Biodiesel Lifespan Quality Performance

Monitoring and Assessment

Evaluation of Quality Changes in Biodiesel along the Logistics Chain

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In Cooperation with:

Total Deutschland GmbH

OMV Refining & Marketing GmbH

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Contents

Index of Abbreviations

ASG	Acylated Steryl Glycosides
FAME	Fatty Acid Methyl Ester
csFBT	Cold Soak Filter Blocking Tendency
FBT	Filter Blocking Tendency
SG	Steryl Glycosides

1. Introduction

Blending Diesel fuel with Biodiesel (FAME) is prevalent practice in Europe today. In Germany, Diesel FAME blends have been used nationwide since 2004; due to the introduction of the quota obligations with only few exceptions to this rule, practically only Diesel fuel containing 7 % V/V FAME (B7) is to be found at German filling stations.

While in every-day consumer practice the use of those fuels is mainly unproblematic, in the past there have been problems in individual cases due to filter plugging at filling stations. The trouble-shooting for the causes could not be finalized so far. While in some cases microbes could be identified as cause, in other cases steryl glycosides (SG) and acylated steryl glycosides (ASG) for example – which are a natural part of vegetable oil and of which proportions may also be identified in Biodiesel after transesterification and product cleaning – may also be considered a possible cause. Since their solubility in Biodiesel is low, they crystallize after the Biodiesel's synthesis thus possibly plugging fuel lines.

A study, jointly organised and realised by *Verband der ölsaatenverarbeitenden Industrie e.V. (OVID)* and *Arbeitsgemeinschaft Qualitaetsmanagement Biodiesel e.V. (AGQM)*, showed which processing steps lead to a decrease of the SG and ASG contents in rapeseed and soybean oils thus enabling the production of a feedstock with little SG and ASG contents for the production of Biodiesel [1].

[1] J. Haupt, G. Brankatschk, T. Wilharm, "Sterol Glucoside Content in Vegetable Oils as a Risk for the Production of Biodiesel", **2010,** www.agqm.de

With another AGQM project the influence of the Biodiesel production process on the SG and ASG contents was examined[2]. So far it has not been possible in any case to detect a correlation between SG and ASG contents of FAME and the filterability of Biodiesel and/or the blend fuels produced thereof.

Bearing that in mind AGQM carried out another project monitoring the fuel quality along the Biodiesel supply chain from production to storage, transport and finally fuel blending. The objective of the study was to detect potential problems caused by the presence of minor components like steryl glycosides and to especially assess the risk of an impact on the filterability of B7 fuels. Two mineral oil companies as well as their individual Biodiesel suppliers could be won for that project.

Now after tests with vegetable oils and Biodiesel, the study at hand shows how the quality of Biodiesel changes between production, storage and transport up to fuel blending and how the filterability of B7 blend fuels is influenced by the Biodiesel's filterability. Parameters which are not part of the standards for Biodiesel (EN 14214) or Diesel fuel (EN 590) – like the content of steryl glycosides and the particle size distribution – were tested in particular in order to identify significant changes of minor components or parameters which may influence the filterability.

2. Objectives

The following problems were subject of tests for this study:

- Is it possible to detect and maybe even avert any potential risks related to minor components from the Biodiesel's production to its processing as blend fuel?

- What is the impact of the Biodiesel filterability on the filterability of B7 blend fuel?

In order to find answers to those questions, parameters which may alter from the beginning to the end of the supply chain were tested during the entire Biodiesel life span.

3. Realisation

Apart from the SG and ASG contents and the filterability, for samples from Total Deutschland GmbH (supply chain 1) the particle size distribution, the water content, the oxidation stability and the fatty acid methyl ester composition as well as the proportions of glycerol, monoglycerides, diglycerides, triglycerides and total glycerol in the Biodiesel were determined. SG, ASG and glycerides were not determined in B7 blend fuels because the results are expected to be below the detection limits (SG/ASG) or cannot be determined at all (glycerides).

For OMV Refining & Marketing GmbH samples (supply chain 2) the SG and ASG contents were determined.

