

# Biodiesel Tech

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## OILSEED PREPARATION FOR FOOD AND BIODIESEL PRODUCTION

Summarized from *Biodiesel Production Technology*, NREL/SR-510-36244, J. Van Gerpen, B.Shanks, R. Pruszek, D. Clements, and G. Knothe, 2004

Most oils from oilseed crops are used as food products, which require considerable processing before they are considered edible. Fully refined oils are also known as RBD oils where RBD stands for refined, bleached, and deodorized. High quality biodiesel can be produced from crude oilseed oil, but the use of partially or fully refined oil simplifies the biodiesel production process.

Oil is obtained from oilseeds by two techniques: mechanical crushing and solvent extraction. Mechanical crushing is preferred for smaller plants because it requires a smaller investment and provides flexibility. Typically, it is used by plants that process less than 100,000 kg of seed/day. If plants need to process more than 300,000 kg/day, solvent extraction is more commonly used.

Before the oil can be extracted, the seeds must be cleaned to remove foreign materials. This involves screening to remove stems, leaves, stones, sand, dirt, and weed seeds. Magnets can remove iron particles.

After cleaning, the seeds are often dehulled. The hulls are abrasive and contain little oil. Removing the hulls reduces the wear on the screw press and also increases the extraction yield of oil because the hulls absorb oil. For oilseeds with thick shells, dehulling typically involves cracking the shells and then separating the shells by screening or aspiration. With some oilseeds, dehulling generally includes removal of residual fine particles.

Oilseeds that are to be processed by solvent extraction are usually flaked to increase the exposure of the oil to

the solvent. Hexane in a percolation extractor is the most common technology used today. This type of extractor drips the solvent down on the flaked oilseeds so it can dissolve the oil in a manner similar to a coffee percolator. The oil-laden solvent, or miscella, is then filtered and removed from the extractor. The miscella is then heated to vaporize the solvent. This gets the miscella to about 5% solvent. The remaining solvent is stripped by injecting steam into the oil. The steam and hexane vapors are condensed and, since the two fluids are insoluble, they can be separated in a settling tank.

The alternative to solvent extraction is mechanical extraction or crushing. The oilseeds are generally preheated to destroy enzymes that cause problems with using the meal as animal feed. One popular way of cooking the oilseed is to use an expanding extruder. This device compresses the oilseed with high pressure so that the temperature rises to 150- 160°C, which is high enough to deactivate the enzymes. The extrusion also frees the oil and produces a frothy liquid that quickly solidifies as the water flashes to vapor and reduces the moisture level. After cooking or extrusion, the oilseed is usually fed to a screw press where it is crushed. Drainage slots are provided for the oil to leave the press. Moisture control is important. If the moisture level is too high, then the oilseed will be soft and the oil will contain excessive solids. If the moisture level is too low, then the oilseed will overheat. If the oilseed has not been extruded, then pre-pressing may be used to break the cell walls and release the oil from the oilseed.

After extrusion from the oilseed, the crude oil contains impurities that are either insoluble or soluble with the oil. The oil-insoluble impurities consist of seed fragments and meal fines, free water, and long-chain hydrocarbons or waxes that cause cloudiness when the oil is refrigerated. Most of this material can be removed by filtration. The oil-soluble materials include phosphatides, free fatty acids (FFA) and other compounds. Some of these compounds can cause the oil to have an unpleasant taste and smell. Other compounds are actually desirable and can remain in the oil. For example, the food-grade oil typically contains 0.5-1.5% non-oil compounds that are known collectively as unsaponifiable matter.

Processing of crude seed oil further to food-grade oil generally consists of three steps: refining (degumming and caustic refining), bleaching, and deodorization.

### Refining

The refining step is designed to remove the phospholipids and FFA from the crude oil. The main reason to remove phospholipids is that some of these compounds are strong emulsifiers. If these compounds are still present during the



later alkali neutralization step, they will inhibit the separation of the soaps and lower the yield of neutral oil. Phospholipids will also react with water to form insoluble sediments that are not desirable when cooking with the oil.

The first step of oil refining is degumming. In this step, crude oil is mixed with 1-3% water and the mixture is agitated mechanically for 30-60 minutes at 70°C (158°F). This hydrates phospholipids and gums into compounds that are insoluble in the oil, which can be separated from oil by settling, filtering, and/or centrifuging. The compounds of water degumming are byproducts that have value as a feedstock for lecithin production.

