

2. Listing of feed materials

The main raw materials processed by the EU Biodiesel industry are rapeseed oil, soybean oil, sunflower seed oil and palm oil in combination with methanol.

The Catalogue of the European Union for Feed Materials provides a common system in the EU for the description and labeling of feed materials. The Catalogue includes, for each feed material listed, the name of the product, an identification number, a description of the feed material including - if appropriate - information on the manufacturing process, and the particulars replacing the compulsory declaration for the purpose of article 16 (1) (b) of Regulation (EC) No 767/2009.

Hereby listed the biodiesel related feed materials within Reg. 68/2013 (adapted for vegetable origin):

Glycerine, crude	13.8.1	<p>By-product obtained from:</p> <ul style="list-style-type: none"> – the oleochemical process of oil/fat splitting to obtain fatty acids and sweet water, followed by concentration of the sweet water to get crude glycerol or by transesterification (may contain up to 0,5 % methanol) of natural oils/fats to obtain fatty acid methyl esters and sweet water, followed by concentration of the sweet water to get crude glycerol; – the production of biodiesel (methyl or ethyl esters of fatty acids) by transesterification of oils and fats of unspecified vegetable origin. Mineral and organic salts might remain in the glycerine (up to 7,5 %). <p>May contain up to 0,5 % Methanol and up to 4 % of Matter Organic Non Glycerol (MONG) comprising of Fatty Acid Methyl Esters, Fatty Acid Ethyl Esters, Free Fatty Acids and Glycerides;</p> <ul style="list-style-type: none"> – saponifications of oils/fats of vegetable origin, normally with alkali/alkaline earths, to obtain soaps. <p>May contain up to 50 ppm Nickel from hydrogenation.</p>
Glycerine	13.8.2	<p>Product obtained from:</p> <ul style="list-style-type: none"> – the oleochemical process of (a) oil/fat splitting followed by concentration of sweet waters and refining by distillation (see part B, glossary of processes, entry 20) or ion-exchange process; (b) transesterification of natural oils/fats to obtain fatty acid methyl esters and crude sweet water, followed by concentration of the sweet water to get crude glycerol and refining by distillation or ion-exchange process; – the production of biodiesel (methyl or ethyl esters of fatty acids) by transesterification of oils and fats of unspecified vegetable origin with subsequent refining of the glycerine. Minimum Glycerol content: 99 % of dry matter; – saponifications of oils/fats of vegetable origin, normally with alkali/alkaline earths, to obtain soaps, followed by refining of crude Glycerol and distillation. <p>May contain up to 50 ppm Nickel from hydrogenation.</p>

2.1 Processing aids possibly used during treatment and processing

Water

Aluminium Sulphate

Citric acid

Ferric Chloride

Hydrochloric acid

Potassium hydroxide

Sodium Hydroxide

Sodium Methoxide

Sulphuric acid

Phosphoric acid

Toluene Sulphonic acid

This list is non-exhaustive

3. Process Description of Biodiesel

Biodiesel consists of Fatty Acid Methyl Esters and is produced by the chemical reaction of Oils and Fats with monoalcohols, typically methanol. A catalyst, usually sodium or potassium hydroxide and/or methylate, is utilized to accelerate the formation of alkyl esters. This production process is generally known as transesterification.



This section will describe the process of biodiesel production from vegetable oils where the triglyceride oil is broken into alkyl (biodiesel) and glycerine by reaction with a mono alcohol. The biodiesel and glycerine phases are then separated and purified. Production processes contain the same stages, irrespective of the production scale, although the differences in equipment may be significant.

3.1. Reception of Vegetable Oil

Vegetable oils delivered to biodiesel plants are partially refined vegetable oils. Pre-delivery (as per FEDIOL sector reference document)¹ vegetable oils undergo a number of refining steps to remove various impurities, such as phosphatides, free fatty acids, waxes, tocopherols or colorants, which could impede the biodiesel process reaction.

Most if not all feedstocks undergo some form of pre-treatment for impurities or free fatty acid (FFA) reduction prior to being utilised as a raw material in the manufacture of biodiesel.

On delivery, the partially refined vegetable oils are segregated to pre-processing storage tanks. The safety and quality of the incoming material is assessed.

The content of free fatty acids, water and non-saponifiable substances are key parameters in achieving high conversion efficiency in the transesterification reaction. The results of the oils parameters will influence the quantity of raw material, alcohol and catalyst ratio in the transesterification process.

3.2. Reaction Stage/ Transesterification:

The objective of the reaction stage is to convert the free fatty acid (FFA) fraction of the feedstock into biodiesel. When the FFA content is high the reaction stage is usually conducted in two steps: esterification and transesterification.

¹ Fediol Sector Reference Document: appendix 4 to the European Code to good practice for the manufacture of safe feed materials:
http://www.efisc.eu/data/1342020514Sector%20document%20oilseed%20and%20proteinmeal%20industry_version-2-2-1_16-02-11%20update%20hyperlinks%209_7.pdf
Sector reference document on the manufacturing of safe feed materials from biodiesel processing

Following the pre-processing analysis of the incoming vegetable oil, the alcohol and catalyst is mixed and sent to the reaction vessel where upon the vegetable oil is added (Figure 1 Flow Chart). This stage is known as esterification and is a pretreatment step to the transesterification which reduces the FFA content of the oil.

The complete transesterification process is closed to the atmosphere to prevent any loss of alcohol. Excess alcohol is normally used to ensure total conversion of the oil to its esters.

3.3. Separation Stage

Once the reaction is complete, two major products exist: glycerine and biodiesel. Each has a substantial amount of the excess methanol that was used in the reaction. The reacted mixture is sometimes neutralized at this step if needed. The glycerine phase is much more dense than biodiesel phase and the two can be gravity separated with glycerine simply drawn off the bottom of the settling vessel. In some cases, a centrifuge is used to separate the two materials faster.

3.4. Acidulation and FFA separation

Typically, the glycerine after the separator is usually 50% glycerine, 40% methanol and 10% soap and catalyst. The catalyst is neutralized and soaps are split to fatty acids and salts. Free fatty acids and methanol are removed and recovered.

3.5. Glycerin Neutralization

The glycerine by-product contains unused catalyst and soaps that are neutralized with an acid and sent to storage as crude glycerine. In some cases the salt formed during this phase is recovered for use as fertilizer. In most cases the salt is left in the glycerine. Water and alcohol are removed to produce approx. 80% pure glycerine that is ready to be sold as crude neutralised glycerine.

Refined Glycerine: Crude glycerine in some cases maybe further refined to pharmaceutical or technical grades or feed grades by removing water and salts by distillation.

3.6. Methyl Ester Wash

Once separated from the glycerine, the biodiesel is sometimes purified by washing gently with warm water to remove residual catalyst or soaps, dried, and sent to storage. In some processes this step is unnecessary. In some systems the biodiesel is distilled. This step is optional and increases biodiesel purity.

3.7. Storage

Glycerine is stored in suitable storage tanks.

3.8. Transport

Transportation is required for the processed co-product glycerine. The transportation of the co-product has to be in line with EU and National legislations, existing transportation codes, customer requirement and the requirements in this code 'Sector reference document on the manufacturing of safe feed materials from biodiesel processing'.

6. Biodiesel Production Process Flow Chart

This chart describes a very general biodiesel process