Table 1 shows the sampling scheme at Total Deutschland GmbH.

- Sample A: FAME, loading at Biodiesel production plant (5 different samples)

[2] J. Haupt, J. Fischer, K. Witt, T. Wilharm, „Steryl Glycosides and Acylated Steryl Glycosides in Vegetable Oils and Fatty Acid Methyl Esters – Effects on the Filterability of Biodiesel", **2011**, www.agqm.de

- Sample B: FAME, Biodiesel discharged at Total Deutschland GmbH (3 different samples)
- Sample C: FAME, sample from Biodiesel tank at Total Deutschland GmbH (1 sample)
- Sample D: fossil Diesel component (4 different samples)
- Sample E: Blend fuel (3 different samples)

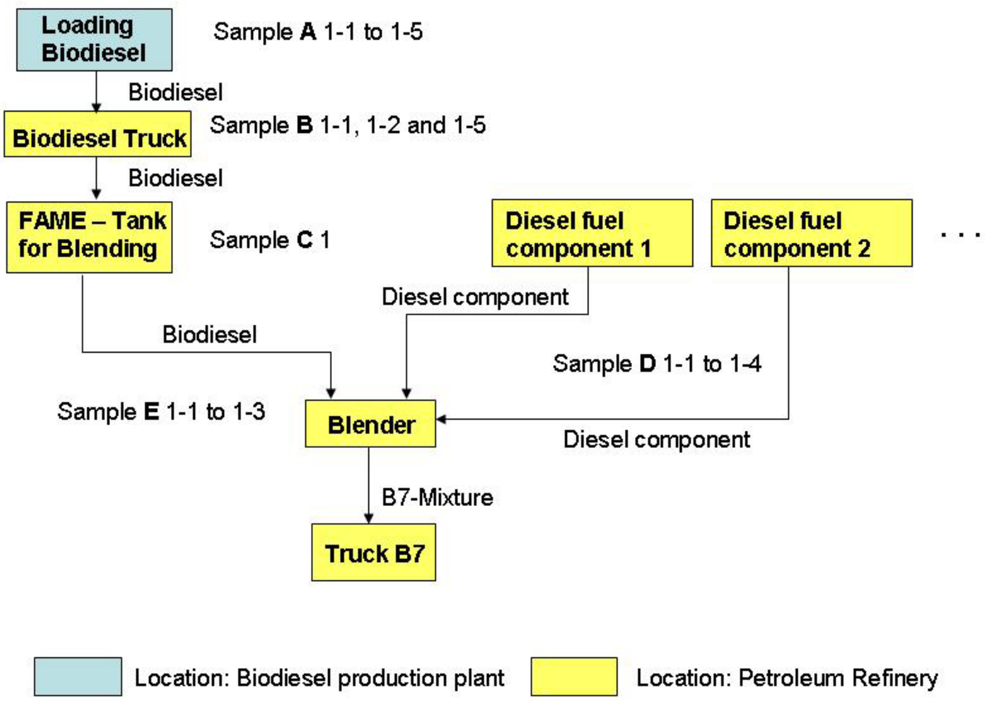

Fig. 1: Diagram of sampling points at Total Deutschland GmbH (supply chain 1)

Samples ‘A’, ‘B’ and ‘C’ are pure Biodiesel samples (B100); their analyses provide indications whether any changes occurred along the supply chain from the Biodiesel production plant to the Biodiesel used at the refinery. All Biodiesel samples were tested for SG, ASG, filterability, water content, oxidation stability, glycerol and glycerides as well as particle size distribution. The determination of the particle size shall reveal whether there is a correlation between the particle size distribution and the SG and ASG contents and the filterability. Changes such as particle size growth along the supply chain shall also be subject of testing.

Sample ‚D’ stands for the blend components of the mineral Diesel forming the base of the finished fuel. In this case the final product is a blend of four different mineral oil components mixed with Biodiesel in the blending facility. Therefore, every mineral oil stream was sampled separately. The samples were tested for filterability, water content and particle size distribution.

The ‚E’ samples are B7 blend fuel samples free of any brand additives. A sample each was taken from the upper, middle and lower part of the tank in order to determine whether there are layers in the tank.

