanol) to form methyl or ethyl fatty acid esters.

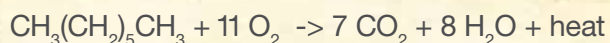
FAME is less viscous and more volatile than triglycerides. FAMEs are also a much better solvent for various synthetic plastics, such as polystyrene.

The process of transesterification basically makes an aliphatic hydrocarbon chain with a carboxylic acid group at one end. The presence of the carboxylic acid terminal allows living organisms to metabolize the normally inert hydrocarbon chain, for example, it makes the molecule biodegradable.

Petroleum Fuel

Fossil fuels, such as gasoline and diesel, are basically linear hydrocarbon chains (carbon atoms linearly linked together), each of which has two hydrogen atoms attached with terminal methyl groups (CH_3 - $(\text{CH}_2)_n$ - CH_3). For example, if there are 8 carbons in the chain, this molecule is called octane. Petroleum is generally a mixture of molecules that have different chain lengths, may contain carbon-carbon double bonds, and may be branched or cyclic. They release considerable amounts of energy when oxidized, a process known as combustion or oxidation (reaction with oxygen).

Combustion is the main energy process used in modern automotive engines. During the combustion process, each carbon and hydrogen atom reacts with oxygen atoms making water (H_2O) and carbon dioxide (CO_2) and release heat energy. For example, when heptane is completely oxidized the reaction is written as:



This states that every molecule (or mole) of heptane oxidized requires 11 molecules (moles) of oxygen (O_2) and produces 7 molecules (moles) of carbon dioxide (CO_2) and 8 molecules (moles) of water (H_2O) plus thermal energy. The energy released by the oxidation reaction is converted into thermal energy of the molecules, which raises their temperature and pressure. The chemical reaction of oxidation of the reactant liquid fuel also results in gaseous carbon dioxide and water products, which have a much greater volume than the original liquid fuel. So if this reaction is conducted in a piston-cylinder (as in a combustion engine, see 4 stroke engine diagram) the increase in gaseous volume causes the piston to move, which results in mechanical motion.

[Note: A mole is defined as 6.023×10^{23} molecules of a substance. Since different substances are made of different compositions of atoms, it is convenient to define moles in terms of the total atomic mass of the molecule. Hence, the molecular weight is the number of mass units in 1 mass unit-mole of a substance, e.g. 1 kg-mole of carbon dioxide (molecular weight $12 + 2 \times 16 = 44$) is equal to 44 kg of carbon dioxide. Alternatively, 1 lb-mole of carbon dioxide is 44 lbs of carbon dioxide (different weight unit). See <http://library.thinkquest.org/12497/molar.htm> if needed]

Since methyl esters are very similar to hydrocarbons in chemical structure, they can also be oxidized in similar fashion. Information on calculating the amount of energy released by oxidation is provided in the appendices.



LESSON 2

Scheduling this Lab Experiment

Due to the long reaction and settling times, careful scheduling of this lab should be conducted to ensure that both reaction and settling can occur by the next time the class meets. During the first session, the students will begin the reaction. In the following session, they will decant the glycerol layer and proceed with the analysis. For example, this will require the instructor to stop the reaction (by turning off the stir plates) either at the end of the day if the class takes place in the morning; or by stopping the reaction the following morning if the class takes place later in the afternoon. The reaction should be allowed to proceed for a minimum of 4 hours, and the settling a minimum of 8 hours. Longer reaction and settling times do not negatively affect the reaction.

Suggestions:

If the lesson is being taught in an organic chemistry course, it may be helpful to illustrate:

- The structure of the triglyceride molecule and a comparison with straight chain hydrocarbons
- Similarities between fatty acids and petroleum hydrocarbon chains
- The definition of an ester linkage and its role in the triglyceride
- The properties of esters and their role in the natural world (i.e. pentyl acetate and the smell of oranges, ethyl acetate and the smell of bananas, etc.)

If the lesson is being taught in an earth and atmospheric science course, it may be helpful to illustrate:

- The similarities between fatty acid/ester chains and linear petroleum hydrocarbons
- The carbon cycle, specifically carbon loading in the atmosphere due to petroleum combustion
- The carbon cycle as it relates to the growth and death cycle of plants
- The integration of using plant-based fuels to reduce atmospheric carbon loading
- The environmental impact oil spills and how biodiesel and other compound can reduce these effects

If the lesson is being taught as a general chemistry class teaching analytical skills, it may be useful to include:

- A discussion and calculation of the molar weight for esters and triglycerides
- The role of limiting reagents and driving a reaction using excess reagents
- Accuracy and precision in measurement
- A discussion of the role of catalysts



Lab Protocol and Instructions

IMPORTANT:

The FAME made in this experiment WILL NOT meet ASTM specifications for biodiesel. The FAME from this reaction will be “Crude” and contain significant amounts of catalyst, methanol, and un-reacted material which can damage a diesel engine. ASTM specification FAME undergoes extensive purification steps post reaction in order to provide a safe and clean biodiesel product. Typically, these purification steps include neutralization, liquid extraction or washing with a suitable solvent and/or distillation of the FAME under heavy vacuum. UNDER NO CIRCUMSTANCES SHOULD THE CRUDE ESTER FROM THIS EXPERIMENT BE USED IN A DIESEL ENGINE.

General Safety and Preparation Suggestions:

This lab involves the use of methanol, as well as potassium hydroxide, both of which present potential hazards to the students. Follow the standard protocol for safety and handling of these reagents in the laboratory.

Reagents

Food grade soybean oil (commonly sold as vegetable oil), available from any grocer or kitchen supplier may be used in the experiment.

Methanol must be technical grade (99 percent minimum purity). Presence of water or other contaminants will cause destruction of the catalyst and reaction failure.

Potassium Hydroxide must be technical grade as well, and free of any moisture. It should be of pellet or powder form, suitable for division to 0.05 grams.

Equipment

The equipment provided in this kit is intended for student groups of two.

This kit will provide the 250 ml reaction vessels (polyethylene bottom draining bottles) for use in the lab. These vessels will allow the students to easily decant the separated glycerol after the reaction has been completed, as well as allowing for visual inspection of the phase structure.

All other materials listed on the first page of the student handout should be provided by the instructor (graduated cylinders and electronic balances need not be provided for each group).

IMPORTANT: FAME will eventually permeate polyethylene of the bottle, causing the exterior to film and the bottle to soften. The reaction should be carried out for no longer than 24 hours, and allowed to separate for no more than an additional 24 hours. As soon as the decanting procedure is completed and the FAME removed, have the students submerge the vessels in a sink full of soapy water. Allow the vessels to soak overnight, and wash and dry them the following day. Inspect the bottles for soft spots or leaks before reuse.



LESSON 2

Breakdown of the Procedure (1st session):

In this experiment, the students will react soybean oil (vegetable oil) with methanol to create Fatty Acid Methyl Esters (FAME). Potassium hydroxide will be used as a catalyst.

The students will be instructed to mix the proper amount of alcohol and catalyst in an Erlenmeyer flask, and then combine the oil and alcohol/catalyst mixture in the reaction vessel. The same magnetic stir bar will be used for both mixing the catalyst and the reaction.

Care should be taken to ensure that the reaction is not over agitated as to cause splashing or spilling of the reactants. However, it is necessary to maintain a healthy vortex in the reaction vessel to provide phase contact for the alcohol and triglycerides.

The reaction should be carried out for at least 4 hours up to 24 hours. At room temperature and the specified ratio of alcohol, the reaction will proceed to 80 percent within one hour, with the remaining time needed to achieve approximately 95 percent completion. In this manner, it will be necessary for the instructor to turn off the stirrers after the class period is over. However, at least 8 hours of settling time must be provided before the next lab experiment may be conducted on the reaction products. This will require proper scheduling on the part of the instructor.

Extension Suggestions:

In order to illustrate the role of agitation in the progress of a chemical reaction, if there are a sufficient number of students in the class, have several of the groups stop their reaction within the first hour, for example at 10, 20, 30 and 40 minutes into the reaction. Have the students note the time allowed for the reaction. In subsequent analysis, these groups should observe signs of an incomplete reaction.

Breakdown of the Procedure (2nd session):

In the second session of the experiment, the students will make observations about the contents of the reaction bottle, and then decant the glycerin layer from the bottom of the reaction vessel and transfer the FAME to a separate flask. There is a series of data analysis and discussion questions that the students will then perform to evaluate the experiment.

Disposal of Chemicals:

The FAME and the glycerol created in this experiment will contain methanol and KOH. Central collection points for the glycerin and FAME should be set up for the students. After all of the experiments which use the FAME are complete, it should be disposed of as a flammable lab waste.



TRANSESTERIFICATION OF SOYBEAN OIL TO FATTY ACID METHYL ESTERS

Name _____ Class _____ Date _____

INTRODUCTION

Soybean oil is a desirable raw material for alternative diesel fuels since it is widely available from soybeans, a renewable agricultural crop. Soybean oil is made up of molecules called triglycerides, which are formed in the seed of the soybean plant. Triglycerides have very high energy content, almost equal to that of petroleum diesel. However, the high viscosity of soybean oil makes it difficult to burn in diesel engines. Transesterification is the process of converting soybean oil triglycerides into smaller molecules known as fatty acid methyl esters (FAME). In this process, the glycerol molecule in the triglyceride is replaced by 3 alcohol molecules, typically methanol, hence the name methyl esters. A schematic of his reaction is shown in figure 2.4 in this handout.

In this experiment, you will learn how to make crude FAME and separate the crude glycerin from the FAME. This crude FAME is further refined in commercial operations to produce biodiesel.

OBJECTIVES

1. To make crude Fatty Acid Methyl Esters (FAME) from soybean oil
2. To separate the glycerol and FAME

EQUIPMENT

- | | |
|-----------------------------------|------------------------------------|
| 2 safety goggles | 1 electronic balance |
| 1 magnetic stir plate | 1 125 or 250 ml Erlenmeyer flask |
| 1 magnetic stir bar | 1 100 or 250 ml graduated cylinder |
| 1 250 ml reaction bottle with cap | 1 25 ml graduated cylinder |
| 1 weigh dish | |



Figure 2.8 Soy cooking oil which can be modified through transesterification to produce biodiesel

MATERIALS

- Soybean Oil (Vegetable Oil)
- Methanol (CH_3OH)
- Potassium Hydroxide (KOH) Safety

1. Wear safety goggles.
2. Potassium hydroxide is corrosive and can cause severe injury. If you spill potassium hydroxide on yourself, flush the affected area with water for 5-10 minutes and notify the teacher.
3. Methanol is highly flammable and poisonous if ingested. Methanol can irritate the skin, and can cause dizziness and nausea when exposed to the fumes. Handle pouring and measuring of the methanol in a fume hood. If methanol should get in your eyes, begin flushing with water immediately and continue for 10 minutes. If you should spill methanol on yourself or in the laboratory, immediately notify the teacher.



