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The FOOLPROOF way to make biodiesel

Updated! (6 April 2002)

By Aleks Kac

Free fatty acid to ester conversion

This is a FOOLPROOF way to make biodiesel. No titration is required, and no extra equipment -- a thermometer's handy, but NO pH meter!

This is a two-stage procedure, acid first-stage, base second-stage. It is based on the highest free fatty acid (FFA) content found in used cooking oil, but it can be used with any waste vegetable/animal oil or fat, whether or not it has a high FFA content. The process increases yields dramatically. Use it as your standard method.

Introduction

To make biodiesel fuel efficiently from used vegetable oils and animal fats we have to avoid one major problem: soap formation. Soap is formed during base-catalyzed transesterification (using lye) when sodium ions combine with free fatty acids present in used (and some virgin) vegetable oils and animal fats. The soaps diminish the yield because

UPDATE

Since Aleks first posted the Foolproof Method at Journey to Forever a year ago, it's been used to make many thousands of gallons of high-quality biodiesel from all sorts of feedstock.

Feedback has been most appreciative -- the method is now being used all over the world, by both seasoned biodiesellers and first-timers, as well as several commercial operations, a women's cooperative, student groups, a Third World village, a volunteer fire brigade, and many others.

In laboratory tests the product has tested at Spec. grav. (density) 886g/l, Kin. visc. 4.7 mm2/sec, CFPP –9 deg C, and with an IR spectrum equal to control samples from commercial producers in Austria -- very good fuel! See National standards for biodiesel

This newly updated version includes improvements developed over the last year.

they bond the methyl esters to water. The bonded esters get washed out at the washing stage but make water separation more difficult and increase water consumption. This process takes care of the free fatty acids.

In one early test I used a mixture of 50% heavily used cooking oil and 50% pork lard. The result was a pure product with absolutely no trace of soap! The biodiesel looked nice, and smelt nice, as if made from virgin oil.

This is a simple procedure. The first-stage process is not transesterification, but pure and simple ESTERIFICATION. Esterification is followed by transesterification, but under acid conditions it's much slower than under caustic conditions and it won't do a complete oil-to-methyl ester conversion as the reaction is much more equilibrium-sensitive. Without methanol recovery, the alcohol overdose required would make the price of your fuel jump, and even with recovery it would still be much more expensive. Hence the second base-stage.

Aleks (background) and friend Matevz making biodiesel in Matevz's living room.

For the first stage you'll form a compound out of an acid and an alcohol. The alcohol is still methanol, but instead of using lye (sodium hydroxide), the CATALYST in this reaction is sulfuric acid ("battery acid"). It needs 95% sulfuric acid (battery acid is around 50%). Sulfuric acid is one of the commonest chemicals on Earth, just like lye. More concentrated sulfuric acid -- 98% and above -- costs more, but 95% works just fine if you

follow these directions. Other acids won't work: it must be sulfuric acid. The second stage uses lye, as usual -- but it only uses about half as much as other methods.

The sulfate ion in the sulfuric acid combines with the sodium ion in the lye during the second-stage reaction to form sodium sulfate, which is a water-soluble salt and is removed in the wash. No sulfur remains in the biodiesel fuel product.

Equipment

A bottom-drained reactor vessel is best, closed on top. Tall, narrow containers work better than wide, shallow ones. Use a circulating pump for mixing rather than a mechanical stirrer. The pump should take the mixture from near the bottom of the reactor and return it via the top, to splash down on the surface. For a 35-litre reactor, a 100-W washing-machine pump will do, along with a 1.5kW washing-machine immersion heater to heat the mixture (get a heater that's coated with stainless steel). You could use a thermostat to control the temperature, but they're expensive: just use a thermometer and switch on the heater as required.

Ordinary iron and steel implements and containers will eventually corrode because of the acid used in this process. However, you can still use the usual 55-gal drum. The proportion of acid used in this process is very low -- you should be able to use an uncoated drum for a year or more before the rust gets out of control. I use a polypropylene plastic reaction vessel. Any plastic that won't deform at 100 deg C (212 deg F) or a bit more is fine. Stainless steel is also fine. Use an immersion heater with plastic containers. With steel containers you can use propane heaters to heat the oil, then switch to an immersion heater before adding the methanol.

