Universität Rostock Traditio et Innovatio

Final report

Project:

Deposit Formation and Prevention of Deposit Formation Biodiesel (EN 14214)

Short title: Prevention of Deposit Formation Biodiesel

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Dr. Ulrike Schümann

Prof. Dr.-Ing. Bert Buchholz

Chair of Piston Machines and Internal Combustion Engines

University of Rostock

Albert-Einstein-Str. 2

D-18059 Rostock

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1 Research Topic

As part of the JFTOT Diesel I and II projects run by the Research Association for Combustion Engines e.V. [Forschungsvereinigung Verbrennungskraftmaschinen e.V., FVV], the University of Rostock has developed the Diesel Deposit Formation Test (DDFT) to examine the deposit formation tendency of diesel fuels. This testing method is based on commercially available analysis devices, such as Jet Fuel Thermal Oxidation Tester and/or the commercially available since 2018 Diesel Thermal Oxidation Tester (DTOT) manufactured by PAC, and an ellipsometer to determine the layer thickness of the deposit generated by the DTOT. Modifications have been made in the JFTOT device to the hardware (adapted to the higher viscosity of diesel fuels and/or biodiesel), the biodiesel resistance of the sealing materials (included in the DTOT scope of delivery), and the evaluation software in parts. For the ellipsometer measurements, an optical model adapted to the deposits that occur in diesel fuel is required. The DDFT method also uses an evaluation method for deposits generated by the DTOT; this method was developed at the University of Rostock and allows a temperature-dependent evaluation of deposits [1, 2].

This DDFT allows an advance screening of fuels with regard to their deposit formation tendencies, as well as statements on the thermal resistance of diesel fuels and/or the additives used. This method can reduce the number of bench and engine tests usually conducted in relation to this issue, which in turn means considerable cost savings.

The Prevention of Deposit Formation Biodiesel project aimed to adapt testing conditions for biodiesel and its blends, as well as to conduct a testing programme to examine the deposit formation tendency of various qualities of biodiesel in accordance with EN 14214 and of diesel-biodiesel blends in accordance with standards including EN 590, EN 16734 and EN 16709. Furthermore, the project aimed to obtain findings on the deposit formation tendencies of various FAME (RME, SME, TME and UCOME), as well as blends thereof.

At present, for example, an engine test (the XUD9 test)* is conducted as part of the AGQM No-Harm tests. This test examines the tendency toward nozzle coking and lacquering and could potentially be replaced by the DDFT method developed here.

The Prevention of Deposit Formation in Biodiesel project focussed on the following tasks:

1. Adapting the DDFT method to biodiesel and biodiesel blends
2. Deposit tests using various fatty acid methyl esters
3. Deposit tests using FAME blends
4. Deposit tests using diesel-biodiesel blends
5. Deposit tests using aged fatty acid methyl esters
6. Deposit tests using stabilised B10
7. Deposit tests using B10 with the addition of various additives
8. The impact of diesel as a blend component on deposit formation

*Note: The authors are aware that the XUD9 engine test describes the behaviour of fuels in relation to the formation of deposits on injection nozzles in indirect injection engines and is thus only significant for external diesel injector deposits (EDID). The test has different deposit conditions and mechanisms compared with the DDFT method. However, the objective was to examine whether the same deposit precursors are responsible for nozzle fouling and if there potentially is a correlation to the DDFT.

The original aim was to examine the deposit formation tendencies of fuel samples used in the No-Harm test and to analyse the correlation of these results with those gained in the XUD9 engine test. Unfortunately, no notable samples could be provided over the course of the project. The impact of diesel as a blend component on deposit formation was therefore added to the test programme.

2 Current State of Knowledge

The introduction of increasingly stringent emission laws requires the continuous development of diesel combustion methods. In relation to the use of high EGR concepts to further reduce nitrogen oxide, it is absolutely necessary to raise injection pressures to control the emission of carbon-particulate matter and the degree of efficiency. Taking the diesel injection systems with maximum permissible system pressures of around 2,000 bar currently in volume production as a starting point, system and engine manufacturers are currently focussing on the development of 2,500-3,000 bar injection systems. Due to the fact that pressure increases inevitably mean higher required driving power, whilst simultaneously, demands for carbon dioxide reduction in combustion engines need to be met, the aim is to develop extremely efficient, low-loss injection components. The use of complex injection strategies with up to five injections per cycle further necessitates extremely precise proportioning of smaller and larger injection volumes, as well as excellent repeat accuracy.

Realising these requirements results in increasingly complex components, the secure functionality of which can only be guaranteed by adhering to extremely narrow tolerance limits – particularly when it comes to guides for moving parts. As nozzle-needles in passenger car and commercial vehicle CR injectors have a clearance of approx. 2-4 µm, one can imagine that deposits of merely a few µm can already have a considerable impact on the operational behaviour of the injector. Conceivable consequences include increased emissions and rough engine running caused by damage to the injector’s timing and/or changed injection volumes, as well as severe engine damage caused by injectors that consistently inject incorrectly or by jammed needles/valves (in CR systems, there is a risk of extremely high injection volumes).

Internal diesel injector deposits (IDID) can be divided into three types, that often emerge in combinations that differ depending on the region, fuel and additive. Figure 2-1 shows an overview of the deposits observed and examined in and on the injectors, as well as the causes for these deposits. In Figure 2-1, typical IDID have been highlighted in grey.

It is apparent how complex deposit formation can be, and that a number of deposit forming components are responsible for causing said deposit formations. The causes range from the impact of the basic fuel to the numerous interactions of additives. As a result, the development of diesel injection systems must go hand in hand with the development of fuels.

Diesel fuels require additives that are adequate for use in modern diesel engines with fuel lubricated injection systems. Globally, there is a continuing tendency towards desulphurisation and thus the necessity for an increase in additives. In future, fuels will furthermore contain components that differ more strongly than previous components in terms of their polarity. Especially in blends of polar and non-polar components, interactions among the individual blend components, as well as interactions with metallic surfaces and with surface-active additives, must be taken into account. The evaluation of these interactions in relation to the formation of internal diesel injector deposits (IDID) has become so complex that it can no longer be achieved in full by means of elaborate engine tests.

