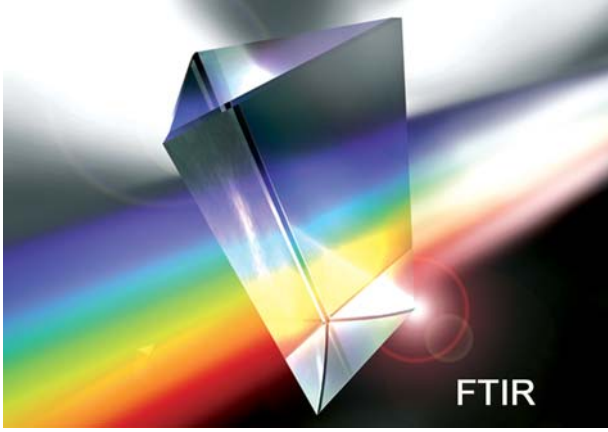
Application Note



Infrared Spectroscopy differences between biodiesel prepared from rapeseed and the edible rapeseed oil

Identification of Ester and especial FAME in different oils

European agriculture produces a lot of plants containing oil which will be used for commercial purposes. For the food market the rapeseed oil is used as salad oil or as main part of margarine.
Actual the rapeseed oil is also a discussion point for the biodiesel aspects.
The quality of such biofuel and the blends of it are controlled by the Biodiesel regulation.
One aspect of the control is the FAME (Fatty acid methyl ester) contents.
Biodiesel like rapeseed oil is rich on fat molecules which are treated in a transesterification process to contain a variety of esters. The transesterification is necessary because motors cannot run with the natural rapeseed oil which is too viscose.
In the figure1 the chemical treatment is shown in a short graph. The huge molecule of a tri-glycerol is cracked into the glycerine molecule and diverse FAME.
To visualize the change of the natural product the FTIR Spectroscopy was investigated to show the effect of the transesterification on infrared spectra of an edible rapeseed oil and a biodiesel prepared from rapeseed. The difference should be visible regards triglycerides and the FAME as shown in figure 2A and 2B