Test batches

Whenever you're trying a new method, it's always a good idea to make small test batches of a liter or less first to familiarize yourself with the process before moving on to bigger batches. Most people use kitchen blenders for this -- but don't use it for food again afterwards!

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Foolproof pictures

Aleks made a couple of mini-batches in glass jars for these photographs -- one batch of heavily used cooking oil and fat, and one of pure fat.

1. 30% fats and 70% oil -- lumps of semi-hard fat remain after water boil-off.

The process

1. Filter the used cooking oil first as usual.

2. For a successful reaction the oil must be free of water. Here are two methods of removing the water content:

(a) Settling the water out: This method saves energy. Heat the oil to 60 deg C (140 deg F), maintain the temperature for 15 minutes and then pour the oil into a settling tank. Let it settle for at least 24 hours. Make sure you never empty the settling vessel more than 90%.

(b) Boiling the water off: Less-preferred method as it uses more energy and helps to form more FFAs in the oil. Heat the oil to 100 deg C (212 deg F). As the heat rises water separates out and falls to the bottom -- drain it off to avoid steam explosions. Maintain the temperature until no more steam bubbles rise.

First stage

3. Measure the volume of oil/fats to be processed (preferably in liters).

4. Heat the oil to 35 deg C (95 deg F) -- make sure that all solid fats are melted.

5. Methanol: use only 99%+ pure methanol. Measure out the methanol -- 0.08 liters of methanol for each liter of oil/fats (8% by volume). Add the methanol to the heated oil.

CAUTION

Sulfuric acid is a DANGEROUS CHEMICAL. Take full safety precautions, wear safety goggles, gloves and apron and eye protection. Have running water nearby. Don't inhale fumes!

If you run out of sulfuric acid NEVER try to make up the required volume with nitric acid. It may form small quantities of nitroG (nitroglycerine) -- even the smallest amount can cause horrible accidents. See: **High Explosives**

Methanol can cause blindness and death, and it is absorbed through the skin. Sodium hydroxide can cause severe burns and death. Together these two chemicals form sodium methoxide. This is an extremely caustic chemical. These are dangerous chemicals -- treat them as such! Always have water running nearby when working with them. The workspace must be thoroughly ventilated. No children or pets allowed.

2. Fat-oil mixture after melting.

3. Suspension formation after methanol introduction (note murkiness).

4. End of acid stage (note darker colour).

5. 100% semi-solid fats-melted fats.

6. Mix for five minutes -- the mixture will become murky because of solvent change (methanol is a polar compound, oil is strongly non-polar; a suspension will form).

7. For each liter of oil/fats add 1 milliliter of 95% sulfuric acid (H2SO4). Use a graduated eyedropper, a graduated syringe or a pipette. TAKE CARE when handling the concentrated sulfuric acid!

8. Mix gently at LOW rpm (don't splash!) while keeping the temperature at 35 deg C. The rotation of your stirrer should not exceed 500 to 600 rpm -- speed is not crucial and splashed oil is a mess to clean.

9. Maintain the temperature at 35 deg C for one hour then stop heating. Continue stirring.

10. Stir the unheated mixture for another hour, a total of two hours, then stop mixing. Let the mixture sit for at least eight hours, overnight is better.

11. In the meantime prepare the sodium methoxide: measure 0.12 liter of methanol for each liter of oil/fat (12% by volume) and weigh 3.1 grams (up to 3.5 grams if purity is in doubt) of sodium lye (sodium hydroxide, NaOH) per liter of oil/fat. Mix the lye into the methanol until the lye is completely dissolved.

Sodium methoxide is a DANGEROUS CHEMICAL. Take full safety precautions when working with methanol, lye and sodium methoxide, wear safety goggles, protective gloves and clothing, have running water nearby.

NOTE: This process uses only about half the usual amount of lye as there is less fat left to transesterify. Use 99%+ pure sodium hydroxide lye. After

Methoxide the easy way

Mixing lye with methanol creates an exothermic reaction, generating heat. It's nasty stuff and it's not easy to mix -- and it must be thoroughly mixed before you use it, with all the lye dissolved. This is a safe and easy way to do it. The disadvantage is that you have to do it in advance, but that's easily arranged.

