

Evonik for Biodiesel

Biodiesel from sunflower oil



Laboratory Process

Starting materials

Compound		CAS	Hazard Symbols	Quantity [g]
Sunflower Oil, dewaxed SFO	SFO	[8001-21-6]	-	400.0
Methanol	MeOH	[67-56-1]	F, T	63.0
Sodium Methylate	NM30	[124-41-4] in [67-56-1]	F, T, C	8.0
Hydrochloric Acid (aqueous, 32 %)	HCl	[7647-01-0]	C	0.6
De-ionized Water	H ₂ O	[7732-18-5]	-	250.0



The quality of the oil was checked by determining the water and free fatty acid (ffa) content:

- Water: 0.02 %;
- Acid number (ffa): 0.05 mg KOH/g;
- Appearance: clear, light brown liquid.

Transesterification

A 1l glass reactor (oil-heated, double-walled jacket) is charged with 400 g (ca. 0.45 mol) sunflower oil, 45 g (1.41 mol) methanol, and 6.0 g (refers to 0.03 mol of catalyst) of a 30 % methanol solution of sodium methylate (NM 30).

The mixture is heated to 65 °C, once this temperature is set the mixture is stirred for 2 h. Hereafter the twophase mixture obtained is cooled to 40 °C with slow stirring. The stirrer is switched off and the phases are allowed to settle for 10 min. The lower viscous glycerol phase is removed. 39.7 g glycerol phase is obtained (viscous liquid, solidifies upon cooling without any further treatment). The remaining upper phase is combined with 18 g methanol and 2.0 g NM 30.

The mixture is again heated to 65 °C and stirred for 2 h. After this step the mixture is cooled to 45 °C and the second phase separation is carried out where 9.0 g of a glycerol phase is collected.



Work-up

The crude biodiesel is transferred to a separation funnel and intensively mixed with 50 g preheated (ca. 90 °C) water and 0.6 g concentrated (32 %) hydrochloric acid. The aqueous layer is removed (it should have a neutral pH). Hereafter the organic layer is washed with ca. 100 ml hot water (90 °C) twice.

Drying

The biodiesel is dried with a rotary evaporator for 90 min (90 °C, 20 mbar). A light-yellow clear liquid is obtained.

Stabilization

A few 100 ppm of an antioxidant is added.

Yield

396 g, 99 % (based on oil quantity).

Key Analyses

Parameter	unit	determined	spec
Water	% (weight)	0.02	≤0.05
Acid number	mh KOH/g	0.37	≤0.5
Mono-glycerides	% (weight)	0.44	≤0.8
Di-glycerides	% (weight)	0.14	≤0.2
Tri-glycerides	% (weight)	<0.1	≤0.2
Free glycerol	% (weight)	0.015	≤0.02



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