

Feedstock Quality Issues for Biodiesel Production

Biodiesel Series

Innovative and practical information on biodiesel for the homeowner, farmer and small business owner.

Quality of the raw oil that serves as the feedstock for biodiesel production plays a crucial role in determining the quality and quantity of the end product.

Impurities in the oil significantly affect the conversion process. Under similar environmental conditions, you can expect 67 percent to 84 percent conversion into biodiesel esters when using crude vegetable oils compared to 94 percent to 97 percent using refined oils. The lower conversion rate for crude oil is because impurities like free fatty acids in the oil interfere with the catalyst and favor soap formation.

Concentrations of these free fatty acids depend on the source and original use of the oil. Virgin oil contains approximately 0.3 percent free fatty acids, but used vegetable oil has high concentrations of free fatty acids due to high heating during the frying process causing hydrolysis of oil (triglycerides).

Similarly, different long chain fatty acid components in the oil have different physical properties. For instance, rapeseed oil contains higher levels of unsaturated fatty acids, which keeps biodiesel a liquid at ambient temperatures. Tallow and hard fats, on the other hand, have higher levels of saturated fats that creates a product that would solidify below 68 degrees Fahrenheit, which would affect the cold flow properties of biodiesel product.

Biodiesel feedstock should be screened for:

Free Fatty Acids

Free fatty acids are formed when vegetable oil is subjected to high temperature during frying. One molecule of oil can yield three molecules of free fatty acids.

Free fatty acids are not favored by transesterification reaction. As the name indicates, a free fatty acid is an acid and will react with a base forming salt (soap is a salt of fatty acids – the saponification process). The levels of free fatty acids in feedstock play a critical part in the biodiesel reaction and if too high or improperly handled could lead to waste of part or all of a batch.

The ranges of free fatty acids commonly found in biodiesel feedstock are: refined vegetable oils, less than 0.05 percent; crude vegetable oil, 0.3-0.7 percent; restaurant waste grease, 2-7 percent; animal fat, 5-30 percent; and trap grease, 40-100 percent.

The transesterification reaction can easily assimilate free fatty acid levels less than 1 percent, and levels less than 0.5 percent can be ignored. For free fatty acid levels above 1 percent, however, extra alkali catalyst is added to neutralize the free fatty acids by forming soap while still leaving enough to act as the reaction catalyst. The additional amount is based on grams of catalyst required to neutralize free fatty

acids in 1 liter of feedstock. The free fatty acid level is determined by an acid number/titration test before making biodiesel so you can compensate for the catalyst that will eventually be used to neutralize the free fatty acids. For extremely high free fatty acid levels, acid esterification is the most favored reaction to esterify the free fatty acids into biodiesel.

Water

Water is one of the three important reactants for making soap – with those being water, base and vegetable oil. In that process, water would break apart the base molecule, producing free potassium ions, which could then combine with free fatty acids to produce soap. If the potassium ions are bound up in potassium methoxide, however, when the vegetable oil is broken apart, the methyl group in alcohol will preferentially bond with the fatty acid, which will result in biodiesel rather than soap.

The presence of water in the feedstock has greater negative effects than free fatty acids. Water content kept below 0.06 percent ensures the highest yield from the process. Particular attention is therefore required for the most efficient uses of waste vegetable oils and crude oils since they generally contain water and free fatty acids.

The amount of moisture in the feedstock is determined by centrifugation or by the weighing and heat

test. The weighing and heat test is simply heating the oil sample to evaporate the moisture. The easy way to detect the presence of water is the bubbling. That's fairly hard to do in big containers, but using a small sample in a glass beaker/container helps you make an easy determination.

An easy way to remove the water from the feedstock is to employ the forces of Mother Nature by using gravity settling. Letting the oil settled in a tank, preferably a cone-bottom tank, will help to separate out at least 85 percent of the water and solids in two days. Solids and moisture settle to the bottom and leave the clean oil toward the top (Figure 2). The drop from any level of moisture down to 0.08 percent in three days (about 94 percent removal of moisture) has been observed at the LSU AgCenter's W.A. Callegari Center.

To avoid soap formation, it is necessary to dry the oil as much as possible. The process can easily assimilate 1 percent water. The drying process can be accomplished by heating the oil to about 150 degrees Fahrenheit for six hours before the reaction. If using methanol, the temperature of the feedstock has to be less than 147 degrees to avoid boiling of methanol from the reaction mixture.

The other major problem associated with water contamination is that it contributes to microbial growth.