The entire sampling from shipment to blending the B7 fuels was arranged within one week and it was coordinated so that the Biodiesel supplied was also the blend component for the B7 blend fuel.

At one of the refineries of OMV Refining & Marketing GmbH there is also a direct pipeline link from a Biodiesel production plant to the Diesel fuel blending facilities. Therefore, this special FAME supply was integrated into AGQM's research programme. Sampling is illustrated in table 2.

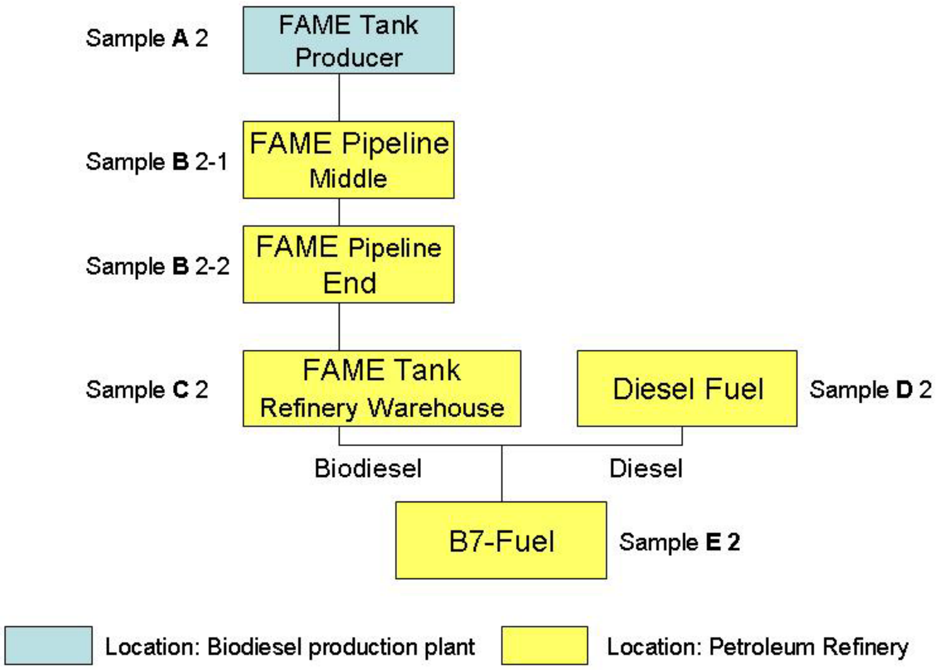

Fig. 2: Diagram of sampling points at OMV Refining & Marketing GmbH (supply chain 2)

Sample 'A2' was taken from the producer's Biodiesel tank; sample 'B2' was taken at two different sections of the pipeline; sample 'C2' came from the Biodiesel tank of the refinery's warehouse. Sample 'D2' is a sample of the fossil Diesel component without any FAME blends. Sample 'E2' is a B7 blend fuel sample. All samples were tested for SG and ASG.

4. Methods of Analysis

The parameters also subject of DIN EN 14214 were determined by the relevant test methods: water according to DIN EN ISO 12937, oxidation stability according to DIN EN 14112; free glycerol, glycerides and total glycerol according to DIN EN 14105. The fatty acid profile was determined according to DIN EN 14103.

Steryl glycosides and acylated steryl glycosides were determined according to the HPTLC method (High Performance Thin Layer Chromatography).

The filtration properties were determined according to IP 387, method B (FBT) and IP PM_EA/08 (csFBT). According to both methods a defined sample volume is filtrated. As soon as the entire sample volume is filtrated or a defined pressure is reached, the filtration process is stopped and the pressure actually achieved or the filtrated sample volume is determined to calculate the FBT index. Values of less than 1.41 represent samples the volumes of which could be filtrated completely. If the value exceeds 1.41 the defined pressure was reached before the entire volume was filtrated. For the determination according to csFBT a sample is stored at +4° C for 16 hours and filtrated at room temperature afterwards. The comparison of the FBT and csFBT values shows, whether low temperatures can cause any crystallization of individual components in the sample. Increased values for csFBT allow the conclusion that temperature has a crucial impact on the filterability of fuels.