Procedure (1st Session)

1. As you perform the experiment, record your data in Table 2.1 and your observations in Table 2.2.
2. Measure the mass of the reaction bottle without the cap and record the value to 0.01 grams.
3. Measure out 150 +/- 5 ml of soybean oil using the graduated cylinder.
4. Pour the soybean oil into the reaction bottle and record the mass of the bottle and oil to 0.01 grams.
In Table 2.2 note the color of the soybean oil and how it behaves when poured.
5. Measure the mass of the 125 ml Erlenmeyer and record the value to 0.01 grams.
6. In the fume hood, measure out 35 +/- 1 ml of methanol using a graduated cylinder.
Pour the methanol into the 125 ml Erlenmeyer flask and record the mass to 0.01 grams.
7. Measure out .75 +/- .05 grams of KOH in the weigh dish.
8. Place the magnetic stir bar in the Erlenmeyer flask containing the methanol and place it on the magnetic stir plate.
9. Turn on the stir plate to a medium setting being careful not to splash the methanol.
10. Slowly pour the KOH into the methanol and continue stirring until ALL of the KOH has dissolved.
Stop agitation and remove from the stir plate.
11. Slowly pour the methanol/KOH mixture and the stir bar into the soybean oil being careful not to splash.
Note that there are two phases of liquid.
12. Place the reaction bottle on the stir plate.
13. Begin agitating by turning on the stir plate to a medium setting. Describe any changes you observe in the color and phase behavior during the first 10 minutes of agitating and record your observations.
Continue mixing for 3 to 24 hours (see instructor). Record the total time of your reaction in Table 1.2.
14. Turn off mixing bar and allow reaction vessel to sit undisturbed for 8-24 hours.
(Your instructor may do this.)

Procedure (2nd Session)

1. As you perform the experiment, record your data in Table 2.1 and your observations in Table 2.2.
2. Examine the contents of the reaction vessel. Compare the color and appearance of the contents to the beginning reagents and record your observations in Table 2.2.
3. Measure the mass of the 100 ml beaker and record the value to 0.01 grams.
4. Screw the cap onto the reaction vessel firmly, being careful to avoid squeezing the vessel when handling.



Figure 2.9 Separating glycerol layer



- Place the tip of the spout of the reaction vessel in the 100 mL beaker. While tilting the bottle to keep the dark glycerol layer at the bottom, squeeze the bottle to decant the glycerin layer. Release pressure on the bottle very slowly to avoid mixing the layers. See Fig. 2.9 for details. Fig. 2.3 Separating glycerol layer
- When the entire glycerol layer has been removed, measure the mass of the beaker and glycerol and record the value to 0.01 grams.
- Measure the mass of the reaction bottle and methyl esters and record the value to 0.01 grams.
- Label a clean, dry 500 mL flask with your group members' names.
- Pour your methyl esters from the reaction vessel into the 500 mL flask for storage. Cover the vessel with a watch glass or weigh dish to avoid contamination. You will use your methyl esters for the following labs.

Table 2.1 Transesterification Data

Item Weighed	Mass (grams)
Empty reaction bottle (with cap)	
Reaction bottle and vegetable oil	
Empty dry Erlenmeyer flask	
Erlenmeyer flask and methanol	
Potassium hydroxide used	
Empty 100 mL beaker	
Beaker and glycerol removed by decanting	
Reaction bottle and methyl esters (with cap)	

Table 2.2 Observations

Step	Observations
Session 1	
Step 4	
Step 11	
Step 13	
Session 2	
Step 2	

STUDENT DATA ANALYSIS

Name _____ Class _____ Date _____

INTRODUCTION

Determine the following:

Mass of soybean oil used

Mass of methanol used

Mass of glycerine produced

Mass of FAME produced

Calculate the number of moles of soybean oil using a molecular weight of 875g/mol.

Calculate the number of moles of methanol CH₃OH (hint: find the molecular weight of methanol)

Calculate the molar ratio of methanol: vegetable oil used in the reaction.

Determine the theoretical molar ratio methanol: soybean oil triglycerides for this reaction by using Figure 2.10.

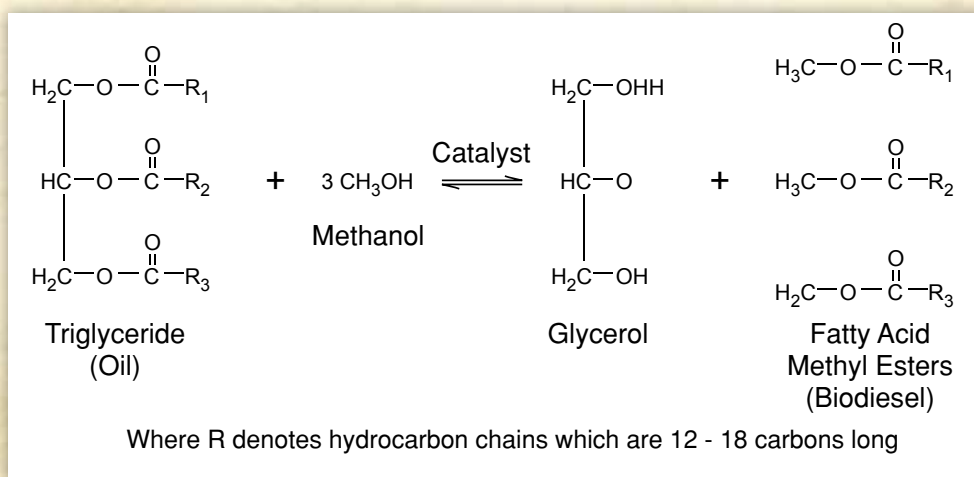


Figure 2.10 Transesterification reaction



ANALYSIS

1. From the number of moles found in the data analysis, determine the limiting reagent to this experiment.

2. Compare the theoretical molar ratio of methanol: vegetable oil triglycerides, with the molar ratio used in the experiment. What is the difference? Why might this difference be useful in performing this reaction?

3. Compare the sum of the mass of the reagents to the sum of the mass of the products. Is there a loss of mass? How might this be explained?



PHYSICAL AND CHEMICAL PROPERTY DIFFERENCES OF SOYBEAN OIL AND FATTY ACID METHYL ESTERS





TEACHERS' BACKGROUND MATERIAL

INTRODUCTION

This series of experiments is designed to provide students with qualitative confirmation of the transesterification reaction by allowing them to test the soybean oil vs. the Fatty Acid Methyl Esters (FAME) produced in the previous experiment.

Suggestions

Nearly any lecture material relating to physical property change or chemical change as it relates to chemical reactions can be presented prior to this material, however, specific topics which may be useful are:

- Intermolecular forces
- Effect of molecule size on viscosity
- Definition and properties of solvents and solutes
- Oxidation (as it applies to combustion)
- Capillary action
- Mass transfer (and effect of molecule size)

General Safety and Preparation

Except for the use of glass plates in the viscosity experiment, there are no dangerous reagents or equipment used by the student groups. FAME can cause mild skin irritation, which can be cured by flushing the affected area with water.

The combustion experiment should be performed at the class level, either by the instructor or by one or two students under the close observation of the instructor.

Reagents

Food grade soybean oil (commonly sold as vegetable oil), available from any grocer or kitchen supplier may be used in the experiment.

The FAME produced in the previous experiment will be used in this comparison.

Equipment

The equipment provided in this kit is intended for student groups of two, except for the combustion test which will be conducted on the class level.

The instructor must provide glass plates and a means of elevating them, e.g. textbooks. (See the example in the student lab handout.) The actual size of the glass plate is not critical, as long as it is large enough to be elevated and allow a sufficient amount of time for the students to observe the difference in viscosity.

This kit will provide the 50 ml tubes needed for the solvent properties test, as well as the lamps to be used in the combustion test. All other materials listed on the first page of the student handout should be provided by the instructor.

BREAKDOWN OF THE PROCEDURE (VISCOSITY EXPERIMENT)

In this experiment, the students will compare the time it takes for samples of soybean oil and FAME to flow down an inclined plate. It should be stressed to the students to have the glass plate as clean as possible, to spot the plate on a level surface and to move the plate in a swift but controlled manner during the experiment.

Extension suggestions:

Have students carefully heat or cool samples of soybean oil and FAME, and conduct the same viscosity experiment, to illustrate the effects of heat on viscosity.



LESSON 3

BREAKDOWN OF THE PROCEDURE (SOLVENT PROPERTIES EXPERIMENT)

In this experiment, the student will compare the effects of soybean oil and FAME on polystyrene. FAME act as a mild solvent and will rapidly dissolve the polystyrene and form a stable mixture. Be sure to remind the students to have the caps fitted tightly to avoid release of the liquids.

Extension Suggestions:

Using a stopwatch, have the students record the time it takes to dissolve each subsequent Styrofoam peanut in a measured quantity of FAME. The students should observe the time increasing as the FAME reaches its saturation point.

Encourage critical thinking about the possibility of using materials such as FAME to collect and process waste Styrofoam.

BREAKDOWN OF THE PROCEDURE (COMBUSTION EXPERIMENT)

In this experiment, the instructor will demonstrate to the class a combustion comparison on soybean oil and FAME using a modified alcohol lamp. The purpose of this experiment is to show the difference in volatility between soybean oil and the FAME, as well as demonstrating the effect of viscosity on the combustion characteristics of the fuel. It should be noted that if the lamps are burned for an extended period of time, both flames may reach a similar height. However, the primary purpose of the experiment is to show difference in the ease of ignition and flame size and color of the FAME. It is therefore suggested to burn the lamps for a short period of time ~5 minutes or less. The following steps should be taken to ensure a satisfactory experiment:

1. Measure both wicks and cut them at exactly $\frac{1}{2}$ inch above the brass wick retainer.
This will ensure that both fuels have the same amount of surface area on which to burn.
2. Clearly label both lamps.
3. Fill the vessels about 75% full using soybean oil in one and one or more of the student groups' FAME in the other.
4. Light both lamps using the same method (match, Bunsen burner, etc.). Allow the students to observe how long it takes each lamp to begin burning.
5. Allow the lamps to burn for approximately 5 minutes, allowing the wicks to become charred and for the mass transfer up the wick to reach steady state. Have the students observe the state of the flames of each lamp throughout this period.
6. Extinguish the lamps and then relight them, and have the students note which lamp relights easier.
7. After the experiment is complete, remove the top of the lamp and the wick, wash out the lamp bodies, and fill the lamps with a suitable alcohol, i.e. isopropyl.
8. Relight the lamps and burn until a uniform color and flame height is achieved, and no smoke is visible.
This will indicate a majority of the oils have been removed from the wick.
9. Store the lamps with alcohol until the next experiment is to be performed.

Disposal of Chemicals:

The FAME polystyrene solution created in the solvency experiment should be disposed of as a hazardous lab waste.

PHYSICAL AND CHEMICAL PROPERTY DIFFERENCES OF SOYBEAN OIL AND FATTY ACID METHYL ESTERS

Name _____ Class _____ Date _____

INTRODUCTION

In the previous experiment, you converted soybean oil triglycerides into fatty acid methyl esters (FAME) and glycerine using a process called transesterification. It is important to understand the effects of this reaction on the chemical structure and the resulting changes in behavior. These changes make FAME easily combustible in modern diesel engines, as well as act as biodegradable solvents. In this experiment, you will examine some of the physical and chemical differences between the original soybean oil and the FAME.



Figure 3.1 Fatty Acid Methyl Esters can be used in existing equipment without modification due to the physical and chemical changes that occur during transesterification of vegetable oil.

OBJECTIVES

1. Compare the viscosity of FAME and soybean oil
2. Compare the solvent properties of FAME with soybean oil on polystyrene
3. Compare the combustion characteristics of FAME and soybean oil

EQUIPMENT

- | | |
|---|-----------------------------------|
| 2 safety goggles | For Class: |
| 1 glass plate | 2 wick lamps with fiberglass wick |
| 1 text book of 1-2 inches thickness | 1 metric ruler |
| 2 50 ml plastic centrifuge tubes and caps | Matches/butane lighter |
| 2 eyedroppers | |
| 3-4 Polystyrene (Styrofoam) peanuts | |
| 1 felt tip marker | |

MATERIALS

- Soybean Oil (vegetable oil)
- Fatty Acid Methyl Esters (from previous experiment)

SAFETY

1. Wear safety goggles.
2. Use caution when handling glass in the inclined plate viscosity experiment.
3. Fatty acid methyl esters can cause skin irritation. If you get FAME on your hands wash thoroughly with water.



