Biodiesel Safety and Best Management Practices for Small-Scale Noncommercial Use and Production

PENNSTATE

1855

COLLEGE OF AGRICULTURAL SCIENCES

SAFETY GEAR SUMMARY

The following gear should be on hand each time you produce biodiesel:

- Chemical-resistant gloves (butyl rubber is best for methanol and lye)
- Chemistry goggles (indirect vented) and face shield
- Dust mask or cartridge respirator
- Eyewash bottle with saline solution
- Small spray bottle with vinegar for neutralizing lye spills
- Access to running water
- Telephone in case of emergency and emergency telephone numbers
- Fire extinguishers (ABC or CO_2)
- Absorbent material and spill-containment supplies

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INTRODUCTION

This publication addresses processing and safety issues associated with making biodiesel fuel. Biodiesel is a clean, renewable fuel that can be made from various biomass oil feedstocks such as waste vegetable oil, yellow grease, animal fats, and virgin vegetable oils. Small-scale biodiesel production has been growing due to higher fuel prices, a desire for energy independence, and interest in environmentally friendly renewable fuel production.

Although the biodiesel manufacturing process is fairly straightforward, there are several aspects of biodiesel production that need careful attention to detail for a productive, safe, and environmentally sound practice. First, some chemicals used could pose serious risks to the operator or to the environment, unless the proper precautions are taken for storage, process safety, handling, ventilation, and use. Second, disposal of glycerol by-product and waste water generated from biodiesel production could cause environmental harm, unless approved practices are used. Finally, operators need to pay close attention to the quality of the biodiesel produced and proper storage to avoid costly engine problems or excessive emissions during use.

Most enthusiastic newcomers to biodiesel production will find that successfully running a safe and responsible operation is not as easy as it looks. Production of biodiesel on a small-scale carries inherent risks, and careless producers are likely to have mishaps. While the obvious goal of all producers should be to minimize mistakes, it is also important to know how to deal with these mistakes and respond appropriately. Knowledge, attention to safety, and advanced planning are the best approaches to preventing serious accidents.

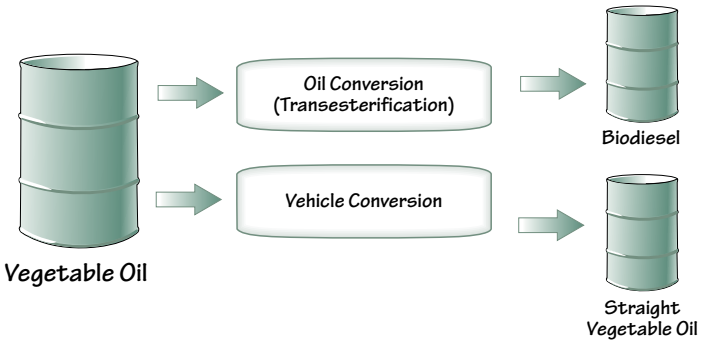
The objective of this publication is to review some of the accepted practices associated with small-scale biodiesel production to make it a safe, environmentally sound practice that generates a quality product. Prospective biodiesel producers are also encouraged to use this information as they decide whether or not to begin making biodiesel. For the preparation of this document, we have solicited input from a variety of experts including chemists, agronomists, environmental engineers, regulators, and educators in the field of small-scale biodiesel production and use. Please note that this booklet provides references to state regulations for Pennsylvania; biodiesel producers in other states are encouraged to check with their state regulators to ensure that they are in compliance with all regulations. A glossary is provided at the end of the booklet which defines terms used throughout the publication.

FUEL OPTIONS FROM BIOMASS OIL FEEDSTOCKS

While early diesel engines demonstrated an ability to run on crude plant-based oils, modern diesel fuel systems are designed for efficiency and low engine emissions and burn fuels of a relatively low viscosity. At average fuel tank temperatures, unconverted vegetable oils are too viscous (too thick) to properly burn in modern diesel engines (especially those with common rail fuel injection systems). Those who wish to run their diesel equipment on raw vegetable oil, used cooking oils, animal fats, or fuels derived from these have the following options: (1) convert the oil to biodiesel, or (2) convert the diesel's fuel system to run heated oils as illustrated below (Figure 1).

Note: For simplicity, the general term "vegetable oils" is used and includes virgin vegetable oils, used cooking oil, and rendered animal fats. Some researchers have experimented with blending unconverted vegetable oils into diesel fuel as a fuel additive or extender. While some diesels may tolerate low percentage (5 percent) blends of vegetable oil in diesel, this information is engine-specific, and higher-percentage blends are not advised. In a different approach, some commercially available products promote blending used cooking oils with unleaded gasoline and additives to reduce the viscosity. This practice is not recommended by the authors of this booklet. Numerous studies by the Department of Energy and others have demonstrated that the use of high-viscosity unconverted oils at levels as low as 10 percent may cause engine wear, injector deposit formation, and premature engine failure. Anecdotal reports by diesel service personnel also report several cases of problems with injector pumps and other parts linked to the use of high-percentage blends of unconverted vegetable oils with diesel fuel. Engine manufacturers may not honor warranties if raw oils or any other fuel products not meeting fuel quality specifications are used as fuel.

FIGURE 1



Biodiesel—Converting the Oil

Biodiesel fuel must not be confused with crude, untransesterified vegetable oil. Transesterification is a chemical reaction wherein oil molecules react with alcohol in the presence of a base catalyst such as lye. This somewhat-reversible reaction results in alkyl esters, also known as biodiesel, and a crude glycerol by-product. The American Society for Testing and Materials (ASTM), a consensus organization of users, producers, and manufacturers, clearly specifies fuel quality standards for biodiesel.

Some people use the two terms "soy-diesel" and "biodiesel" interchangeably. In certain regions of the United State, soy-diesel refers to crude soybean oil that is filtered and may be clarified, while in other regions, both terms refer to biodiesel fuel that has been properly processed (transesterified) from soybean oil. In industry literature, FAME is an acronym that is often used for Fatty Acid Methyl Esters (biodiesel). In this document, biodiesel will refer only to transesterified oil from a variety of feedstocks.

Biodiesel fuel can be burned in any diesel engine without major modifications to the equipment. Biodiesel blends readily with petroleum diesel. Fuel blends are referred to by the percentage of biodiesel in the mix (e.g., B20 is 20 percent biodiesel). Biodiesel fuel can also be blended with diesel fuel oil for use in heating appliances. Those who own diesel equipment under warranty are advised to check with their manufacturers regarding recommended percentage limits for biodiesel blends. Manufacturers require that any biodiesel fuels used meet the ASTM D6751 specifications for valid warranties.

"Straight Vegetable Oil"—Converting the Diesel Equipment

The option known as straight vegetable oil (SVO) conversion requires altering the fuel system of the diesel engine to heat the vegetable oil, thereby reducing its viscosity. There are a number of commercially available kits, as well as homemade conversions, most of which function on the same basic principles. This approach is described in Appendix A, but research is ongoing to review potential engine problems and environmental concerns (increased emissions). Although this booklet describes safety and environmental issues pertaining to small-scale biodiesel production and use, some of the regulations and safety information will be useful to SVO system users.

Both biodiesel and SVO systems begin with vegetable oil. In the case of biodiesel, the oil is modified to suit the engine. In the case of SVO, the fuel system of the engine is modified to suit the oil.

USING BIODIESEL—BEST MANAGEMENT PRACTICES FOR VEHICLES, MACHINERY, COLD WEATHER, AND STORAGE

Transitioning to Biodiesel

Whenever switching to biodiesel or a biodiesel blend fuel in an engine or storage tank that had been running on petroleum diesel, it is recommended to monitor or change fuel filters on a more frequent basis at least for the first six months after the transition. Biodiesel is an excellent solvent and, as such, it will readily dissolve many of the deposits in a diesel engine, fuel injectors, fuel supply lines, and storage tanks that have accumulated over the years of engine operation with petroleum diesel. Continue changing fuel filters on an as-needed basis until the system has been cleaned of the deposits. When high-percentage blends of biodiesel are used in road vehicles, it is wise to carry a spare fuel filter and the tools and knowledge to change it on the road.

Fuel Storage

Most of the standard storage and handling procedures used for petroleum diesel can also be used for biodiesel. The fuel should be stored in a clean, dry, dark environment. Recommended materials for storage tanks include aluminum, steel, polyethylene, polypropylene, and Teflon, but not concrete-lined storage tanks. If possible, the storage tank should not include any copper, brass, lead, tin, zinc, or rubber fittings if possible (practically speaking, brass ball valves are used by many with no major ill effect). Since biodiesel is an organic liquid, the use of an algaecide or fungicide additive is recommended whenever the fuel is stored during warm weather. Storage time for biodiesel and petroleum diesel should be limited to six months for best performance. Refer to the sections covering Regulatory Issues and Storage Issues for information on regulations that apply to storage tanks.

Biodiesel in Vehicles/Machinery

Burning ASTM 6751 biodiesel is a recommended, accepted practice consistent with many engine warranty agreements. As discussed later in this booklet, the biodiesel that you produce may not meet this standard. Marginal-quality biodiesel may eventually affect engine emissions, longevity, and performance.

Biodiesel can be blended with petroleum diesel fuel in blends up to 20 percent (B20) with relatively few risks. Penn State is doing long-term studies with tractors using B100 (100 percent) biodiesel. The potential for problems with using B100 is expected to involve the rubber seals and hoses in the fuel system. As mentioned above, biodiesel is a better solvent than petroleum diesel and may soften or dissolve rubber parts, especially older formulations of rubber. Replacing old rubber lines with newly manufactured rubber is often sufficient. Since biodiesel is a good solvent, it is necessary to quickly wipe any biodiesel spills from painted surfaces to avoid paint removal.

Biodiesel can also be used in residential oil furnaces or boilers, especially if it is blended with petroleum fuel oil. The same issues that apply to vehicle tanks and storage tanks apply here—the deposits in the bottom of the fuel tank may dissolve with the addition of biodiesel. It is recommended to start with a low concentration of biodiesel relative to petroleum diesel, changing the fuel filter more frequently to ensure that any dissolved tank-bottom sludge is captured by the fuel filter. Fuel pump seals that are made of rubber will have the same issues of degradation that vehicles and machinery will, particularly at higher blends. Biodiesel should be blended with petroleum diesel prior to filling the fuel tank to ensure adequate mixing. If this is not practical, at a minimum the biodiesel should be added to the tank after the petroleum fuel, as it is slightly denser and some blending will occur as it settles to the bottom of the tank. Since biodiesel burns with a cleaner flame than heating oil, it may confound flame-sensing components in heating appliances. Users who wish to experiment with high-percentage blends are advised to consult a professional heating contractor before doing so. See the References section for more information.

Cold-Temperature Issues

Biodiesel and its blends with petroleum diesel will cloud and gel at higher temperatures than normal winterized petroleum diesel fuel. As temperatures drop, wax-like crystals form in the fuel. These crystals will potentially clog fuel filters or fuel-line inlet screens, causing stalls or failure to start. Plugged fuel filters may strain injection pumps, possibly reducing the longevity of these expensive components. Users are advised to prepare for cold temperatures in advance in order to avoid vehicle and equipment problems associated with cloudy or solidified fuel. The petroleum diesel used in cold climates should be winterized diesel fuel.

A fuel's cloud point (CP) is the temperature at which it first begins to appear cloudy, as crystals start to form. The pour point (PP) is the minimum temperature at which the fuel can be considered a liquid—below this temperature the fuel will be fully gelled. The cold filter plugging point (CFPP) is usually closely related to the cloud point, thus testing for cloud point may be the most practical way to predict a fuel's winter performance. Commercial fuel producers are required to notify customers of a given fuel's pour characteristics, and small-scale producers are advised to be aware of pour points in the biodiesel they produce. The recent introduction of ultra-low sulfur fuel (ULSF) makes this more important, since early indications are the high ULSF can also contribute to the low temperature problem.

Cloud point and pour point of biodiesel will vary according to feedstock. Of the vegetable oils commonly available in the U.S., canola oil is the best for producing winter biodiesel fuel, with a cloud point in the 20- to 30-degree range (F). Soy biodiesel typically clouds around 32 degrees Fahrenheit (0 degrees Celsius). Biodiesel made from animal fats, tallow, or hydrogenated (creamy) vegetable oils will cloud and gel at temperatures above freezing, and thus these fuels perform best in southern regions or summertime.

Freezer Test

Small producers who use oils from multiple sources are advised to test each batch of biodiesel fuel for cold weather performance. A simple test using jars, a freezer, and a thermometer is effective to determine proper winter blending rates. Blends of biodiesel and winterized petroleum diesel fuel of varying proportions (e.g, B10, B20, B50) are made up in small jars and then placed in the freezer. By frequently checking the temperature of each jar, a producer can roughly determine the temperature at which clouding and gelling will occur for each blend. Knowing the expected low temperature, users can then predict which blend will be trouble free. Extreme care must be taken not to spill diesel or biodiesel blends in a freezer that is used for food storage. Use a plastic tub for secondary containment, and place in a secure, level spot. Do not leave fuel in freezer where others may disturb it accidentally.

A rough version of this test can be performed by leaving marked blended jars of fuel in an outdoor, unheated location during the winter. It is best to start blending biodiesel with petroleum diesel conservatively well before winter to prevent filter plugging in unexpected cold snaps.

Additives

Several fuel additives are available for winterizing petroleum diesel fuel, and a handful are specifically designed to winterize biodiesel blends. A 2005 study at the University of Idaho found several commercial biodiesel additives to be effective at reducing the pour (gel) point of blends, but they had little effect on the pour point of B100. Furthermore, the additives did not significantly reduce the

cloud point (key to prevent filter plugging) of biodiesel blends, even at several times the recommended usage rates. Some individuals do report successful reduction of cloud points with various unconventional additives, but users are advised to test blends before using in their equipment and to err on the side of caution.

Preparing for Winter Trouble

It is advisable to be familiar with the fuel system in vehicles and machinery using biodiesel in case any winter troubles arise. It is always helpful to carry a spare fuel filter and the tools to change it, especially in winter. If equipment stalls due to fuel starvation, exchanging the waxed filter for a fresh one filled with petroleum diesel may get it back in service. A spare five-gallon tank of fresh winterized petroleum diesel (or kerosene for off-road equipment) can be quite handy. If temperatures drop unexpectedly below the cloud point of biodiesel in the equipment's fuel tank, adding fresh diesel to top off the tank may help prevent clouding and filter plugging. This is especially effective if the diesel fuel is stored in a heated space. Block heaters are effective at keeping the engine warm enough to start, but if a gel layer forms in the fuel tank, problems will persist. When all else fails, the vehicle can be towed to a heated garage to thaw out. For more information on winter preparations, see References.

Blending for the Cold Season

It is best to blend biodiesel with winterized petroleum diesel fuel (cut with kerosene by the fuel distributor). Small producers will typically "splash blend" by simply pouring the two fuels into a tank at the desired proportions. Biodiesel will mix readily with diesel fuel simply through the agitation generated by driving a vehicle. For stationary tanks, it is best to add biodiesel on top of petroleum diesel, as biodiesel is denser and will settle to the bottom, blending on its way down. When blending, both biodiesel and petroleum diesel should be at temperatures above the biodiesel cloud point, as experience has shown that fuels blended at cold temperatures may not stay mixed. For this reason, small producers working in unheated shops may want to blend up a large batch of winter fuel before outdoor temperatures drop in the late fall. If biodiesel should cloud or gel, it will need to be warmed to well above its cloud point to fully dissolve waxy crystals that formed at low temperatures. Note: When blending fuels, tanks should be grounded to avoid static charge buildup (see Chemical Handling).

BIODIESEL—SHOULD I MAKE IT MYSELF?

We begin this section assuming that you are already committed to using biodiesel, and are deciding if you should make it yourself. Making biodiesel fuel yourself offers many challenges. For some people, the enjoyment of the process and the reward of making your own fuel outweigh the challenges. This is for each person to determine. While converting one gallon of oil into biodiesel is simple and fairly easy to learn, processing and finishing hundreds of gallons of waste fryer oil into fuel will present new challenges with each different batch of oil. Reading to become familiar with the finer details of processing, safety protocols, and new developments in quality testing is essential. This section provides some things to consider when deciding if you want to proceed with small-scale production of biodiesel.

Economics

It is common for new producers to underestimate the time they will invest in the myriad aspects of fuel production. An analysis can be performed of the cost of inputs versus the resultant value of the fuel produced. Whether or not labor cost will be accounted for depends on the individual production facility, and whether biodiesel is made as a hobby, or in place of other income-generating work.

Costs to consider in determining the economics of small-scale biodiesel production include:

- Capital investment in equipment—this may include purchasing new equipment and acquiring a location to produce and store the biodiesel. Capital costs may be negligible if production primarily uses recycled materials in an existing well-ventilated space.
- Feedstock acquisition—procuring the oil, whether free for pickup or purchased
- Chemicals—these may be bought in bulk or small quantities
- Disposal cost of by-products
- Permit fees
- Electricity/energy—this includes the electricity to run the process, but may also include heat and ventilation for the facility
- Labor—you may or may not choose to include this in your costs

Since costs and availability of feedstocks vary, your cost should be compared to quality commercial biodiesel fuel before deciding to make your own fuel.