The particle size distribution was determined according to method ISO 4406/SAE 4059 in order to obtain information on the particle sizes and number in fuels. This method is actually intended for the inspection of lubricants; however, it can be used for Biodiesel and Diesel fuel despite the different medium. So far there is no corresponding standard test method for fuel, nor is there any comparable data for the particle sizes in those media.

The results were also assessed analogically to ISO 4406/SAE 4059:
The particles are divided into three size classes (particle > 4 µm, > 6 µm and > 14 µm) and the number of particles per 100 ml is used for the classification of the cleanliness levels.

Cleanliness Level	Particle Number / 100 ml	
	From	**To**
10	500	1000
11	1000	2000
12	2000	4000
13	4000	8000
14	8000	16000
15	16000	32000
16	32000	64000
17	64000	130000
18	130000	260000
19	260000	500000
20	500000	1000000
21	1000000	2000000
22	2000000	4000000
23	4000000	8000000

Table 1: Cleanliness Levels according to ISO 4406 (extract)

Every particle size range is listed separately so that three figures are given for the assessment of the sample (e.g. 21/18/15). The assessment according to this method shall illustrate in particular when the differences in particle numbers of two samples can be considered significant.

5. Assessment

5.1 Total Deutschland GmbH

5.1.1 Biodiesel Samples

Samples of five Biodiesel suppliers were taken directly at the production plants during loading (A 1-1 to 1-5). Three of the shipments were also sampled (B 1-1, 1-2, 1-5) on arrival at the refinery. Another sample representing a mix of the different shipments (C 1) was taken from the Biodiesel tank.

The data concerning SG and ASG, filterability, water content and oxidation stability are compiled in table 2.

Analysis of FAME Samples, Part 1

Sample	SG [mg/kg]	ASG [mg/kg]	FBT	csFBT	Water [mg/kg]	Oxstab [h]
A 1-1 (FAME)	11	< 5	3.63	1.34	196	10-4
A 1-2 (FAME)	30	6	5.79	6.67	123	10.0
A 1-3 (FAME)	19	< 5	7.68	2.55	207	8.7
A 1-4 (FAME)	12	< 5	1.06	1.09	223	10.0
A 1-5 (FAME)	9	< 5	1.25	1.00	190	9.7
B 1-1 (FAME)	8	< 5	3.79	1.63	211	9.5
B 1-2 (FAME)	31	7	8.74	3.18	156	10.3
B 1-5 (FAME)	17	< 5	1.75	1.19	221	9.6
C 1 (FAME)	27	< 5	5.02	2.59	197	9.5

Table 2: Total Deutschland GmbH, part 1

Steryl Glycosides and Acylated Steryl Glycosides

On comparing the measuring results of the SG and ASG contents of samples A 1-1, A 1-2 and A 1-5 with the corresponding samples ‚B' one realises that, with regard to the precision of the test method, the results of samples 1-1 and 1-2 hardly differ, whereas the SG results of samples 1-5 differ by 8 mg/kg. Maybe batch 1-5 was not entirely homogenous with regard to the particle number so that for sample B 1-5 a somewhat bigger proportion of particles was taken from the batch. This may also result in the higher SG value in this sample, when steryl glycosides crystallized and thus accumulated in the particles.

The SG and ASG values for FAME samples A, B and C show maximum values of 31 mg/kg for SG (sample 1-2) and 7 mg/kg for ASG (sample B 1-2) which is comparable to the data gained in comprehensible field tests. Sample C represents the Biodiesel used for the production of the blend fuel at blending.

Filterability

Even though the individual deliveries differ considerably concerning their filterability, no significant changes would be detected in the transport chain. Samples A 1-4, A 1-5 and B 1-5 show low FBT as well as csFBT values of less than 2.00; while the values of samples A 1-2, B 1-2 and A 1-3 are clearly higher. There are samples which are practically identical with regard to FBT and csFBT (A 1-4, A 1-5, B 1-5) as well as others with a significantly higher FBT value (A 1-4, B 1-1). All values determined for filterability and steryl glycoside contents tend to correspond to those of the study mentioned above[3].