INCLINED PLATE VISCOSITY EXPERIMENT

Name _____ Class _____ Date _____

BACKGROUND

Viscosity is a measure of a liquid's resistance to flow. Viscosity is strongly related to interaction forces between the fluid molecules. Stronger interaction makes a fluid more resistant to flow. Viscosity is important in a diesel engine because the fuel injection system must spray a fine mist of fuel into the engine cylinder in order to achieve a complete combustion. If the viscosity is too high, the fuel will not disperse properly and will result in poor fuel economy and possible engine damage. It is therefore desirable to have a fuel viscosity as close to petroleum diesel fuel as possible. One way to measure viscosity is to place equal amount of liquid samples on a smooth surface and tip the surface, causing the fluids to flow. By comparing the reactions of the two fluids, a general idea of their viscosity can be obtained. The inclined plate experiment will compare the viscosity of soybean oil and fatty acid methyl esters made from soybean oil.

PROCEDURE

1. Using the felt marker, draw an even line on the glass plate approximately 4 cm from one end (this will depend on the size of your plate).
2. Use a paper towel to remove any oils or films on the plate.
3. Following steps 1-4 in Figure 3.2, pay careful attention to the progress of the samples down the plate.

ANALYSIS

Compare the behavior of the two samples. Which liquid moved more quickly down the plate?

Based upon the background information above, which liquid is best suited for burning in a diesel engine?

What else did you notice about the behavior of the two fluids? Which fluid deposited more material as it moved down the plate?



1. Use 1.7 ml transfer pipette to place equal amounts of samples on plate along line.



2. Firmly grip plate on sample end.



3. Quickly but carefully slide the plate onto the paper towel as shown.



4. Observe the progress of the two samples.

Figure 3.2 Inclined plate viscosity tipping experiment



SOLVENT PROPERTIES OF FATTY ACID METHYL ESTERS

Name _____ Class _____ Date _____

BACKGROUND

In addition to acting as a fuel, FAME also act as a mild solvent. A solvent is a substance which can dissolve other materials. Solvents are commonly used to remove dirt, oils and finishes from surfaces or to remove other liquids from solid-liquid mixtures. Many fluids act as solvents. Most commercial solvents are derived from crude oil. These include gasoline, ether, hexane and mineral spirits. In addition to being flammable and dangerous to handle, some petroleum-based solvents have been shown to cause cancer. Solvents based on FAME are beginning to become more popular because they are very stable, far less flammable than petroleum solvents and non-toxic. The following experiment will demonstrate the difference between the solvent effects of soybean oil and FAME on polystyrene.

PROCEDURE

1. Pour approximately 20 ml each of soybean oil and FAME into separate 50 ml tubes.
2. Place two or three polystyrene (Styrofoam) peanuts in each tube.
3. Replace the lids and shake both tubes.
4. Stop every 15 to 20 seconds and observe the state of the peanuts in each tube.
When the peanuts in one of the tubes becomes completely dissolved, stop shaking the tubes and answer the questions below.

ANALYSIS

What effect did the soybean oil have on the polystyrene? The FAME?

Can you think of a possible use for this type of solvent property?

Why would a petroleum solvent such as gasoline not work as well in this application?



COMBUSTION PROPERTIES OF FATTY ACID METHYL ESTER

Name _____ Class _____ Date _____

BACKGROUND

This experiment will demonstrate the combustion properties of FAME vs. soybean oil. Special wick lamps will be used to burn samples of both soybean oil and FAME. This demonstration is designed to provide a visual means of evaluating the combustion properties of the two liquids. In an oil lamp, more viscous fluids will not be transported as easily up the wick of the lamp, resulting in smaller flame and more difficult lighting.

PROCEDURE

This experiment is designed to be conducted in a group under the supervision of your instructor. Obtain the proper instructions and record any observations you make during the experiment.

ANALYSIS

Which lamp lit more quickly?

Which lamp burned with a larger flame?

Briefly describe the quality of the flame from each lamp and describe any other interesting observations about the difference in the flames.

What property of the fuel is being demonstrated in this experiment? Why is this important in diesel engine operation?



THIN LAYER CHROMATOGRAPHY TO EXAMINE THE BIODIESEL REACTION





TEACHERS' BACKGROUND MATERIAL

GENERAL NOTES

This experiment will allow students to use thin layer chromatography to analyze the product of the transesterification reaction performed in Lesson 2.

Lecture Suggestions

A discussion of other chromatography techniques may also be introduced (column chromatography, liquid chromatography, etc.)

General Safety and Preparation

IMPORTANT: The solvent solution used in this lab is extremely flammable and can cause nausea and unconsciousness if inhaled in large quantities. Take the proper precautions to warn students of potential dangers.

General Suggestions

Due to the delicate procedure used to spot the chromatography plate, it is highly recommended that the instructor complete this experiment at least once prior to the student's lab period. It is also highly recommended to demonstrate how to use the micropipettes to the class during the lab period.

Reagents

The chromatography solvent should be prepared prior to the class period from technical grade reagents. The proportion of reagents on a parts volume base is:

85:15:1 Petroleum Ether Diethyl Ether: Acetic Acid

This solvent should be stored in a brown glass bottle and only be poured while wearing a fume hood.

Each group of students will use between 5 and 10 ml of solvent per experiment, so the instructor should prepare a corresponding amount of solvent.

The iodine used in this experiment should be of technical grade. Only a small amount should be required per student group. Illustrate to students the corrosive properties of iodine and its ability to stain skin and clothing.

Equipment

The equipment provided in this kit is intended for student groups of two.

The chromatography plates provided with the kit should be carefully cut into 2.5 cm x 6.5 cm pieces for use by the students. This will allow 24 slides per 20x20 cm plate (enough for 48 students). Use a pencil to mark the plates for cutting. The plates are delicate and some of the silica will dislodge during cutting.

This kit will provide the 50 ml tubes needed for the iodine and chromatography solvent.

The micro capillary pipettes needed for spotting the plates are also provided.



LESSON 4

BREAKDOWN OF THE PROCEDURE

The students will measure out small quantities of the chromatography solvent and iodine, mark and spot the TLC plate and then elute the plate in the solvent and develop the plate in iodine gas in another container. It should be stressed to avoid breathing the solvent fumes. The students will then compare their slides and draw conclusions on how far their reaction has gone to completion.

After the students have completed the experiment, have them empty the chromatography tubes of solvent and iodine into suitable waste containers.

Disposal of Chemicals

Remaining chromatography solvent should be disposed of as an explosive/flammable lab waste in a suitable container.

Waste iodine should be collected and disposed of as a hazardous lab waste.

THIN LAYER CHROMATOGRAPHY TO EXAMINE THE BIODIESEL REACTION

Name _____ Class _____ Date _____

INTRODUCTION

Thin layer chromatography (TLC) is a method used to visualize the presence of different compounds in a solution. TLC uses molecular interactions between two phases (solid plate and liquid solvent) to separate compounds in a solution. This technique is useful in detecting for contaminants or by-products from a reaction. In this case, thin layer chromatography will be used to examine the purity of the fatty acid methyl esters produced in Lesson 2.



Figure 4.1 A Gas Chromatograph allows scientists to examine complex mixtures of chemicals accurately. *Photo courtesy of Shimadzu Inc.*

OBJECTIVES

1. To prepare a thin layer chromatography plate.
2. To develop the plate and perform analysis on the results.

EQUIPMENT

- | | |
|-------------------------|----------------------------------|
| 2 safety goggles | 2 small beakers (100 ml or less) |
| 2 pairs of lab gloves | 2 microcapillary pipettes |
| 1 forceps | 1 metric ruler |
| 1 chromatography slide | 1 pencil |
| 2 50 ml tubes with lids | 1 felt tip marker |

MATERIALS

1. Chromatography solvent (prepared by instructor)
2. Iodine Crystals
3. Soybean oil (vegetable oil – Triglyceride)
4. Fatty acid methyl esters (from Lesson 2)

SAFETY

1. Wear safety goggles.
2. Wear gloves when handling slides to avoid contamination.
3. The chromatography solvent used in this lab contains chemicals which are highly flammable and can cause headache, nausea and unconsciousness. Pour the solvent only while in the fume hood and immediately replace the cap when opening vessels which contain the solvent outside of the hood. Avoid breathing the fumes. Should the solvent get in your eyes, begin flushing with water immediately and continue for 10 minutes. If you should spill the solvent on yourself or in the laboratory, immediately notify the teacher.
4. Iodine is corrosive and can cause severe skin irritation and burns. Avoid skin contact with iodine crystals and wash hands thoroughly before leaving the lab.



PROCEDURE (SEPARATION)

As you perform the experiment, record your observations in the spaces provided on the next page.

1. Using the ruler and felt pen, mark a depth of 1 cm above the top of the cone portion of one of the 50 ml tubes.
2. In the fume hood, transfer the chromatography solvent to the 50 ml tube until it reaches the 1 cm mark. Immediately replace the lid on the tube and the solvent container and tighten them firmly to avoid evaporation.
3. In the other 50 ml tube, add two to three medium sized iodine crystals. Replace the lids on the tube and iodine bottle immediately to avoid evaporation.
4. While wearing gloves, use the pencil and ruler to lightly mark the chromatography slide as shown in Figure 4.4.
5. In one of the small beakers, pour 5-10 ml of soybean oil. Pour 5-10 ml of your fatty acid methyl ester in another beaker.
6. OBSERVE A DEMONSTRATION OF MICROPIPETTE USE BY YOUR INSTRUCTOR. Then, use the micropipettes to place a small amount (about the size of a pencil tip) of soybean oil and methyl esters on the chromatography slide 1.5 cm from the bottom as indicated in figure 4.2. Use different micropipettes for each sample to avoid contamination of the samples.
7. Using the forceps, carefully place the chromatography slide sample end down into the glass jar containing the chromatography solvent. The solvent should be below the sample line. Immediately replace the lid.
8. Observe the progress of the solvent moving up the slide. When it reaches just below the top of the slide, remove the slide from the solvent bath and quickly mark the highest point the solvent reached with your pencil. Immediately replace the lid of the solvent tube. Allow the slide to dry for 2-3 minutes.

PROCEDURE (DEVELOPMENT)

1. Take the dry slide and place it sample end down into the glass jar containing the iodine crystals. Be sure that you can observe the side of the slide on which you spotted the sample. Immediately replace the lid.
2. Allow the slide to develop for 10-15 minutes. You should observe spots beginning to form like the ones in Figure 4.3. If spots are not clearly defined after 15 minutes, allow them to develop further.
3. Using the forceps, remove the slide from the jar and immediately replace the lid. Using the pencil, outline areas which have become dark. Refer to Figure 4.3 for an example. After removing the slide from the iodine, the spots will begin to fade.
4. Following the directions of your Instructor, dispose of the chromatography solvent, iodine crystals and tubes in appropriate containers.