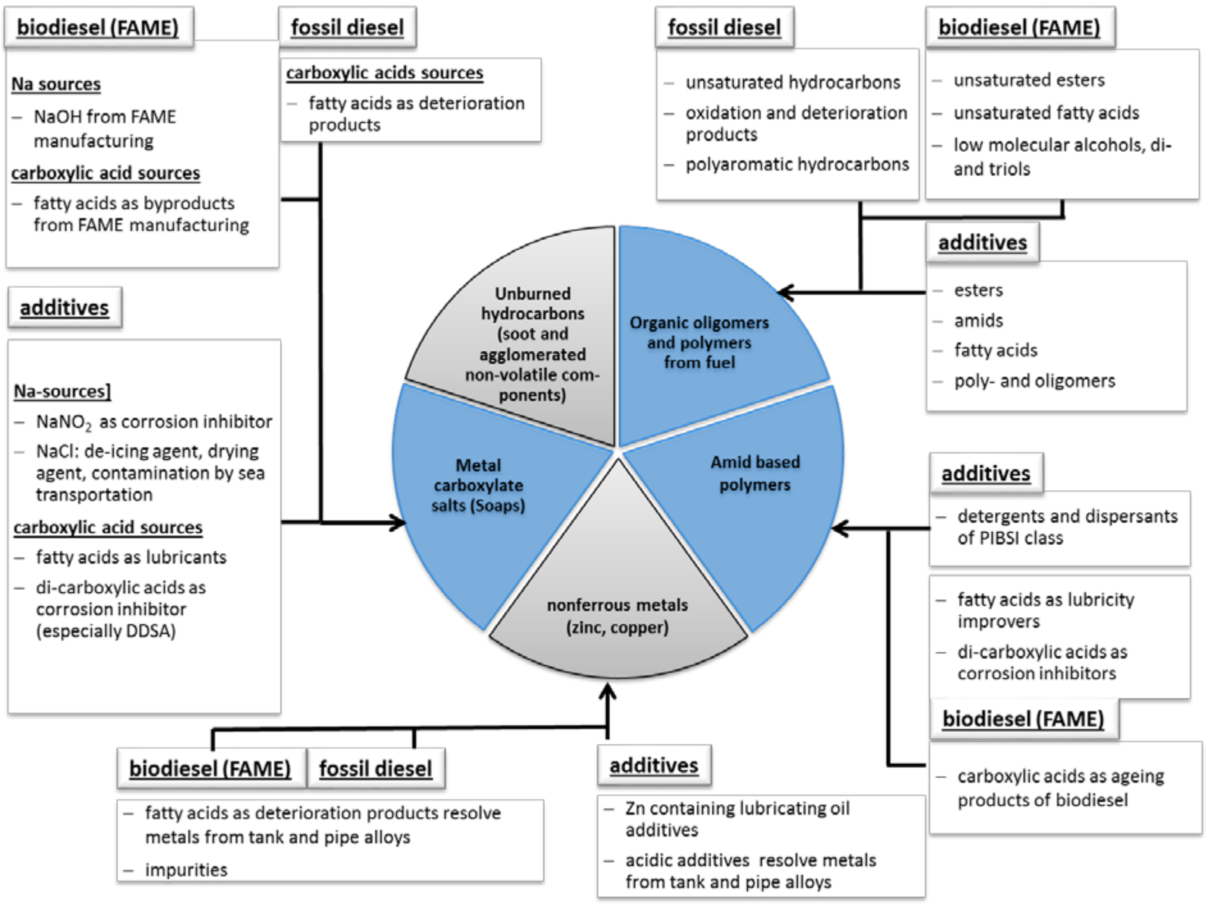


Figure 2-1: Deposit types and sources [3]

There is no conclusive evidence on the role that biodiesel (FAME) plays in the occurrence of IDID. However, it is known that the oxidation stability of biodiesel alone is not decisive for deposit formation; interactions with additives [3-5] and the formation of soap caused by the increased sodium content in FAME and/or the formation of carboxylic acid as a secondary reaction product in FAME autoxidation [3] also have a significant impact. As an oxidatively more unstable fuel component, FAME furthermore acts as an initiator in deposit formation [6]; however, due to the solubility in the blends, the injectors irregularly “wash away” the deposit in certain operating conditions, meaning no FAME structures could be identified in the deposits.

The mechanism of deposit formation in engine operation using FAME corresponds to the conventional mechanism of hydrocarbon autoxidation using peroxide. Autoxidation is defined as a radical chain reaction under the effects of oxygen, which can be initiated by light, heat or the presence of oxidation promoters (catalytically effective metals such as copper) [7]. Various methods were also used to identify the oxidation products and the related polymers created by pyrolysis at high temperatures on metal surfaces present in the deposits [8, 9, 10].

The high polarity and the high molecular weight of the formed oligomers leads to increased sediment formation in blends containing non-polar, fossil diesel fuels, particularly with FAME concentrations of between 5% and 20%. This effect has only been observed in ultra-low sulphur diesel (ULSD) and is known as the B20 effect [9].

Over the course of an ageing study, laboratory tests conducted by the University of Rostock also verified the dependence of sediment mass (% of the total fuel mass) on the FAME content. In these tests, the fuel and fuel blends are aged at 150 °C without aeration; an air cooler is used to condense and return volatile components to the sample. In B7 blends, an increase in sediment mass was observed compared with B0 fuel. In the B30 blend, however, the level of residue sank below the B0 value (see Figure 2-2).

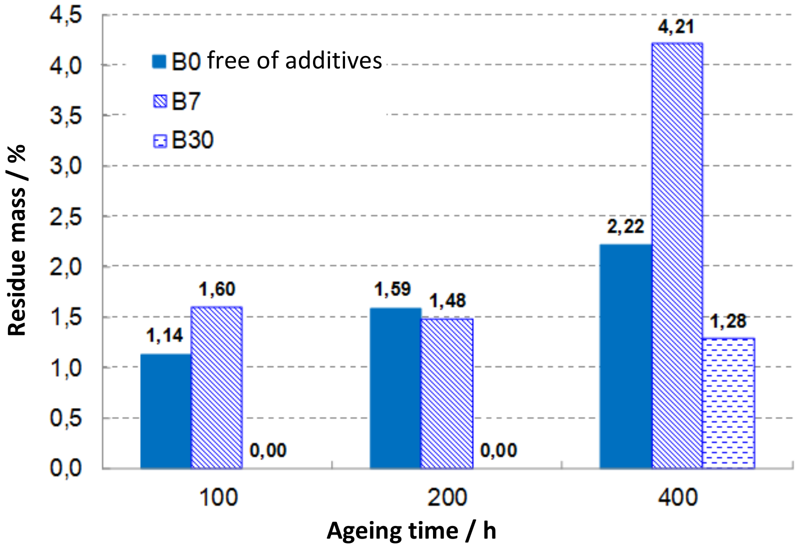


Figure 2-2: Residue mass in aged fuels in dependence on FAME content [3]

For further considerations, however, it is necessary to differentiate the terms sediment and deposit. In the laboratory tests conducted during the project, sediments and surface deposits formed two different phases in the aged fuel. The observed sediments are a temperature-dependent, chemical balance of fuel degradation products that dissolve in the aged fuel when heated and can be filtered out when cooled.