The FBT and csFBT values of the sample pairs A 1-5/B 1-5, A 1-1/B 1-1 and A 1-2/B 1-2 match quite well within the precision of the method; however, it must be considered that due to the poor precision of this method the deviations of high FBT values are much bigger than those of low ones. Overall no changes of the filterability were detected along the transport chain, though it becomes apparent that the mixture of the different deliveries in the tank (C1) shows relatively poor filterability. Mixing different qualities thus results in a 'mixed value' for FBT.

Water Content

The water contents of the Biodiesel samples range between 123 mg/kg and 223 mg/kg which is significantly below the limit of 500 mg/kg stipulated by DIN EN 14214; the AGQM requirements – which for AGQM members stipulate a limit of 220 mg/kg for producers and

[3] J. Haupt, J. Fischer, K. Witt, T. Wilharm, „Steryl Glycosides and Acylated Steryl Glycosides in Vegetable Oils and Fatty Acid Methyl Esters – Effects on the Filterability of Biodiesel", **2011**, www.agqm.de

300 mg/kg for trading companies – are also met. A change of the water values along the transport chain cannot be identified.

Oxidation Stability
The samples show oxidation stability between 8.7 and 10.4 hours thus being significantly above the required minimum value of 6 hours. A change in the oxidation stability during transport and storage could not be observed.

Glycerol and Glycerides
The glycerol and glyceride contents of the FAMEs tested are listed in table 3 with a special focus on monoglycerides; saturated monoglycerides can crystallize at low temperatures thus impairing the filterability.

Analysis of FAME Samples, Part 2

Sample	Glycerol [%(m/m)]	Monoglycerides [% (m/m)]	Digylcerides [% (m/m)]	Triglycerides [% (m/m)]	Total Glycerol [% (m/m)]
A 1-1 (FAME)	0.01	0.67	0.14	0.03	0.21
A 1-2 (FAME)	< 0.01	0.62	0.19	0.01	0.19
A 1-3 (FAME)	0.01	0.47	0.09	0.02	0.15
A 1-4 (FAME)	0.01	0.42	0.07	0.01	0.13
A 1-5 (FAME)	< 0.01	0.54	0.08	< 0.01	0.15
B 1-1 (FAME)	0.02	0.71	0.15	0.03	0.22
B 1-2 (FAME)	< 0.01	0.61	0.19	0.01	0.19
B 1-5 (FAME)	< 0.01	0.52	0.08	< 0.01	0.15
C 1 (FAME)	0.01	0.51	0.13	0.06	0.17

Table 3: Total Deutschland GmbH, part 2

The contents of glycerol, monoglycerides, diglycerides, triglycerides and total glycerol meet the relevant requirements of DIN EN 14214; they are typical for Biodiesel produced in Germany. As expected, an impact of the supply chain is not identifiable.

Fatty Acid Profile
The fatty acid profile of the Biodiesel batches used is typical for Biodiesel produced from rapeseed oil. Therefore, the tabulating of the individual data was dispensed with.

5.1.2 Mineral Oil Components

At the Total Deutschland GmbH refinery the final B7 fuel is mixed in the blender with different Diesel fuels and Biodiesel. Therefore, it was necessary to beforehand individually sample the four different components the mineral oil share is mixed of (samples D 1-1 to D 1-4). Their filterability, water contents as well as the particle size distribution were determined. The data is compiled in table 4.

Analysis of the Fossil Blend Components

Sample	FBT	csFBT	Water [mg/kg]
D 1-1 (B0)	1.00	1.00	31
D 1-2 (B0)	1.03	1.03	52
D 1-3 (B0)	1.02	1.00	54
D 1-4 (B0)	1.01	1.00	57

Table 4: Total Deutschland GmbH, part 3

With FBT as well as csFBT values around 1.00, the filterability of the different components is very good; the water contents ranging between 31 mg/kg and 57 mg/kg are low. All results are typical for mineral oil fuels.