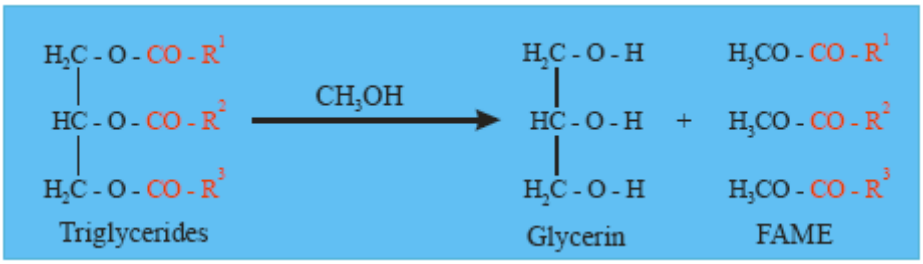


Fig. 1: Transesterification of tri-glycerides to FAME

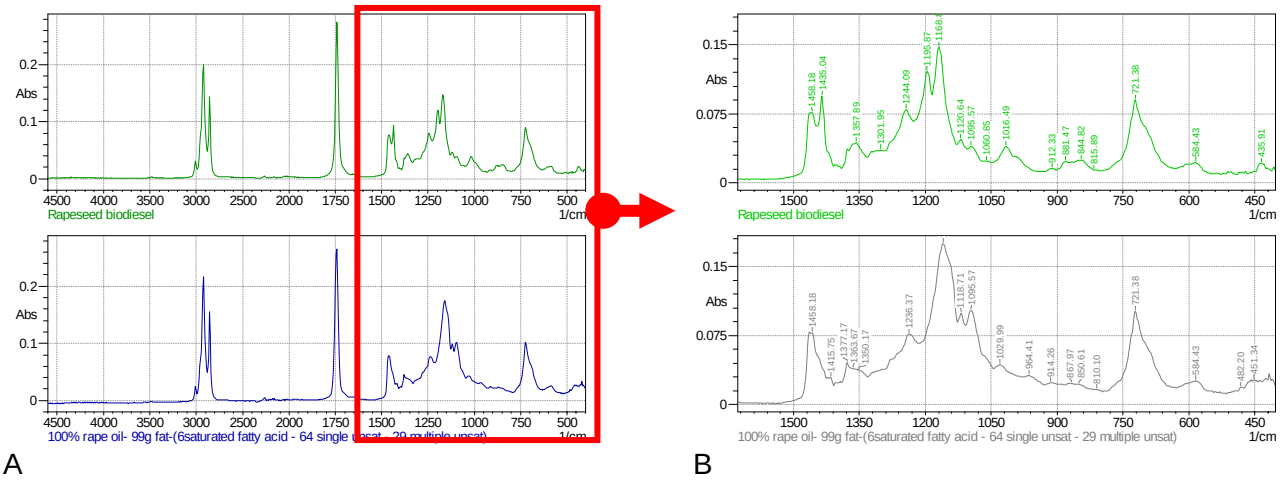


Fig. 2: Typical infrared absorbance spectra of pure rape oil (black) and a pure biodiesel (green) in A, visible are the differences in region at 1600 to 400 cm^{-1} in B.

The peak analysis of both spectra shows significant differences. The differences are effected by the ester groups. The change from ester groups to concrete methyl ester has the strongest impact in the infrared spectrum. All aspects regards the carbonyl groups are visible. These are inductive and mesomeric effects which needed more or less strong energy to get the special group into vibration. The ester group we are talking about is in common described as R_1-C(OR)=O in edible oils and as R_1-C(OCH_3)=O in biodiesel. R_1 represents long chains of hydrocarbons. Additional chains representing Palmitic, Stearic, Oleic and Linoleic acid are visible in both spectra with the -CH_2 hydro carbon part. Signal groups belonging to the vibrations are visible and fixed in both (table1). But all groups regards the CH_2-O- are reduced and new signals are visible belonging to CH_3-O- vibrations in the biodiesel. The most influence as result from transesterification is to see in the new signal at 1435 cm^{-1} which is definitely the methyl ester group with its deformation vibration. The next visible transformation is in the ester control signal area approx. 1200 cm^{-1}. The strong, broad signal at 1159 cm^{-1} in edible oil will separate into two concrete signals at 1168 and 1195 cm^{-1}. The averaging of the energy over the triple ester group of the triglycerides is gone. Same happens to signal at 961 cm^{-1} which is the CH_2 wagging frequence (a deformation vibration of the RO-CO- group).

Sample preparation

Instead of the classical infrared transmission cell the single reflection technique was used for the measurements of the infrared spectra. One drop of the oil was placed on the measurement window, measured and later removed simply with a tissue and cleaning of remaining parts with a drop of solvent. System will be ready for measurement of next sample within one minute.

Table 1: Discussion of infrared spectra based on rape seed oil – comparison of methyl ester and triclycerol ester

Edible Rape oil		**Biodiesel based on rapeseed**		**Basic oil**
Vibration [cm^{-1}]	**Remark**	**Vibration [cm^{-1}]**	**Remark**	**Vibration [cm^{-1}]**
584.43		584.43		584.43
721.38	-CH2 rocking	721.38	-CH2 rocking	721.38
914.26	-OH (oop)	912.33	-OH (oop)	
964.41	-CH2 wagging in RCOCO-	-		
1029.99		1016.49		
-		**1060.85**	-O-CH2-C	
1095.57		1095.57		1095.57
1118.71		1120.64		
1159.22	(1137 shoulder) average of signals	**1168.86**		
-		**1195.87**	C-O	
1236.37	C-CO-O-	1244.09	C-CO-O-	
1415.75		**1435.04**	**(CO)-O-CH3**	
1458.18		1458.18		1458.18
1743.65	C=O Ester	1741.72	C=O Ester	
2852.72	-CH2	2852.72	-CH2	2852.72
2922.16	-CH2	2922.16	-CH2	2922.16
3007.02	-CH	3007.02	-CH	3007.02

Instrumentation:

IRPrestige-21 with IRsolution Software
DuraSamplIR with KRS-5 crystal
Penetration into sample surface: approx. 2 µm
Sample preparation time: 30 sec.
Measurement technique: single reflection ATR