Feedstock Availability

Each finished gallon of biodiesel produced will require slightly over one gallon of feedstock, either new vegetable oil or filtered waste fryer oil from restaurants. Prospective biodiesel producers should consider the amount of fuel they hope to produce, and then be certain that they will be able to obtain and handle the necessary feedstock oil on a regular basis. As production volumes increase, the logistics of collecting large amounts of waste fryer oil may become more cumbersome. In addition, as more people produce biodiesel, there may be competition for waste fryer oils. Lastly, it should be noted that working with used cooking oil is somewhat more complicated than virgin oils, due to the varying acidity, moisture content, and quality of used oil.

Compliance with Environmental Regulations

Biodiesel production facilities may be subject to regulation by the Department of Environmental Protection (PADEP) and other entities, depending on their size and commercial status. Currently, small-scale, noncommercial facilities are exempt from PADEP permitting, but will be subject to "discretionary enforcement" if problems or complaints arise. Other aspects of small-scale biodiesel production may be regulated. A summary of the regulations which may apply to your facility is given in Regulatory Issues. We strongly encourage anyone who is considering small-scale biodiesel production to carefully review the regulatory requirements in order to avoid any violations of the law.

Time Commitment

While biodiesel that is made from free used cooking oil has a reduced materials cost, the time commitment for processing should not be overlooked. To responsibly operate a small-scale biodiesel facility, you should allow time for all of the following:

- Fabrication and maintenance of biodiesel equipment
- Oil collection
- Securing chemicals
- Fuel processing
- Methanol recovery
- Water washing of the fuel or other finishing techniques
- Quality testing
- Disposal of waste products

Those who do not have ample time in their schedule for a new project are not advised to begin biodiesel processing purely as a means of saving money.

Messiness

Despite careful handling, biodiesel processing will commonly result in some "greasy spots on the driveway" or in the farm yard. Collections of oil jugs and barrels can become unsightly unless strictly managed. Leaking or seeping occurs in some plastic jugs over time. In addition, there may be an objectionable odor associated with spoiled used fryer oil. Vehicles used to collect fryer oil, buildings used for housing biodiesel gear, and clothing worn during processing are also likely to be affected by sticky fryer oil over time. Keeping the work area clean adds labor cost to the production of the fuel, but is a worthwhile safety practice.

Chemical, Waste, and Safety Issues

Chemical Exposure and Safety

Methanol (a flammable, toxic alcohol) and lye (a corrosive, caustic base) are two hazardous chemicals required to convert vegetable oil into biodiesel. Overexposure to methanol can cause neurological damage and other health problems; and it presents a serious fire risk. Lye can cause skin and lung irritation. Both methanol and lye can cause eye damage or blindness. Rigorous precautions are necessary to avoid personal poisoning, fire, and contamination of soil and water resources. Before beginning a biodiesel project, please consider whether or not you can responsibly follow the safety protocols outlined in Process Hazards, Safety Precautions, and Handling Issues.

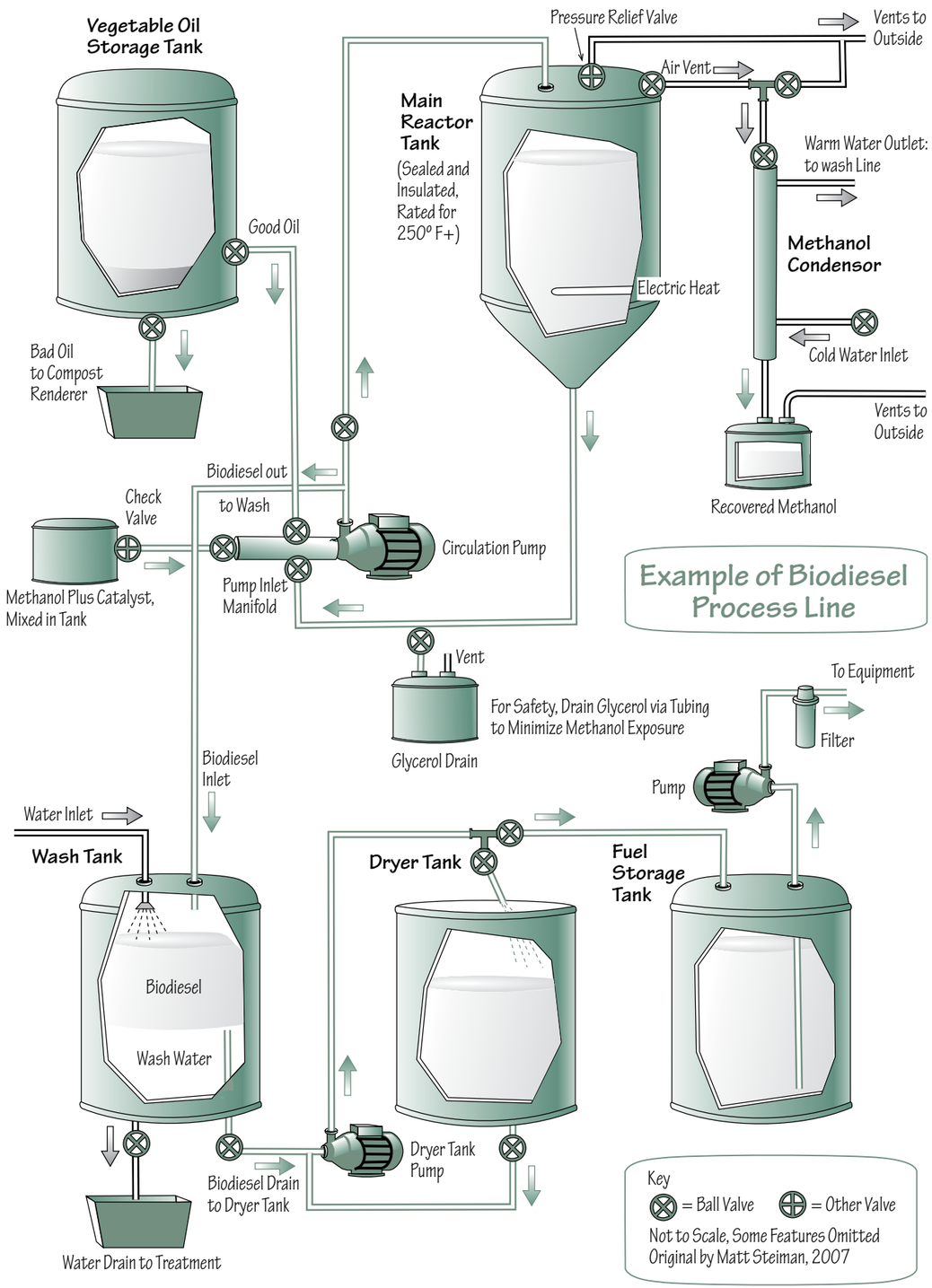
Handling of By-Products

Biodiesel processors generate substantial quantities of crude glycerol by-product (about one gallon of waste product containing glycerol for every five gallons of biodiesel produced). Most processors also use water for fuel purification, and may generate as much as three gallons of waste water for each gallon of fuel produced. Both glycerol and waste water require handling and disposal consideration (see By-Product Handling and Disposal for further information).

Responsibility for Vehicle/Equipment Failure

The moment that a small producer pours the first gallon of homemade fuel into a fuel tank, that producer takes responsibility for the future performance of the equipment in question. While there are thousands of examples worldwide of small producers successfully powering diesel equipment with homemade fuel, users should also understand that problems can and do occur. Careful attention to production chemistry and fuel-quality

FIGURE 2



testing is a must, as is care in developing climate-specific winter blends. A basic understanding of the mechanics of diesel equipment and a readiness to address any fuel system problems that may arise are advisable. Small-scale producers are cautioned to develop their production skills while using their fuel in older, inexpensive equipment, before running their biodiesel in newer, more expensive, and essential equipment. Homemade (non-ASTM certified) biodiesel fuel should not be used in equipment that is under warranty if you expect the warranty to remain valid.

BEST MANAGEMENT PRACTICES FOR SMALL-SCALE BIODIESEL PRODUCTION

What follows is a summary of practices for small-scale biodiesel production developed with the goal of producing high-quality biodiesel fuel in a manner that is safe, environmentally responsible, and labor efficient. This section is not meant as a "how-to" manual, but instead a summary of regulations and guidelines for small-scale production in Pennsylvania. See References to find information on how to produce biodiesel.

Process Description

While one commonly followed version of the biodiesel process is presented here (Figure 2), there are a multitude of alternative methods through which small producers successfully transform feedstock oils into quality biodiesel. Producers are encouraged to stay up-to-date with Internet and printed resources, as new practices for safe and efficient processing are constantly being devised by the small-scale production community.

Simplified Overview of Processing Steps

1. Collect feedstock: waste oil from restaurants, pressed oil from oilseed crops, render animal fats, trap grease, etc.
2. Check oil for water content (see Biodiesel Quality Test Methods) and de-water oil if necessary. (Settling collected oils in large bulk tanks/drums, then drawing oil for processing from the top of the tank/drum may be sufficient in warmer weather.)
3. Filter oil to remove food particles and debris.
4. Fill processing equipment with oil and begin heating.
5. Test oil for free-fatty acid content via titration to determine the amount of catalyst (NaOH lye or KOH) to be used per liter of oil (see Biodiesel Quality Test Methods).
6. Make a mini batch with the heated oil (500 mL or 1 liter) to test the accuracy of the recipe being used before proceeding to a large batch. Mason jars make cheap and effective test batch vessels, provided they have tight-fitting lids.
7. When oil has reached the reaction temperature of 120 to 130 degrees Fahrenheit (50 to 55 degrees Celsius), proceed to mix the large batch using the recipe from a successful test batch. Carefully mix lye with methanol, add chemicals to oil, mix for up to two hours, and then allow glycerol by-product to settle.
8. Make accurate records of all variables in each batch of fuel: oil source, mixing time, temperature, quantities of reactants, any other processing variables.
9. Drain glycerol by-product and label for further processing, temporary storage, or disposal. The glycerol will contain significant methanol and should be handled as such (flammable and toxic).
10. Recover excess methanol from raw biodiesel. If methanol is not recovered from biodiesel, the raw fuel should be treated as if it were methanol (flammable and toxic).
11. Pump biodiesel to wash tanks and wash.
12. Allow water to settle out over several days or dry biodiesel via "active drying."
13. Pump through a filter to fuel storage barrels.
14. Test fuel for quality parameters and compare with processing records.
15. Recover methanol from glycerol once sufficient quantities are on hand.
16. Dispose of glycerol and wash water in a safe and responsible manner (see By-product Handling and Disposal).

Regulatory Issues

There are regulations, both state and federal, which cover various aspects of biodiesel production, storage, and disposal. Knowledge of which regulations apply to you is essential. Furthermore, you may be subject to specific requirements from your local municipality, so it is important to check the local regulations and ordinances. You should check with your local County Emergency Management Agency, fire officials, and your local Department of Environmental Protection office prior to initiating any chemical storage, transportation, or handling practices on your premises, and before disposing of any waste materials. The regulations change frequently for these types of activities.

It is critical to realize that your goals and those of the regulators are the same—to protect the environment, your

health, your property, and the general public. Don't be afraid to report a spill or to ask for assistance.

The following list gives a summary of federal and state requirements which may apply to your facility. The acronyms for the regulatory agencies are defined below. Contact information for each agency is listed in References, as is the Web address for Pennsylvania's regulations.

- Environmental Protection Agency (EPA)
- Occupational Safety and Health Administration (OSHA)
- Pennsylvania Department of Environmental Protection (PADEP)
- Pennsylvania Department of Labor and Industry (PADL&I)
- Pennsylvania Department of Revenue (PAREV)

Issues and Regulations That May Apply

Biodiesel production from used cooking oils or other used animal fats

- PADEP requires a commercial processor (an entity that produces biodiesel for sale) of used cooking oil, yellow grease, oils and fats from rendering plants, etc., to have a general permit for beneficial use under the residual waste regulations. Farm or home production of biodiesel that is not for sale is exempt from these requirements.

Storage tanks

- EPA requires a Spill Prevention, Control, and Countermeasures Plan for aboveground storage of oils (any type including vegetable, petroleum, animal fats, etc.) of 1,320 gallons or more or underground storage of 40,000 gallons or more (Title 40, Code of Federal Regulations, Part 112).
- PADEP regulates aboveground storage tanks that contain hazardous substances (such as methanol) of 250 gallons or more or underground storage tanks of 110 gallons or more (Title 25 Pa. Code Chapter 245). They also regulate storage tanks at residences or farms that contain biodiesel or blends of biodiesel and petroleum diesel if they contain more than 1,100 gallons. If tanks contain waste cooking oils which were generated by a residence, municipal building, institution, or commercial establishment (such as a restaurant) and the biodiesel is made by a small-scale noncommercial producer, the Municipal Waste Management storage regulations apply. These require that the aboveground tanks be properly constructed and have secondary containment. Underground tanks must be checked annually for leaks. (Title 25 Pa. Code 285.122). If the oil was generated by a food processing plant or was an agricultural waste product such as on-farm renderings, then Residual Waste Management storage regulations apply. These tank regulations are considerably more complex and biodiesel producers are advised to contact the DEP (Title 25 Pa. Code 299.122).
- PADL&I regulates storage tanks of 30 gallons or more that contain blends of B60 or lower (flashpoint 200 degrees Fahrenheit [93 degrees Celsius] or lower). Tanks that contain fuels used for heating or generators that are less than 3,000 gallons are exempted. (Note: In Allegheny and Philadelphia counties, this regulation is under the jurisdiction of the county fire marshal, not PADL&I). See Title 37 Pa. Code Chapters 11, 13, and 14.

Oil and hazardous-material storage

- PADEP may require a Preparedness, Prevention, and Contingency Plan if hazardous materials and/or oil are stored in quantities of 55 gallons or more without secondary containment and they have the possibility of entering the environment (soil, stormwater drains, surface water, etc.) if spilled or leaked (see References for detail).

Oil transportation

- Used cooking oil that has been generated by a residence, municipal building, institution, or commercial establishment (such as a restaurant) must be transported as Municipal Waste (25 Pa. Code 285.201). Used oils generated by food-processing plants must meet the same requirements but must be labeled "Residual Waste" (25 Pa. Code 299.201). See the Storage Issues section for more details.

Safety

- OSHA regulates employee safety and provides standards for workplace hazards.

Oil and hazardous-substance spills

- EPA regulates spills of 40 gallons of oil or more (this includes used cooking oil and biodiesel) to waterways or where a visible sheen is present; spills of this size must be reported to the National Response Center (Title 40, Code of Federal Regulations, Part 110). Spills of methanol that exceed 5,000 lbs (758 gallons) and potassium hydroxide or sodium hydroxide that exceed 1,000 lbs must also be reported to the National Response Center (Title 40 Code of Federal Regulations, Part 302).

- PADEP regulates spills of hazardous substances and oil that could pollute the "waters of the Commonwealth." This is interpreted to include groundwater and surface water; all spills must be reported to the regional PADEP office. Prevention of spills from waste materials in storage (including containers) are regulated by Title 25 Pa. Code 285.121

Air quality

- PADEP regulates air quality and requires a plan approval for facilities that will have methanol emissions of one ton or more (see the Tracking Emissions section for a discussion of methanol emissions). A Request for Determination (RFD) can be made through the PADEP's Web site (see the References section). In addition, no malodors can leave the property (Title 25 Pa. Code Chapter 127).

Disposal of waste products

- PADEP regulates the waste products generated by the biodiesel process as at least residual wastes, and in some cases, such as pure methanol, as hazardous wastes. Any waste that meets the characteristics of a hazardous waste under Title 25 Pa. Code 261.20 has to be handled as a hazardous waste. The waste generator is responsible for appropriate chemical testing and recordkeeping.

Wash Water

- PADEP does not allow this to be discharged into on-lot septic systems or into storm sewers. It is at least a residual waste and may be a hazardous waste, depending upon its characteristics (see above). Disposal to a municipal wastewater treatment plant must be approved by the operating agency of that facility.
- Local municipalities may regulate disposal of wash waters to the treatment plant; best management practices to neutralize this wash water are given in the By-Product Handling and Disposal section and must be approved by the treatment plant prior to disposal.

Glycerol

- PADEP does not allow this material to be disposed of by either land application or composting if it contains methanol. It is regulated as a residual waste or possibly a hazardous waste, pending characteristic test results under Title 25 Pa. Code 261.20, and must be disposed at a permitted landfill pending test results and approval from the Landfill Operating Authority and the Waste Management Facilities Permitting Section of the Regional PADEP Office. If the methanol has been removed, the glycerol may be composted or used as a dust suppressant on roads, but a permit from the DEP is required. See By-products Handling and Disposal, for further discussion.

Methanol, Lye

- EPA and PADEP regulate these materials as hazardous wastes once they are determined to be wastes (Note: This is *not* household hazardous waste, it is from a chemical reaction producing combustible fuel, and has nothing to do with a normal household activity). See Process Hazards, Safety Precautions, and Handling Issues for proper storage and disposal of hazardous wastes.

Bad vegetable oils

- PADEP regulates this as a residual waste. It may be disposed of in a permitted landfill (with approval from the Landfill Operating Authority) or composted. Consult the Waste Management Facilities Permitting Section of the Regional PADEP Office.

Public Highway Road Tax

- PAREV regulates the use of any fuel used in a diesel engine on the road. If a producer makes less than 1,000 gallons of biodiesel per year for road use, the "road tax" is prepaid for 1,000 gallons and reduced at the end of the year based on the actual volume produced. If 1,000 gallons or more are produced per year, the producer must be licensed and bonded and pay monthly fees.