5.1.3 B7 Blends

At first the B7 production tank was checked for homogeneity of the products in order to avoid faults at sampling. For that purpose samples were taken from the top, middle and bottom layers and then analysed. The results are listed in table 5:

Analysis of the B7 Fuel

Sample	FBT	csFBT	Water [mg/kg]
E 1-1, top (B7)	1.08	1.03	53
E 1-2, middle (B7)	1.08	1.03	53
E 1-3, bottom (B7)	1.07	1.00	57

Table 5: Total Deutschland GmbH, part 4

With values of just above 1.00, the filterability of the B7 fuel is very good and the water content of 53 mg/kg is low. There is no indication for any layer-forming in the tank. Blending Biodiesel does not noticeably raise the water content.

The quality of the B7 fuel was not influenced even though its blend component was a Biodiesel showing limited filterability with an FBT value of 5.02 and csFBT value of 2.59. This allows the conclusion that the used Diesel exclusively determines the filterability of a blend; still, in the past contrary results were also observed in other tests.

5.1.4 Particle Numbers

Particle numbers are not part of the common test scope of fuels, so AGQM has no comparable date at hand. The particle numbers for FAME, fossil Diesel components and B7 blend fuel are listed together in table 6. The number of particles is indicated for 100 ml samples.

Particle Size Distribution in FAME, B0 and B7 Fuels

Sample	< 2 [µm]	2-4 [µm]	4-6 [µm]	6-14 [µm]	14-21 [µm]	21-38 [µm]	38-70 [µm]	70-90 [µm]
A 1-1 (FAME)	4878	679	580	85	19	2	1	0
A 1-2 (FAME)	457171	15452	4901	899	94	2	0	0
A 1-3 (FAME)	36455	12749	29793	7583	737	2	0	0
A 1-4 (FAME)	39890	2657	876	37	10	2	0	0
A 1-5 (FAME)	2389	860	1485	132	19	2	0	0
B 1-1 (FAME)	8071	1951	1906	109	27	5	0	0
B 1-2 (FAME)	78490	48541	115411	4749	432	3	0	0
B 1-5 (FAME)	26931	13413	30552	4233	622	3	0	0
C 1 (FAME)	53328	15946	29705	9136	2666	17	0	0
D 1-1 (B0)	14790	2618	1318	36	5	1	0	0
D 1-2 (B0)	7451	4400	3115	705	432	92	5	1
D 1-3 (B0)	979	406	324	44	14	6	1	0
D 1-4 (B0)	3325	1260	868	49	12	2	0	0
E 1-1 top (B7)	7692	998	872	73	11	1	0	0
E 1-2 middle (B7)	5703	631	593	52	6	1	0	0
E 1-3 bottom (B7)	9364	1319	1221	121	34	7	1	0

Table 6: Total Deutschland GmbH, part 5

For every measured particle size the samples were given a relevant cleanliness level according to table 1 to simplify their comparison. Figure 3 shows the cleanliness levels for the different samples of FAME, fossil Diesel and B7 fuel with regard to the various particle sizes. In order to be able to graph particle numbers of less than 500, samples with particle numbers < 500 were assigned to level 9.