The layers of deposit on metallic surfaces examined in this project are insoluble in fuel and adhered tightly to the surface of the test specimen.

Temperature and oxidation conditions are key factors that define the chemical composition, volume and structure of fuel-related deposits.

There is, for example, a difference in the decomposition products of fatty acid methyl esters (FAME) in the presence and absence of oxygen [3]. Many publications have determined a direct correlation between temperature and deposit mass and/or layer thickness. Oxidation products that arise during fuel degradation contribute considerably to deposit formation. If the oxidation of fuel is prevented (e.g. by degassing), deposition decreases dramatically, as verified by laboratory tests conducted at the University of Rostock [11].

In general, high fuel temperatures, good oxygen availability in the fuel, as well as basic fuels with a high content of heteroaromatic compounds (particularly carbazoles, pyrroles, indoles) thus increase the deposit formation tendency.

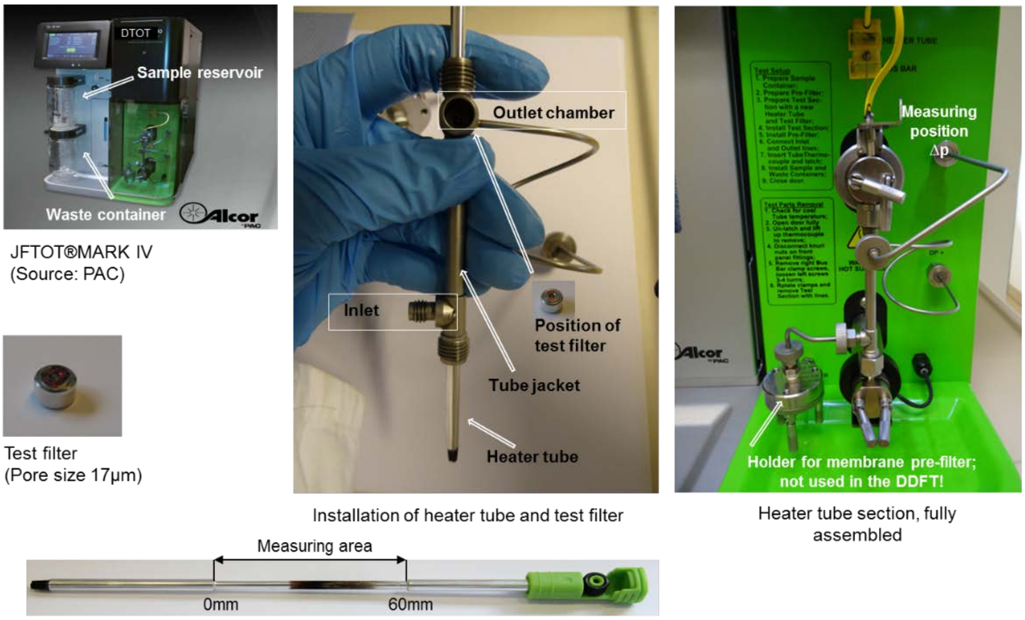
The work of Lin et al. [12] examines the reaction kinetics of the thermal decomposition of biodiesel. The results show no significant thermal decomposition of biodiesel at temperatures below 250 °C. Therefore, the temperatures in the interior of the injector (150 °C-180 °C) cannot cause thermal decomposition with sufficient speed. This means that the decomposition products of biodiesel identified in internal injector deposits are caused by oxidative decomposition.

3 Method Description

The main steps necessary to conduct the DDFT are summarised in brief in the following: The specially made and commercially available aluminium heater tube is installed in the heating tube covering, the test filter is assembled (see Figure 3-1) and the thermal element is placed in the interior of the heater tube. A new heater tube and a new test filter are used in each test. Potential deposits are formed on a cylindrical measuring surface (Ø 3.175 mm x 60 mm length) between the shoulders of the rod. The fuel enters the heater tube at the 0 mm position and is emitted at 60 mm.

At the beginning of the test, the fuel sample (600 ml) is filled into the storage vessel and dried air is passed through the sample for 6 minutes (preliminary test to condition the specimen – uniform oxygenation). The fuel system is then pressurised with a pressure of 34 bar and the heater tube is tempered to the set-point temperature of 240 °C selected in the programme. The heater tube is heated resistively. The temperature is regulated by a thermal element that is positioned in the interior of the heater tube (control temperature T_{max} at 39 mm). An isocratic pump pushes the fuel through the measuring arrangement at a defined flow rate (3 ml/min) (Figure 3-1). The fuel flows around the heater tube and is channelled through the test filter (precision filter) to the refuse bin. The test duration is 150 minutes. After the test has been completed, the measuring arrangement is disassembled and the pipes and testing rod are rinsed in solvents, dried (see Table 4-1) and then evaluated optically.

In the DDFT method, test results are evaluated in dependence on temperatures by using an ellipsometer to measure the layer thickness of the deposit on the test surface of the aluminium heater tube (see the measuring range of the aluminium heater tube in Figure 3-1 and the 2D thickness map in Figure 3-2). In addition, the speed at which the test filter becomes clogged is registered via the drop in pressure. The test filter has a pore size of 17 µm and is installed in the test arrangement (see Figure 3-1).



JFTOT®MARK IV
(Source: PAC)

Test filter
(Pore size 17µm)