Federal Excise Tax

- The IRS requires that biodiesel producers (including small-scale production for personal use) register (Form 637), report, and pay excise tax for biodiesel that is produced for use in on-road registered motor vehicles. The vehicle operator is liable for the federal excise tax of $0.244/gallon on the B100 fuel using Form 720. A biodiesel producer may claim the biodiesel blenders credit if the fuel is mixed with at least 0.01% diesel and/or may claim the biodiesel producer's credit. For both of these credits the biodiesel must meet the ASTM requirement D6751 and the requirements of the Clean Air Act. In order to meet the Clean Air Act requirements, a small producer would have to join the Nation Biodiesel Board, which is likely too cost prohibitive for most small-scale non-commercial producers. These biodiesel fuel credits may be claimed using Form 8864.

Process Hazards, Safety Precautions, and Handling Issues

Safety should be the top priority in any biodiesel operation, above all other goals. Accidents involving chemicals and or large volumes of vegetable oil, biodiesel, or by-products can cause injury, loss of life, property damage, or environmental contamination. Those who hope to avoid future government prohibition of small-scale biodiesel production would do well to consider that regulation often follows accidents. By following "best practices" for safety, small producers as a group will continue to remain in compliance with officials responsible for public and environmental health.

A comprehensive approach to safety begins with a whole-system consideration of all potential areas for risk, followed by thorough plans for accident prevention. As a backup, preparations should also be made for response to any accident that may occur.

As a general rule, users should obtain and read a copy of the Material Safety Data Sheet (MSDS) for methanol and lye, and be familiar with the safety considerations for each chemical. A "best practice" is to create a clearly labeled "safety station" within the processing facility, where the MSDSs are kept readily accessible. This will allow workers and fire or emergency personnel to readily locate chemical safety information in case of an accident.

It is recommended that small-scale producers contact their local fire station to advise them of the processing and chemical storage that may be occurring on site. This will serve to warn safety personnel for their protection in case of a fire, as well as to give fire officials a chance to help small-scale producers address any potential areas of concern. Those who are unwilling to invite fire safety personnel to their facility would do well to consider their ability to responsibly produce biodiesel without incident.

Accidents tend to occur when operators are tired, distracted, or hurried. Producers are advised to work slowly and thoughtfully, and to avoid juggling too many tasks at once in the biodiesel plant. Temporary rigging of equipment and shortcuts are high-risk behaviors that often lead to unforeseen consequences.

Chemical Handling

Methanol is toxic and must be handled and used in a well-ventilated area. Inhalation or ingestion of methanol can be very harmful at higher concentrations and can lead to death or blindness. It is especially damaging to the eyes; safety goggles, chemical-resistant clothing, and gloves must be worn whenever handling the material. If concentrations in the air exceed 200 ppm, air-supplied respirators are required, preferably with a full-face mask. (Canister respirators are not effective for regular use in methanol vapors). The level at which the odor of methanol is perceptible is greater than 200 ppm. Thus, if one "smells" methanol in the processing facility, unhealthy personal exposure is already occurring. Producers are advised to alter any processing activity that results in perceptible methanol odors.

There have been many instances where methanol was ingested purposely because of its mildly intoxicating effect (much like ethanol). Ingestion has led to numerous cases of death or blindness, and precautions must be taken to keep this chemical away from children and animals. Methanol ingestion, inhalation of high concentrations, and any contact with the eyes requires immediate medical attention. Short-term exposure to methanol vapor can irritate the eyes, nose and throat, and cause headache, nausea, vomiting, dizziness, and trouble breathing. Other common symptoms of drunkenness, such as lightheadedness, giddiness, blurred vision, and dilated pupils, might also appear. The symptoms depend on the level and length of exposure and can vary from person to person.

Methanol will readily dissolve into water and if released without proper treatment, can get into the water table.

Methanol is flammable and presents a fire hazard. It is heavier than air and can travel a substantial distance to find an ignition source with subsequent flashback to the processing unit or methanol storage tank. Methanol's flash point is a rather low 52 degrees Fahrenheit (11 degrees Celsius) which is the point whereby sufficient vapor is released to form a potentially flammable mixture. At biodiesel processing temperatures (110 to 140 degrees Fahrenheit), there is enough methanol vapor generated to sustain a serious fire if air is allowed to mix with the methanol.

Sodium hydroxide (NaOH) and potassium hydroxide (KOH) are corrosive and may be fatal if ingested. (These chemicals are both referred to as "lye" or "catalyst" elsewhere in this booklet.) Skin contact can cause severe burns and the area affected should be thoroughly flushed with water or a dilute vinegar solution. Inhalation of the solid NaOH or KOH is possible if the material is reduced to dust-sized particles. Any of these situations is critical, requiring immediate medical attention. Sodium and potassium hydroxide must be stored away from water since water will inhibit the biodiesel reaction as well as cause heat release due to mixing, which can potentially cause a fire in adjacent material. NaOH, KOH, and concentrated solutions should never contact aluminum as they will create explosive hydrogen gas.

Proper safety gear for working with NaOH or KOH includes elbow-length gloves, chemical safety goggles, a dust mask or respirator, long pants, and shoes. An eyewash station and/or emergency shower within 25 feet of the workspace are also highly recommended. (A homemade eyewash station can be as simple as a dedicated garden hose or faucet that runs a constant, gentle stream of water up into the eyes. Design for "hands-free" operation so that an affected person can use the hands to keep eyes open while flushing.)

When measuring anything but very small quantities of NaOH or KOH, wearing a dust mask or cartridge respirator is very important to prevent inhalation of caustic particles. A spray bottle of vinegar is handy for neutralizing any small residual catalyst spills in the workplace. Fine particles of NaOH or KOH will produce holes in clothing, thus a protective apron or jumpsuit is also advised. It is also helpful for each person working in the biodiesel shop to keep a spare change of clothes on hand, so that accidentally contaminated clothing can be quickly shed if needed.

Sodium methoxide or potassium methoxide is made during the process by combining the methanol and sodium hydroxide. It has many of the same corrosive and toxic characteristics as methanol and sodium hydroxide, and should be handled similarly. Pure sodium or potassium methoxide is not very stable and can ignite on contact with water or moist air.

FIGURE 3

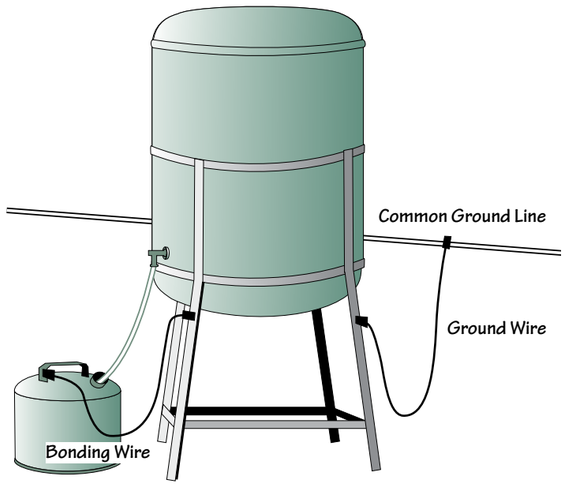
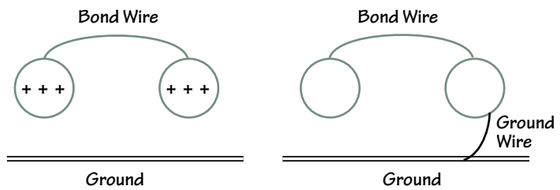


FIGURE 4



Bonding and Grounding

Static electricity can build up when fluid flows through a pipe or from an opening into a tank. Bonding and grounding prevents static electricity from causing a spark that could ignite vapors from a flammable liquid (Figure 3).

Bonding physically connects two conductive objects together with a wire to eliminate the difference in static charge potential between them. A bond wire must be provided unless a metallic path between them is otherwise present.

A grounding wire eliminates a difference in static charge potential between conductive objects and the ground. Although bonding will eliminate a difference in potential between objects, it will not eliminate a difference in potential between these objects and earth unless one of the objects is connected to earth with a ground wire (Figure 4).

General Fire Safety: No flames, smoking or sparks anywhere near the production area.

Spontaneous Combustion

Biodiesel production will routinely produce rags saturated with oil or biodiesel. It is also common (but not advised) for sawdust or other fibrous materials to used as an absorbent for spilled biodiesel or vegetable oil. These materials present an inherent fire risk, as oily rags or sawdust can easily spontaneously combust. In 2007, a Pennsylvania barn burned to the ground when sawdust soaked with vegetable oil caught fire on a sunny day. These materials must not be allowed to accumulate in or around the workspace, including in open trash cans.

Oily rags should be kept in an air-tight metal container, a bucket of water, or sealed in evacuated plastic bags and properly disposed of in the trash. Free liquid should be squeezed into an appropriate container before disposal of saturated rags. When disposing of saturated sawdust in a dumpster, be sure to scatter the material to avoid any piles that may combust and cause a dumpster fire.

Electrical equipment should be installed by qualified service personnel in accordance with the national electrical code and local regulations. Temporary rigging of electrical equipment is inherently dangerous and poses a serious fire risk. Heating elements used in processing fuel should be regulated by appropriate thermostats. Care should be taken when running power to heavy electrical loads (such as heating elements) through extension cords, so that the amperage or wattage rating is not exceeded. If an electrical cord ever feels warm, it is overloaded and should be unplugged immediately. When in doubt, consult a qualified electrician.

Fire extinguishers should be conveniently located throughout the biodiesel shop, and all users should be familiar with their operation. Avoid blocking access to extinguishers with clutter or equipment. Remember the acronym PASS:

Pull the pin from the fire extinguisher

Aim the nozzle at the base of the fire

Squeeze the trigger device

Sweep from side to side of the flame as you extinguish the fire

A twenty-pound ABC fire extinguisher is recommended. It is a good idea to run occasional "fire drills" in the biodiesel shop to be prepared in case of an actual emergency.

Safety Gear Summary

The following gear should be on hand each time you brew biodiesel:

- Chemical-resistant gloves (butyl rubber is best for methanol and lye)
- Chemistry goggles (indirect vented)
- Face shield
- Dust mask or cartridge respirator
- Plumbed eyewash station
- Small spray bottle with vinegar for neutralizing lye spills
- Access to running water
- Telephone in case of emergency and emergency telephone numbers
- Fire extinguishers (20-lb ABC)
- Absorbent material and spill-containment supplies

Process Safety and Handling Issues

The Production Facility

An ideal space for biodiesel production will be well-ventilated yet protected from weather, and lockable to prevent untrained people from accessing chemicals and equipment. Adequate ventilation is an absolute must due to serious health hazards associated with methanol fumes and lye dust. Ventilation can be provided mechanically, via fans or chemical fume hoods, or by working outdoors in the open air. Storage space for vegetable oil, chemicals, and processing gear should be ample, and should not cramp the working space. Electrical and water service are also required for most processing systems. A secondary containment system is advisable in case of large spills of oil, biodiesel, wash water, etc.

A "best practice" is to have a separate space dedicated to biodiesel production, apart from other buildings that serve multiple purposes. Biodiesel processing in existing multi-use buildings risks the loss of assets in case of an unforeseen fire or other accident. Users are cautioned to absolutely avoid producing biodiesel in quantity inside their personal residences. Making biodiesel inside a production greenhouse or livestock barn is also not advised. Fires due to biodiesel production may not be covered by home or farm insurance policies.

A typical solution for small-scale producers would entail erecting an appropriately sized shed apart from other buildings on the property. Inexpensive modular metal buildings of various sizes (such as those sold as carports) are now commonly available throughout Pennsylvania. Such buildings, when equipped with retractable walls and doors, would make an ideal site for small-scale biodiesel production. It is essential that the facility not be located immediately adjacent to waterways that could be impacted by spills of chemicals, oil, or biodiesel.

A good processing safety plan starts with a schematic flow chart for the proposed facility. Where and how will lye and methanol be stored and in what quantity? How will they be handled? Where will vegetable oil enter the building and where will it be stored? How will the materials involved in various processing steps move through the building? How will fuel and by-products exit the facility and where will they be stored? Be sure to note the location of safety gear. Once a schematic is made, it can be applied to the physical space under consideration for biodiesel production.

Processing Equipment

All of the problems associated with processing biodiesel can be handled with a properly engineered design. However, some Web sites suggest that you can build something with "what's lying around the house." This can be very dangerous and should be avoided. Please consult with experienced biodiesel producers or industrial personnel when designing your facility.

While there are a wide variety of processor designs that will effectively convert vegetable oil into biodiesel, for safe and energy efficient production, a closed system that minimizes opportunities for escape of methanol vapors into the workspace or environment must be used. Although early books on biodiesel popularized the use of open-top barrels for mixing oil with methanol and lye, these irresponsible designs result in increased fire risk, increased worker methanol exposure, and reduced fuel quality due to evaporation of methanol from the reaction tank. Additionally, any pouring of large quantities of methanol or mixtures that contain methanol should be avoided in the process design. For this reason, a closed system, wherein oil, chemicals, and end products can be safely transferred using pumps, tubing, and valves is ideal. Use of devices with sparking electric motors, such as drills or paint mixers, near open containers of methanol also presents a fire risk and must be avoided.

One successful closed-system design that is widely used is the water-heater-based processor (popularly known as the "Appleseed" reactor). Electric water heaters, whether purchased new or recovered from plumber's salvage yards, have the following desirable features: they are sealable, factory insulated, and come equipped with electric heater elements, thermostats, a drain, and several inlet ports at the top of the tank. Reliable information and plans for building a biodiesel reactor from a water heater can be found in References. The water heater reactor uses a centrally located pump for recirculation of the reacting biodiesel. This eliminates the need for a mechanical mixer/stirrer, simplifying the system while improving overall safety.

Sealed processors must be equipped with a temperature pressure-relief valve that is compatible with the tubing used to plumb the reactor (most water heaters are sold with 150-PSI valves, and these should be replaced with a 30-PSI valve if using any poly or plastic tubing). A manually operated vent valve, plumbed to the outdoors, is also essential, as described below. A sealed system based on a metal tank also allows for the recovery of excess methanol from biodiesel fuel and the glycerol by-product, which saves money and reduces handling and environmental complications after processing.

When plumbing a reactor of any design, stainless-steel or black iron tubing should be used wherever possible, as they are most compatible with the chemicals involved in processing. Galvanized fittings and any copper parts will reduce the oxidization stability (shelf life) of biodiesel, while standard PVC tends to break down over time. Aluminum should also be avoided, as it will violently react with lye to form explosive hydrogen gas. Many systems use brass ball valves with no major ill effects.

Wherever plastic, poly, or HDPE is used in biodiesel equipment, its proximity to heat sources should be considered. For example, early works on small-scale equipment popularized the use of conical-bottomed plastic tanks for the main reactor vessel. While they do possess some advantages, plastic vessels present an increased fire and spill risk in the event of an unforeseen overheating situation, structure fire, or other accident. In the event of a fire in or near the processing space, any plastic tanks will likely rupture and release their contents, potentially adding fuel to the fire. Heating elements should never be installed in plastic tanks. Most conical-bottomed plastic tanks are not 100 percent airtight, and thus will result in some methanol release if used as a main reactor. These tanks also will not tolerate the high temperatures needed to use the reactor as a methanol recovery device.

While homemade wooden stands are commonly used to support biodiesel equipment, these also present a weak link in overall plant fire safety. In the event of a fire in the plant, a wooden, oil-soaked stand will likely burn, dumping whatever liquid is in the tank it supports into the fire. An ideal fire-safe biodiesel plant will use metal tanks supported on secure metal stands.

Ventilation, Methanol Fumes, and "Make-up Air"

The main reactor and other processing equipment will be laden with methanol vapors at various times during biodiesel production. Whenever fluids are added or drained from a sealed reactor, make-up air must be allowed to flow into or out of the reactor to prevent pressure or vacuum buildup. Air leaving the reactor should be vented through tubing to the outdoors at a minimum, as it may contain toxic or flammable vapors. Simply running a temporary flexible hose from a valved port on the reactor to the outdoors may be sufficient, while hard-plumbed venting is desirable for regular processing. A best practice is to vent all methanol vapors through a condenser for collection, as described in Methanol Recovery.