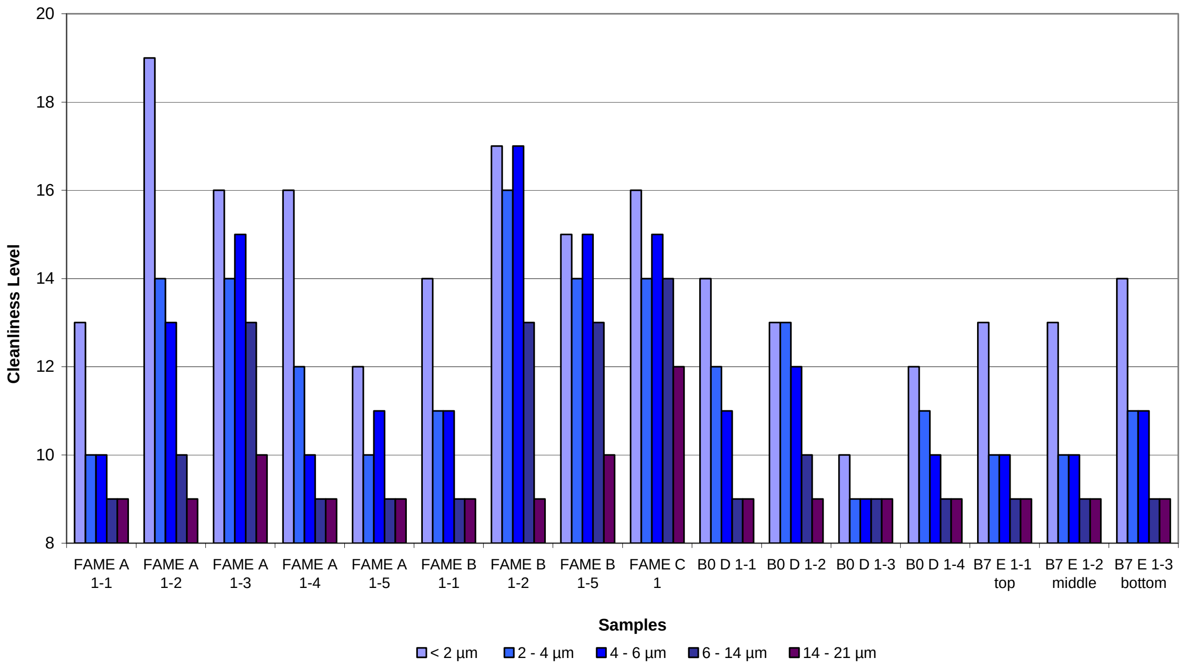

Fig. 3: Classification into Cleanliness Levels of FAME, Fossil Diesel and B7 Fuel Samples

When a sample's cleanliness levels are compared, it is noticeable that in most cases the largest particle numbers are to be found in the range of up to 2 µm and then gradually decrease as the particle sizes increase. In all samples only few particles are found which are larger than 14 µm. For sample pairs A 1-1/B 1-1, A 1-2/B 1-2 and A 1-5/B 1-5 it can be observed that in most cases the particle number increases from A to B. Sample 1-2 in the range of up to 2 µm is an exception; there the particle number decreases from A to B. So in this case the small particles either grew or dissolved again. In the ranges of 2 µm up to 14 µm sample C shows similar particle numbers to those of samples B 1-2 and B 1-5; just in the range of 2 µm to 14 µm a slight increase is noticeable. During storage the numbers of particles in Biodiesel do not change significantly.

The particle numbers of the fossil blend components and B7 fuels range in comparable dimensions, whereas some FAME samples show larger particle numbers. Therefore, the particle number of the blend fuel is determined more by the one of the fossil Diesel component than by that of the FAME proportion.

5.1.5 Filterability

With 30 and 31 mg/kg samples A 1-1 and B 1-1 show the highest SG content; their filterability is relatively bad and in comparison with the other samples they have the largest particle number. Sample C 1 shows similar values. Examples for well filterable samples with low SG contents are A 1-5 and B 1-5, whereupon the particle number of sample A 1-5 is lower than that of sample B 1-5; however, that does not influence the filterability.

Samples A 1-3 and A 1-4 show a similar particle size distribution, except for the range of 4 to 6 µm in which sample A 1-3 shows clearly more particles. The filterability of sample A 1-3 is worse than that of sample A 1-4 but sample B 1-5 shows a similarly large particle number in this range without any deteriorating filterability. Therefore, no inference can be made with regard to the particle number influencing the filterability.

The filterability of C 1 does not have an impact on the filterability of the blend fuel.

5.2 OMV Refining & Marketing GmbH

5.2.1 Sampling and Analysis

Sampling at OMV Refining & Marketing GmbH was modified due to special local conditions because at this particular refinery the mineral oil company is directly linked to a Biodiesel production plant via pipeline. Sample A 2 was taken from the Biodiesel producer's storage tank, samples B 2-1 and B 2-2 were taken at different sections of the Biodiesel pipeline.