Take FULL SAFETY PRECAUTIONS when

6. Change after adding methanol: murky fat (normal -- this is procedure).

7. First-stage glycerine.

8. Second stage -- finished esters.

9. Second-stage glycerine.

opening the container, close it again as quickly as possible to prevent moisture getting in. Weigh the lye carefully -- using too much will complicate the washing process later.

12. After settling for eight hours, or the next morning, pour half of the prepared methoxide into the unheated mixture and mix for five minutes. This will neutralize the sulfuric acid and boost the base catalysis. If you've used solid fat, it probably solidified during settling -- gently melt the mixture first.

Now you can continue with the normal procedure with the lye as the second stage.

Second stage

This is the base-catalyzed stage.

13. Heat the mixture to 55 deg C and maintain for the whole reaction.

14. Add the second half of the prepared sodium methoxide to the heated mixture and start mixing at the same low speed of not more than 500 to 600 rpm.

TAKE CARE when handling the sodium methoxide -- full safety precautions!

15. If your reactor allows for it, start draining glycerine from the bottom 20-25 minutes after the start of the base stage. (Pump-mixing works best for this.) Repeat every 10 minutes -- take care, the glycerine is quite hot and caustic! Set aside -- see step 18.

working with methanol, lye and sodium methoxide!

Use a tough, thick, container made of HDPE (High-density Polyethylene -- usually marked "HDPE" on the bottom, with the international code "2"), with a tight stopper and a screw-on lid. Measure out the methanol into the container. Add the required amount of lye -- if you're doing large quantities, add it bit by bit rather than all at once, give the container a swirl in between (replace stopper and lid first). Once it's all added, replace the stopper and the lid, and swirl the mixture about for a few seconds. Then let it stand. Do that a few more times, every few hours or so (at least 4-6 times in all). It will be thoroughly dissolved in 24 hours, or maybe a bit longer.

The proportion of lye to methanol used in making biodiesel is low, particularly with the Foolproof method. If for some reason you're using much higher proportions of lye, then don't do it

this way.

16. All users: Take regular samples in a 1" to 1.5" diam. glass container. Watch for a straw yellow color of the ester portion. Glycerine (brown and sticky) will settle at the bottom of the jar. When this color is reached (usually in 1.5-2.5 hours) turn the heat and mixer off. Instead of taking out samples to check the color you could use translucent braided tubing for the pump.

17. Allow to settle for one hour.

18. For easier washing: Drain off the glycerine. Measure off 25% of the total glycerine (including previously drained glycerine if you followed step 15) and mix with 10 milliliters of 10% phosphoric acid (H3PO4) for each litre of oil/fat processed. The mixing can be done with a wooden spoon in a plastic container. Pour the acidified glycerine back into the reactor and stir for 20 minutes, unheated. Allow to settle for at least six hours and then drain the glycerine fraction completely.

THIS IS IT. During the first stage, free fatty acids were esterified and some triglycerids were transesterified. The base-catalyzed stage does only transesterification, but it's much quicker and more complete.

Washing

19. Use the bubblewash method, but no need to monitor pH anymore. Just add a little 10% phosphoric acid (H3PO4) to the washing water first, 10 millilitres per gallon, just to be on the safe side -- I don't want ANY lye floating around my fuel pump.

If you are curious about the results of your wash, use ordinary litmus paper, it will tell you the rough pH level (acidity/alkalinity). The end result should be neutral (pH7) or just below neutral.

20. Use one-third the volume of water as the amount of biodiesel to be washed. Make sure both the water and biodiesel are roughly the same (room) temperature. Pour your biodiesel into the vessel with the water, throw in the aquarium stone and start the air pump. Let it bubble for 24 hours minimum. Turn the pump off and let the mixture settle for half an hour. The water will fall to the bottom, turning completely white, and the fuel you made will be much

lighter in color now. Drain the water, repeat the procedure two more times. Remove the biodiesel from the vessel, taking care not to get any water with it.

21. Let the biodiesel stand for about three weeks and use only when it becomes crystal clear; take a sample in a large marmalade jar and wait until it is completely cleared. Put it on your window shelf and enjoy looking at it while it clarifies.