Installation of heater tube and test filter

Heater tube section, fully assembled

Aluminum heater tube after test with oxidative deposits

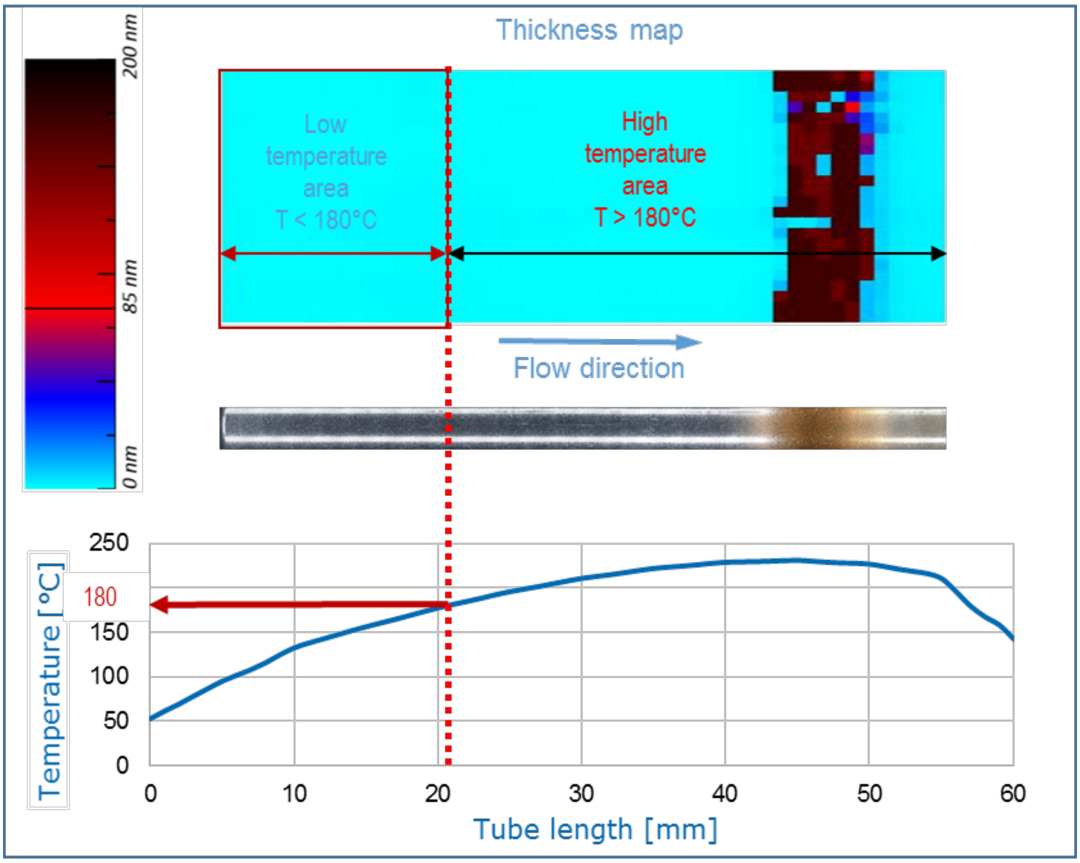
Figure 3-1: Description of the measuring system

Oxidative deposits are generally observed at the hottest area of the heater tube, which lies between the 30 mm and 50 mm positions. Saponaceous deposits are primarily located in the low temperature area (up to 180°C). Deposits (fuel degradation products) that have been washed away by the flow of fuel clog the test filter.

Literature contains numerous methods for quantifying deposits formed on the test rods. Ellipsometry is one of the most frequently used measurement methods [13, 14, 15, 16].

The ellipsometer measures the changing polarisation state of the light reflected on the surface of the test rod [16]. Browne et al. [13] describe this measurement method as a suitable option for measuring the layer thickness, especially as it measures independently of the temperature, material and fuel parameters. In this project, the layer thickness was measured using the OptiReader ellipsometer (manufactured by PAC).

Test results are evaluated as shown in Figure 3-2:



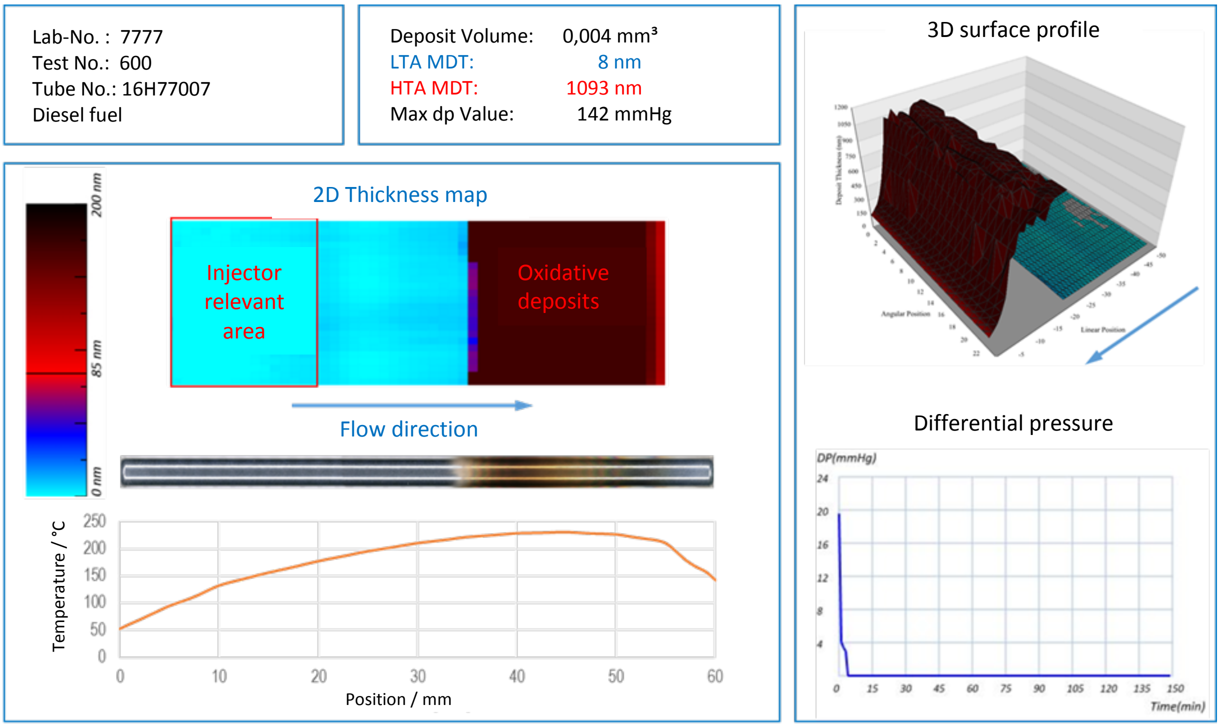
Low Temperature Area: 95°C - 180°C; Parameter LTA MDT (max. Deposit Thickness = mean value of the six highest measuring points)

High Temperature Area: >180°C – 240°C Parameter HTA MDT (max. Deposit Thickness = mean value of the six highest measuring points)

Figure 3-2: Temperature-dependent evaluation of deposits

The cold zone of the heater tube is in the temperature area of between 95 °C and 180 °C. This is the relevant zone for internal diesel injector deposits (IDID). Unlike external diesel injector deposits (EDID), which are formed by fuel oxidation/pyrolysis at the nozzle tip or in the spray holes of the injectors, these deposits form in the interior of the injectors. Temperatures of 240 °C and above are relevant for deposit formation at the nozzle tip. The hot zone of the heater tube, which is in the temperature area of > 180 °C – 240 °C, thus represents the zone of beginning fuel oxidation.

A typical DDFT report contains the following depictions and parameters (Figure 3-3):



Lab-No. : 7777
Test No.: 600
Tube No.: 16H77007
Diesel fuel

Deposit Volume: 0,004 mm^3
LTA MDT: 8 nm
HTA MDT: 1093 nm
Max dp Value: 142 mmHg

Figure 3-3: Presentation of the results of a DDFT

Here, the maximal differential pressure parameter is taken from the DTOT.

