Titration kit for biodiesel production

Filtertechnik

Filtration, Purification &
Separation Solutions

Biodiesel titration kit

Using this kit will enable you to accurately determine the amount of Free Fatty Acid (FFA) in your vegetable oil. Knowing this will allow you to accurately determine the amount of catalyst required for a complete reaction.

The following items are included in your kit:
1 x 2.5L plastic bottle of 2-Propanol
1 x 1L bottle of 0.1M sodium hydroxide standard
1 x 100ml bottle of phenolphthalein indicator solution
1 x pack of 5, 10mL glass bulb pipettes
1 x 15ml self filling automatic burette with base
1 x 250ml plastic measuring cylinder
1 x 250ml glass conical flask
1 x green pipette pump

Please Note: Place the green pipette pump onto the pipette before starting out.

Safety

In the main the chemicals supplied in this kit are safe for general use provided a number of simple precautions are taken. 2-propanol is a flammable liquid and as such should be keep in a cool location away from any naked sources of heat. The other products should also be stored in a cool location to prolong their shelf life. For the 0.1M sodium hydroxide standard it is important to keep the lid tightly sealed to make sure no impurities are present. As standard when handling with all chemicals please make sure that you avoid an exposure to the eyes.

For reference the relevant hazard safety sheets are included with each kit.
Please contact with your reseller in the event of any questions.

Procedure

The acidity amount or % of Free Fatty Acids (FFA) is required to determine how much catalyst to add to the oil in the manufacturing process. The % FFA is determined by accurately titrating the oil with a Sodium Hydroxide (NaOH) solution of known concentration. An indicating solution is added to the oil which changes colour to pink above pH 8.7, that is known as the "end point" of the reaction. The amount of solution added to the oil is then used to calculate the amount of catalyst to add per litre of oil.

1) Measure 10ml of oil into the conical flask using a 10ml pipette and the pipette pump. Draw the oil up to the line at the top of the pipette, this is exactly 10ml
2) Add 100 ml of 2-propanol solution and swirl the flask to mix the contents
3) Pour the 0.1M standard NaOH into the bottle at the bottom of the burette. Squeeze this bottle to fill the burette, it will automatically zero itself
4) Add 2 drops of the phenolphthalein indicator solution to oil/2-propanol mixture and swirl to mix. The solution won't change colour.
5) Place the flask under the burette and start dispensing the NaOH into the flask, using the button. As the NaOH is added you will see the solution changing to a pink colour. Keep swirling the contents and the pink colour will eventually fade back to a light green colour. .
6) As you get closer and closer to the end point the pink colour will remain for a longer period of time. At that time flick the toggle up over the button and use the screw to finely add the NaOH drop by drop. The end point is reached at the precise time the solution remains a pink colour.
7) Note the volume of NaOH added to the nearest 0.1ml. If you have to add more than 15ml of NaOH, squeeze the bottle to refill it and continue.

Calculation

The calculation is very simple. The amount of NaOH you have to add to the solution is equal to the amount of "extra" catalyst that you need to add per litre of used oil. The only difference occurs if you use Sodium or Potassium Hydroxide as your catalyst, for example:

Sodium Hydroxide Catalyst
ml titrant added + 5 = x grams of NaOH to add per litre of oil
e.g. 8.8ml + 5 = 13.8g per litre. So for a batch of 48L the amount sodium hydroxide required is 662.4g
Potassium Hydroxide Catalyst
ml of titrant added + 7 = x grams of KOH to add per litre of oil
e.g. 8.8ml + 7 = 15.8g per litre. So for a batch of 48L the amount of potassium required is 758.4g

Biodiesel titration kit

What do I need to titrate the oil?

Titration is a test for the free fatty acids (FFA's) in used cooking oils.

Each titration will take about 30 seconds (once you have made your initial bottle of Reference Tester Solution (RTS)) Titration is done by reacting a small sample of FFA (free fatty acid) which happens to be 1ml of used cooking oil, with a measured amount of catalyst (lye or KOH), using the pH to tell us when the FFA is all used up.

The biodiesel reaction needs alkaline lye (NaOH) or KOH, as a catalyst. This is because methanol and vegetable oil wont react to make biodiesel on their own.