A portion of the phospholipids are not hydratable by contact with water alone. The addition of citric or phosphoric acid will hydrate those remaining. Citric acid is preferred if the byproduct is to be used for lecithin. The phosphoric acid hydration is accomplished by adding 0.05-0.2 wt% of concentrated (85%) phosphoric acid to the crude oil at 60-85°C. The residence time varies from a few seconds to 1-2 minutes depending on the type and quality of the oil. The extent of gum removal by these techniques is related to the strength of the acid treatment.

The second step of oil refining is neutralization, or caustic refining which removes the FFA present in the crude oil. An alkali solution, usually sodium hydroxide (NaOH), is added to react with the FFA to produce soap. The soap is insoluble in the oil and easily separated by water washing. The alkali solution also neutralizes any acid remaining from the degumming step.

The alkali will also react with the triglycerides of the oil, so the neutralization parameters (type of alkali, solution strength, temperature, agitation, and time) must be optimized to minimize the yield loss, or additional losses can be resulted from emulsification and suspension of oil droplets in the soap solution. A byproduct of caustic refining is a mixture of soap, water and oil known as soapstock. This has been considered as a feedstock for biodiesel production, but its high water content and the difficulty of converting the soap to methyl esters are significant obstacles to cost-effective utilization.

### **Bleaching**

The primary purpose of bleaching is to remove the color pigments from the oil. It also helps to remove remaining soap, trace metals, phospholipids, and sulfur compounds. Hydroperoxides, the initial products of oxidation, are broken down during the bleaching process and some of the final oxidation products are removed.

Bleaching involves mixing bleaching clays, consisting of naturally occurring bentonite and montmorillonite clays, with the oil under agitation for 10-30 minutes. The oil is usually heated to 90-120°C and the process occurs under a slight vacuum to exclude oxygen. A consequence of the vacuum and high temperature is that residual water from the neutralization and water washing is removed. This is

not always desirable because the effectiveness of the clays is usually enhanced by the presence of a small amount of water. The clays are usually activated before use with a mineral acid, such as sulfuric acid, to remove some of the minerals from the clay and produce a larger volume of micropores and smaller clay particles.

During bleaching, the clays are mixed with the oil, either directly or by premixing with a small amount of oil to produce a slurry and then adding the slurry to the oil. After the adsorption has reached equilibrium, the clay is filtered out.

### **Deodorization**

Deodorization is the final processing step before the oil is ready for use in food products. The deodorization removes the trace components that give the oil an unpleasant taste and odor. Deodorization is essentially a distillation process that occurs at high temperature (200-260°C) and low pressure (2.5-9.2 mbar). The odor causing compounds are more volatile than the oil and will be removed by distillation.

The first step of deodorization is deaeration, in which the dissolved oxygen is removed so that the oil does not oxidize at the high temperatures used in deodorization. In batch deodorizers, the deaeration occurs naturally while the oil is heated from the typical inlet temperature of 40-80°C to the deodorization temperature range of 200-260°C. If adequate vacuum is available, the oil should be deaerated by the time the oil reaches 100-120°C. Preheating is sometimes identified as the second step of deodorization. In continuous-flow processes, a portion of the heat required for heating the oil comes from cooling the deodorized oil, and as much heat as possible is transferred from the final product to incoming oil. After preheating, the oil is heated to its final temperature by steam or another heating fluid such as Dowtherm A.

After the oil has been held at the set temperature for sufficient time (2-5 hours in a batch deodorizer), it is cooled to 120°C where it is common practice to add 50-100 mg/kg of citric acid to chelate trace metals in the oil so that they can be removed by filtration. Chelation is a chemical process where a compound, known as a chelating agent, binds to a metal compound and makes it unavailable for further reaction. Then the oil is cooled further to its storage or bottling temperature. The final stage of bleaching, known as polish filtration which removes the clay and any other particles, occurs immediately before the oil leaves the processing plant.

Biodiesel can be produced from oil at any stage in the various processes. The compounds removed during refining, bleaching, and deodorization mentioned above are mostly removed by water washing or collect in the glycerol phase in biodiesel production. Biodiesel produced from crude oil retains somewhat more color than biodiesel from refined oil but phosphorus and FFA levels seem to be equivalent.