NOTE: A deposit forms in the bottom during settling -- don't let it get in your fuel tank!

Acid-stage questions

A question will probably arise: why not mix the methanol with the sulfuric acid before adding them to the oil/fats? Two major reasons: (a) the reaction between methanol and concentrated H2SO4 is quite violent and it could splash, which doesn't happen if you mix it as described; and (b) dimethyl ether can form. Mixing alcohols with concentrated H2SO4 is a way to dry the alcohols (which is good) and also a way to make di-alcohol ethers, not good: dimethyl ether is a gas, colorless and highly explosive.

Base-stage questions

The second-stage product should be quite murky. This is no problem, as it will wash out.

After the processed oil/fat has turned straw-yellow (step 16), you've let it settle for an hour and drained the glycerine, you should have a total of about 120 ml of glycerine per litre of oil/fat used. If it's less than about 100 ml/litre oil, something's wrong, even if the color is right -- the process hasn't gone far enough.

This will almost certainly be due to carbonated lye. Lye has a really limited shelf life: CO2 from the air neutralizes it and forms sodium carbonate. Carbonated lye is much whiter than pure lye, which is almost translucent. The carbonate in the lye won't harm the reaction, but you'll have to use more lye.

The solution: Repeat the procedure from step 13. Prepare a fresh batch of methoxide with 0.03 liters of methanol and 0.75 grams of

lye for each liter of oil/fat. Reheat the biodiesel to 55 deg C, add the fresh methoxide and mix as before. No need this time to remove glycerine during the processing as in step 15, and don't worry about the color. Mix for one hour, settle, drain off the extra glycerine, and proceed from step 18.

If you plan to continue using the carbonated lye, make sure to increase the amount by 25% next time you make biodiesel. Store lye at room temperature, in dry conditions if possible, with the container lid really tightly closed.

Methanol recovery (optional)

To keep costs down, even amateur biodiesel producers try to salvage the unreacted methanol. There are two major methods to do this: heat extraction and vacuum/heat extraction.

Heat extraction

Heat the second-stage product to 70 deg C in a sealed boiler/vessel and lead the fumes into a condenser. Intercept the condensed methanol in a liquid trap. Take great care because methanol is highly flammable and the fumes are explosive.

Vacuum/heat extraction

This is basically the same as heat extraction, but it requires less energy. The drawback of this method is that you need a special vessel and equipment to do this. A good example is Dale Scroggins's reactor:
http://home.swbell.net/scrof/Biod_Proc.html

When building your reactor it may be a good idea to take one step at a time. Build the reactor, get confident with the process and eventually upgrade to methanol recovery.

At least a quarter of the methanol used can be recovered -- ie, 50+ ml per litre of oil/fat. Mix it with fresh methanol for preparing the next batch of methoxide.

Quality

Diesel engines require quality fuel. You just can't pour poor-quality biodiesel into the tank and expect the engine to go on and

on without problems. You have three very dangerous enemies: free glycerine, poorly converted oils/fats and lye. Free glycerine and mono-, di- and triglycerids (poor ester conversion) will form gum-like deposits around injector tips and valve heads, lye can damage the injector pump. The key to good fuel is to just do it right and finish it! Use pure chemicals (sulfuric acid, sodium lye and methanol) and measure them accurately, and follow the instructions carefully -- this will take care of poor conversion. Proper washing will get rid of the glycerine and neutralize any remaining lye.

There are also kits available for various quality tests. I was told in a letter from one of the visitors to our site of a test used by the motor industry for determination of glycol in motor oil. This should work for free glycerine determination.

"For glycerine analysis I suggest that you get a test kit for determining ethylene glycol in motor oil. This test is simple and it generates a purple colour if substantial free glycerol is present. Just analyze the biodiesel as if it were motor oil. Used-car dealers use the test to determine if there are leaks in the cooling system. Glycol and glycerol give the same result in the test." (With thanks to Martin Reaney)

Paper chromatography and thin layer chromatography will tell you the conversion rate, and titration may tell you about any remaining lye.

(See also **Biodiesel and your vehicle**.)

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