Name _____ Class _____ Date _____

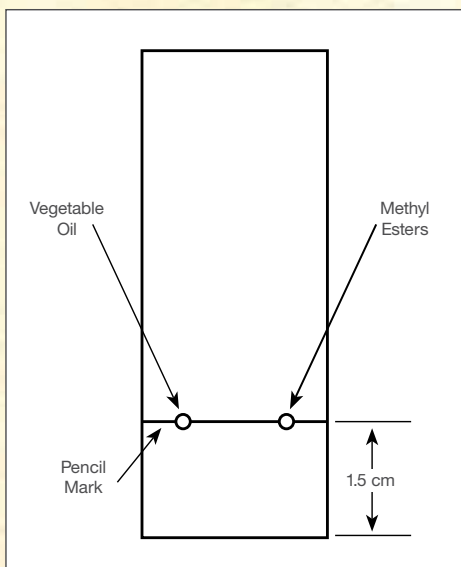


Figure 4.2 Slide diagram

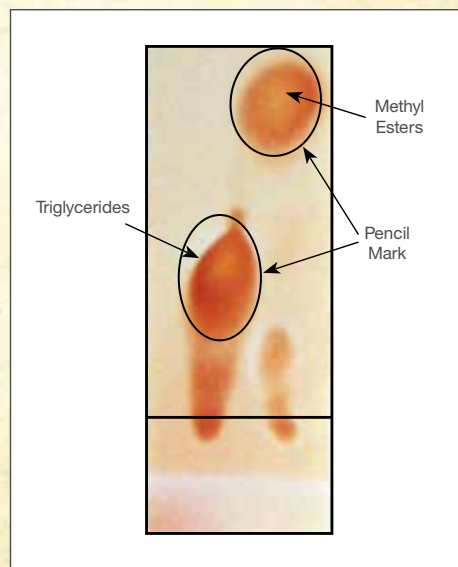


Figure 4.3 Example slide

ANALYSIS

The methyl ester should move faster along the slide than the triglyceride due to the molecular size (see Fig. 4.3).

1. Did you detect the presence of compounds in places other than those shown in the example slide above? If so, where?
2. What could be some of the possible causes for an incomplete transesterification reaction? On the plate diagram to the right, sketch what an incomplete reaction slide might look like on a slide.
3. Based on the results of your slide, which compound would you estimate as being more soluble in the chromatography solvent, triglycerides (soybean oil) or fatty acid methyl esters?

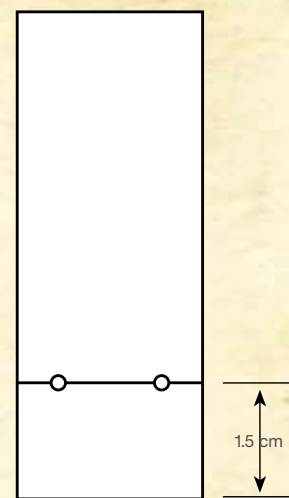


Figure 4.4 Lightly mark slide with pencil



Name _____ Class _____ Date _____

THIN LAYER CHROMATOGRAPHY SLIDE DEVELOPMENT COMPARISON

You may use the figure below to roughly evaluate whether or not your reaction has gone to completion. The figure below shows Thin Layer Chromatography results for blends of 100 percent fatty acid methyl esters and soybean oil. Compare the FAME side of your chromatography plate with the figure below and estimate the completeness of your reaction.

What is your estimated completeness of your reaction?

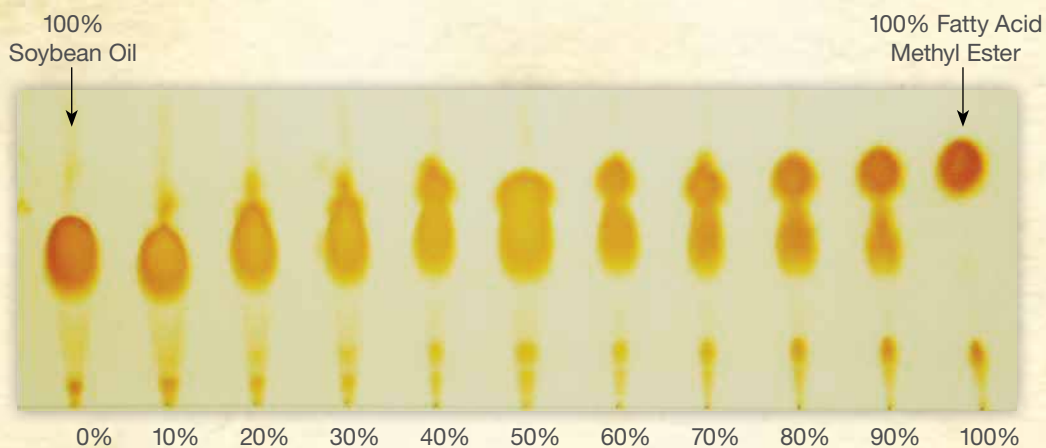


Figure 4.5 Percent of fatty acid methyl esters (in soybean oil)



DETERMINING BIODIESEL CONCENTRATIONS IN FUELS





TEACHERS' BACKGROUND MATERIAL

In this lesson, the students will learn about the detection of biofuel in diesel mixtures. This lab is designed to be performed over two class periods. In the first class period, the students will perform the reaction between sulfuric acid and FAME in the samples. After a minimum 4-hours settling period, the students will observe and analyze results in the 2nd class period.

Background of the Reaction

This reaction is based on charring using sulfuric acid, a common reagent used in charring. When concentrated acid is exposed to organic compounds, it removes the hydrogen and oxygen, leaving only carbon. When in solution, the carbon creates a very fine precipitate and may cause a dark color in the liquid. The FAME present in a mixture of diesel fuel will be charred by sulfuric acid and will form a precipitate/become a darker color. (Note: Pure diesel fuel would not show a reaction. However, commercial mixing and delivery of diesel fuels results in almost all diesel fuels containing some FAME, so it is very difficult to obtain 'pure' diesel fuel these days.)

Mixing of the fuel samples with sulfuric acid should result in rapid color change, darkening to a brown or black color. It is likely that 'pure' diesel fuel from the gas station will react with the sulfuric acid since it likely contains FAME. Initially, the samples will likely be indistinguishable in the amount of darkness due to large amounts of precipitate formed. The samples should be allowed to settle for at least 4 hours under a hood or an appropriately vented location. Approximately four hours is necessary for settling, but the tubes can be left overnight or even a couple days if that is more convenient.

After settling, most of the large precipitate particles will have settled and a slight color difference between the solutions containing different FAME concentrations should be evident. It is possible that the solution FAME concentrations may need to be adjusted so that there is a greater difference in the concentrations of the two solutions in order to see a difference in color. The instructor may want to try different concentrations to determine what works best for their diesel fuel prior to making solutions for the students. The main point to emphasize is that as the concentration of FAME in solution increases, the color should be darker.

In commercial practice, the idea behind this test is to quantify the level of FAME in biodiesel fuels. This would normally be done using a colorimetric chart or optical transmission device to quantify the color difference, rather than the simple visual differences shown by this experiment.



LESSON 5

Reagents

1. Concentrated Sulfuric Acid
2. Diesel fuel, which can be obtained from any gas station

Videos and pictures included with this kit used diesel with red dye in it. (Red dye is added to off-road diesel fuels for taxation purposes.) Diesel from gas stations should be clear. Whether the diesel used in the experiment is dyed or not, the coloration change due to the different solutions with biodiesel will be possible to distinguish.

Also, the diesel fuel obtained from the gas station will almost certainly contain up to 5% biofuel already. This means that when a 12.5% and 25% v/v FAME solution is made, the actual concentration of biofuel in the solution is not accurately represented by 12.5% and 25%. That will be fine for this experiment because the purpose is to differentiate between the two, not identify exact concentrations.

Safety

Sulfuric acid is very corrosive. Wear gloves and goggles and work under the hood when preparing samples for the students. Take caution not to spill or splash on clothing.

Explain to the class that when working with concentrated acids, the acid should always be added into the other liquid. DO NOT pour/add any liquid into concentrated acid. Heat is generated when concentrated acids mix with other liquids. If this heat is not rapidly dissipated, it may cause rapid vaporization/boiling of the mixture, which could cause concentrated acid to be sprayed out of the container. By adding the concentrated acid into the liquid, the concentrated acid is rapidly diluted, minimizing any localized heating/mixing.

Preparation

Put 4 drops of sulfuric acid into centrifuge tubes. Each group will need 3 of these tubes.

Make 2 solutions of diesel with FAME, one at concentrations of 12.5% volume/volume (v/v) FAME and the other at 25% v/v FAME solution. For example, to make 1 L of 12.5% v/v, add 125 ml of FAME to 875 ml diesel. Make enough of each solution so that each group of two students will have 40 ml of each solution.



DETERMINING BIOFUEL CONCENTRATIONS IN DIESEL

Name _____ Class _____ Date _____

INTRODUCTION

Biofuel (FAME) is often added to petroleum diesel fuel in order to increase the use of renewable resources vs. non-renewable petroleum. It is often desirable to know if and how much biofuels is contained in a fuel blend. This experiment will demonstrate a colorimetric detection method used to identify if FAME is present in a diesel fuel blend. The intensity of color is related to the concentration of the FAME in the fuel.

When fuel blend containing FAME is mixed with concentrated sulfuric acid, a reaction occurs which causes the liquid to turn dark brown or black. Pure petroleum-based diesel fuel will not change color. Even small amounts of FAME (1 percent or less) in diesel fuel will result in a visible color change. The color change is due to charring, a chemical reaction which produces very fine carbon particles which result in a darkening color change.

OBJECTIVES

1. To determine the presence or absence of FAME biofuel in a diesel sample from the gas station
2. To compare the FAME content of two diesel fuel blends

EQUIPMENT

- | | |
|-------------------|---|
| 2 safety goggles | 4 empty 50 ml plastic centrifuge tube and cap |
| 2 pairs of gloves | 1 felt tip marker |
| 3 100 ml beakers | 3 2 dram sample vials |

MATERIALS

- Standard diesel fuel
- Two samples containing different amounts of added FAME
- Concentrated sulfuric acid

SAFETY

1. Always wear safety goggles and gloves.
2. Sulfuric acid is corrosive and can cause severe injury and damage to clothing. If you spill sulfuric acid on yourself, immediately flush the affected area with water for 5-10 minutes and notify the teacher.
3. Diesel fuel is highly flammable and toxic if ingested. Diesel fuel can irritate the skin, and can cause dizziness and nausea when exposed to the fumes. All handling of diesel solutions should be done in a fume hood. If you should spill diesel on yourself or in the laboratory, immediately notify the teacher.



PROCEDURE (1ST SESSION)

1. As you perform the experiment, record your observations in Table 5.
2. Get four 50 ml tubes. Label the tubes 'Reference', 'Diesel', 'Unknown #1', and 'Unknown #2'.
3. Pour diesel fuel into the Reference tube up to the 40 ml mark. This will be the color reference tube. Record color observations in Table 5.
4. Pour diesel fuel into the tube labeled 'Diesel' up to the 40 ml mark. Add 4 drops concentrated sulfuric acid. (Note: Some classrooms may require that only the teacher add the acid.) Put the cap on tightly. Invert the tube multiple times in order to ensure mixing and a complete reaction. Observe any color change in Table 5.
5. Repeat Step 4 with the tubes labeled Unknowns #1 and #2.
6. Store the tubes upright at room temperature for at least 4 hours, and do not mix or shake the contents during that time. This allows the larger carbon precipitate particles in the solution to settle to the bottom so color differences in the liquid are more easily distinguished.

PROCEDURE (2ND SESSION)

1. Using a transfer pipette transfer approximately 5 ml from each 50 ml centrifuge tube into separately labeled 2 dram sample vials. The purpose of using the smaller vial is to more easily observe any color differences. Be careful not to get any precipitated solids in your sample as this will change the color by blocking light.
2. Observe the color of the tubes noting any differences in color/darkness. It may be necessary to switch the tubes around or adjust your viewing angle in order to distinguish the difference. Record your observations in Table 5.