A titration test is therefore required to calculate how much FFA is present in the oil and to determine how much to compensate for it by adding more lye. The titration performs the lye/free fatty acid reaction on a very small scale, and we use pH to measure the colour change point of phenolpthalien indicator.

How to use this information?

The titration test will give us a number (technically referred to as acid value.

Every 1ml titration result will tell us to add an extra 1 gram of catalyst (lye or KOH for each litre of oil/ffa you are using to make biodiesel.

What equipment do you need?

- Isopropyl alcohol: 99% proof
- Phenolphthalein indicator
- 3 x 10 ml syringes
- An accurate burette
- I litre of distilled water DW (de-ionized water).
- An empty , clean bottle to store RTS (Reference test solution)
- 3 x 50 ml glass beaker
- Sodium Hydroxide (NaOH) or Potassium Hydroxide KOH
- Accurate scales

Biodiesel titration kit

Step 1.

Make your Reference Tester Solution
A base reference solution is essential for accurately titrating oil.

Materials required:
a) Sodium Hydroxide (NaOH) OR Potassium Hydroxide KOH and
b) I litre of distilled water DW (de-ionized water).

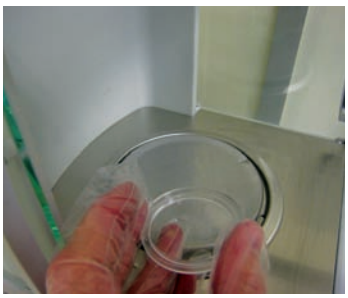
a. NaOH crystals

b. De-ionised water

Firstly, we need to prepare our Reference Tester Solution. This will be a 0.1% NaOH in water solution. 1 gram of catalyst (lye or KOH) dissolved in 1 litre of distilled water. Try and be as accurate as possible when measuring the 1 gram of lye. You can improve the accuracy of adding 3 grams of lye to 3 litres of distilled water. If you work with KOH as a catalyst in your biodiesel production process, use that KOH for your solution. If you use NaOH as your catalyst in your reaction process, then use NaOH as your test solution.

Every 1ml of this solution will now contain 1/1000 of a gram of lye. This is too small an amount to weigh, hence the use of water.

THIS BASE WILL NOW BE REFERRED TO AS REFERENCE TESTER SOLUTION (RTS)



Place a beaker onto accurate scales

0 the scales

Add 1 gram of catalyst

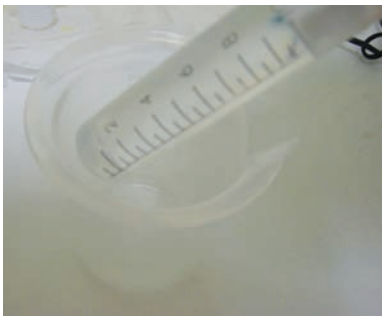
Measure 1000 ml (1 litre) of de-ionised water. Mix in the crystals thoroughly.

Once you have made this solution you can use it for many tests.
KEEP IT AIRTIGHT AND DO NOT CONTAMINATE THE SOLUTION!

Place your RTS into a accurate dispenser

Step 2.

Titrating oil – Pour 10 ml of isopropyl alcohol into the 50 ml glass beaker. This doesn't have to be exact.

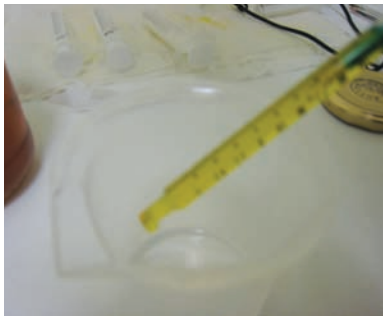


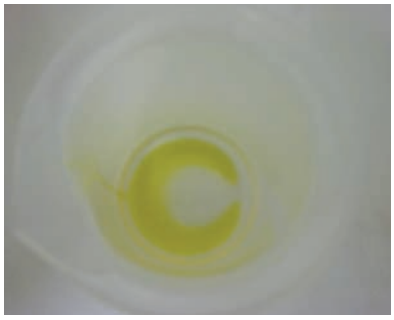
Measure a 10 ml alcohol sample

Step 3.

Measuring an oil sample.

Using a syringe measure exactly 1 ml of your oil and put into the 50 ml beaker. If you use syringes you MUST clean or use separate ones for each different chemical. It is more accurate to fill the syringe with 2 or 3 ml and then dispense 1 ml into the beaker.