Sample C2 was taken from the Biodiesel tank at the refinery warehouse. Sample D 2 is a B0 sample whereas E 2 is final B7 blend fuel. All samples were tested for their SG an ASG contents. All results are listed in table 7.

Sample	SG [mg/kg]	ASG [mg/kg]
A 2 (FAME)	18	< 5
B 2-1 (FAME)	24	< 5
B 2-2 (FAME)	20	< 5
C 2 (FAME)	16	< 5
D 2 (B0)	< 5	< 5
E 2 (B7)	< 5	< 5

Table 7: OMV Refining & Marketing GmbH

5.2.2 Assessment

The SG and ASG values of all four Biodiesel samples are comparably low with maximum values of 24 mg/kg for SG and values below 5 mg/kg for ASG; and – as is the case for Total Deutschland GmbH – this is the range which was assessed in the study for steryl glycosides in Biodiesel[4]. No significant change of the SG contents can be observed from production up to the tank at the refinery warehouse. As anticipated, the SG content of B7 blend fuel is below the detection limit as a result of dilution with fossil Diesel fuel.

6. Summary

The objective of this study was the inspection of any potential changes of the blend component 'Biodiesel' along its entire lifespan and likely impacts resulting thereof on the quality of the final product. For that purpose Biodiesel samples of different producers, various fossil Diesel components and the B7 fuel produced thereof were examined in cooperation with Biodiesel and mineral oil companies.

Two different distribution chains were analysed for this study:

1) *FAME supply to the refinery by different Biodiesel suppliers, subsequent intermediate storage of the Biodiesel in a joint storage tank and production of B7 by mixing those components.*

[4] J. Haupt, J. Fischer, K. Witt, T. Wilharm, „Steryl Glycosides and Acylated Steryl Glycosides in Vegetable Oils and Fatty Acid Methyl Esters – Effects on the Filterability of Biodiesel", **2011**, www.agqm.de

For that, there were checks of the Biodiesel when loaded on and unloaded from the tankers and during storage in the storage tank, of the mineral oil components as well as of the final B7 blend fuel.

Apart from the filterability different Biodiesel parameters were checked which may have an impact on the filterability of Biodiesel such as the steryl glycosides (SG) and acylated steryl glycosides (ASG) as well as the content of monoglycerides.

Some of the individual Biodiesel batches differed significantly with regard to their filterability although the additional analysis of the FAMEs gives no indication as to the causes. The samples taken from the refinery tank show an average value of the individual FBT results. However, the filterability of the final product was obviously not influenced negatively; the FBT values of the finished fuel correspond to those of the basic fuel components.

During transport particle sizes and size distribution undergo changes. An increase of the particle size could be observed as well as of the particle number in combination with an increase of the SG concentration for which an influence due to sampling cannot be ruled out.

Significant changes of the other tested properties during transport and storage could not be determined.

2) *Direct supply to the refinery via pipeline from the Biodiesel production plant*

In this case the Biodiesel was sampled at the Biodiesel producer’s tank, at the start and the end of the pipeline as well as at the refinery’s Biodiesel storage tank; additionally the basic Diesel fuel (B0) and the final B7 blend fuel were analysed.

The fuels were tested for both SG and ASG contents. During the Biodiesel lifespan their concentrations stayed constantly at a low level and did not change significantly from the Biodiesel’s production up to the storage tank in the refinery warehouse. Like in the other case, changes which may be considered critical could not be observed.

As anticipated, the SG and ASG contents in blend fuel are below the detection limit as a result of dilution with fossil Diesel fuel.

A correlation between the filterability of Biodiesel and the filterability of the blend fuel produced thereof cannot be derived from the results of the presented research project. Even in case of high FBT values in FAME, the fossil Diesel component is apparently decisive; the filterability of the B7 fuel was a good one in the case inspected. The filterability properties of the Biodiesel do apparently not have any negative impact on the quality of the final product.

A negative impact of the Biodiesel on the quality of the blend fuel cannot be assumed from the data at hand. From production to storage and transport up to blending critical changes of the contents of by-products, the particle size distribution or the filterability could not be detected.