The deposit volume, the 3D surface profile, the 2D thickness map and the data on layer thickness of a measurement (24 x 50 measuring points, thickness plot not depicted here) are taken from the ellipsometer report. Using computational fluid dynamics (CFC), the fuel temperature gradient of the heated aluminium tube was calculated for a set-point temperature of 240 °C; this gradient changes at other set-point temperatures.

The LTA and HTA MDT parameters are generated in the thickness plot by means of a MATLAB routine.

The presentation of results is completed by an overview image of the heater tube, taken with a digital microscope.

The colours, structures and temperature areas of the deposits, as well as the rise in differential pressure, if applicable, allow an assessment of the type and origin of the deposits, as well as of potential risks for the functionality of the injection system.

4 Results

4.1 Adaptation of the Test Method for Biodiesel/Biodiesel Blends

The commercially available DTOT (see Figure 3-1) has already been adapted for tests using biodiesel. In the course of the JFTOT Diesel I project, PAC already adapted the optical model required for evaluating diesel deposits; this model is now available for the OptiReader ellipsometer used here (see Figure 4-1). However, a new method for evaluating deposits in dependence on temperature had to be developed, as the Standard Spot Thickness parameter developed for jet fuels (see ASTM D3241) has not proven useful in diesel fuels.

Figure 4-1: OptiReader ellipsometer (source: PAC)

Table 4-1 shows the measurement conditions for the DDFT and the modified test conditions for examining biodiesel (highlighted in red):

Table 4-1: Overview of the test method and DDFT development steps

Conditions	Description
Pre-treatment fuel	No filtration with pleated filter / recommended in the case of fuel inhomogeneities (particles or incomplete additiv tests - check for not completely solved add ons)
Pre-filter in DTOT device	No
Temperature	240°C; 280°C recommended for comparative measurements of FAME and FAME blends
Test duration	150 min
Flow	3 ml/min
After-treatment tube	Flushing steps: 2x 10 ml n-heptane afterwards 10 ml pentane, drying
After-treatment apparatus	Flushing internal metal surfaces and heater tube section with "disolvent" **
By operation with FAME or FAME-Blends after series or each days testing	DTOT has to be flushed with non – FAME diesel by starting a four minute test and abort

*If deposits are flowable, heater tube rinsing steps may need to be repeated after the ellipsometer measurements have been taken, if necessary. This must be documented. Both measurement results must be indicated in the report.

** Dissolvent = mixture of 50% toluene by volume and 50% isopropanol by volume

When applying the DDFT method to biodiesel and biodiesel blends, the changes to the test procedure highlighted in red are recommended. This particularly applies to sufficient homogenisation of the sample prior to the test and the flushing of the system after every test. In some cases, filtering the sample can also make sense and be conclusive (e.g. in the event of solubility problems in certain additives, flowable deposits).

In this project, the DDFT method was generally applied as described in Table 4-1. Any deviations from the test procedure have been indicated separately.

The JFTOT Diesel I project showed that biodiesel made of rapeseed oil methyl ester (RME) in accordance with EN 14214 and biodiesel blends made of diesel and RME do not form deposits at a temperature of 240 °C during the DDFT. Therefore, preliminary tests were conducted to find out whether extending the test period and/or increasing the temperature leads to more conclusive test results (see Table 4-2 and Figure 4-2).

Measurement series 1 – Variation of test period and set-point temperature

<u>B10 fuel:</u> 10% RME + 90% reference diesel fuel 1 (RF 79-07)

Table 4-2: Test conditions and differential pressure in the DTOT – Measurement series 1

Test number	Temperature [°C]	Time [min]	DP [mmHg]
338	240	150	0
348	240	450	0,1
350	270	150	0
352	280	150	0
349	300	150	0

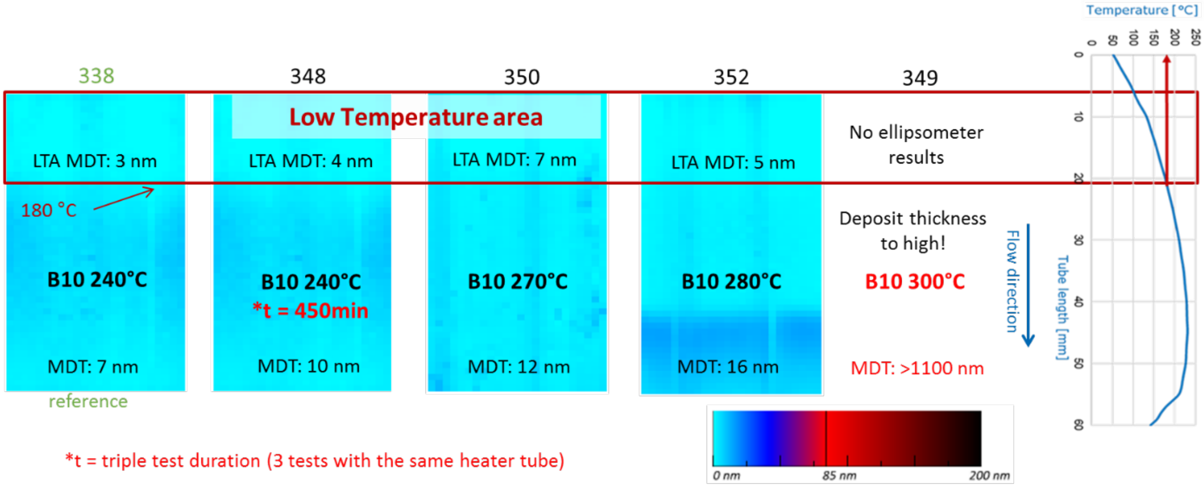


Figure 4-2: Thickness maps and DDFT results for a B10 fuel with extended test period and/or increased test temperatures

To extend the test period, the heater tube was left in the system at the end of the test, and the DDFT was restarted twice. No increase in deposits was observed in the subsequent measuring of layer thickness (see Figure 4-2).

The set-point temperature was gradually increased in Test Nos. 349 to 352. The results show that no increase in deposits is observed at temperatures < 270 °C. At 280 °C, a slight increase in deposits can be observed in the high temperature area. At 300 °C, extremely high deposits are registered in the high temperature area (strong fuel oxidation/thermal decomposition). The deposits were already so high that they reached the measuring limit on the ellipsometer and the device discontinued the test (see Figure 4-2). No rise in differential pressure was registered in the DTOT (see Table 4-2).

For comparative measurements of various FAME types or FAME blends, increasing the test temperature appears to be the only option.

Despite this fact, the project group decided to conduct all further measurements at 240 °C, as this corresponds to actual conditions in today’s injection systems and thus allows a comparison with the deposit formation tendency of EN 590 diesel fuels used in the JFTOT Diesel I and II projects.