Sample 1 ml of oil

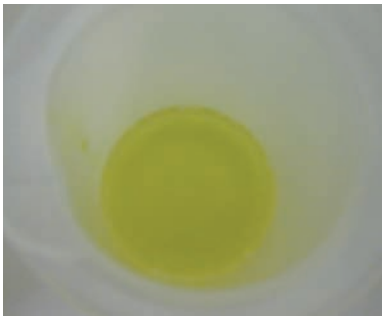
Add this 1ml of oil into a beaker

Step 4.

Introduce 4-5 drops of phenolphaleine indicator with the eye-dropper
Swirl the beaker to dissolve everything. A yellow, murky colour will form.

TIPS:

- If you are using solid oils or in very cold weather, warm the oil before dissolving.
- When producing a batch of WVO biodiesel, mix the oil in your processor properly and then take an oil titration sample to "balance" out the titration results.
- Phenolphthalein has a shelf life of about a year, it is very sensitive to degradation by light so after a while it will start giving erroneous readings.



Step 5.

- Add and measure the RTS (Reference test solution)
- Add your RTS, a small amount at a time. Add in tiny increments and keep a note of how much you are adding. WATCH FOR A COLOUR CHANGE.
- Keep swirling the contents until it turns the fluid in the beaker to lavender or pink.
- This pink colour should last 30 seconds or longer.
- Don't mix up your oil and RTS syringes. Clean them with alcohol if you make a mistake.

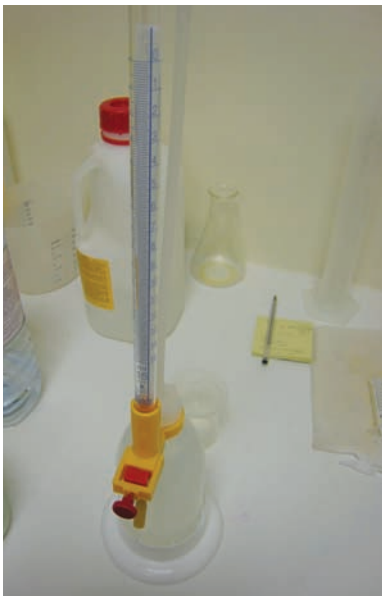
You MUST note of how many millilitres (ml) you add. Gently agitate the beaker whilst doing this. You are watching for a colour change from murky yellow to pink/purple.

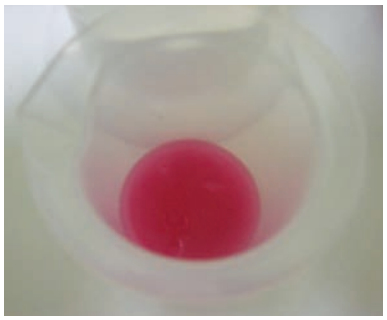
Once the mix reaches this point record how many millilitres of KOH solution you added to the oil/phenolphthalein mix WRITE IT DOWN! This figure is IMPORTANT as it is the titration level of the oil.

Most oil that is of a reasonable standard should titrate between 2 and 5, which means you would have added 2 and 5 ml of solution respectively.

REPEAT THIS PROCESS 3-5 TIMES with clean and dry the equipment

YOU DO NOT NEED TO REMAKE THE REFERENCE SOLUTION AS MENTIONED IN STEP 1.





Step 6:

Calculate your catalyst levels for the process
How much RTS (Reference Test Solution) did you use? This is the point that all the FFA's were neutralized with RTS.

Each ml of RTS relates to an extra gram of catalyst (NaOH or KOH) you will need to add per litre of oil in your production batch. If you oil uses more than 4ml of lye RTS or 5ml of KOH RTS, do not use it until you get more experienced.

Average restaurant oil will require ± 3ml on a titration. Very bad WVO may require 10, which will be impossible to convert into biodiesel using this approach.

Please note that titration should be done with the same catalyst you are going to us. See notes on RTS.

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Working with batches of larger than 1 litre:
Measure the capacity in litres of your biodiesel processor.

To find out how much catalyst to use you will multiply the following:
For NaOH (Use a base of 3.5)

The base number of 3.5 NaOH is required if you are using new oil. When using UCO we need to then add on your titration level results in ml and multiply it by the number of litres being processed.