Table 5	Observations
Session 1	
Reference	
Diesel	
Unknown #1	
Unknown #2	
Session 2	
Reference	
Diesel	
Unknown #1	
Unknown #2	



THIN LAYER CHROMATOGRAPHY TO EXAMINE THE BIODIESEL REACTION

ANALYSIS

1. From your tests, do you detect any FAME biofuels in the standard petroleum diesel from the gas station? Explain your reasoning for this answer.
2. The tubes labeled as unknowns contain either 12.5% FAME or 25% FAME.
Can you tell which one contains each concentration immediately after mixing (prior to settling)?
3. After the precipitate had settled, can you identify which tube contains 12.5% FAME and which contains 25% FAME?
4. Why might it be important to be able to identify/quantify the concentration of FAME in diesel fuel mixtures?



UREA INCLUSION FRACTIONATION OF BIODIESEL





TEACHERS' BACKGROUND MATERIAL

INTRODUCTION

This experiment is designed to provide students with an example of a separation process, which changes the fatty acid methyl ester compositions and physical properties. Students will be separating saturated fatty acid methyl esters from un-saturated fatty acid methyl esters that are present in soybean-based biofuels.

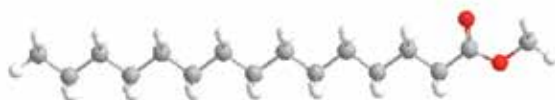
Biodiesel from most normal oil/fat sources are composed of a mixture of different composition of saturated and unsaturated fatty acid methyl esters. Saturated fatty acid methyl esters have much higher melting points than unsaturated esters. Using a biofuel with a high concentration of saturated esters during cold conditions is not recommended because they may solidify and clog fuel filters or solidify in the fuel tank/lines. This separation process produces winterized biodiesel which does not contain saturated fatty acid methyl esters and has a melting point suitable for cold conditions.

Biodiesel is a mixture of different types of fatty acid methyl esters. The composition depends on the source of the oil/fat used to make the biodiesel. Most common fats/oils are composed of 5 types of fatty acids:

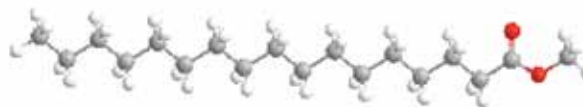
palmitic acid	$C_{16}H_{32}O_2$ (saturated 16:0)
stearic acid	$C_{18}H_{36}O_2$ (saturated 18:0)
oleic acid	$C_{18}H_{34}O_2$ (1 double bond 18:1)
linoleic acid	$C_{18}H_{32}O_2$ (2 double bonds 18:2)
linolenic acid	$C_{18}H_{30}O_2$ (3 double bonds 18:3)

The term saturated means that there are no carbon-carbon double bonds, so all the carbons are "saturated" with hydrogen atoms (alkane structures). Myristic (14:0), palmitic (16:0), and stearic (18:0) are all saturated fatty acids. Conversely, unsaturated means that there are one or more carbon-carbon double bonds present (alkene structures). Oleic acid (18:1) is a monounsaturate, and linoleic (18:2) and linolenic (18:3) are polyunsaturates (di- and triunsaturates).

The physical molecular shape of the different fatty acid methyl esters changes with the presence of carbon double bonds. The normal single carbon bond angle is 109.5° . For saturated fatty acid esters, there are no carbon double bonds.



Methyl palmitate (16:0)



Methyl stearate (18:0)



LESSON 6

For unsaturated fatty acid methyl esters, the molecular structure contains carbon double bonds, which have slightly larger bond angles.

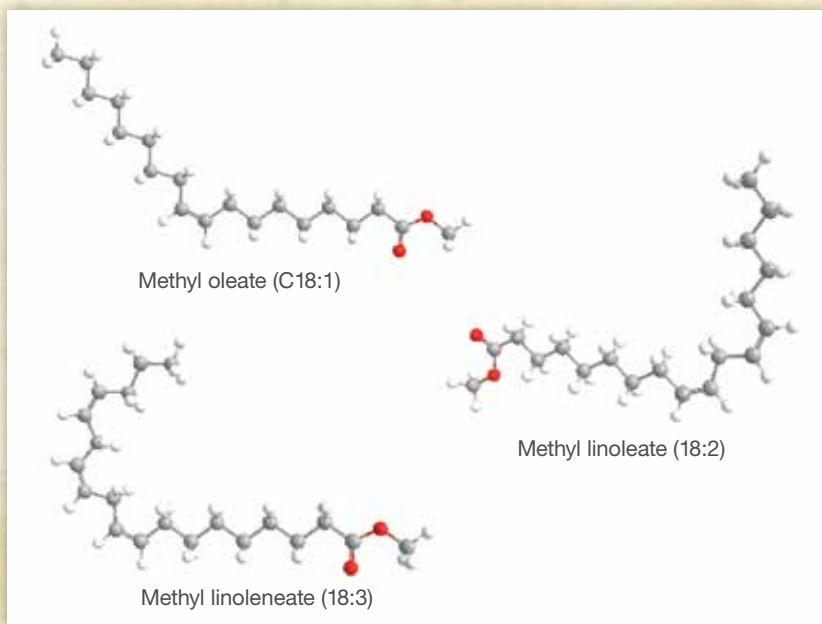
The structure/composition of the fatty acid methyl esters strongly affects their physical properties. Longer carbon chains generally tend to crystallize at lower temperatures.

Linear saturated (alkane type) fatty acid esters tend to crystallize much more easily than non-linear unsaturated (alkene type) esters. So the saturated fatty acid esters tend to crystallize/solidify at much higher temperatures than unsaturated fatty acid esters. See table below where x:n refers to the carbon chain length (x)

and number of carbon double bonds (n). You will probably recognize that oils from beef, palm, and butter (which contain more saturated fatty acids) are solids at room temperature, whereas sources such as soybean, olive, and canola (which contain more unsaturated fatty acids) are liquids at room temperature.

Fatty Acid composition of Different Fat sources and methyl ester melting points (from Bailey's Industrial Oil and Fat Products, 5th ed.) Values shaded in orange are saturated fatty acids. Values shaded in green are unsaturated fatty acids.

When biodiesel is cooled, the components with high melting points will begin to form crystals. If this occurs, it can clog fuel lines/filters resulting in stopping the engine.

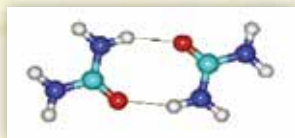


SOURCE	SATURATED					UNSATURATED			
	10:0	12:0	14:0	16:0	18:0	18:1	18:2	18:3	20:n
Coconut	7	48	17	8	3	6	2		
Palm			1	46	5	39	9	1	
Soybean				11	4	22	54	8	
Olive			1	15	3	67	13	1	
Canola				3	5	45	27	9	
Beef			3	16	22	42	2	0.2	
Butter	6	6	11	26	10	28	2	1	
Jatropha				16	7	42	33	1	
Fish	6		16		3	5	2	1	14
Melting Temperature (°C)		+6	+18	+31	+39	-19	-35	-57	



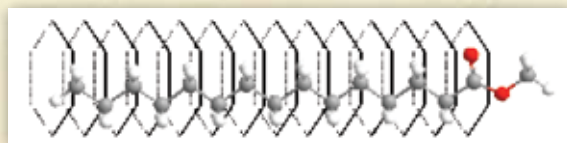
UREA CLATHRATION/INCLUSION COMPOUNDS

Urea ($\text{CO}(\text{NH}_2)_2$) is a common chemical made by mammals (urine) and used to make fertilizer, plastics, and adhesives. Two urea molecules can associate to form a hexagonal structure (see figure below).



Hexagonal Structure

When mixed with a linear hydrocarbon structure, urea molecules form a linear hexagonal host-guest complex with the hydrocarbon inside called a clathrate or inclusion compound (see figure below).



Clathrate/Inclusion Compound

These clathrate structures are generally insoluble, so they form a precipitate/solid which can be separated from the original liquid. Due to the properties of urea, it can only form clathrates with linear hydrocarbon molecules.

This experiment applies urea inclusion to separate the saturated and unsaturated components of soybean biodiesel. The linear saturated fatty acid methyl esters form clathrates more readily than the non-linear unsaturated esters. Therefore, urea inclusion can be used to separate saturated and unsaturated esters. By removing the saturated esters from the biodiesel fuel, the temperature at which solid crystals start to form (cloud point) is reduced. So this produces a winter biofuels that does not cause fuel line/filter clogging problems.

In summary, this experiment uses urea clathration to remove high melting methyl ester components from biodiesel to allow better functioning at winter conditions.

COURSE MATERIAL SUGGESTIONS

Nearly any lecture material relating to physical and chemical properties and organic chemistry as it relates to the molecular interactions that occur in this experiment can be presented prior to this material, however, specific topics which may be useful are:

- Chemical structure
- Hydrophobic and hydrophilic characteristics
- Definition and properties of solvents and solutes
- Definition of miscible and immiscible fluids



LESSON 6

GENERAL SAFETY AND PREPARATION

It is important to keep in mind that methanol and urea are used in this experiment and caution should be used when handling them. Use methanol under a bench hood. Methanol is a volatile, colorless, flammable liquid that is highly toxic if ingested. It is important to keep in mind that FAME can cause mild skin irritation. Proper eyewear and gloves should be worn by the students when conducting this experiment.

This experiment should be performed at the group level with close monitoring by the instructor at all times.

General Suggestions

It is recommended that the instructor perform this lesson prior to class in order to have a full understanding of the time requirement. The experiment should be broken up into two days. This is due to the time required for the water soluble contents to separate from the non-water soluble contents after washing. In order for the formation of two immiscible fluid layers upon cleansing you will need to allow the contents to sit overnight. To obtain a more purified product it is necessary to continue the cleansing process two additional times, but it is not necessary for the purpose of this lab.

The analysis section of this lesson will be best utilized if each group collectively answers the questions and then a class discussion is held on the topics presented. You should focus the students on the environmental and economic aspects of biodiesel and the separation process covered in this lesson.

Reagents

Methanol and urea pellets are to be used in this experiment.

The FAME produced in Lesson 2 will be used for the exercise.

Equipment

All equipment must be provided by the instructor except for the reaction bottles which are included in the kit. Access to a freezer is also needed.

It is necessary for the instructor to prepare one ice bath for each group. The ice bath must be able to chill a 50 ml Erlenmeyer flask. A simple example is filling a large beaker full of crushed ice.

BREAKDOWN OF THE PROCEDURE

The students will measure out small amounts of FAME, urea, and methanol. The FAME and urea will be added to methanol and heated via a stir plate until fully dissolved. Once a uniform solution is formed it will be chilled using an ice bath, forming a precipitate (clathrate) is present. A solid/liquid separation will be performed using a reaction bottle. After separating the liquid from the solid, the liquid solution must be washed with water to remove residual methanol and urea. The resulting liquid biodiesel will have reduced saturated ester composition and hence have a much reduced freezing point.

Disposal of Chemicals:

All chemicals used in this experiment should be disposed of using the proper protocol established by the school and be treated as hazardous chemical waste.



UREA INCLUSION TO FRACTIONATE BIODIESEL

Name _____ Class _____ Date _____

INTRODUCTION

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The physical molecular shape of the different fatty acid methyl esters changes based on the presence of carbon double bonds. For unsaturated fatty acid methyl esters, the molecular structure contains carbon double bonds, which have slightly larger bond angles. These create a non-linear ‘kink’ in the molecular structure.

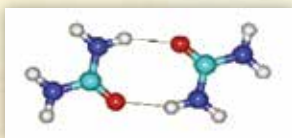
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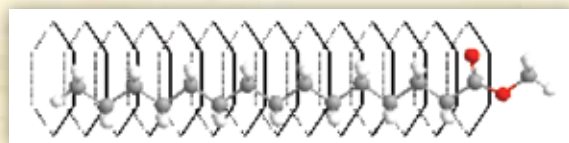
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Hexagonal
Structure

When mixed with a linear hydrocarbon structure, urea molecules form a linear hexagonal host-guest complex with the hydrocarbon inside called a clathrate or inclusion compound (see figure below).