4.2 Tests on the Deposit Formation Tendencies of Various Types of Biodiesel

In Germany, biodiesel is largely produced using domestic rapeseed. Besides rapeseed, other oleaginous plants also serve as a source of raw materials for biodiesel. Soya oil, for example, is extracted from soy beans, whilst palm oil is extracted from the pulp of the fruits of oil palms. The cultivation of these plants for use in the production of biofuels is subject to strict sustainability criteria. According to the Federal Office for Agriculture and Food [Bundesanstalt für Landwirtschaft und Ernährung, BLE], in 2016 the consumption of biodiesel made out of used cooking oil methyl ester (UCOME) exceeded the consumption of biodiesel made out of rapeseed oil methyl ester (RME) for the first time, with 0.87 million tonnes, and is increasingly gaining in importance. Tallow methyl ester (TME) has a certain relevance at EU level and should therefore be included in testing. Like the oils and fats they are made of, the various types of FAME exhibit different fatty acid patterns (see Table 4-3). They exhibit varying degrees of saturation and thus various oxidation stabilities/thermal stabilities.

Table 4-3: Examples of typical distributions of fatty acids in various vegetable oils, %(m/m), excerpt from [17]

Fatty acid / oil type		Palm oil	Rape seed oil	Soybean oil	Sunflower oil
		%(m/m)	%(m/m)	%(m/m)	%(m/m)
Lauric acid	C 12:0	0,1 to 0,3	0,1	0,1	0,1
Myristic acid	C 14:0	1,0 to 1,4	0,1	0,2	0,1
Palmitic acid	C 16:0	41,0 to 44,0	3,5 to 5,5	8,0 to 13,5	4,0 to 6,0
Palmitoleic acid	C 16:1	0,2 to 0,4	0,2 to 0,4	5 0,2	0,1 to 0,2
Stearic acid	C 18:0	4,2 to 4,6	1,1 to 5,0	2,0 to 5,4	2,9 to 3,5
Oleic acid	C 18:1	38,5 to 40,5	55,0 to 65,0	17,0 to 30,0	21,0 to 33,5
Linoleic acid	C 18:2	9,0 to 12,0	17,9 to 25,0	48,0 to 59,0	56,0 to 60,0
Linolenic acid	C 18:3	0,2 to 0,5	7,8 to 10,0	4,5 to 11,0	0,1 to 0,6
Arachic acid	C 20:0	0,3 to 0,4	0,4 to 0,7	0,1 to 0,6	0,2 to 0,3
Gadoleic acid	C 20:1	0,1 to 0,2	1,1 to 1,5	≤ 0,5	0,1 to 0,3
Behenic acid	C 22:0	≤ 0,1	0,3 to 0,4	≤ 0,7	0,7 to 0,9
Erucic acid	C 22:1	≤ 0,1	0,3 to 0,5	≤ 0,3	5 to 0,1

The composition of UCOME differs depending on origin and has not yet been examined with regard to deposit formation tendencies.

Table 4-4 and Figure 4-3 present the results of tests on various types of biodiesel.

Measurement series 2 – Variation of types of biodiesel fuels:

RME	Rapeseed oil methyl ester
UCOME	Used cooking oil methyl ester
TME	Tallow methyl ester
SME	Soy oil methyl ester

In principle, the used FAME met the biodiesel requirements according to EN 14214, with the exception of the oxidation stabilities for UCOME and SME, which did not conform to standards. The TME was a biodiesel admixture.

Table 4-4: Fuel parameters of various types of biodiesel and differential pressure in the DTOT – Measurement series 2

Test No.	Fuel	Note	Oxidation stability at 110°C [h]	Acid number [mg KOH/g]	DP mmHg]
335	RME	according to EN 14214	8,6	0,64	0
340	TME	S-content: 13,9 mg/kg; CFPP: +10 °C	15,3	0,49	0
341	SME	-	6,3	0,51	0
358	SME	-	6,3	0,51	0
359	UCOME	-	2,2	1,11	0

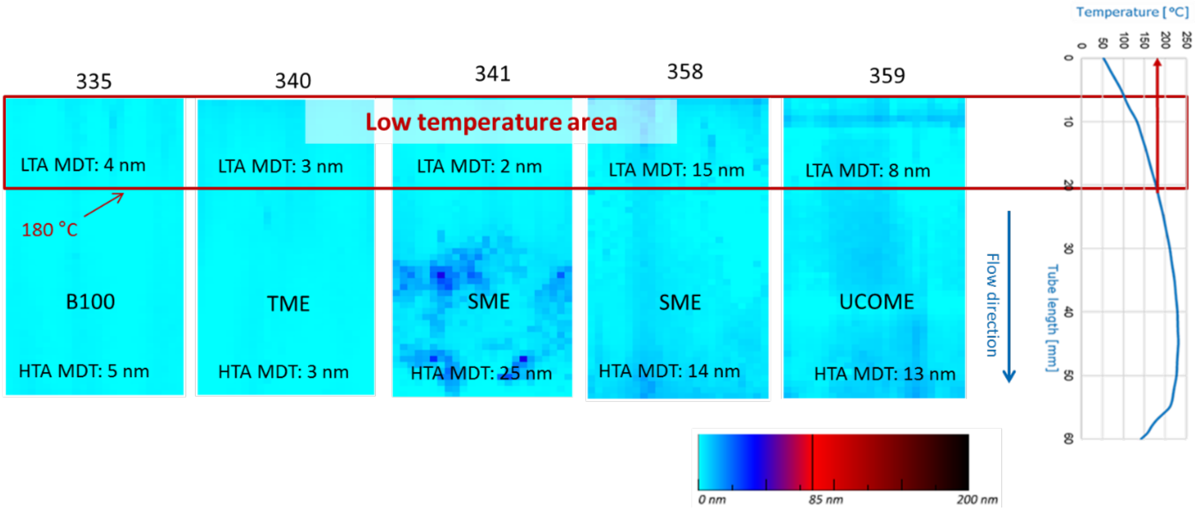


Figure 4-3: Thickness maps and DDFT results of various types of biodiesel

The results presented in Figure 4-3 show that B100 merely forms low layers of deposit, regardless of the FAME type. The project group was unable to reproduce the slight increase in the maximum layer thickness that occurred during the SME test (Test No. 341) in a retry; presumably, this was the result of particulate contamination.