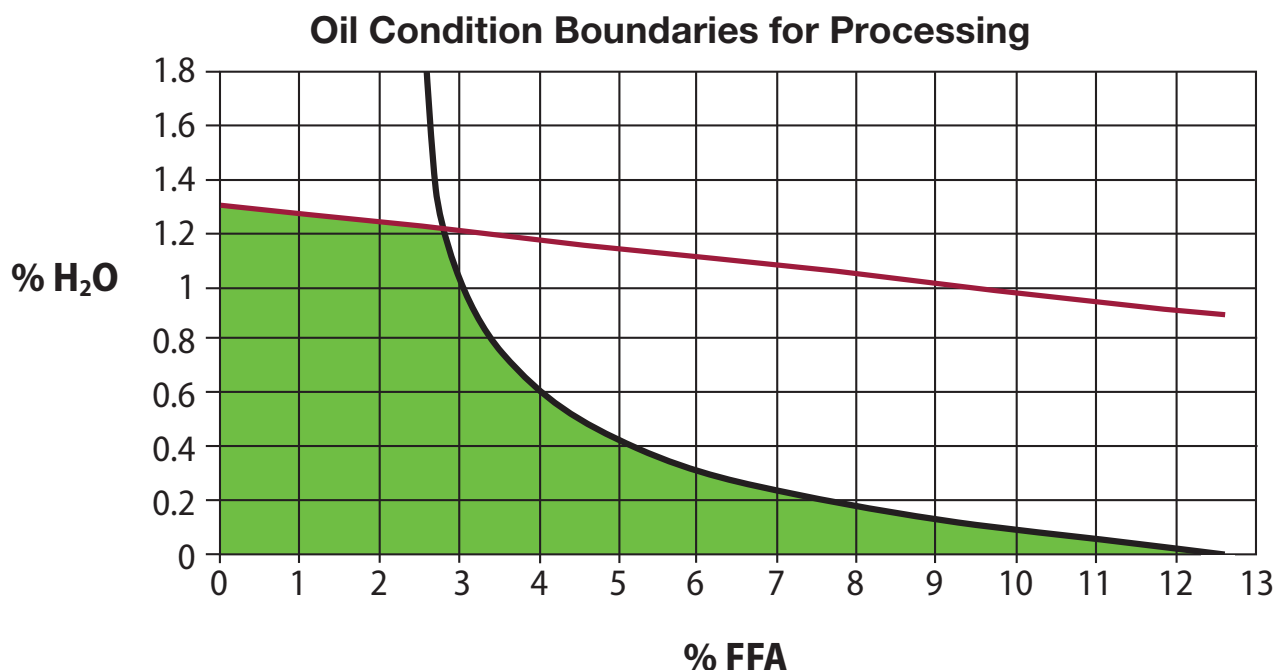


Figure 1. Free fatty acid content versus water content in the feedstock favored for biodiesel production (Source: Springboard Biodiesel)

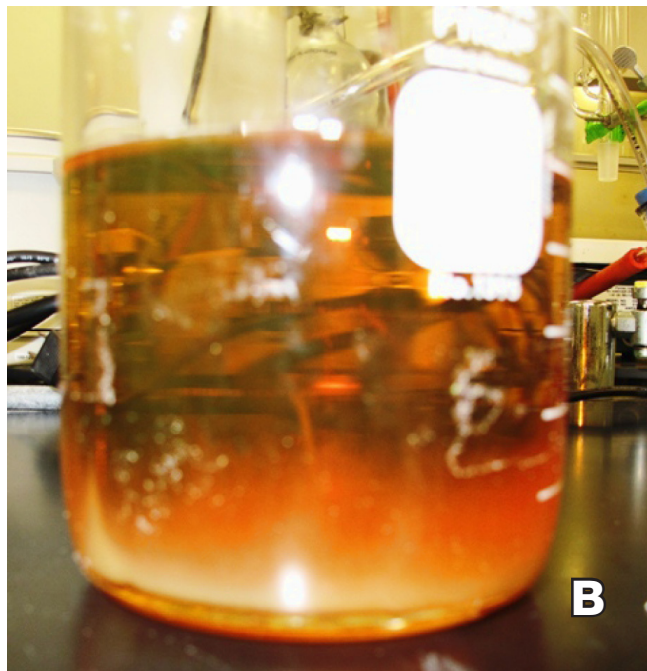
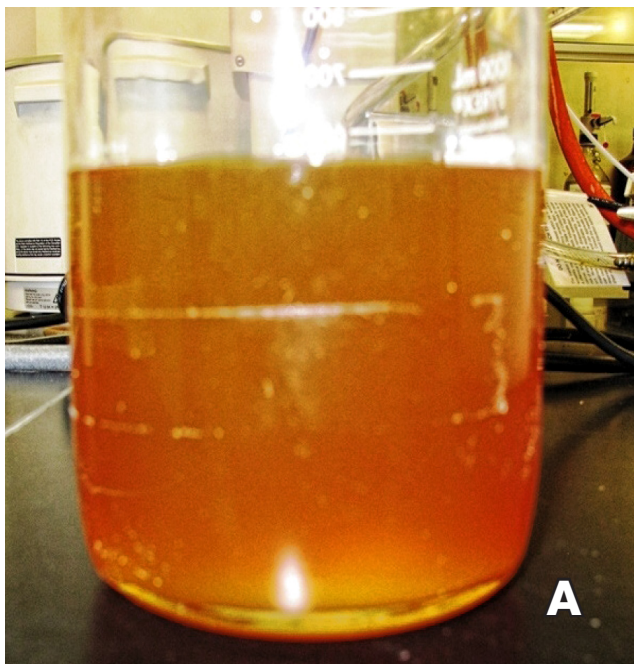