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Compound

These clathrate structures are generally insoluble, so they form a precipitate/solid which can be separated from the original liquid. Due to the properties of urea, it can only form clathrates with linear hydrocarbon molecules.

This experiment applies urea inclusion to separate the saturated and unsaturated components of soybean biodiesel. The linear saturated fatty acid methyl esters form clathrates more readily than the non-linear unsaturated esters. By removing the saturated esters from the biodiesel fuel, the temperature at which solid crystals start to form (cloud point) is reduced. So this produces a winter biofuels that does not cause fuel line/filter clogging problems.

OBJECTIVES

1. To separate saturated fatty acid methyl esters from unsaturated fatty acid methyl esters by urea inclusion.
2. To compare freezing point (formation of solid crystals) in biodiesel containing different levels of saturated fatty acid esters.

EQUIPMENT

- 2 safety goggles
- 2 pairs of lab gloves
- 1 250 ml beaker
- 1 250 ml reaction bottle
- 2 50 ml Erlenmeyer flasks
- 1 50 ml graduated cylinder
- 1 25 ml graduated cylinder
- 2 magnetic stir bars
- 1 stir/hot plate
- Access to a freezer

MATERIALS

- 1 FAME (previously prepared by instructor)
- 2. Ice bath (prepared by instructor)
- 3. Urea
- 4. Methanol

SAFETY

1. Wear safety goggles.
2. Wear gloves when handling materials to avoid contamination.
3. Stir plate may be hot.



PROCEDURE

Forming urea-methyl ester clathrates (steps 1-11)

1. Measure out 5 +/- .05 grams of urea in a weigh dish.
2. Measure out 20 +/- .5 ml of FAME using the 25 ml graduated cylinder.
3. Measure out 32 +/- .5 ml of methanol using the 50 ml graduated cylinder.
4. Pour the methanol into the 50 ml Erlenmeyer flask.
5. Pour the FAME into the 50 ml Erlenmeyer flask.
6. Place the magnetic stir bar into the Erlenmeyer flask containing the fame and methanol and place it on the magnetic stir plate.
7. Turn on the stir plate to a medium setting being careful not to splash the contents.
8. Turn the heat on the stir plate to a medium setting.
9. Slowly pour the urea into the solution and continue stirring and heating until the urea has dissolved.
Note: Methanol can evaporate during this process, so work in a hood or well-ventilated area.
You can put a watch glass or glass plate on top of the flask to minimize/prevent evaporation.
10. Place the Erlenmeyer flask containing the solution into an ice bath and cool, observing the formation of a precipitate (clathrate).
11. Perform a liquid/solid separation by decanting the liquid into a 250 ml reaction bottle leaving the magnetic stir bar with the solid. You may also use filtration or centrifugation to separate the solids, if the equipment is available. Discard the solids, which contain urea, fatty acid methyl esters, and traces of methanol by appropriate disposal means.

Washing the fractionated methyl esters (steps 12-19)

The purpose of these steps is to wash/remove residual methanol and urea from the methyl esters. If separatory flasks/funnels are available, these can be used instead of the reaction bottle to wash the methyl ester solutions.

12. Add 45 ml of deionized water to the solution in the reaction bottle.
13. Place a clean magnetic stir bar into the reaction bottle and place it on the magnetic stir plate.
14. Turn on the stir plate to a medium setting being careful not to splash the contents.
15. Mix contents for 2-3 min.
16. After mixing turn off the magnetic stir plate and allow contents separate until two immiscible fluid layers are clearly visible. This may take several hours so you may wish to do this overnight or multiple lab periods.
17. Screw the cap onto the reaction vessel firmly, being careful to avoid squeezing the vessel when handling.
18. The lower layer is water containing residual urea and methanol.
19. Holding the bottle upright, carefully squeeze the bottle to remove the water layer (discard).

Repeat steps 12-19 two more times to remove and residual urea.

20. Checking the cloud point of the fractionated methyl esters
21. The washed methyl esters should now be composed of primarily unsaturated esters, whose cloud point/freezing temperature is much lower than the original biodiesel. You will now compare this by putting samples of both the original biodiesel and the fractionated biodiesel into the freezer.
22. Place samples (approx. 5 ml) of the original biodiesel and the fractionated biodiesel into a freezer.
23. After allowing the samples to reach freezer temperature (Approx. 15 to 30 minutes) examine both samples for the presence of solids.

BENEFITS OF BIODIESEL

ENVIRONMENTAL BENEFITS

- Biodiesel is biodegradable, nontoxic, and essentially free of sulfur.
- Biodiesel reduces most regulated emissions.
- A DOE/USDA life cycle analysis shows biodiesel reduces lifecycle carbon dioxide by 78 percent compared to diesel. The Environmental Protection Agency recognizes that GHG emissions could be cut by as much as 85 percent.
- Biodiesel's GHG reductions make it America's first commercially available, domestically produced advanced biofuel.
- Biodiesel has the highest energy-in, energy-out ratio of any domestic transportation fuel. For every one unit of fossil energy needed to produce biodiesel, 5.5 units of energy are gained.
- Biodiesel production reduces wastewater by 79 percent and hazardous waste by 96 percent compared to diesel. A to-go latte takes 26 times more water to produce than a gallon of biodiesel.
- Biodiesel protects human health, reducing particulate matter by 47 percent and cancer-causing compounds by 80 to 90 percent compared to regular diesel.

PERFORMANCE BENEFITS

- Blends up to B20 can be used in diesel engines with few or no modifications. The majority of U.S. engine manufacturers accept up to B20.
- Biodiesel has the highest energy content (BTUs) of any alternative fuel. It has higher average cetane than diesel and similar fuel economy, horsepower, and torque.
- Biodiesel enhances lubricity, which protects engine life by preventing premature wear and tear.

FOOD SECURITY

- Soybeans have two components – oil and protein meal. Soybeans are grown mostly for their meal, used in food and livestock feed. Biodiesel uses only the oil portion of the soybean, leaving the meal intact to nourish livestock and humans.
- The increased use of biodiesel can have a positive effect on meat and dairy prices. Greater demand for soybean oil makes feed for livestock cheaper than it otherwise would be.



THE BIODIESEL INDUSTRY

The biodiesel industry in the United States consists of a network of over 130 production plants, with an annual capacity of 1.5 billion gallons in 2011. The production of and distribution of biodiesel has contributed \$4.2 billion to the U.S. economy. The industry employs an estimated 50,000 people throughout the United States. Soybean oil the main feedstock for making biodiesel in the U.S. However, due to the wide variety of feedstocks available, biodiesel can be produced nearly anywhere oil or fat is available.



Alaska Green Waste Solutions – Anchorage, Alaska



Pacific Biodiesel – Honolulu, Hawaii

Alaska Green Waste Solutions and Pacific Biodiesel Oahu are two examples of multi-feedstock biodiesel plants operating in Alaska and Hawaii. The wide availability of different feedstock means all regions of the United States can benefit from the production of biodiesel.



CAREERS IN BIODIESEL

A great variety of careers are available in different areas of the biodiesel industry, from university and private research on new feedstock and production methods, agricultural and recycling jobs associated with the production and collection of biodiesel feedstock, as well as technical jobs in building and operating biodiesel plants. Biodiesel industry jobs are available at every skill level, in many different supporting industries and all over the United States. Examples of careers include:

Biodiesel Research

- Chemists, Biochemists, Agricultural, Chemical, Mechanical and Biological Engineers
Feedstock Production and Collection
- Farmers, Truck Drivers, Salespeople, Commodity Traders and Brokers, Technicians
Building Biodiesel Plants
- Agricultural, Chemical, Civil, Electrical, Mechanical Engineers, Welders, Fabricators, Construction Workers
Operating Biodiesel Plants
- Technicians, Chemists, Agricultural, Chemical, Mechanical Engineers, Salespeople

For more information on the biodiesel industry including career opportunities and plant construction, visit the following websites

NBB Website – www.biodiesel.org

Pacific Biodiesel Website – www.biodiesel.com

Biodiesel Jobs Website – www.biodieseljobs.com

BIODIESEL AND SUSTAINABILITY

Biodiesel has a significant role to play in the production of truly “sustainable” renewable fuels. Biodiesel’s high positive energy balance (3.24 BTU output/ 1BTU input) is one of the highest of any commercially available renewable fuel. The feedstock used to produce biodiesel can come from a wide variety of sources, all with their own unique properties and benefits. Soybeans, the primary feedstock for biodiesel produced in the United States, are an advanced commodity crop, with many new innovations being developed each year to improve sustainability through reducing cropping inputs and improving yields. New oilseed crops are also being explored worldwide to make biodiesel without impact of food production. Examples of these new crops include jatropha, camelina, seashore mallow, jojoba and castor oil. Second use feedstocks such as used cooking oil also provide economic and environmental benefits by recycling waste oil resources into biodiesel. With an estimated 2 billion pounds of these waste greases available in the U.S. each year, second use feedstocks are expected to grow as a sustainable feedstock for biodiesel production. An exciting new



area of research focuses on low input production of algae to make oil for biodiesel. Autotrophic algae could potentially be grown in large ponds or photobioreactors to produce oils on a large scale using sunlight, water, and carbon dioxide from the air or a point source such as powerplant exhaust. For more information on biodiesel and sustainability visit:

Sustainable Biodiesel Alliance — <http://www.sustainablebiodieselalliance.com>

Biodiesel Sustainability Blog — <http://www.biodieselsustainability.com>

OTHER USES OF BIODIESEL

Due to their low flammability, excellent biodegradability, and strong solvent properties, methyl esters have found use in a wide variety of industrial and commercial applications beyond use as a fuel. With increasing pressure to improve safety and reduce the health effects of industrial solvents, biodiesel is making inroads in many uses including:

- Adhesive removers
- Paint removers
- Degreasing products
- Sealers
- Release Agents

In addition, methyl esters are increasingly replacing petrochemicals in the production of soaps, personal care products, laundry detergents, and other oleochemicals. With increasing environmental stewardship as a focus of the oleochemical industry, this trend is expected to continue.

USES OF GLYCERIN

Glycerin, the byproduct of biodiesel production, is one of the most versatile platform chemicals used today. It is found in thousands of commercial and industrial products, with new applications being developed each year as supplies increase. Examples of products containing glycerin include resins, explosives, soaps, lotions, pharmaceuticals, emulsifiers for food products, deicers, and heat transfer fluids.



BIODIESEL PRODUCTION

The production of biodiesel, or alkyl esters, is well known. There are three basic routes to ester production from oils and fats:

- Base catalyzed transesterification of the oil with alcohol.
- Direct acid catalyzed esterification of the oil with methanol.
- Conversion of the oil to fatty acids, and then to alkyl esters with acid catalysis.

The majority of the alkyl esters produced today is done with the base catalyzed reaction because it is the most economic for several reasons:

- Low temperature (150°F) and pressure (20 psi) processing.
- High conversion (98 percent) with minimal side reactions and reaction time.
- Direct conversion to methyl ester with no intermediate steps.
- Exotic materials of construction are not necessary.

The general process is depicted below. A fat or oil is reacted with an alcohol, like methanol, in the presence of a catalyst to produce glycerine and methyl esters or biodiesel. The methanol is charged in excess to assist in quick conversion and recovered for reuse. The catalyst is usually sodium or potassium hydroxide which has already been mixed with the methanol.