Methanol fumes will also be present when glycerol is drained from the reactor, and when raw biodiesel is pumped from the reactor to wash tanks, especially if these fluids are drained while hot. Gas analysis in

FIGURE 5

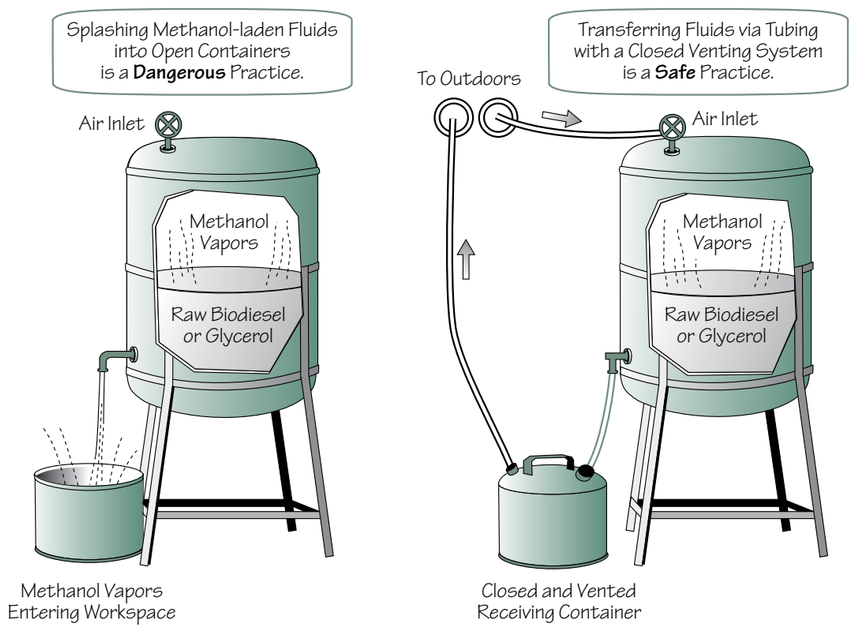


TABLE 1

Step	Heat	Pump	V1	V2	V3	V4	V5	V6	V7	V8	V9	V10	V11
Oil in		◆	◆	◆		◆		◆			◆		
Heat oil	◆	**	◆	***		◆			◆				
Inject methoxide		◆	◆	◆		◆			◆	◆			
Reaction stage		◆	◆			◆			◆				
5% prewash		◆	◆	◆		◆			◆	◆			
Drain plumbing			◆	◆		◆							◆
Settle glycerol													
Drain glycerol			◆	◆					◆				◆
Distill methanol	◆	◆		◆	◆								
Drain biodiesel		◆	◆	◆			◆		◆				
Drain site tube			◆						◆		◆		◆

◆ = OPEN or ON
** = Pump should only run periodically
*** = Release pressure periodically

one Pennsylvania biodiesel facility found methanol concentrations in excess of the lower explosion limit (and personal exposure limit) when splash-pumping warm biodiesel into an open wash tank. Remember, if you can smell methanol in the workspace, there is a problem with your process design to be corrected. A best practice is to transfer all methanol-laden fluids via tubing into closed vessels, and to vent all vessels to the outdoors, (Figure 5). One innovative design drains glycerol into a sealed container, which has air-vent tubing temporarily plumbed to the main reactor, so that air pressures between the two vessels can stabilize without methanol release into the workspace or environment. Periodic venting of the reactor to the outdoors is still necessary due to thermal expansion and contraction associated with heating and cooling. However, if this occurs through a condenser, methanol release is minimized. Such a "fumeless" design is very attractive for air-quality considerations. For further information on fumeless processing, see References.

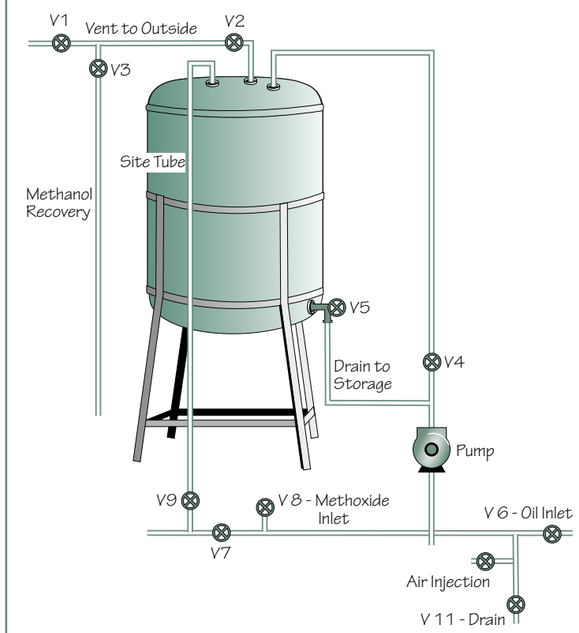
Operator's Quick Reference

Basic processing equipment developed in the home or farm shop can become complicated with multiple valves and switches for various purposes. Even experienced operators can make mistakes, and opening (or failing to open) certain valves during processing stages may result in spills or accidental release of dangerous chemicals into the workspace. A best practice is to develop a well-thought-out process diagram, including step-by-step guidelines for the state of valves and switches during different stages of production (Table 1 and Figure 6). This diagram should be posted on or near fuel-making equipment, to serve as a reference for all trained operators. In the sample diagram below, each "X" represents an open valve, or electrical component turned on during a given process step.

Step-by-Step Process Protocol

In biodiesel shops where multiple operators use shared equipment (co-ops, educational facilities, etc.), it is helpful to print off and post a standard processing protocol. Such a document ensures that all personnel follow proper safety and fuel quality procedures during each step. In addition, a "batch step checklist" that follows a batch of fuel from tank to tank on a clipboard is a handy way for multiple plant operators to communicate exactly what is happening in each tank in the shop. Also, a batch record sheet that documents the inputs and processing of a specific batch should be maintained as part of the record-keeping system. Examples of a batch checklist and batch record sheet can be found in Appendix B.

FIGURE 6



Washing Fuel

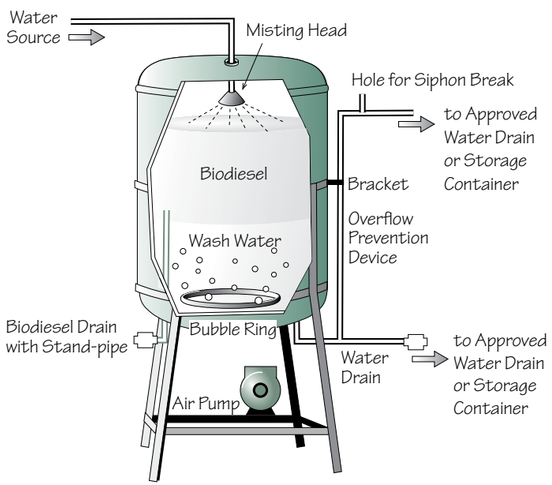
Wash tanks are considered essential equipment by most producers. They are used to "finish" biodiesel by cleaning it with water. Raw biodiesel straight from the reactor typically contains soap, residual catalyst, traces of glycerol, and residual methanol if a methanol recovery stage was not performed. While a few small-scale producers run unwashed fuel in their equipment, for high quality biodiesel that approaches the ASTM standard, washing or other soap removal methods are strongly recommended.

An inverted 55-gallon closed-head barrel with the bottom cut out makes an affordable wash tank: 25 to 35 gallons of fuel can be washed in each barrel, and drains and valves are easily plumbed into the fittings at the bottom. A stand-pipe such as the one pictured in Figure 7 is helpful for separating fuel from water in the wash tank.

Washing fuel is achieved by gently adding water to the biodiesel: gentle addition is important in order to avoid emulsification of fuel and water, especially in the first wash of a batch of fuel. Likelihood of emulsion is increased by the presence of glycerol or soap in the fuel, cold temperatures, and violent agitation. It is common to warm biodiesel and/or wash water when washing in cold weather.

Gentle addition of water to fuel is best achieved by either mist washing (mist nozzles rain very fine water

FIGURE 7



droplets onto the surface of the fuel and the water settles to the bottom of the tank drawing impurities with it) or bubble washing (air bubbles are pumped into clean water at the bottom of the wash tank, where they rise and pull the water into the fuel, gently circulating the water until it is saturated with impurities.) Bubble washing is an effective way to reduce the total amounts of water used and waste produced, since it recirculates water through the fuel several times over.

Poorly designed washing systems are a common cause of spills in biodiesel production facilities. In the case of mist washing, water may be added to the tank over the course of several hours, and the risk of fuel overflow in a forgotten wash tank is high. A simple overflow prevention device such as that pictured above can be easily built for a few dollars. The use of an open-topped tee for a siphon break at the top is essential to prevent accidental draining of the tank. Affordable water shutoff timers or more sophisticated float switches are also advisable to prevent accidental fuel overflow; plumbing two garden water timers in series provides redundancy in case one should fail. Threaded (or soldered) secure connections should be used between the water supply and misting device to prevent accidental separation, leading to rapid tank overflow.

Soiled wash water should be discarded in a manner consistent with suggestions in By-Product Handling and Disposal. If methanol was not recovered from the biodiesel fuel prior to washing, the waste water from the first wash will contain significant methanol and should be handled with care.

Prewashing Fuel for Soap Reduction

In order to reduce the volume of water used in washing, and to reduce time that fuel spends in the wash tank, some producers utilize a prewash step while the fuel and glycerol are still in the main reactor. Water is added to the fuel and glycerol mixture immediately following the initial mixing. Typically, warm water equal to 5 percent of the original volume of oil will be added to reaction, then mixed for another 15 minutes or more. (For example, in a 50-gallon batch reaction, 2.5 gallons of water would be added at the end of the normal mixing time). When the fuel, glycerol, and water mixture is allowed to settle, most of the water will settle in the glycerol layer, taking soap with it. The resulting biodiesel may be easier to wash, requiring less water, and producing less waste water for disposal. (**Note:** The water *must* be added while glycerol is still in the reactor, or else emulsification will result from vigorously mixing water and biodiesel together.) Adding water to the reaction will complicate methanol recovery steps, and achieving high-purity (dry) methanol becomes more of a challenge. See References for further discussion.

Drying Fuel

During the washing process, biodiesel will become cloudy with suspended water droplets (sometimes referred to as having an "orange-juice-like" appearance). Washed fuel must be thoroughly "dried" before use in equipment or transfer to storage. Water in fuel can cause serious damage to diesel system components, and wet fuel may also promote algae growth in storage tanks. Suspended water droplets may settle out of washed fuel over the course of several days, depending on ambient temperatures. Passive drying may be achieved by allowing washed fuel to sit in barrels exposed to a breeze for several days, though success will depend on air temperatures and relative humidity. Washed and dried fuel should be transparent, sparkly, and free from any haze. Wet fuel may appear to clarify on warm days, but will become cloudy again as it cools. Water testing information can be found in Biodiesel Quality Test Methods.

In "active drying," the fuel is separated from any standing water either by draining the water or pumping the fuel to a separate "dry tank." The fuel is then pumped through a circulating loop that allows it to fall through the air and back into the dry tank, giving the fuel intimate contact with the air. Increased air flow can be achieved with a small fan directed into the drying tank. Warming the fuel will speed the drying process, especially on humid days.

While use of a showerhead or similar device in the drying tank will increase air contact (smaller fuel droplets), the possibility for fuel atomization presents a serious fire and health risk. Successful drying can be achieved by simply directing a stream of fuel against the side of a tank and blowing air across the fuel. "Bubble drying," in which air bubbles are passed through an open-topped fuel drum for several hours, takes longer than the process described above, but is typically a cleaner, more energy efficient means of drying fuel. Active pump drying presents another potential spill point: all connections and mounting of drying apparatus must be secure to prevent accidental pumping of fuel onto the shop floor.

Due to the open-topped nature of many wash and dry tanks, it is advisable to place a filter in-line with the pump used for drying and transfer of fuel from the dry tank to fuel storage barrels. If the outlet hose from fuel storage barrels is equipped with a 10-micron water-blocking diesel fuel filter, it will catch any last particles or water before fuel is pumped into diesel equipment. A 1-micron filter may be advised for newer, high-pressure injection diesels, as their longevity may be affected by 2- to 5-micron-sized particles.

Waterless Soap Removal

Due to the inherent challenge of proper wash water disposal in rural areas, waterless washing methods may also be attractive. Some small-scale producers have managed to achieve ASTM-quality fuel with no washing. This process is most effective following the "prewash" technique described above for initial soap reduction. The majority of the contaminating soap, residual catalyst, and glycerol are not soluble in biodiesel, but are suspended in biodiesel because they are soluble in methanol. If methanol is driven out of the fuel (see Methanol Recovery), soap and other contaminants will settle out of the fuel over a period of several days. Soap separation from biodiesel happens more reliably if sodium hydroxide is used as the reaction catalyst (rather than KOH). After the soap is removed, the fuel can then be passed through a series of filters and put directly into storage without water washing. A soap test (described in Biodiesel Quality Test Methods) is helpful to determine that the process is working correctly.

Ion-exchange resins, such as Amberlite or Purolite beads, are used by some commercial producers for removal of impurities instead of water washing. These beads, which absorb soap, water, glycerol, and some methanol, are packed in columns through which unwashed biodiesel fuel is passed. After flowing through a series of columns, the biodiesel is then filtered and pumped to storage, ready to use. A single load of beads may process numerous batches of biodiesel, depending on fuel quality. Bead longevity may be extended by preceding the bead columns with the methanol recovery or soap separation method described above. While the cost of the ion-exchange beads has mostly kept this process in the realm of larger plants, small facilities may find them attractive as a way to avoid producing a waste water problem. For more information on waterless soap removal and ion-exchange beads, see References.

Some small producers report adequate success with simply allowing fuel to settle in barrels or tanks for a month or more following glycerol separation, which also promotes soap settling via gravity. In this case, fuel should be drawn off the top of the barrel, leaving the soapy layer behind. Fuel "cleaned" by settling alone should be considered of suspect quality, and monitored carefully for any equipment problems.

Storage Issues

Oil Collection

Before collecting oil from any restaurant, be sure to ask permission from the management. Oil that has been placed in a rendering company barrel technically belongs to that company, and should not be taken despite permission from the restaurant. To avoid legal trouble, provide your own barrel, or ask the restaurant to save the used oil in the plastic jugs that are emptied each time they add fresh oil to the fryers. Barrels should be clearly labeled "used cooking oil." If you enter into a grease collection agreement with a restaurant, it is essential to reliably collect oil on a schedule that works for the restaurant.

Small-scale biodiesel production is easiest with high-quality oil that is free from water and excessive food particles, does not smell rancid, and is low in acid due to regular fryer changes. Outdoor oil storage at restaurants should be securely covered to prevent water and vermin from entering containers.

The PADEP regulates the transportation of used cooking oil. Each vehicle used in collection must bear a label (decal or magnetic sign) reading "MUNICIPAL WASTE" or "RESIDUAL WASTE" (if it is from a food-processing plant) plus the name and address of the vehicle owner. All letters on the sign are required to be six inches in height, or readable from a reasonable distance. In addition, the collection vehicle must carry a log that indicates where oil is collected and the number of gallons per pickup. A fire extinguisher must also be carried in the collection vehicle. The same vehicle cannot be used to

transport food or animal feed.

Spill-free collection and transportation will ensure a lasting good relationship with restaurants and regulatory agencies. Keeping a bucket of an absorbent "oil dry" material and a spill kit in the collection vehicle is advised in case of spills. Secondary containment in the collection vehicle is desirable, as is securing any buckets or barrels to prevent spills during transportation. Take care not to overload the rated hauling capacity of your vehicle with heavy oil drums or totes. While a vehicle may physically carry more weight than it is rated for, braking ability may be increased when overloaded, presenting an accident hazard.

Oil can be stored for six months to a year before processing, ideally stored in a container with minimal air space. Large heavy duty plastic totes (300- to 350-gallon cubes) equipped with drains at the bottom make handy oil storage containers. (These must be well-supported; 300 gallons of oil weighs about 2,100 pounds without adding the weight of the container.) Oil can be rough-filtered by pouring it through a screen when transferring from the collection vehicle to storage. Water and food particles will naturally fall to the bottom of the container over time, and can be periodically drained. Oil for processing should be drawn off of the top of the container to avoid any watery layer below.

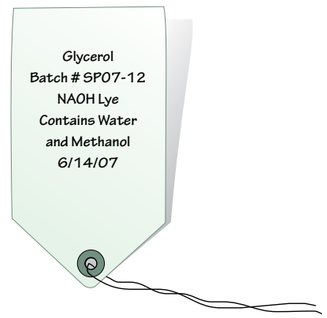
Chemical Storage

All hazardous chemicals (and any contaminated processing gear) should be kept in locked storage containers when not in use. A best practice is to use an approved metal fire cabinet for storage of flammable liquids. Heavy duty 5-gallon plastic carboys are handy for methanol storage and transportation. Handling of methanol is reduced if using carboys that can be plumbed directly to the biodiesel reactor for chemical addition to the feedstock oil. Chemical storage containers should be clearly identified with safety labels to advise fire or safety personnel and others in case of an emergency.

Labeling Stored Fluids

A best practice is to label all storage containers, from buckets of glycerol by-product to large tanks of used cooking oil. It is very easy to lose track of which "unidentified brown fluid" may be in an unmarked container. Labeling prevents accidental misuse of fluids, and will help emergency-service personnel know what they are dealing with in case of an accident. Paper tags with strings or wires sold at office-supply stores are easily affixed to jug and bucket handles. (Pencil or permanent marker is the most reliable for marking tags in oily conditions).

FIGURE 8



Container labels should indicate the type of fluid, any distinguishing characteristics, and the date the fluid was produced or stored. Dating waste containers is particularly important to be sure that storage does not exceed the one-year limit regulated by the PADEP. Methanol containers should be labeled "flammable" and lye or KOH containers should be labeled "corrosive." Stickers for labeling may also be available from chemical suppliers.

Spills

Spills of hazardous materials or oil are easiest to clean up if they are kept to paved or concrete areas. If the spill occurs on these areas, great effort should be made to keep it on these surfaces and not allow it to migrate to soil, into storm sewers or manholes, or other surface water. If a spill enters surface water or the storm water system, it becomes a high-impact spill and must be reported to 911 for emergency cleanup response. Typically this will involve the county-designated Hazmat team.