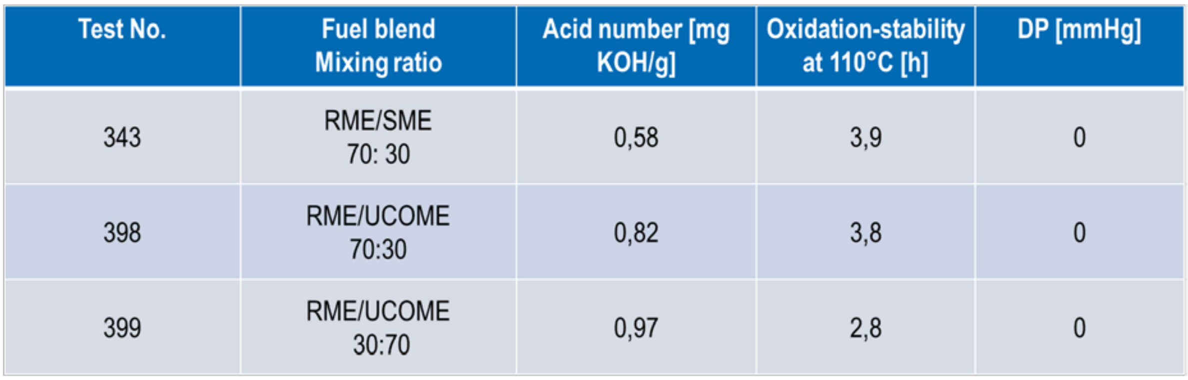
4.3 Tests on the Deposit Formation Tendencies of FAME Blends

Due to the supply of raw materials, as well as the manifold sources of supply combined with international trade routes, blends using various types of FAME are common. To date, it has not been examined whether different blends impact deposit formation in injectors. The following tests take this consideration into account (see Table 4-5):

Table 4-5: Biodiesel blends

Biodiesel blend	Mixing ratio
RME/SME	70 : 30 (% V/V)
RME/UCOME	70 : 30 (% V/V)
RME/UCOME	30 : 70 (% V/V)

Table 4-6: Fuel parameters and differential pressure in the DTOT – Measurement series 3



Test No.	Fuel blend Mixing ratio	Acid number [mg KOH/g]	Oxidation-stability at 110°C [h]	DP [mmHg]
343	RME/SME 70: 30	0,58	3,9	0
398	RME/UCOME 70:30	0,82	3,8	0
399	RME/UCOME 30:70	0,97	2,8	0

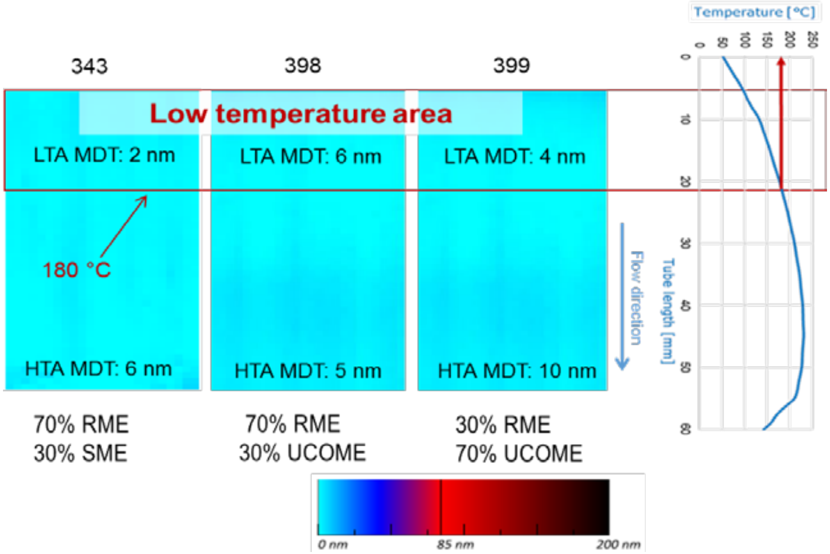


Figure 4-4: : DDFT results for various types of biodiesel at 240°C

The results presented in Table 4-6 and Figure 4-4 show that biodiesel blends using various FAME blends do not form deposits during the DDFT.

4.4 Tests on the Deposit Formation Tendencies of Diesel-Biodiesel Blends

In Europe, a variety of liquid diesel fuels are used, with very different compositions. The fuels differ significantly in their oxidation and thermal stabilities, as well as in their polarity, and thus in their dissolving capacity towards oxygen, water, additive components and polar degradation products.

It is therefore important to test standard and future diesel-biodiesel blends with regard to their deposit formation tendencies. Practice has shown that some blends can exhibit extremely unexpected behaviour with regard to deposit formation. To date, tests on UCOME in particular have not been conducted using this method. Therefore, the following fuel blends are to be tested in the following WP (see Table 4-7):

Table 4-7: Diesel-RME and diesel-UCOME blends

Fuel blend	Fuels
B7	RF 79-07 / RME
B10	RF 79-07 / RME
B20	RF 79-07 / RME
B30	RF 79-07 / RME
B100	RF 79-07 / RME
B10	RF 79-07 / UCOME
B20	RF 79-07 / UCOME

Table 4-8: DTOT results and fuel parameters – Measurement series 4

Test No.	Mixing ratio blends [%(V/V)]	Acid number ASTM D664 [mg KOH/g]	Oxidation stability at 110°C [h]	DP [mmHg]
339	B7	0,26	27,3	0,1
338	B10	0,32	17,5	0
337	B20	0,34	16,0	0
336	B30	0,40	12,6	0
335	B100	0,64	8,2	0