Nothing is wasted

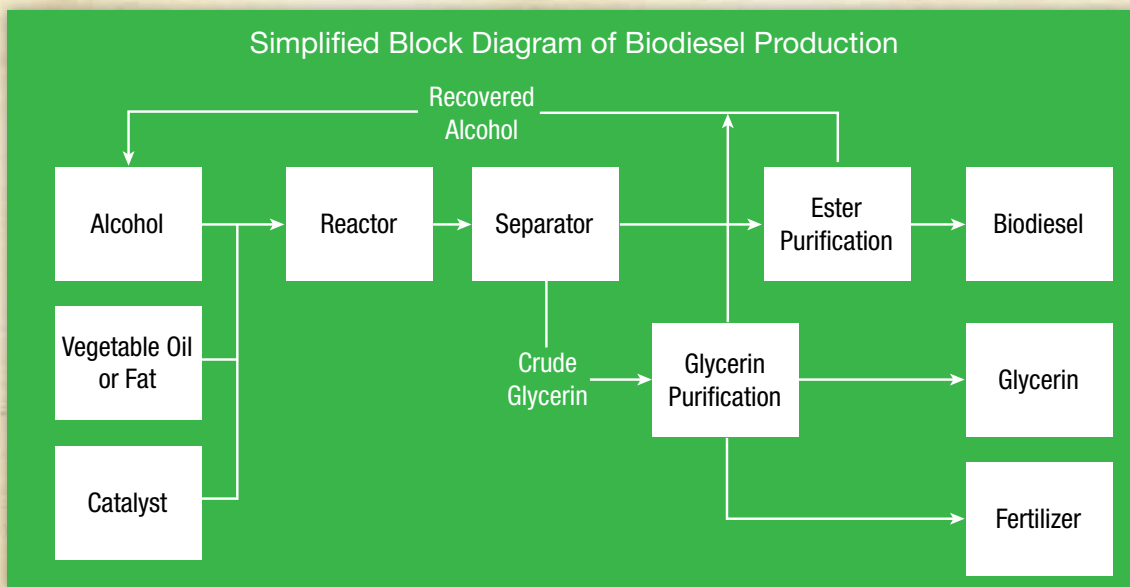
Process Input Levels = Process Output Levels



■ Alcohol 12%
 ■ Catalyst 1%
 □ Oil 87%



■ Alcohol 4%
 ■ Fertilizer 1%
 ■ Glycerine 9%
 ■ Methyl Ester 86%





CHEMICAL REACTION AND ENERGY CALCULATIONS

CHEMISTRY OF TRANSPORTATION FUELS

Most modern transportation today (cars, buses, airplanes, boats) depend on liquid fuels, such as gasoline or diesel fuels. These fuels are combined with air (oxygen) and quickly burned (oxidized) in what are called internal combustion engines, which are arrangements of pistons and cylinders (see section on diesel engines). The oxidation chemical reaction releases heat energy, which is converted into mechanical energy to power the vehicle.

Petroleum-based liquid fuels are called hydrocarbons since they are composed of hydrogen (H) and carbon (C). The main component of liquid fuels are linear hydrocarbons which have the general chemical formula C_nH_{2n+2} . This class of chemical structures are called alkanes (use 'ane' suffix in nomenclature) and uses the Greek nomenclature for numbering.

NAME	FORMULA	STRUCTURAL
methane	CH_4	CH_4
ethane	C_2H_6	CH_3CH_3
propane	C_3H_8	$CH_3CH_2CH_3$
butane	C_4H_{10}	$CH_3CH_2CH_2CH_3$
pentane	C_5H_{12}	$CH_3(CH_2)_3CH_3$
hexane	C_6H_{14}	$CH_3(CH_2)_4CH_3$
heptane	C_7H_{16}	$CH_3(CH_2)_5CH_3$
octane	C_8H_{18}	$CH_3(CH_2)_6CH_3$
nonane	C_9H_{20}	$CH_3(CH_2)_7CH_3$
decane	$C_{10}H_{22}$	$CH_3(CH_2)_8CH_3$

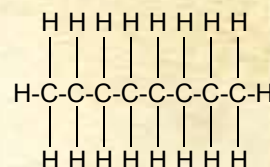
For example, heptane is a linear hydrocarbon containing 7 carbon atoms and 16 hydrogen atoms.

This can be written as:

$CH_3CH_2CH_2CH_2CH_2CH_2CH_3$
or in short hand form as:

$CH_3(CH_2)_5CH_3$.

Often, these structures are drawn as stick figures, such as:



where the lines refer to covalent chemical bonds.

A short hand graphical form of this is presented as:

where the intersection points are CH_2 groups and the terminal points are CH_3 groups.

If the carbon-carbon linkages contain a carbon double bond, the molecular nomenclature of the structure is called an alkene, where the suffix 'ene' is used to denote the presence of a carbon double bond in the structure. The number of the carbon linkage is used to designate the position of the double bond(s) within the molecule.

A more complete set of nomenclature rules for hydrocarbons is given at http://www.acdlabs.com/iupac/nomenclature/79/r79_5.htm

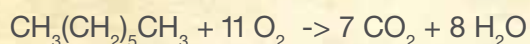


APPENDIX 3

Transportation fuels are generally a mixture of different molecular structures with a range of carbon chain lengths. For example, gasoline is composed of hydrocarbons with carbon chain length range of approximately 6 and 10, i.e. C_6H_{14} to $C_{10}H_{22}$. You may have heard of the term 'octane' in reference to gasoline quality. This is because the combustion behavior of gasoline is usually compared to the combustion properties of pure octane, C_8H_{18} .

Diesel fuel is composed of mixtures of hydrocarbon molecules with carbon chain length range of approximately 14 and 20, i.e. $C_{14}H_{30}$ to $C_{20}H_{42}$. Similar to gasoline, the combustion properties of diesel fuel are compared to the combustion properties of pure hexadecane (also called cetane), $C_{16}H_{34}$. The reason hydrocarbons are used is that they release high amounts of thermal energy when they are combusted with oxygen (oxidation) to form carbon dioxide and water. See Lesson 2 and Appendix on chemical reactions and energy calculations for more details/information on the oxidation reactions and calculating the energy released by oxidation of hydrocarbons and biodiesel.

The reason hydrocarbons are used as transportation fuels is that they release high amounts of thermal energy when they are combusted with oxygen (oxidation) to form carbon dioxide and water. For example, when heptane is completely oxidized the reaction is written as:



This states that every molecule (or mole) of heptane requires 11 molecules (moles) of oxygen (O_2) to be completely oxidized and produces 7 molecules (moles) of carbon dioxide (CO_2) and 8 molecules (moles) of water (H_2O). [Note: A mole is defined as 6.023×10^{23} molecules of a substance. Since different substances are made of different compositions of atoms, it is convenient to define moles in terms of the total atomic mass of the molecule. Hence, the molecular weight is the number of mass units in 1 mass unit-mole of a substance, e.g. 1 kg-mole of carbon dioxide (molecular weight $12 + 2 \cdot 16 = 44$) is equal to 44 kg of carbon dioxide. Alternatively, 1 lb-mole of carbon dioxide is 44 lbs of carbon dioxide (different weight unit). See <http://library.thinkquest.org/12497/molar.htm> if needed.

For any alkane of carbon length n , the molar amount of oxygen (O_2) required for complete oxidation is $(3n+1)/2$. For example, oxidizing undecane ($C_{11}H_{24}$) would require $(3 \cdot 11 + 1)/2 = 17$ molecules of O_2 [$CH_3(CH_2)_9CH_3 + 17 O_2 \rightarrow 11 CO_2 + 12 H_2O$]. Can you calculate the molecular amounts of carbon dioxide (CO_2) and water (H_2O) formed by oxidizing an alkane of carbon chain length n ? (Answers: n carbon dioxide and $(n+1)$ water)

Energy of Oxidation

A chemical reaction is the process of taking apart the atoms that compose a molecule and reforming them into different arrangements. For example, the oxidation of hexane (C₆H₁₄)

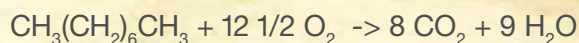


breaks the bonds between carbon/carbon, carbon/hydrogen and oxygen/oxygen atoms and reforms them into new bonds to make carbon dioxide and water. (Note: The process of breaking existing bonds requires energy. The process of forming new bonds releases energy.) The net energy of the reaction (energy required to break the old bonds minus the energy released when forming new bonds) is called the energy of reaction (or enthalpy of reaction). Each chemical bond between atoms has a specific energy. The energy of reaction is obtained by summing up the energies of all bonds broken minus all the bonds formed by a chemical reaction.

In the case of hydrocarbons, the energies of the various bonds are shown below.

BOND	Energy(kJ/mole)
C-C	347
C-H	410
O=O	494
C-O	360
C=O	799
O-H	460
C=C	611
H-H	432

For example, if octane is oxidized



the numbers of bonds broken are:

$$7 \text{C-C} = 7 \times 347 \text{ kJ/mole}$$

$$18 \text{C-H} = 18 \times 410 \text{ kJ/mole}$$

$$12.5 \text{O=O} = 12.5 \times 494 \text{ kJ/mole}$$

Total energy required to break bonds = 15984 kJ/mole

The numbers of new bonds formed are:

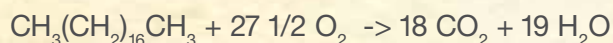
$$16 \times \text{C=O} = 14 \times 799 \text{ kJ/mole}$$

$$18 \text{O-H} = 18 \times 460 \text{ kJ/mole}$$

Total energy released by forming new bonds = 21064 kJ/mole

So the energy of reaction of this reaction is 15984 - 21064 = -5080 kJ/mol. The minus sign indicates that energy is released by this reaction, i.e. an exothermic reaction or more energy is released from forming product bonds than required to break reactant bonds.

As the carbon chain length of the hydrocarbon increases, the amount of energy released by oxidation per mole also increases. Also, the amount of carbon dioxide and water formed also increases. For example, if octadecane is oxidized,



The energy of reaction is -11180 kJ/mol.

Reactant bonds	product bonds
17 C-C bonds@347 kJ/mole	36 C=O bonds@799 kJ/mole
38 C-H bonds@410 kJ/mole	38 O-H bonds@460 kJ/mole
27.5 O=O bonds@494 kJ/mole	

Total energy required to break bonds = 35064 kJ/mole

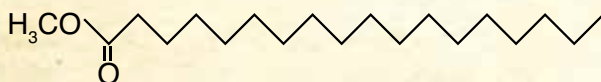
Total energy released by forming new bonds = 46244 kJ/mole

Energy of reaction = -11180 kJ/mole

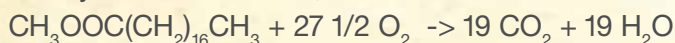


Biodiesel reaction energy

Methyl esters (biodiesel) are very similar to hydrocarbons, but at one end they have a carboxylate group (CH_3OOC) instead of a methyl group (CH_3). Therefore, a methyl ester is partially oxidized since it already has oxygen in its structure. For example, the methyl ester equivalent of octadecane is $\text{CH}_3\text{OOC}(\text{CH}_2)_{16}\text{CH}_3$.



If octadecane methyl ester is oxidized, the reaction is



(Note the stoichiometry changes vs. octadecane since one of the carbons is already oxidized and there is an additional CH_3 group added.)

The energy of the reaction is calculated below.

The numbers of bonds broken are:

$$17 \text{ C-C} = 17 \times 347 \text{ kJ/mole}$$

$$38 \text{ C-H} = 38 \times 410 \text{ kJ/mole}$$

$$1 \text{ C=O} = 1 \times 799 \text{ kJ/mole}$$

$$2 \text{ C-O} = 2 \times 360 \text{ kJ/mole}$$

$$27.5 \text{ O=O} = 27.5 \times 494 \text{ kJ/mole}$$

$$\text{Total energy required} = 36583 \text{ kJ/mole}$$

The numbers of new bonds formed are:

$$38 \text{ C=O} = 38 \times 799 \text{ kJ/mole}$$

$$38 \text{ O-H} = 38 \times 460 \text{ kJ/mole}$$

$$\text{Total energy released} = 47842 \text{ kJ/mole}$$

So the net energy released by this reaction is $36583 - 47842 = -11259 \text{ kJ/mole}$.