Figure 2. Gravity settling takes care of moisture and solids in waste vegetable oil collected from restaurants – A shows before and B after 48 hours of settling.

There are species of yeast and bacteria that will grow at the bottom of a storage tank. The organisms produce sludge and slime and the water can turn acidic. Some of the organisms can convert the sulfur to sulfuric acid, which can corrode metal, if any, in the dispensing line. Frequently draining the water from storage tanks, ensuring that vents and seals do not allow rainwater to enter and not drawing from the bottom of the tank should prevent large amounts of free water from entering the system.

Solids

Solids in waste vegetable oil are considered contaminants because of their indirect effect on biodiesel process – particularly their tendency to hold moisture. There are different approaches used to remove solids and moisture from the feedstock. Filtering is the most common method, but an inexpensive and clean method is to use gravity settling.

With gravity settling, collected oil is transferred to a big tank where it is allowed to settle for two or three days. Solids and moisture sink to the bottom of the tank while the supernatant oil is ready to use for biodiesel production. The longer the settling time, the cleaner the oil, but the oil needs to be monitored for microbial growth.



Figure 3. Sample collected from the bottom of a settling tank separates over time, depending on the ambient temperature.

Almost 100 percent of the solids settle to the bottom in two days, ensuring a good biodiesel product that meets ASTM specifications. Settling time for solids and water depends on the temperature of the oil and quality of the oil collected. For example, oil with higher mono- and diglycerides would take longer to settle because of their higher affinity toward moisture compared to pure oil containing only triglycerides.

During the frying process, animal fats are dissolved in the oil. The cloud point of these fats is around 54 degrees Fahrenheit, and they tend to form wax at lower temperatures. Centrifuging a sample collected from the bottom of a settling tank indicated 65 percent oil and 35 percent solids (Figure 3). That shows the oil was still in the process of separation. Heating the solid part to 95 degrees F yielded 60 percent oil, which, in turn, showed at

least 85 percent of the settled solids could easily be converted into biodiesel at higher ambient temperature, if the moisture is boiled off first.

Gravity settling also takes care of types of oil that tend to gel at lower temperatures even before processing into biodiesel. Biodiesel made from such oil also gels at lower temperatures.

Summary

For efficient transesterification reaction to produce biodiesel, it is important to screen the feedstock for quality issues. Good quality feedstock means better quality biodiesel. Better quality biodiesel can meet ASTM specifications and hence ensure the best performance of your vehicle's engine. Although there are many quality parameters, free fatty acids, water content and solids in the feedstock are the most important factors for your biodiesel brewing.



Authors

Javed Iqbal, Lab Manager
W. A. Callegari Environmental Center

Shelly Martin, Lab Associate
W. A. Callegari Environmental Center

William A. Carney Jr.
Associate Professor and
Head W. A. Callegari Environmental Center



Visit our website

www.LSUAgCenter.com

Louisiana State University Agricultural Center
William B. Richardson, Chancellor

Louisiana Agricultural Experiment Station
John S. Russin, Interim Vice Chancellor and Director

Louisiana Cooperative Extension Service
Paul D. Coreil, Vice Chancellor and Director

Pub. 3166B (online only) 3/2011

The LSU AgCenter is a statewide campus of the LSU System and provides equal opportunities in programs and employment.