For smaller spills and releases (those that can be safely handled by the biodiesel producer and do not enter storm sewers, manholes, or other surface water), the following procedures should be used:

- Identify the problem.
- Stop the source of the spill/release.
- Contain the spill/release by using absorbent socks, or prevent the spill/release from migrating to other areas such as soil, drains, or surface water by using the absorbent socks or constructing a dike.
- Recover the spilled/released material and affected soil or gravel by covering it with absorbent materials (pads, pillows, or loose absorbent material). Other materials can be used as needed including shop rags, sawdust, etc. All free product must be absorbed. If the spill/release is to soil or gravel, excavate all material that is affected. Use both visual observations and odors to determine the extent of material to be excavated.
- Properly dispose of the used absorbent materials and excavated soil/gravel. Used cooking oil spills and small wipe-ups of hazardous materials such as methanol may be disposed of as municipal waste. Spills of methanol, or other chemicals that saturate the absorbent material, should be disposed of as hazardous waste.
- Report the spill to the PADEP if it occurs outdoors or enters any storm-water drains, manholes, or surface water. Provide information on the location of the spill, the material spilled and its volume, and the affected media (water, soil, etc.).

It is helpful to have a spill kit on hand to easily deal with spills. The kit should contain some loose absorbent material such as kitty litter, as well as pads that are designed to absorb oils and other chemicals. Secondary containment around oil and fuel storage areas is a highly recommended management practice in anticipation of spills. A short painted or sealed concrete curb or dike around oil storage tanks should be sized to accommodate potentially spilled oil, or a sturdy wooden enclosure lined with pond liner or heavy-duty plastic may suffice for a temporary solution. For small-scale producers, plastic children's pools are an affordable option for spill prevention around barrels. Containment of any kind should be sized to accommodate at least 110 percent of the volume of the single largest container in the containment area. Using the figure 1 gallon equals 7.48 cubic feet, you can calculate the appropriate size of your containment area for your largest tank. It is possible to make the entire shop floor into one large containment area using a short wall around a concrete floor.

By-Product Handling and Disposal

Glycerol

The answer to the question "What do you do with the glycerol and wash water?" is a moving target. Rules and recommendations regarding legal and environmentally responsible by-product disposal are likely to change over time as research is carried out, and the regulatory agencies consider the applications of the law. What follows is the authors' best possible synopsis of the available information at the time of publication. Biodiesel producers are advised to stay in contact with their local PADEP office and biodiesel production networks for the latest on this evolving issue. Suffice it to say that responsible glycerol disposal is a major sticking point for small-scale producers. The opportunity for innovators to take advantage of this "waste" product for a yet-to-be-determined beneficial use is profound.

Biodiesel production will result in significant quantities of residual glycerol by-product. For each gallon of biodiesel produced, approximately 1/5 gallon of glycerol will result. Refining crude biodiesel glycerol into pure, marketable glycerin is technologically difficult, expensive, and is not considered practical on a small-scale. New producers should give ample consideration to how they will dispose of their by-product before beginning a biodiesel facility. Storage of glycerol by-product should be done in accordance with applicable sections of Title 25 Pa. Code Chapters 285 and 299, as it presents a health and fire risk. Glycerol stored in plastic fryer oil jugs will leak over time. Storage cannot exceed a one-year period, as this is considered on-site disposal. See References for a link to the Pennsylvania Code, which provides the regulations regarding storage of residual and municipal waste materials.

Crude glycerol by-product is contaminated with methanol (approximately 25 percent by volume) and as such may be considered hazardous waste. Contrary to popular belief, methanol will not evaporate from stored glycerol at ambient temperatures sufficiently to consider the glycerol uncontaminated. Raw, methanol-laden glycerol by-product should be handled as if it were methanol. This includes wearing gloves and goggles and avoiding any concentrated vapors.

Land application of glycerol that is contaminated with methanol may result in methanol leaching into ground water, leaving biodiesel producers liable for any damages to drinking water sources. Land application of contaminated glycerol will certainly result in air emissions of methanol. Methanol is a listed hazardous air pollutant by the EPA and PADEP due to its impact on human health and other organisms.

A best practice is to recover the methanol from the glycerol by-product via distillation, prior to disposal or further use. Methanol recovery reduces environmental pollution and allows producers to reuse methanol, thereby reducing costs and improving energy balance. Methanol recovery has some inherent safety concerns, and should only be carried out by advanced producers who fully understand the safety issues involved with handling methanol and hot liquids. However, the authors of this booklet strongly recommend that all biodiesel producers incorporate methanol recovery systems into their long-term plant designs. New producers may consider stockpiling a modest amount of glycerol until they are ready to advance

to methanol recovery. A critical flaw of the small-scale biodiesel community is that the hazards of methanol-contaminated glycerol have been casually brushed aside, and methanol recovery has been considered an option rather than a requirement for a mature production facility. For more information on methanol recovery, see Methanol Recovery and References.

Crude glycerol has a high pH due to a residual NaOH or KOH catalyst from the biodiesel reaction. If acid is added to the by-product to neutralize the pH, this may allow the recovery of some additional free fatty acids for processing into biodiesel. Salts such as sodium chloride or potassium phosphate (depending on acid and catalyst choices) will result from acidulation. This may be considered waste processing and would require a permit from PA DEP.

Glycerol Contaminated with Methanol

Currently no easily attained options for disposal of glycerol with methanol are allowed by the PADEP. Some options are under investigation and may be allowed as more data become available. In other states, use as a feedstock for on-farm composting or anaerobic (biogas) digestion may be allowed, as well as incorporation into landfills. Please check with regulators in your state for the latest information and permitting requirements.

- Anaerobic Digestion: Crude by-product has been added to anaerobic biogas (methane) digesters with success in several locations, including farm-scale manure digesters and municipal waste water treatment plants. In controlled anaerobic digestion, microbes decompose animal manures and/or human waste, producing methane gas that may be used for power generation. These microbes are capable of breaking down methanol and glycerol into more benign compounds. The addition of biodiesel glycerol by-product may actually boost biogas production. The glycerol by-product typically has a high pH and a high carbon to nitrogen ratio, which may also make it attractive as a digester additive. Dilute additions of small amounts of glycerol by-product should be tested while carefully monitoring the response of the digester. For a listing of farm-based manure digesters in your area, contact your county Penn State Cooperative Extension office. This use is not currently approved by the PADEP. Please contact your local office to request a permit should you desire to pursue this further.
- Industrial Combustion: Glycerol by-product may be allowed for use as a blended fuel in industrial burners and boilers, such as cement and lime kilns if it is classified as a co-product. Testing to ensure that the flash point exceeds 140 degrees Fahrenheit (60 degrees Celsius) must be done on the waste material to ensure it is not classified as a hazardous waste, which would prevent its use in a fuel-using system not permitted for hazardous waste incineration. High-temperature burning is imperative to avoid the formation of highly toxic acrolein, which results from burning glycerol at temperatures less than 536 degrees Fahrenheit (280 degrees Celsius). Acrolein, a suspected carcinogen, is dangerous to living things at concentrations as low as two parts per million. For this reason (and considering the fire and health hazards of handling methanol-contaminated by-product in the home), home-scale burning of glycerol is not recommended. The Air Quality section of the PADEP needs to be apprised of any fuel-burning situations with this material.

Glycerol After Methanol Recovery

- Industrial Combustion as described above. The Air Quality section of the PADEP needs to be apprised of any fuel-burning situations with this material. The Waste Management section should also be consulted for permission to designate glycerol as a co-product for use as fuel.
- Composting. After methanol removal, glycerol can be safely composted with reduced concern for environmental contamination. Glycerol by-product is a high-carbon, high-pH, wet feedstock, and should be combined with a bulking agent (straw, hay, leaves) and some nitrogenous materials (manures or fresh green yard wastes) for proper composting. Glycerol should be added to compost piles in dilute amounts to prevent wet saturation leading to anaerobic conditions. Frequent turning of piles will promote speedy aerobic decomposition and an even distribution of glycerol into the mix. Glycerol compost piles should be constructed far away from any water source to prevent runoff contamination. Covering piles with commercially available compost fabrics will prevent water saturation and reduce runoff potential. Soils where compost is applied should be tested regularly for any salt buildup from residual biodiesel catalyst in the glycerol by-product. The PADEP requires a permit for this method of disposal, so consult your local office. Until further research into the action of crude biodiesel glycerol in the compost pile can be conducted, the authors do not recommend adding glycerol to compost piles used to fertilize food gardens.

- Soap. Glycerol can be processed into a crude hand or body soap using simple recipes (see References). It is imperative that all methanol has been removed from the glycerol before making soap. To be certain, glycerol should be heated slowly to 212 degrees Fahrenheit (100 degrees Celsius), which is the boiling point of water. If bubbling occurs in the glycerol pot before 212 degrees Fahrenheit (100 degrees Celsius) is reached, then some methanol is still present. Heating should continue until no bubbling occurs at temperatures below 212 degrees Fahrenheit. When making soap, use only stainless steel, glass, or ceramic pots (no aluminum). Heating should occur outdoors over an electric burner to prevent any concentration of fumes or ignition of stray methanol vapors.
- Degreaser. A simple but effective engine degreaser or floor cleaner can be made by making a 50/50 blend of water and glycerol after methanol removal. Individual biodiesel producers report a strong demand for glycerol degreaser in some locations.
- Dust Suppressant. After methanol recovery is complete, glycerol by-product has been blended with water and sprayed on roads and dirt tracks as a dust suppressant. Glycerol tastes sweet to animals and may draw wildlife to roads, so caution is advised. Avoid spraying near any waterways to prevent contamination. Any land application of waste material must have permission from your Regional PADEP Office and the township or state if they have jurisdiction of the road.
- Animal Feed Additive. Early experiments show some success with adding glycerol to animal feed after methanol recovery. Glycerol is a sweet, high-carbohydrate energy source that may be added to grain feeds in dilute quantities. Further research is needed to determine proper blending rates and the possible effects of potassium or sodium salts and high pH of the by-product. For more information, see References.

Wash Water

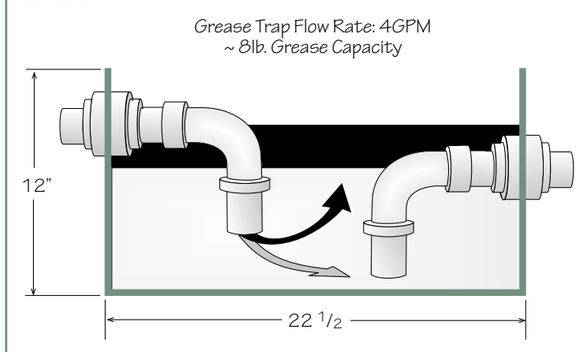
Wash water waste from biodiesel processing presents a significant disposal and handling challenge in rural areas. Those who have access to a public sanitary sewer may be permitted to dispose of wash water by their local water treatment authority. Contact your water treatment authority for permission before disposing of wash water to the sanitary system.

Wash water may be contaminated with any of the following: methanol (first wash, if methanol recovery step was not carried out on fuel), soap, excess catalyst, glycerol, vegetable oils, free fatty acids, and biodiesel. Wash water will typically have a high, alkaline pH. The pH of the water should be tested prior to disposal in a sanitary sewer. Local regulations vary, but any liquid with a pH higher than 9.5 or 10 is of concern due to its corrosive nature. The alkalinity of wash water may be neutralized in batches using vinegar or dilute muriatic acid. However, "pretreatment" or neutralizing a waste prior to disposal is regulated under PADEP Permit-By-Rule regulations for neutralization, pending test results for waste determination as hazardous or residual. Each case will be handled separately by the PADEP, depending on the test results of the waste and the needs and type of treatment plant involved. At the very least, the treatment facility operator and local PADEP Water Quality section should be consulted prior to sewering wash water.

Methanol-laden wash water will have a high biological oxygen demand (BOD), which is of concern to water treatment plants. Some treatment plants are very wary of flammable liquids in sewer water. Local tolerances for methanol in sewer water may vary.

Vegetable oil or biodiesel in wash water can also confound water treatment and sewer systems—many plants have very low tolerances for fats, oils, and grease. A best practice is to pass wash water (and water from sinks where biodiesel lab ware is cleaned) through a grease trap. Commercially available grease traps will prevent excessive oil contamination of the waste water stream. An effective grease trap can also be made from plumbing parts and a large plastic tote (Figure 9).

FIGURE 9



Note that the outlet pipe exits the trap slightly lower than the inlet pipe, and that the outlet pipe draws water from near the bottom of the tub. Baffles may be added along the bottom to encourage fats to rise. A small vent hole should be drilled in the upper portion of the outlet pipe to prevent siphoning. Fats will collect at the surface of the liquid in the trap, and must be skimmed off the top from time to time.

If wash water is allowed to settle for several days, most oil in the water will rise to the surface, where it can be skimmed off and returned to the wash tank. Some users also add Epsom salts (magnesium sulfate) while aerating wash water, which will cause soaps to precipitate out of solution.

Responsible disposal of wash water is a combination of the above techniques (methanol recovery, neutralization, oil separation, and soap removal.) Some small-scale producers report success with biological filtration of wash water in a series of aerated tanks serving as constructed wetlands, in which plants are grown to accumulate any contaminants. Composting or dilute field application may be allowed as a normal farming practice, provided that the farmer generates the waste and is making the biodiesel for noncommercial use on the farm. Heavy land application of concentrated, raw wash water will negatively affect soil organisms due to the oils and high pH. Compost piles and fields should be monitored for any unusual salt or nutrient buildup. These processes lend themselves to a potential Permit-by-Rule from the PADEP; so contact your local office.

Wash water should be prevented from entering waterways, storm sewers, ground water, and other water runoff channels at all costs. Biodiesel producers who contaminate waterways with wash water or other wastes may be held accountable by law.

Given the many challenges of proper wash water disposal on farms, many producers may find waterless soap removal as described in Process Safety and Handling Issues to be attractive.

"Bad" Vegetable Oil

Small producers working with used cooking oil will inevitably encounter some material that is unusable for production of biodiesel. Tank bottoms, oil that is excessively laden with food particles, rancid oil with a foul odor, and oil/water emulsions are examples of what may be considered "bad" oil. The following are disposal options tentatively approved by the PADEP. A permit may be required for some options. Please consult your local PADEP office to ensure compliance with the latest regulations. Any land application that results in foul odors affecting neighboring properties is likely to be prohibited.

- Application to gravel/dirt farm roads for dust suppression and "paving." Many farmers report effective control of dust using dilute additions of vegetable oil to unimproved roads. Care must be taken to avoid application where runoff could enter any storm sewer, stream, or other water source. Vegetable oil may draw pets or wildlife to roads. Keep accurate records of the date, time, location, and quantity of vegetable oil applied to roads. Consult your local PA DEP office prior to application.
- Compost. Vegetable oil will compost if small amounts are added to absorbent material (straw, hay, leaves, wood, etc.) in a large compost pile. Oils tend to slow the composting process, but may also help to tie up soluble nitrogen. Keep accurate records of the date, time, location, and quantity of vegetable oil applied to compost piles.
- Burning in bonfires/wood furnaces. This use may require a permit from the air quality section of the PADEP.
- Mix with absorbent material and landfill. Consult with your landfill administrator or trash hauler before disposing of large quantities of oil in this manner. Oil-soaked sawdust presents a dangerous spontaneous combustion fire hazard and must not be allowed to linger in or around buildings. Oily sawdust should be wetted down with water and packed in sealed containers to avoid starting a dumpster fire.

Tracking Emissions: Where Does the Methanol Go?

Methanol is an air pollutant of concern for the PADEP and the United States EPA due to its potentially harmful impact on humans and other organisms. In Pennsylvania, facilities that emit into the air one ton (2,000 lbs) or more of methanol per year are required to obtain a permit from the PADEP. Facilities that emit less than one ton per year are currently exempt from permitting. As a best practice, small producers can monitor and record their methanol emissions to ensure that they fall well below the exempt levels. This section discusses some factors involved in tracking methanol emissions.

A gallon of methanol weighs about 6.6 pounds at room temperature. One ton of methanol is therefore equal to approximately 303 gallons. A facility's methanol emissions will depend on annual production volume and processing methods: Does the system include methanol recovery? How much methanol is used in the reaction? Is it a closed system? Table 2, above, can be used as a rough guide to potential emissions. From the chart, we can see that the annual threshold production level below which no air quality permit is currently required is 3,366 gallons for facilities using 22 percent methanol, and 2,525 gallons for facilities using 25 percent methanol. However, producers should not consider this a "pass" to emit methanol into the air. Responsible individuals will work to minimize their emissions of this air pollutant through methanol recovery and good process design. Open-topped reactors will emit significantly more methanol than a closed system.

TABLE 2

Oil processed (gal)	Methanol (percent of oil volume)	Total methanol used (gal)	Methanol recovery?	Potential emissions (gal) *	Potential emissions (lbs)
1,000	22	220	N	90	594
3,366	22	741	N	303	2,000
3,366	22	741	Y	stray emissions only	
1,000	25	250	N	120	792
2,525	25	631	N	303	2,000
2,525	25	631	Y	stray emissions only	

* Potential emissions from glycerol and unwashed fuel based on methanol in the amount of 13 percent of oil volume being incorporated into the fuel.

Biodiesel Chemistry

The methanol required to convert vegetable oil (triglycerides) into methyl esters (biodiesel) and glycerol in the transesterification reaction is approximately 13 percent of the starting oil volume. However, typical base-catalyzed batch reactions use excess methanol in order to achieve full conversion of the oil into fuel. The biodiesel reaction is reversible. Without a surplus of methanol, an equilibrium is reached and some glycerol will remain dissolved in the biodiesel as monoglycerides and diglycerides. Excessive glycerol in the biodiesel will prevent the fuel from meeting ASTM standards, and may harm engine components with extended use. Methanol on the order of 20 percent to 25 percent of the starting oil volume is commonly used to achieve full conversion.