Number of litres of oil x (3.5 Grams + titration results)

Example: If titration using your RTS showed 2ml, this means that you need 2 extra grams for each litre of oil in the batch.
So if we have a 100 litre batch: 100 litres x (3.5+2)
ie 100 litres x 5.5 grams per liter = 550 grams NaOH to make biodiesel with this oil.

For KOH (Use a base of 5.45)
The base number of 5.45 KOH is required if you are using new oil. When using UCO we need to then add on your titration level results in ml and multiply it by the number of litres being processed.

When using KOH do the same thing. The titration results with KOH RTS will be slightly different than you will get with NaOH RTS. They are however used in the same way ie 2 ml of the titration RTS = 2 grams of catalyst per litre to be added to the processor. Using KOH means you will need a slightly different 'base" quantity for the reaction itself ie 5.45 grams instead of 3.5 grams per litre

Example: if titration using your RTS showed 2ml, this means that you need 2 extra grams for each litre of oil in the batch.
So if we have a 100 litre batch: 100 litres x (5.45+2)
ie 100 litres x 7.45 grams per liter = 745 grams KOH to make biodiesel with this oil.

To confuse matters further, KOH comes is a variety of purities. If you are a beginner, it is advisable to use 99% pure KOH. Later on you can adjust your KOH levels to compensate for impurities (ie. If you have got 90% pure KOH you will need to use 10% more of it)

NOTE:
Catalyst quantity:
It is important to get this catalyst quantity correct. The more catalysts you use , the better the quality of the biodiesel….up to a point! Too much catalyst will cause more soap to be produced.

When too much lye is used the result can be a troublesome gel that is tough to do anything with. When not enough lye is used the reaction does not go far enough so some unreacted WVO will be mixed with the biodiesel and glycerine.

This will form three levels with biodiesel on top above unreacted WVO with glycerine on the bottom. If there is too much water in the WVO it will form soaps and settle right above the glycerine forming a fourth level in the container. This layer is not too easy to separate from the unreacted WVO and glycerine layers.

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Preparing the sodium methoxide

Generally the amount of methanol needed is 20% of the WVO by mass. The densities of these two liquids are fairly close so measuring 20% of methanol by volume should be about right. To be completely sure, measure out a half-liter of both fluids, weigh, and calculate exactly what 20% by mass is.

Different WVOs can have different densities depending on what type of oil it originally was and how long it was used in the deep fryer. Example: When transesterifying 100 liters of WVO, use 20 liters of methanol. The methanol is mixed into a solution with the sodium hydroxide (lye), creating sodium methoxide in an exothermic reaction (ie it gets warm from bonds forming). Keep all utensils the lye comes in contact with as dry as possible.

CAUTION:

Treat sodium methoxide with extreme caution! Do not inhale any vapors! If any sodium methoxide gets splashed on your skin, it will burn you without your feeling it (killing the nerves) - wash immediately with lots of water. Always have a hose running when working with sodium methoxide.

Sodium methoxide is also very corrosive to paints. Lye reacts with aluminum, tin and zinc. Use glass, enamel or stainless steel containers -- stainless steel is best.

Trouble Shooting:

Performing a blank titration:
Alcohol sometimes becomes acidic with age, which may cause inaccurate results. So it is advisable to test the alcohol by performing a blank titration from time to time. A blank titration is just like a regular titration but without the oil. A blank titration neutralizes any acids that the alcohol contained, thereby ensuring that the “starting point” is correct.

Blank titration procedure

- Add 10ml of isopropyl into a 50ml container
- Add a few drops of phenolthaleine indicator.
- Swirl the mixture: It will for a yellow colour
- Add lye/water RTS drop by drop and keep swirling

If the isopropyl only needs a very small amount of lye/water solution RTS to neutralize the acid, this would suggest that it is not very acid and therefore suitable for use. If you require a half a ml or more of RTS, you may need to perform a blank titration every time you use this acidic alcohol ie. It provides a more neutral starting point for the real reaction).

- The moment the mixture turns pink/purple STOP. You have now neutralized all the acids in the alcohol. This is your starting point. You will now add oil to the mixture for actual “titration” step.

You may not always need to perform this blank titration, but we need to know that the alcohol is not a variable in our titration results.

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