Comparing this to normal octane oxidation, the oxidation of the octadecane methyl ester reaction produces 79 kJ/mole more energy than octadecane or about 0.7% more kJ/mole. However, recognize that the molecular weight of the methyl ester (298) is greater than the octadecane (254). If the reaction energy is expressed in terms of mass (kg), then octadecane releases $11180/254 = 44.015 \text{ kJ/kg}$ and the octadecane methyl ester releases $11259/298 = 37.782 \text{ kJ/kg}$. So on a mass basis, the methyl ester releases approximately 14.2% less energy per kilogram than octadecane. Hence biodiesel (methyl esters) produces less energy per unit mass than the corresponding alkane due to the partial oxidation of the biodiesel molecules.

How does the reaction energy converted into mechanical energy power a vehicle?

The energy released by the oxidation reaction is converted into thermal energy of the molecules, which raises their temperature and pressure. The chemical reaction of oxidation of the reactant liquid fuel also results in gaseous carbon dioxide and water products, which have a much greater volume than the original liquid fuel. So if this reaction is conducted in a piston-cylinder (as in a combustion engine, see 4 stroke engine diagram) the increase in gaseous volume causes the piston to move, which results in mechanical motion.

The calculations involved in this energy transformation depend on thermodynamic analysis, which is beyond the scope of this kit. However, if interested, these can be found at http://en.wikipedia.org/wiki/Diesel_cycle for an ideal Diesel engine.



EMISSIONS AND SAFETY

Biodiesel reduces the health risks associated with petroleum diesel.

Biodiesel emissions show decreased levels of polycyclic aromatic hydrocarbons (PAH) and nitrated polycyclic aromatic hydrocarbons (nPAH), which have been identified as potential cancer causing compounds. In Health Effects testing, PAH compounds were reduced by 75 to 85 percent, with the exception of benzo(a)anthracene, which was reduced by roughly 50 percent. Targeted nPAH compounds were also reduced dramatically with biodiesel, with 2-nitrofluorene and 1-nitropyrene reduced by 90 percent, and the rest of the nPAH compounds reduced to only trace levels.

ENVIRONMENTAL & SAFETY INFORMATION ACUTE ORAL

Toxicity/Rates

Biodiesel is nontoxic. The acute oral LD50 (lethal dose) is greater than 17.4 g/Kg body weight. By comparison, table salt (NaCl) is nearly 10 times more toxic.

Skin Irritation – Humans

A 24-hr, human patch test indicated that undiluted biodiesel produced very mild irritation. The irritation was less than the result produced by a 4 percent soap and water solution.

Aquatic Toxicity

A 96-hr, lethal concentration for bluegill of biodiesel grade methyl esters was greater than 1000 mg/L. Lethal concentrations at these levels are generally deemed “insignificant” according to NIOSH (National Institute for Occupational Safety and Health) guidelines in its Registry of the Toxic Effects of Chemical Substances.

Biodegradability

Biodiesel degrades about four times faster than petroleum diesel. Within 28 days, pure biodiesel degrades 5 to 88 percent in water. Dextrose (a test sugar used as the positive control when testing biodegradability) degraded at the same rate. Blending biodiesel with diesel fuel accelerates its biodegradability. For example, blends of 20 percent biodiesel and 80 percent diesel fuel degrade twice as fast as #2 diesel alone.

Flash Point

The flash point of a fuel is defined as the lowest temperature at which the vapor above a combustible liquid can be made to ignite in air. Biodiesel's flash point is over 260° Fahrenheit, well above petroleum based diesel fuel's flash point of around 125° Fahrenheit. Testing has shown the flash point of biodiesel blends increases as the percentage of biodiesel increases. Therefore, biodiesel and blends of biodiesel with petroleum diesel are safer to store, handle and use than conventional diesel fuel.



BIODIESEL MYTHS AND FACTS

Myth: Biodiesel is an experimental fuel and has not been thoroughly tested.

Fact: Biodiesel is one of the most thoroughly tested alternative fuels on the market. A number of independent studies have been completed with the results showing biodiesel performs similar to petroleum diesel while benefiting the environment and human health compared to diesel. That research includes studies performed by the U.S. Department of Energy, the U.S. Department of Agriculture, Stanadyne Automotive Corp. (the largest diesel fuel injection equipment manufacturer in the U.S.), Lovelace Respiratory Research Institute, and Southwest Research Institute. Biodiesel is the first and only alternative fuel to have completed the rigorous Health Effects testing requirements of the Clean Air Act. Biodiesel has been proven to perform similarly to diesel in more than 50 million successful road miles in virtually all types of diesel engines, countless off-road miles and countless marine hours. Currently more than 400 major fleets use the fuel.

Myth: Biodiesel does not perform as well as diesel.

Fact: One of the major advantages of biodiesel is the fact that it can be used in existing engines and fuel injection equipment with little impact to operating performance. Biodiesel has a higher cetane number than U.S. diesel fuel. In more than 50 million miles of in-field demonstrations, B20 showed similar fuel consumption, horsepower, torque and haulage rates as conventional diesel fuel. Biodiesel also has superior lubricity and it has the highest BTU content of any alternative fuel (falling in the range between #1 and #2 diesel fuel).

Myth: Biodiesel doesn't perform well in cold weather.

Fact: Biodiesel will gel in very cold temperatures, just as the common #2 diesel does. Although pure biodiesel has a higher cloud point than #2 diesel fuel, typical blends of 20 percent biodiesel are managed with the same fuel management techniques as #2 diesel. Blends of 5 percent biodiesel and less have virtually no impact on cold flow.

Myth: Biodiesel causes filters to plug.

Fact: Biodiesel can be operated in any diesel engine with little or no modification to the engine or the fuel system. Pure biodiesel (B100) has a solvent effect, which may release deposits accumulated on tank walls and pipes from previous diesel fuel use. With high blends of biodiesel, the release of deposits may clog filters initially and precautions should be taken to replace fuel filters until the petroleum build-up is eliminated. This issue is less prevalent with B20 blends, and there is no evidence that lower-blend levels such as B2 have caused filters to plug.

Myth: Biodiesel causes degradation of engine gaskets and seals.

Fact: The recent switch to low-sulfur diesel fuel has caused most Original Equipment Manufacturers (OEMs) to switch to components that are also suitable for use with biodiesel. In general, biodiesel used in pure form can soften and degrade certain types of elastomers and natural rubber compounds over time. Using high percent blends can impact fuel system components (primarily fuel hoses and fuel pump seals) that contain elastomer compounds incompatible with biodiesel, although the effect is lessened as the biodiesel blend level is decreased. Experience with B20 has found that no changes to gaskets or hoses are necessary.



Myth: No objective biodiesel fuel formulation standard exists.

Fact: The biodiesel industry has been active in setting standards for biodiesel since 1994 when the first biodiesel taskforce was formed within the American Society for Testing and Materials (ASTM). ASTM approved a provisional standard for biodiesel (ASTM PS 121) in July of 1999. The final specification (D-6751) was issued in December 2001. Copies of specifications are available from ASTM at <http://www.astm.org>.

Myth: Biodiesel does not have sufficient shelf life.

Fact: Most fuel today is used up long before six months, and many petroleum companies do not recommend storing petroleum diesel for more than six months. The current industry recommendation is that biodiesel be used within six months, or reanalyzed after six months to ensure the fuel meets ASTM specifications (D-6751). A longer shelf life is possible depending on the fuel composition and the use of storage-enhancing additives.

Myth: Engine warranty coverage would be at risk.

Fact: The use of biodiesel in existing diesel engines does not void parts and materials workmanship warranties of any major US engine manufacturer.

Myth: The U.S. lacks the infrastructure to prevent shortages of the product.

Fact: In 2004 there were 20 active biodiesel plants nationwide with production capacity estimated to be 150 million gallons per year. But, with 20 new biodiesel projects in various stages of development, industry capacity could double over the next 12-18 months. In addition to these new biodiesel projects, there is available production capacity for fuel grade methyl esters (biodiesel) within the oleochemical industry. Although few estimates are available to document this surplus capacity, it has been reported that up to 200 million gallons of additional production capacity could be made available.

Myth: Biodiesel will become a complete replacement for diesel fuel.

Fact: Consumption of diesel fuel in the United States is more than 55 billion gallons annually. Estimates by National Renewable Energy Laboratory indicate that current supplies of animal fats and vegetable oils could accommodate 1.7 billion gallons of biodiesel production annually. NREL also estimates that by 2015 potential new feedstock supplies could increase biodiesel production by an additional 1.9 billion gallons. Therefore, biodiesel should be viewed as extender of our petroleum supplies, not as a complete replacement for diesel fuel.

Myth: There is no government program to support development of a biodiesel industry.

Fact: In October 2004, President Bush signed into law a bill containing a biodiesel tax incentive. The incentive went through Congress as part of the bill known as the "JOBS Bill" and provides a federal excise tax credit in the amount of one penny per percentage of biodiesel made from first-use oils blended with petroleum diesel, and a half penny per percentage for biodiesel made from recycled oils.

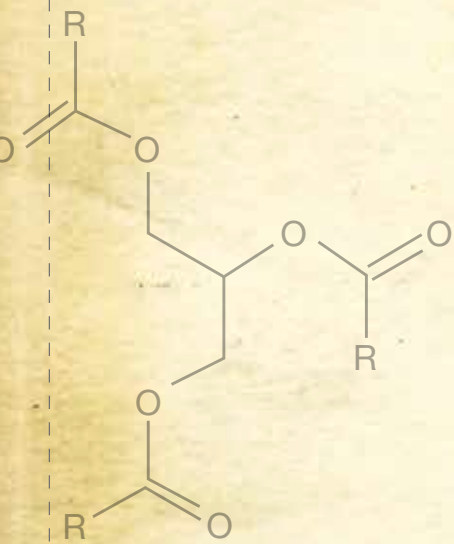


STUDENT SAFETY

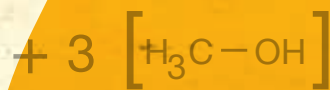
Science should be safe as well as fun, so whenever you're working in the lab with experiments, there are some important precautions you should always keep in mind.

1. Read all the directions before you begin any experiment and if you are not sure about something, ask your teacher!
2. Do not attempt any projects using fire or potentially dangerous substances without your teacher's supervision.
3. Always be careful with chemicals. Never eat or drink anything when you are using chemicals as part of your science experiment.
4. Always protect your eyes by wearing safety glasses.
5. Always clean up after yourself.
6. Always follow your teacher's directions regarding safety.

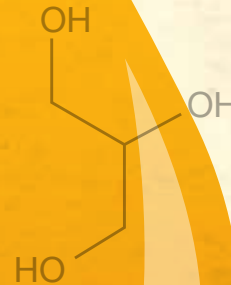
So have fun, be safe, and give yourself a little credit when you collect your Nobel Prize.



Triglyceride

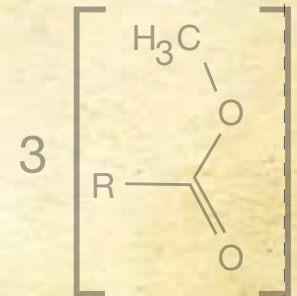


Methanol (3)



Glycerol

+



Methyl Esters



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