If we assume that methanol in the quantity of approximately 13 percent of the oil volume is chemically incorporated into the finished biodiesel, that leaves 7 percent to 12 percent of the oil volume as residual emission-prone methanol. If the reactor is sealed (which is strongly advised for safe and efficient production), unbound methanol amounting to approximately 2 to 4 percent of the oil volume will be present in the biodiesel layer. If methanol is not recovered from the fuel prior to washing, this amount will remain in the water used for the first wash and the wash water should be considered toxic and handled with care. The remaining unbound methanol, amounting to approximately 5 percent to 10 percent of the starting oil volume, will be in the glycerol layer. For example, consider a 50-gallon batch reaction, using 20 percent methanol (10 gallons), with no methanol recovery. Roughly 6.5 gallons of methanol (13 percent of the oil volume) will become a part of the biodiesel molecules and will be burned as fuel. That leaves about 1 to 1.5 gallons of residual methanol in the unwashed fuel, and 2 to 2.5 gallons of methanol in the glycerol by-product.

Fugitive methanol emissions will result from any open vessels that contain methanol or methanol-laden liquids. These may include reactors, wash tanks, storage drums, and carboys. A best practice is to keep all containers sealed except during fluid transfer. Methanol emissions will also escape from open vents, relief valves, etc. Producers are advised to open vents only when necessary, and to route all vented vapors through a condenser if possible.

Each production facility will follow a different set of practices for handling of post-reaction fuel and by-products. Producers can gain a rough idea of their methanol emissions by recording how much methanol they purchase, how much they use to process each batch of fuel, and how much methanol is recovered from biodiesel and glycerol. In addition to ensuring compliance, recording methanol's passage through the shop (Table 3) will help inspire producers to engage in the best management practice of methanol recovery.

TABLE 3

Date	Batch #	Oil Volume	Methanol, %	Methanol, gal	Methanol bonded in fuel, gal (13% of oil)	Free methanol recovered from fuel, gal	Free methanol recovered from glycerol, gal	Potential emissions, gal	Potential emissions, lbs (=gal x 6.6)
11-May	07-001	50	22	11	6.5	0	0	4.5	29.7
30-May	07-002	50	20	10	6.5	0	0	3.5	23.1
10-Jun	07-003	50	22	11	6.5	1.5	0	3	19.8
25-Jun	07-004	50	22	11	6.5	1.5	3	0	0
								Total emissions (lbs)	72.6

Methanol Recovery

Methanol in excess of the actual amount required for the transesterification reaction is added to drive the reaction further to completion. If the excess methanol is recovered, it can be used for processing additional batches. If not recovered, most of the excess ends up in the glycerol and some in the water wash. This is not only wasteful of a valuable component, but can further complicate the disposal of these two waste streams, as discussed above. Using simple distillation technology, it is possible to recover as much as 30 percent of the methanol added to the original reaction, which results in cost savings, increased energy efficiency, and significantly reduced environmental impact. Depending on processing techniques, in a typical 50-gallon batch of biodiesel, there may be 2 to 2.5 gallons of unreacted methanol in the glycerol, and 1 to 1.5 gallons of unreacted methanol in the raw fuel.

Methanol recovery can take place from the fuel after the glycerol is removed, and it can be distilled from the separated glycerol. Fuel or glycerol is heated to temperatures over the methanol boiling point, 148 degrees Fahrenheit (64.6 degrees Celsius), and vapors are directed through a water or air-cooled condenser. In the condenser, methanol gas is chilled to a liquid state, which can then be recaptured. Methanol exiting the condenser will collect in a vessel that must be vented to the atmosphere (outside of the shop). It is important to have an adequate condenser to ensure that all the methanol vapor is condensed. The condenser must have enough surface area, and cooling water temperatures must be low enough to ensure that no gaseous methanol pushes through the collection container. It is also important to keep the temperature in the collection vessel as low as practical to reduce the amount of vapor escaping through the vent.

The rate at which methanol will distill is a function of the temperature and the rate at which heat can be added to the liquid. Methanol can be distilled at atmospheric pressure and at temperatures between the boiling point of methanol (148 degrees Fahrenheit, 64.6 degrees Celsius) and the boiling point of water (212 degrees Fahrenheit, 100 degrees Celsius). Some more complex distillation systems also use vacuum to increase efficiency and reduce the chance of fuel scorching at high temperatures.

An effective water cooled condenser can be made by any moderately skilled plumber using copper pipe and readily available fittings (Figure 10). A popular design appropriate for water-heater based reactors uses a ½-inch copper pipe to carry methanol. This methanol pipe is soldered inside of a ¾-inch copper pipe fitted with a tee at each end. One leg of each tee is adapted to garden hose threads to carry cooling water. Cool water enters the bottom and warm water exits the top (this water can be routed to a wash tank if desired). Hot methanol gas exiting the boiler is directed through the interior copper pipe, where it is cooled by the water jacket, and condenses into liquid methanol before flowing into the collection vessel.

In the design pictured, the interior copper pipe is crimped at 90-degree angles to add turbulence to the flowing methanol, which increases the cooling effectiveness. (Design and photograph are courtesy of Graham Laming.)

For further discussion, see the References section.

FIGURE 10. CONDENSOR ON BIODIESEL REACTOR.

Since there will usually be some water in the reaction mixture, the distilled methanol may contain a small amount of water. Purity of recovered methanol should be assessed to provide feedback on the distillation process. Using a thermometer, graduated cylinder, and the established unit weight of pure methanol at various temperatures (see Table 4, below), one can roughly assess the purity of recovered methanol by weighing a 1 liter sample, then entering the result into the following equation:

$$\frac{1{,}000 - (\text{weight of 1 liter of distillate}) \times 100\%}{1{,}000 - (\text{weight of 1 liter of pure methanol at temperature})} = \%\ \text{purity}$$

Example: A 1-liter sample of recovered methanol weighs 805 grams at 60 degrees Fahrenheit. From the chart, a liter of pure methanol weighs 796 grams at 60 degrees Fahrenheit:

$$[1{,}000 - 805] \div [1{,}000 - 796] \times 100 = [195 \div 204] \times 100 = 95.6\%\ \text{pure}$$

Water contamination is more of a problem if producers are adding water to the fuel and glycerol during the "prewash" step described in Process Safety and Handling Issues. To reduce water contamination, heating of the fuel or glycerol should occur slowly, and care should be used as fuel or glycerol temperatures approach the boiling point of water (212 degrees Fahrenheit). Some producers will separate their "dry" methanol recovered in the early low-temperature stage from "wet" methanol recovered as the boiler temperature nears 212 degrees Fahrenheit. More sophisticated setups may add a reflux column to improve purity. This is essentially a vertical tube packed with some nonreactive material (e.g., stainless steel kitchen scrubbers, glass beads, or marbles) that will cause high boiling point liquids to condense. Hot gases exiting the boiler pass through the reflux column prior to reaching the methanol condenser. Since water has a higher boiling point, water vapor will condense on the packing material and drip back down into the boiler, while methanol vapor will continue to pass through and out to the main condenser. Use of a reflux column may increase the time it takes to distill all of the unbonded methanol from the fuel or glycerol.

Recovered methanol is typically blended with fresh methanol when used for making biodiesel. It is important that the combined fresh and recovered methanol is 95 percent pure or better for making quality biodiesel. Overly wet methanol will create significant heat when combined with

TABLE 4

Temp, degrees Fahrenheit	50	60	75	80	90	100
Mass of 1 liter pure methanol, grams	802	796	787	784	779	773

KOH or NaOH catalyst, which could present a safety hazard. (Water in the reaction also increases soap formation.)

Depending on biodiesel reactor design, the main reactor can also double as a methanol recovery still (provided that it is sealable and the tank and tubing are capable of handling high temperatures). Methanol can be recovered from each batch of fuel immediately after glycerol separation. Glycerol is typically stored in a sealed container until sufficient quantities are collected, then it can be pumped back into the reactor for distillation. More sophisticated plants use separate equipment for distillation, as some distillation units are commercially available.

Extreme caution should be used when distilling methanol to avoid fire and personal exposure. Adequate ventilation and the absence of ignition sources are a must. All fluids and gases should be transferred via tubing rated for the relevant temperatures expected. Methanol collection vessels should be vented to the outdoors to avoid vapor accumulation in the workshop, or recovery gear can be located out of doors with appropriate spill containment. Given the high temperatures involved, accidentally opening the wrong valve at the wrong time could lead to burns or concentrated methanol gas escaping into the workspace. Care must be taken to ensure that untrained personnel cannot inadvertently cause a mishap. Producers are advised to research the design of methanol recovery equipment appropriate to their operation and thoroughly think through all safety considerations. While methanol recovery is no more complicated than the basic biodiesel reaction, due to the inherent safety risks producers should not proceed to this advanced step until they adequately understand how to do so responsibly. For further information on methanol recovery, including equipment examples, see References.

Biodiesel Quality Test Methods

Biodiesel fuel can be made from a number of starting materials (feedstocks) including vegetable oils, used cooking oils, and animal fats. The end product (biodiesel) in the process of transesterification should be of high quality if the user is going to use it in a diesel engine without problems. While diesels will "run well" on a variety of fuels for short periods of time, there is potential for short- and long-term equipment problems if poor quality fuel is used. It is a false economy to damage normally long-lived diesel equipment in pursuit of a cheaper or more environmentally friendly fuel. Low temperature properties and safety (flash point) can also be compromised by poor quality fuels.

There are currently several specifications for biodiesel in the world depending on the location where the product is made. For the most part the standards are similar, but in some cases they are designed around the country's or area's primary feedstock and the temperature zone of the area. In all cases, it would be expensive and economically not feasible for the small manufacturer (farmer, fleet operator, etc.) to conduct a complete analysis of each small batch produced. For example, commercial ASTM testing currently costs in the range of $1,000 per batch. Some labs will test for individual parameters (for example, glycerol content), allowing a producer to pretest a batch of fuel at reduced expense prior to submitting samples for the full set of ASTM tests.

Here are some examples of international standards:

- Europe—EN 14214 and Biodiesel Heating Oil Standard EN 14213
- Brazil—ANP 225 (2003)
- South Africa—provisional standard
- United States—ASTM Biodiesel Standard D6751

The following paragraphs discuss the ASTM test parameters (listed in italic type), reasons for the various testing and some small-scale bench methods (listed in bold type) that may be used to evaluate the quality in an acceptable manner. It must be emphasized that there is no substitute for producing a quality fuel than knowing your feedstock and insuring the process used is the best for that feedstock. Small producers are encouraged to keep detailed production records, including notes on oil sources, catalyst and methanol amounts, mixing time and temperature, and other relevant variables. The small-scale tests are not necessarily quantitative, but they are good indicators as to whether the samples are approaching ASTM quality.

Free Glycerol. The presence of excess glycerol is of concern because it will lead to injector deposits. Free glycerol in fuel will be prevented by water washing and adequate settling of fuel before pumping into storage tanks.

Total Glycerol. Total glycerol is a measure of free glycerol and partially transesterified triacylglycerides (vegetable oil). It is a measure of the mono- , di- , and tri-acylglycerols plus free glycerol. In addition to deposit-forming tendencies, lacquer-like deposits can occur in heated parts of the fuel system. These deposits will affect the combustion spray patterns, resulting in higher emissions and possibly poorer performance. Both free and total glycerol are measured by ASTM Method D6584. The specification for total glycerol is less than 0.240 wt. percent. A goal of all biodiesel producers should be full "conversion," that is, the complete transformation of feedstock oil and methanol into fatty acid methyl esters (biodiesel) and separated glycerol by-product. Incomplete conversion due to poor processing will result in some level of mono- , di- , and triglycerides dissolved in the fuel. These contaminants are not visible in the biodiesel, and thus producers should take care not to assume that they have good quality fuel, just because they have achieved separation. Clear, light-colored biodiesel may still be contaminated to a level that would fail the ASTM test.

Methanol Solubility Test. Conversion is most easily assessed using the Methanol Solubility Test, first attributed to a small producer named Jan Warnqvist. Pure biodiesel will completely dissolve in methanol, while compounds containing glycerol will not. Thus, the ability of a sample of supposed biodiesel to dissolve in methanol is a moderately reliable indicator of the level of conversion.

This test is most easily performed in a tall, narrow container such as a test tube, olive jar, or centrifuge vial. Nine parts methanol are combined with one part biodiesel in the container. (Many small producers use 27 mL of methanol with 3 mL of biodiesel, thus this test is also commonly called the 27/3 test). The container is then inverted a few times to mix the fluids together, after which a visual inspection is performed to check the level of dissolution. This test should be carried out with methanol and biodiesel at roughly 70 degrees Fahrenheit.

A "pass" of this test will result in clear, lightly colored methanol with no residue settling to the bottom. Samples that result in cloudy methanol but no settling of residue may also be considered a "pass." A "fail" will result in some level of cloudiness, which on closer inspection reveals fine droplets of undissolved oil floating in the methanol that gradually settle out into the bottom of the container. The residue that settles out is unreacted glycerides. "Fail" is a qualitative term, and the level of conversion can be roughly assessed by the relative amount of material that settles out between various samples of fuel. Depending on the level of failure, failed batches of fuel may still be run in equipment if blended with diesel fuel at low concentrations. Very poorly converted batches should be returned to the biodiesel processor for further reacting with additional methanol and catalyst (see Reprocess Test below).

It is important that high purity (dry) methanol be used for this test, as the presence of small amounts of water in the methanol will result in inconsistent results. Methanol purity can be checked by weighing a sample of methanol, as described in Methanol Recovery. Accurate measurements are also essential for obtaining consistent results. As always, wear gloves and goggles and work in a well-ventilated, spark-free space when handling any quantity of methanol. While fuel samples that pass the methanol solubility test can be assumed to have achieved a high level of conversion, this is not a guarantee that fuel will meet the ASTM specifications for glycerol content. Complete analysis requires gas chromatograph or other testing by a qualified lab. For a complete discussion of this test, see the References section.

Full conversion of triglycerides to biodiesel is dependent on several factors, including sufficient catalyst (5 grams NaOH or 8 to 9 grams KOH per liter of oil, plus titration), sufficient methanol (22 to 25 percent of the oil volume), sufficient temperature throughout the reaction (120 to 130 degrees Fahrenheit), and sufficient mixing time (90 to 120 minutes or more, depending on reactor design). Producers should compare the results of the methanol solubility test with production records for each batch of fuel, in order to constantly improve their processing in the quest for full conversion. The methanol solubility test can also be performed on unwashed fuel, and as such it may be used on settled samples during processing to determine whether or not the reaction has had adequate time to reach full conversion, before fuel is removed from the main reactor.

It is common for those seeking full conversion to perform a two-stage reaction for the biodiesel process. In a two-stage reaction roughly 80 percent of the methanol and lye mixture are added to hot oil feedstock and mixed for 90 to 120 minutes or more. Glycerol is allowed to settle and then is drained to a storage tank. In the second stage, the remaining 20 percent of the methanol and lye mix is added to the partially converted biodiesel in order to react with any remaining glycerides. After sufficient mixing, the reaction is allowed to settle a second time, after which all remaining glycerol is drained. Producers who consistently fail the methanol solubility test are encouraged to try this two-stage reaction. (NOTE: If you wish to use the two-stage reaction *and* the water prewash method described in Process Safety and Handling Issues, glycerol drained in the first reaction stage must be returned to the reactor prior to adding water, or an emulsion may result.)

Reprocess Test. A somewhat more cumbersome conversion test involves taking a sample of biodiesel and reprocessing it in a small container as if it were vegetable oil. In the reprocess test, only 50 mL of methanol and 1 gram of NaOH or KOH are used per liter of biodiesel to be tested. The reaction is carried out at 120 to 130 degrees Fahrenheit. If residual glycerol is present in the fuel, it will

separate from the biodiesel and settle to the bottom of the container. If no residual glycerol is present, the sample may in fact form a gel or cloud when it is combined with the extra NaOH catalyst. In this test, extra glycerol settling out indicates incomplete conversion during the original processing step, while a gelled sample indicates a "pass" or full conversion. Due to the processing time involved, this test has largely been replaced by the methanol solubility test listed above. If producers desire to reprocess an incompletely converted large batch, the reactant quantities listed above can be extrapolated up to the volume of biodiesel to be reprocessed—running a test reaction on a small sample is recommended before adding more lye to a large batch to avoid gelling or soaping up the reactor.

Viscosity. Viscosity is a measure of the resistance of fluids to flow. The viscosity specification for biodiesel in ASTM D6751 is 1.6 to 6.0 centistokes. Viscosity is controlled to insure proper flow into the combustion chamber. If the viscosity of the fuel is too low, then it leaks by the seals resulting in a loss in power. If the viscosity is too high, sufficient fuel is not injected into the chamber, again resulting in a power loss, and potentially leading to injector coking or damaging carbon buildup in the cylinders. ASTM D445 is the test method for measuring viscosity. Viscosity of biodiesel will be affected by the level of conversion of triglycerides (oil) to methyl esters (biodiesel). If residual glycerol is present in the form of monoglycerides, diglycerides, or triglycerides, the viscosity of the fuel will increase. Viscosity is also greatly affected by temperature, so any viscosity testing must be performed at a constant temperature.

A simple viscometer can be made at home from a plastic soda bottle with the bottom cut out, and a small- (2 mm or less) tube snugly inserted into the lid of the soda bottle. The bottle is inverted, filled with fuel to be tested, and supported in a piece of wood or other stand. "Start" and "finish" lines are inscribed on the side of the bottle, and a stopwatch is used to measure the time it takes for a fuel sample to run through the viscometer.

This simple test can be an effective way to measure qualitative differences between batches of biodiesel made from the same feedstock oil, to assess the success of a particular production method at reaching full conversion. This test is not widely used, as it has been replaced by the methanol solubility test for assessing conversion. For a full discussion of the construction and use of the viscometer, see the References section.

Flash Point. This is an indication of the flammability of the liquid and is the temperature at which the fluid gives off enough vapor to produce a flammable mixture. Washed biodiesel has a very high flashpoint (greater than 130 degrees Celsius) compared to gasoline (-40 degrees Celsius, kerosene, No. 1, and No. 2 diesel fuel (52 to 66 degrees Celsius). It is an important parameter when preparing biodiesel because it gives an indication of alcohol (methanol or ethanol) left in the biodiesel product; the more alcohol remaining, the lower the flash point. With even 1 percent methanol remaining in the biodiesel the flash point would be lowered to a level requiring caution. Most producers increase the biodiesel flashpoint by water washing, which removes methanol. Methanol recovery systems will also sufficiently increase the flash point of the finished product. Unwashed fuel will have a low flash point due to methanol content and should be considered flammable and toxic.

Water. Water is present in fuels as dissolved water or free water. In No. 2 diesel fuel, the amount of absorbed water is about 50 parts per million (ppm) at ambient temperatures. Biodiesel on the other hand, because of its more polar nature, can absorb in the neighborhood of 1,500 ppm. This absorbed water can be corrosive to components of the fuel system. Water can also affect the stability of the fuel and be the media for bacterial growth in storage. Filter plugging can also occur at low temperatures.

Testing for Water. Washed fuel should be properly dried. Dry fuel will be clear enough to see through—this can be casually assessed by holding a jar of fuel in front of a printed sheet of white paper. However, wet fuel may appear clear when warmed, so it is best to assess the clarity of the fuel at or below room temperature (but not very cold, ¬even dry biodiesel will cloud at low temperatures.)

Fuel (and waste vegetable oil feedstock) can be tested for water by carefully heating it past the boiling point of water (100 degrees Celsius/212 degrees Fahrenheit). In this test, a small sample of fuel or oil is heated over a hot plate while stirring. If no bubbles arise in the fuel at the boiling temperature of water, the fuel is dry.

Caution: This test is best done over a hot plate in a safe, fire-proof, well ventilated area. Wet oil or fuel may "spit" if not stirred, or if heated too quickly. Hot fuel may produce smoke. It only takes a few minutes to heat oil to 212 degrees Fahrenheit (100 degrees Celsius), so never walk away while heating a sample of fuel or oil. Never throw water on an oil fire (water will spread an oil fire). Have a fire extinguisher handy.

Cloud Point. Biodiesel fuels have poorer low-temperature properties than petroleum diesel fuels. The cloud point and pour point of fluids are indicators of their low-temperature properties. The cloud point limitation in the biodiesel specification is to "report" the clouding temperature of the fuel according to ASTM Method D2500. This gives an indication of the temperature at which fuel-filter plugging is likely to occur. A new ASTM cold-flow filter plugging test is also published, and may soon be implemented by the biodiesel industry.

When blending biodiesel into winterized diesel fuel, the cloud point should not be a problem at low blend levels (concentrations). The compounds in biodiesel responsible for the formation of crystals at low temperatures will still form crystals at the cloud point temperatures, however, dilution by winterized diesel fuel will minimize the problem. (See Cold Temperature Issues, for further discussion.)

Sediment. Inorganic materials in fuel are undesirable since they can contribute to wear of engine components. Extraneous contamination, oxidation, storage stability, and residual catalysts can contribute to sediment in biodiesel fuels. Filters used in engines take out larger particles according to their nominal size. ASTM D482 and ASTM D874 are used to characterize and measure inorganic materials. ASTM D874 is included in the biodiesel specification ASTM D6751. It converts inorganic materials, including the sodium and potassium catalysts from the transesterification reaction, to sulfated ash. The catalyst residues are the most likely source of ash in the newly processed biodiesel.

In practice, filtration and gravity settling over time provide adequate sediment control for small scale biodiesel production. Particulates settle into the glycerol layer in initial processing, and into the wash water layer in washing stages. It is a good practice to filter fuel as it is passed from tank to tank during processing stages (especially if using open-topped wash tanks, which may accumulate debris or insects). Fuel should be filtered when pumping from the final storage tank to the equipment fuel tank with a water block filter of 10 micron (or smaller) capacity. Standard diesel-fuel filters available at farm or auto parts stores are normally sufficient. Washable cloth "sock" or "bag" filters, in a variety of dimensions, are also commercially available for between tank filtering. A helpful practice is to raise pump intake tubes to avoid pumping from the very bottom of the storage tank, and to remove any residual sediment by periodic cleaning.

Note: Operators running fuel in new diesels with high-pressure injection systems may desire to filter fuel to 1 micron, as particulates smaller than 10 microns in size have been shown to cause premature failure of high pressure injection equipment. Consult the engine manufacturer for specifications.

Soap Test. Residual sodium or potassium catalyst dissolved in biodiesel will be present in the form of soap, which is formed when free fatty acids and water in the feedstock oil react with the catalyst at the initial addition of methoxide. Soap is commonly removed from fuel by water washing, or by settling and filtration after methanol recovery from the fuel. Testing for soap can help producers determine the effectiveness of soap removal methods, and at what point to stop washing or filtering.

The soap test used by small producers is similar to titration for assessing free fatty acids in oil, except that it uses bromophenol blue as an indicator solution, and hydrochloric acid (HCL) as a titration solution.

1. Put a 250 mL flask on an accurate scale.
2. Add 100 mL of Isopropyl Alcohol (e.g., red bottle of HEET) and 12 drops of 0.04 percent Bromophenol Blue.
3. Carefully weigh the flask, alcohol, and Bromophenol Blue, and record the weight.
4. Add 10 grams biodiesel (approximately 11.5 mL) to the flask, using the scale to determine exactly how much biodiesel to add.
5. Place the flask on a stable surface.
6. Slowly add 0.01N HCL keeping track of how much is added. Swirl the flask between additions.
7. When the color just changes to yellow and stays there, the test is done.

Use the amount of HCL added to calculate the soap content of fuel in parts per million (ppm):

ppm soap = mL of 0.01N HCL x soap factor

Soap factor = 304.4 for sodium catalyst, 320.6 for potassium catalyst.

Producers seeking high-quality fuel will wash or filter soap until the levels are found to be below 20 ppm for ASTM spec. Further information on this test can be found in References.

There are currently no commonly used bench tests for the following ASTM specifications:

Sulfated Ash. ASTM D874 is the test method in the biodiesel specification for determining sulfated ash. Sulfated ash was discussed above under sediment. High ash can result in increased rates of wear.

Sulfur (ASTM D5453). The limitation on sulfur in the biodiesel specification is tied directly to the EPA regulations controlling sulfur levels in the oil. The 15-ppm level coincides with the regulation for the sulfur levels of Ultra

Low Diesel Fuel for on-highway diesel fuel that went into effect in 2007. The 500-ppm level regulations are for fuels that after 2007 are used for off-highway vehicles. The main concern with high levels of sulfur is that they contribute to engine particulate emission levels. The sulfur oxides produced can also contribute to acidic corrosion of the engine cylinders if the water jacket temperatures are too low (less than 180 degrees Celsius). Biodiesel fuel typically has a very low sulfur content.

Copper Strip Corrosion (ASTM D130). This is a test used to measure copper corrosion. It is an indication of level of acidic components in the biodiesel product.

Cetane No. Cetane Number (CN) is analogous to Octane Number (ON) in gasoline and is an indicator of the performance level of the fuel. Fuels with high CN have better higher energy content and combustion characteristics in the engine. CN can be determined using ASTM Method D613. Recent studies have shown that higher CN can also result in lower NOx emissions.

Carbon Residue. Carbon residue is an indicator of the tendency of the fuel to form deposits. ASTM Method D4530 is used for this test. Normally, if the fuel meets ASTM Specification D6751, carbon residue should not be an issue.

Phosphorus Content. Phosphorus-containing compounds are present in most vegetable oils as phospholipids. Phosphorus-containing compounds often impart antiwear protection to lubricants. However, phosphorus and sulfur negatively affect after-treatment catalysts and are undesirable in the fuel. ASTM Method D4951 is used to measure phosphorus content.

Distillation Temperature at 90 percent Recovery. High boiling components in a fuel have a tendency to produce deposits and odor. On shutdown, any remaining fuel on the injector tip tends to evaporate, leaving the high boiling components as a heavy residual and possibly odorous component. The high boiling ends tend to form deposits as found in the carbon residue test. The higher boiling components also do not combust as well. ASTM Method D1160 is a vacuum distillation method used to calculate the amount of high boilers above 360 degrees Celsius.

Those who have access to sophisticated laboratory equipment can perform additional tests of their fuel. Some quick tests to further evaluate quality of the biodiesel include viscosity (using a calibrated viscometer), flash point, infrared analysis, and gas chromatography. The tests are not necessarily quantitative but they are good indicators as to whether the samples are approaching ASTM quality.

The "pHLip" test is a commercially available indicator solution (BioSolar-CytocCulture International, Inc., Point Richmond, CA 94801) that is a qualitative visual check (not quantitative) of B100. This test is most commonly used by fuel buyers to determine whether a given delivery of fuel is of acceptable quality, or if it fails for total glycerol, soap content, free glycerol, oxidation, and catalyst contamination.

Titration Test for Used-Fryer-Oil Feedstock: Vegetable oil will develop an acidic condition as it is heated for frying in the presence of water. Extra lye catalyst must be added to account for the catalyst that is neutralized by any acid in the starting oil. The precise amount of extra lye to be added is determined via a simple chemical analysis called titration. This step is essential to making quality fuel as the correct amount of catalyst is necessary to achieve full conversion of vegetable oil into biodiesel.

Materials needed: Isopropyl alcohol (91 percent or better), oil to be tested, pH indicator solution, lye-water solution, small beakers or clean plastic cups, 3cc (3mL) and 5cc (5mL) plastic veterinary syringes without needles.

A lye-water solution is made by adding exactly 1 gram of the NaOH lye (or KOH) that will be used for processing to exactly 1 liter of distilled water. It is helpful to use a high-accuracy digital scale for this step. An effective pH indicator solution can be made by mixing 6 grams of turmeric powder (an Indian spice) to 100mL of isopropyl alcohol, then allowing the powder to settle and decanting the liquid. These stock solutions can be stored for months if they are not contaminated.

Reagents should be at room temperature if possible for successful testing. Cold oil may not dissolve well in cold alcohol.

1. Measure 10 mL of isopropyl alcohol into a small beaker or plastic cup.
2. Add 5 drops pH indicator.
3. Using a 5-mL syringe, add lye-water solution one drop at a time until the alcohol-indicator mix just begins to turn red. This is a "blank titration" and accounts for any acidity in the alcohol.
4. Using a 3-mL syringe, add exactly 1 mL of the oil to be tested to the alcohol-indicator mix, and swirl to dissolve.
5. Note the amount of lye-water solution in the 5-mL syringe and write it down.
6. Carefully add lye-water solution to the beaker, a few drops at a time, and swirl to mix after each addition.
7. When the oil-alcohol-indicator mix turns red or red-orange and stays that way for 15 seconds or more, the titration point has been reached. (Note, the mixture may return to yellow over time if left in the beaker. The initial 15-second color change is all that matters for this test).
8. Note the amount of lye-water solution remaining in the syringe, and subtract that from the amount in step 5, to determine the total lye water added (in mL).
9. The amount of lye-water solution added (in mL) equals the amount of extra lye that should be used for this oil (in grams).

Example: 5 grams of NaOH per liter of oil is commonly used as the necessary base amount for oil with negligible free fatty acids. If 2 mL of lye water was added to a used oil sample, then the total lye amount to be used for this oil would be 5 grams + 2 grams = 7 grams NaOH per liter of oil.

Small producers must become comfortable with accurately performing this simple test, as proper catalyst amounts will impact the quality of the finished product. It is recommended to perform several titrations on oil used in large batch processing in order to obtain an accurate representation.

Some producers will also test restaurant feedstock oils before collecting, to be sure that they are of sufficient quality. In general, oils that titrate below 4 or 5 mL of NaOH lye water are recommended for the simple single stage base catalyzed transesterification practiced by most small-scale producers. At high titration numbers, more soap will be formed during processing as free fatty acids react with extra catalyst added for neutralization. Excessive soap complicates washing, and will result in reduced yield of biodiesel. High titration number oils should be blended with low titration number oils to reach an acceptable average. Very high titration number oils (over 10 mL NaOH titration) are not worth collecting for those using basic processing techniques.

The titration test can be modified to reflect the "acid number" of the finished biodiesel, which is listed in the ASTM specifications:

Acid Number. The acid number is an indicator of the free acid remaining in the fuel. It is usually expressed as mg KOH/gram of fuel. It is often referred to as the "Neut number," or neutralization number. Acid number is of concern for two reasons, the first is that it can lead to acid corrosion and the second is that over time, it can catalyze the formation of additional acid. ASTM Method D664 is recommended to measure acid number.

Those desiring a more thorough discussion of biodiesel quality testing are encouraged to review the National Renewable Energy Laboratory document (see References).

CONCLUSIONS

As we have attempted to show, small-scale biodiesel production can be conducted in a safe and environmentally responsible manner which generates a quality product. It is however, important to ensure that best management practices are followed in order to protect the health and safety of the producer and the environment, and to minimize the risk of vehicle/machinery problems. While we have tried to cover all aspects of small-scale production, each individual's facility will be different, with its own set of risks. Each biodiesel producer must take personal responsibility for his or her own safety and fuel quality. Producers are encouraged to keep up to date with new technologies and to stay informed. There are many different sources of information on biodiesel production, some of which are presented in the next section.

If you are a small-scale biodiesel producer, we congratulate you on your initiative to help mitigate air pollution, climate change, and our nation's energy crisis by using a renewable, alternative fuel. We hope that the small-scale biodiesel community will continue to work with regulatory agencies and academic institutions to cooperate towards the goals of a clean environment, safe workplaces, and a sustainable energy future.

REFERENCES

Regulatory Agencies And Applicable Laws

Environmental Protection Agency (EPA)
US EPA Region 3
1650 Arch Street (3PM52)
Philadelphia, PA 19103-2029
Phone: 800-438-2474

Internal Revenue Service Forms and Publications
http://www.irs.gov/formspubs/
This link provides access to all of the Internal Revenue Service's forms and publications that are needed for registration, payment of excise tax, and any biodiesel producer or blender credits.

Code of Federal Regulations (U.S. Regulations)
www.gpoaccess.gov/cfr/index.html
This link provides access to all of the federal regulations. For those referenced in this publication, browse to the appropriate title (40–Protection of the Environment) and find the appropriate part.

Pennsylvania Department of Environmental Protection (PADEP)
ww.depweb.state.pa.us/dep/site/default.asp

Southeast Region
Bucks, Chester, Delaware, Montgomery, and Philadelphia Counties
2 E. Main Street
Norristown, PA 19401-4915
Phone: 484-250-5900

Northeast Region
Carbon, Lackawanna, Lehigh, Luzerne, Monroe, Northampton, Pike, Schuylkill, Susquehanna, Wayne, and Wyoming Counties
2 Public Square
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Southcentral Region
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909 Elmerton Avenue
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208 W. 3rd Street, Suite 101
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Southwest Region
Allegheny, Armstrong, Beaver, Cambria, Fayette, Greene, Indiana, Somerset, Washington, and Westmoreland Counties
400 Waterfront Drive
Pittsburgh, PA 15222-4745
Phone: 412-442-4000

Northwest Region
Butler, Clarion, Crawford, Elk, Erie, Forest, Jefferson, Lawrence, McKean, Mercer, Venango, and Warren Counties
230 Chestnut Street
Meadville, PA 16335-3481
Phone: 814-332-694

PADEP's Preparedness, Prevention, and Contingency Plan Guidance
www.depweb.state.pa.us/watershedmgmt/cwp/view.asp?a=1437&q=518752&watershedmgmtNav=|
Click on "Technical Information" and then "Guidelines for the Development and Implementation of Environmental Emergency Response Plans."

Request for Determination (PADEP Air-Quality Issues)
www.dep.state.pa.us/dep/deputate/airwaste/aq/permits/rfd.htm

Pennsylvania Code (Pennsylvania's Regulations)
www.pacode.com
This link provides access to all of the Pennsylvania regulations. For those referenced in this publication, browse to the appropriate title (25 for Environmental Protection or 37 for Flammable and Combustible Liquids) and find the appropriate chapter.

Pennsylvania Department of Revenue (PAREV)
www.revenue.state.pa.us
Bureau of Motor Fuel Taxes
717-783-1563

Pennsylvania Department of Labor and Industry, Flammable and Combustible Liquids Section (PADL&I)
Room 1614, L&I Building
7th and Forster Streets
Harrisburg, PA 17121
Phone: 717-705-2787

GENERAL BIODIESEL INFORMATION

Biodiesel Production References and Biodiesel Reactor Designs

- The Collaborative Biodiesel Tutorial: www.biodieselcommunity.org
- The University of Idaho: www.uidaho.edu/bioenergy/
- General Discussion Forum: biodiesel.infopop.cc/6/ubb.x?a=cfrm&s=447609751
- *Biodiesel Smarter* Magazine: www.biodieselsmarter.com/

Biodiesel Quality Testing—National Renewable Energy Laboratory Document
www.nrel.gov/docs/fy04osti/36240.pdf

Biogas (Anaerobic) Digesters: Small-Scale Plans and Discussion
www.biorealis.com

Biodiesel Costs Worksheet

Cost Assumptions						
	Biodiesel Purchase Price ($/gal)	Electricity ($/kwh)	Gasoline ($/gal)	Labor ($/hr)	Feedstock Oil Cost (used cooking oil)	
	3.39	$0.08	$2.79	$10.00	$0.00	
Biodiesel Production and Value						
Batch Size (gal)	Biodiesel Value					
54	$183.06					
Capital Investment Depreciated						
	Capital Cost of Equipment	Number of Batches per Year	Payback period (yrs)	Cost		
	$3,500.00	10	7	$50.00		
Feedstock Acquisition						
Process step	Input	Units	Quantity	Cost		
Cost of oil	Cooking oil	Gallon	55	$0.00		
Collect used cooking oil in truck	Gasoline	Gallon	1	$2.79		
	$2.79					
Chemical Cost						
Chemical	Quantity Purchased	Purchase Price	Quantity Input	Quantity Recovered	Net Quantity Used	Cost
Methanol (gal)	55	$179.30	12	1.25	10.75	$35.05
Lye (gram)	2200	$5.94	1600	N/A	1600	$4.32
	$39.37					
Electricity						
Process Step	Device	Instantaneous power draw (watts)	Time (hours)	Kilowatt hours	Electricity Cost	
Pump oil into storage	suction pump	350	0.33	0.12	$0.01	
Pump oil into reactor	suction pump	350	0.5	0.18	$0.01	
Pump oil into reactor	reactor pump	200	0.5	0.10	$0.01	
Preheat oil for dewatering	Reactor element	4500	1	4.50	$0.36	
Mix during dewatering	reactor pump	200	0.25	0.05	$0.00	
Heat for processing	Reactor element	4500	1.5	6.75	$0.54	
Mix during heating	reactor pump	200	0.5	0.10	$0.01	
Mix during methoxide inlet	reactor pump	200	0.5	0.10	$0.01	
Mix during reaction	reactor pump	200	1.5	0.30	$0.02	
Pump off glycerol	reactor pump	200	0.08	0.02	$0.00	
Heat fuel for methanol recovery	Reactor element	4500	2.5	11.25	$0.90	
Pump fuel to settling tank	reactor pump	200	0.17	0.03	$0.00	
Pump fuel to wash tank	wash pump	200	0.17	0.03	$0.00	
Bubble wash	compressor	15	8	0.12	$0.01	
Pump to dryer	wash pump	200	0.17	0.03	$0.00	
Heat for drying	dry tank element	1500	1.5	2.25	$0.18	
Pump while drying	wash pump	200	2	0.40	$0.03	
Pump to storage	wash pump	200	0.17	0.03	$0.00	
Vent fan, total usage	vent fan	500	0.3	0.15	$0.01	
	$2.12					
Labor						
Process Step	Hours	Cost				
Collect oil	1.5	$15.00				
Titration	0.5	$5.00				
Mini-batch	1	$10.00				
Full-scale batch	5	$50.00				
Fuel testing	1	$10.00				
Waste product disposal	4	$40.00				
	$130.00					
Cost Summary						
	Total Cost	Volume (gal)	Cost/gal			
Cost to make biodiesel	$224.28	54	$4.15			
Cost to buy biodiesel	183.06	54	3.39			
Difference	-$41.22	-$0.76	Extra			
Assumptions						

Constructed Wetlands Small-Scale Plans
www.greywaterguerrillas.com/publications.html

Energy Balance Article
biofuels.coop/education/energy-balance/

Fumeless Processing and Waterless Soap Removal
- "Eco-system": www.london-electronics.com/bd/ecosystem/state_diagram_new.htm
- Discussion: biodiesel.infopop.cc/eve/forums/a/tpc/f/719605551/m/9921000191

Glycerol Feed Additive
- www.regional.org.au/au/gcirc/1/241.htm
- www.uidaho.edu/bioenergy/NewsReleases/Technote06.pdf

Glycerol Soap Recipes
www.b100wh.com/barsoap1.html#black

Heating Appliances and Biodiesel
www.worleyobetz.com

Methanol Recovery
- Discussion forum: biodiesel.infopop.cc/eve/forums/a/frm/f/4441089311
- Copper condenser design: www.graham-laming.com/bd/still/still.htm
- Small condenser design: www.b100wh.com/recovery1.html#condenser
- Slide show: www.b100.org/presentations/MethanolRecovery/

Methanol Solubility Test
biodiesel.infopop.cc/eve/forums/a/tpc/f/9411061471/m/8281092351

Prewashing Fuel
www.biodieselcommunity.org/waterprewash

Soap Test
- www.biodieselcommunity.org/testingforsoap
- www.fryerpower.com/store/page4.html

SVO Conversion Resources
- Fossil-free fuel: www.fossilfreefuel.com
- Discussion forum: biodiesel.infopop.cc/eve/forums/a/frm/f/159605551

Viscometer
biodiesel.infopop.cc/eve/forums/a/tpc/f/419605551/m/590108381/p/1

Winter Preparation
www.coloradobiodiesel.com

GLOSSARY

Agricultural Waste—Waste that is generated in the production and marketing of poultry, livestock, fur bearing animals, and their products. Agricultural waste also includes agronomic, silvicultural, and horticultural commodities grown on farms or other agricultural lands. Waste generated during normal farming operations, including manure and food-processing waste, may be disposed on lands where the materials will improve the condition of the soil or improve the growth of crops. Biodiesel and waste generated during the production of biodiesel, would not be considered agricultural waste because biodiesel production is not a normal farming operation (see also "normal farming operation").

General Permit—A regional or statewide permit issued by PADEP for the beneficial use or processing of solid waste.

Hazardous Waste—Waste that, because of its quantity, concentration, or physical or chemical characteristics may pose a substantial hazard to human health or the environment when improperly stored, transported, or disposed. Characteristic hazardous waste exhibits one of the following characteristics: ignitability, corrosivity, toxicity, or reactivity, or is listed as a hazardous waste under 40 CFR, Chapter 261. Hazardous wastes from biodiesel production may include glycerol or methanol containing water, which may meet the ignitable characteristic for hazardous waste. Pure commercial methanol, if disposed, is a hazardous waste.

Municipal Waste—Waste including solid, liquid, semisolid, or contained gaseous material resulting from the operation of residential, municipal, commercial, or institutional establishments. Municipal waste may include waste or off-spec vegetable oils, used restaurant oil, and yellow grease used for biodiesel production from municipal waste sources as named in this definition.

Normal Farming Operation—The generally accepted activities, practices, and procedures that farms engage in year after year in the production and preparation of poultry, livestock, and their products; and in the production, harvesting, and marketing of agronomic, horticultural, silvicultural, and aquicultural crops and commodities. Biodiesel production is not considered a normal farming operation.

Permit—A permit issued by the department to operate a waste disposal or processing facility, or beneficially use municipal or residual waste.

Permit-by-Rule—A permit which a person is deemed to have for the operation of a specific facility upon compliance with the municipal or residual waste management regulations of Pennsylvania.

Regulated Substance—A substance that, when released into the environment, may present substantial danger to the public health, welfare, or the environment which is either:

1. A hazardous substance
2. Petroleum, including crude oil or a fraction thereof, including fuel oil, oil sludge, oil refuse, oil mixed with other nonhazardous wastes and crude oils, oil, gasoline, and kerosene
3. Other substances determined by PADEP

Residual Waste—Waste including solid, liquid, semisolid, or contained gaseous material resulting from industrial, mining, and agricultural operations. Residual waste may include waste or off-spec vegetable oils, yellow grease, and oils and animal fat from food processing or rendering plants, or on-site rendering waste from farms, used for biodiesel production from residual waste sources as named in this definition. As a by-product of biodiesel production, glycerol and tank bottoms may fall under this definition (see also "hazardous waste").

Solid Waste—Waste including municipal, residual, and hazardous waste.

Storage Tank—An aboveground or underground storage tank that is used for the storage of a regulated substance.

Waste Storage—The containment of any solid waste on a temporary basis. Storage of any waste greater than one year is considered disposal.

APPENDIX A: STRAIGHT VEGETABLE OIL CONVERSIONS

"Straight Vegetable Oil"—Converting the Diesel Equipment
This second viable option for using vegetable oil as fuel requires altering the fuel system of the diesel engine to heat the vegetable oil, thereby reducing its viscosity. There are many commercially available kits, as well as homemade conversions, most of which function on the same basic principles.

A second fuel tank, made from aluminum, plastic, or stainless steel, is added to the equipment; this tank will be filled with filtered, dewatered vegetable oil or used cooking oil. This tank is equipped with a heat exchanger of some kind (aluminum or stainless steel coils, or a small radiator inserted into the spare tank; copper heat exchangers are not compatible with vegetable oil fuel systems). This heat exchanger is plumbed into the motor's cooling system, such that when the motor is hot, high-temperature coolant can be routed through the heat exchanger in the spare fuel tank. A fuel line from the vegetable oil tank is routed to a mechanical tank selector valve upstream of the injector pump. Typically this vegetable oil fuel line is equipped with its own separate fuel filter, and both filter and fuel line are heated by bundling them with the hot coolant line from the engine. Some designs also use electrically heated components throughout the fuel system.

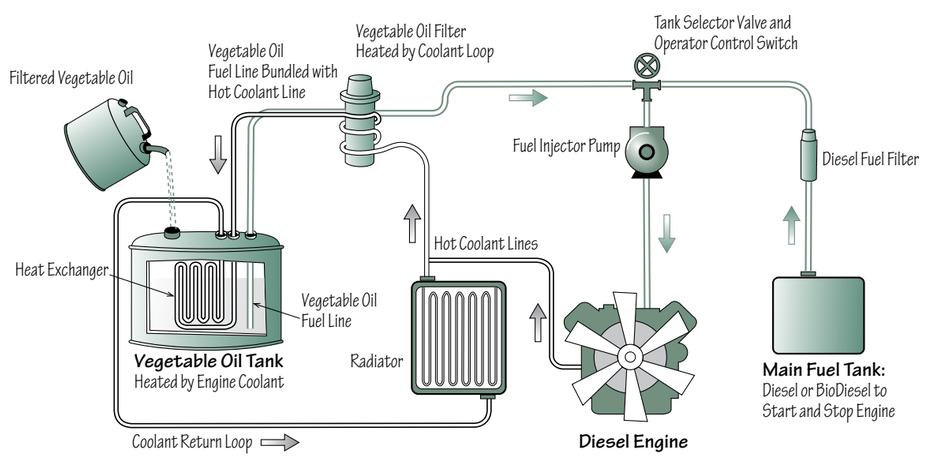
Fuel return lines may be looped back into the injector pump, so that unburned diesel or vegetable oil will not be pumped into the wrong tank. Alternately, the valves can be staggered in their switching to prevent cross-contamination of different fuel tanks, which also allows for shortened purge times and eliminates air entrapment issues sometimes caused by looped return lines.

The engine is started on diesel fuel from its original fuel tank. Once the engine is up to normal operating temperature, the operator throws a switch that controls the mechanical valve, switching the input to the injector pump from diesel fuel over to hot vegetable oil. The engine continues to run on hot (160 degrees Fahrenheit [71 degrees Celsius] is ideal) filtered vegetable oil from the spare tank until just prior to shutting down, at which time the operator switches back to the diesel tank via the mechanical valve. This allows the unconverted vegetable oil to be purged from the injector pump and injectors, so that the motor will be ready for the next cold start. The simplest systems are completely manual, while higher-end kits use sensors and computer-controlled valves to reduce operator input.

Straight vegetable oil conversions are most commonly used on road vehicles, such as diesel passenger cars and trucks. Some individuals have also converted stationary diesels and farm equipment. Prospective users should research the likelihood of success with their particular equipment before proceeding. Older diesels with indirect injection are more likely to be tolerant of a wider fuel temperature range, while modern fuel systems with high pressure "common rail" injection will have tighter tolerances. With enough study and proper design, any diesel system can potentially be converted to run on heated vegetable oil. Conversion of any engine that is under warranty is not advised.

Because the engine must be started and stopped on diesel fuel, SVO systems make sense for equipment that will be operated for long periods at a time, such as field tractors, irrigation pumps, and vehicles that regularly travel more than 20 miles per trip. Other comparative advantages and disadvantages between straight vegetable oil conversions and biodiesel can be summarized as follows:

FIGURE 11



Simplified Example of Straight Vegetable Oil Conversion Concept for Diesel Engine
Not to Scale, Fuel Return Lines Not Shown
Original By Matt Steiman, 2007

SVO Advantages

- SVO systems do not use chemicals to convert the fuel (as in biodiesel). This minimizes the user's input of nonrenewable energy, off-farm inputs, and money. Biodiesel requires an input of purchased chemicals for each gallon produced. Handling of hazardous chemicals and by-products is not an issue with SVO systems.
- If farmers extract oil from their own crops, SVO conversions would potentially allow them to produce their own fuel in complete independence.
- After the initial expense of converting the fuel system, there is minimal additional cost to SVO users except for the periodic purchase of filters. SVO systems can be very cost effective.
- The heated spare tank allows SVO systems to be operated in cold weather, whereas biodiesel must be cut substantially with petroleum fuel or additives in winter to prevent gelling and clogged fuel filters.

SVO Disadvantages

- The fuel system modifications in SVO systems are significant, and will certainly void engine warranties. Service technicians may be unfamiliar with the operation of SVO systems should fuel system troubles arise.
- The custom-fit nature of SVO systems typically requires occasional tinkering under the hood by the user.
- Running equipment on SVO requires the conversion of the fuel system on each engine, and may require a greater initial investment if a farm has several diesels in the fleet. Biodiesel users can build one machine to convert vegetable oil to fuel for any diesel on the farm.
- The user must find room for the on-board spare fuel tank for mobile diesel equipment.
- The effects on the engine of long-term use of SVO are unknown.

For more detailed information on straight vegetable oil conversions, see the References section in this booklet.

BIODIESEL BATCH RECORD SHEET

Batch #:

Date started:			
Name of brewers:			
Quantity of oil used:		Quantity of methanol used:	
Methanol purity:			
Titration # (1)	(2)	(3)	(Avg.)
Catalyst used:			
Quantity of catalyst used:			
Time heat on:			
Time heat off:			
Mixing temp:			
Mixing time:			
5% prewash:	Yes	No	

Washing notes:

Drying notes:

Methanol recovered from fuel?	Yes	No	Quantity recovered:
Methanol recovered from glycerol?	Yes	No	Quantity recovered:

Method of disposal of glycerol:

Quality Testing Notes

27/3:

Water test:

Soap test:

APPENDIX B: SAMPLE BIODIESEL BATCH STATUS CHECKLIST

Batch #:	Oil (gallons):	Catalyst: KOH/NaOH
Start date:	Methanol (gallons):	Avg. titration #:

Processing Step	Date	Initials	Notes
Day 1: Biodiesel Reaction			
Load oil into reactor			
Note oil temp on thermometer			
Heat oil: note start time			
Note heat stop time			
Note pump start time			
Note reaction temp			
Inject methoxide			
Note total mix time			
Note pump stop time			
Note special processing comments			
Check that heat, pump, water, and power are off before departing shop			
Day 2: Methanol Recovery from Fuel			
Connect vent hose to glycerol tank, open glycerol vent valve			
Connect glycerol hose to glycerol tank			
Turn on power and pump glycerol to storage			
Note approx. glycerol amount, add to glycerol tank record			
Attach methanol jug and connect to vent			
Note tank temp			
Turn on heat, note heat start time			
Turn on pump at or before 150°F; note pump start time			
Turn on cooling water at or before 150°F			
Be sure to watch monitor water level in wash tank			
At 190°F tank temp, switch out methanol jug for "wet methanol"			
Weigh a 1-liter sample of recovered methanol; note weight			
Note methanol sample temperature and purity (from chart)			
Stop heat and pump at 200–212°F tank temp; note stop time			
After 5 minutes, turn off cooling water			
Label and store methanol jug in locked cabinet			
Close all valves that should not be open			
Check that heat, pump, water, and power are off before departing shop			
Washing Notes			
Pump to settling tank			
Monitor to avoid overflow			
Wash 1 (Be sure to monitor tank level)			
Note start gallons			
Note stop gallons			
Note pH			
Wash 2			
Note start gallons			
Note stop gallons			
Note pH			
Wash 3			
Note start gallons			
Note stop gallons			
Note pH			
Allow to settle several hours after wash 3.			
Flush water from line between wash tank and pump			
Pump to dry tank			
Note dry tank start temp			
Note heat and pump start time			
Note heat and pump stop time			
Note bubbler start time			
Note bubbler stop time			
Note dry test results			
Pull sample for archive			
Note 27/3 results			
Add batch number to fuel tank log			
Add this sheet to record book			

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