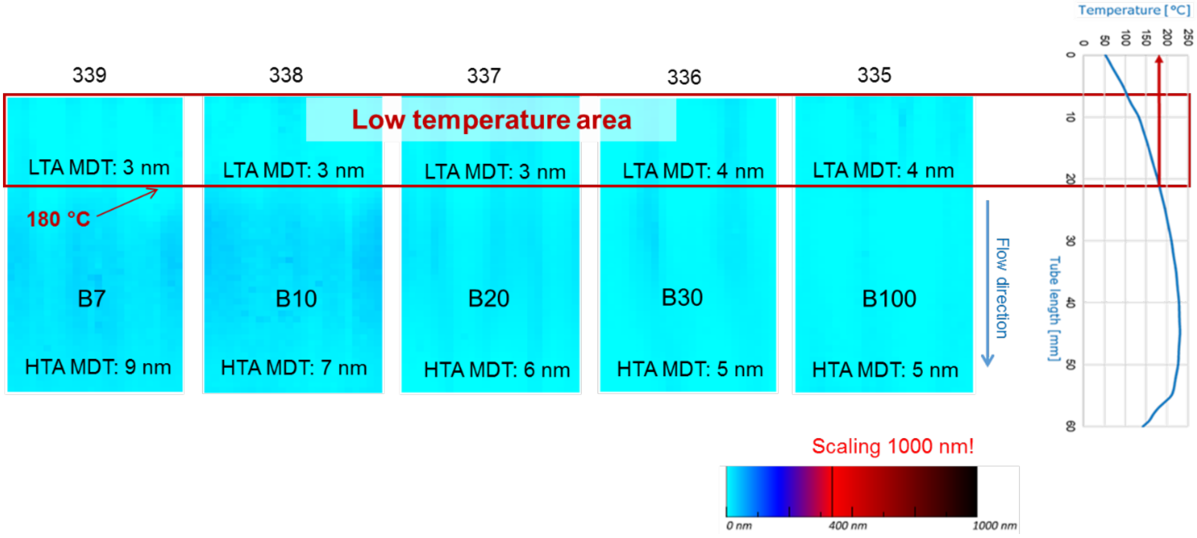


Figure 4-5: DDFT results for various diesel-RME blends at 240°C

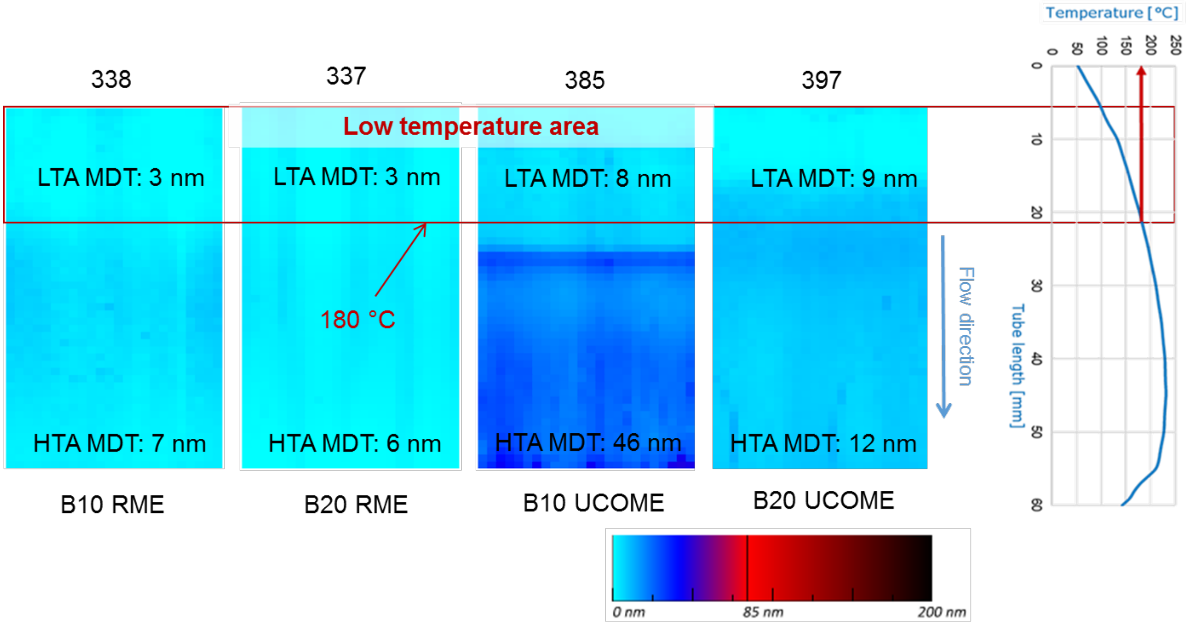


Figure 4-6: DDFT results for various diesel-UCOME blends at 240°C

The results presented in Table 4-8 and Figure 4-5 show that diesel-RME blends do not form deposits during the DDFT. B10 mixed with UCOME forms deposits in the high temperature area, whereas B20 mixed with UCOME, on the other hand, does not exhibit significant deposit thickness (see Figure 4-6). In the course of this project, no explanation could be found for this phenomenon.

4.5 Tests on the Deposit Formation Tendency of Aged Biodiesel

Another focal point is the test series using aged FAME, with the aim of examining the impact of oxidation stability on deposit formation. First, it was necessary to examine whether deposits formed at different stages of fuel degradation are injection system-critical.

The tests used two naturally aged RME samples (storage periods of 2 and 10 years).

Table 4-9: Fuel parameters and differential pressure in the DTOT – Measurement series 5

Test No.	Fuel	Storage duration	Oxidation stability at 110°C [h]	DP [mmHg]
335	B100 (RME1)	fresh	8,2	0
324	B100 (RME 2)	2 years	0,8	0
325	B100 (RME 3)	10 years	2,0	1,9

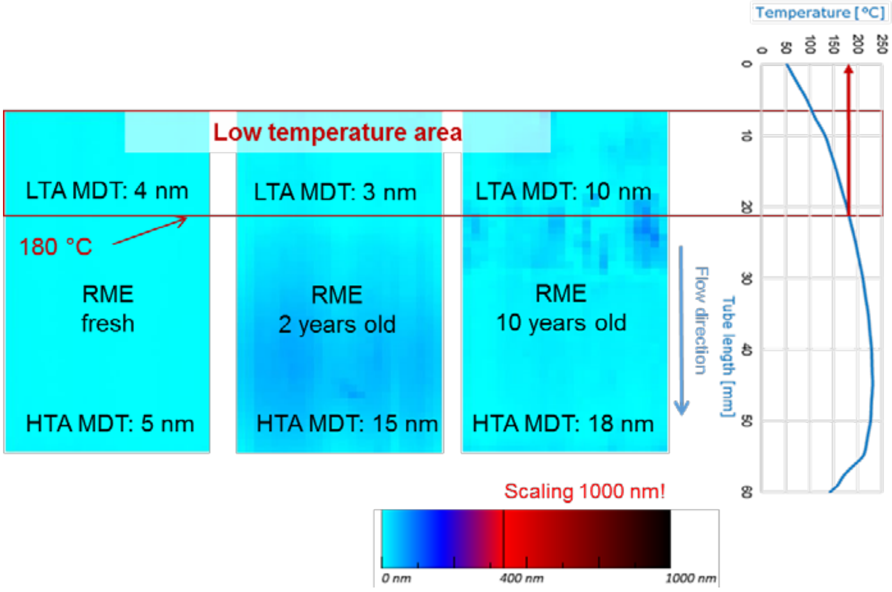


Figure 4-7: DDFT results of naturally aged RME with various storage periods

Neither naturally aged RME sample exhibited deposit formation, despite low oxidation stability. Thus, a connection between oxidation stability and deposit formation during the DDFT can be excluded (see Table 4-9 and Figure 4-7).

The Fuel Changes III project [Kraftstoffveränderungen III, 11] showed that while biodiesel and biodiesel blends do not form deposits in open-loop operation on the injection system test bench, they do exhibit an extreme tendency to form deposits in closed-loop operation (closed-loop circulation of the injected fuel, which is thus subject to thermal stress, see Figure 4-8 and Table 4-10). As a result, there were extreme deviations in the performance of the injector.

This type of RME ageing was used to create a reference fuel with an extreme deposit formation tendency under realistic ageing conditions for use in the subsequent additive tests.

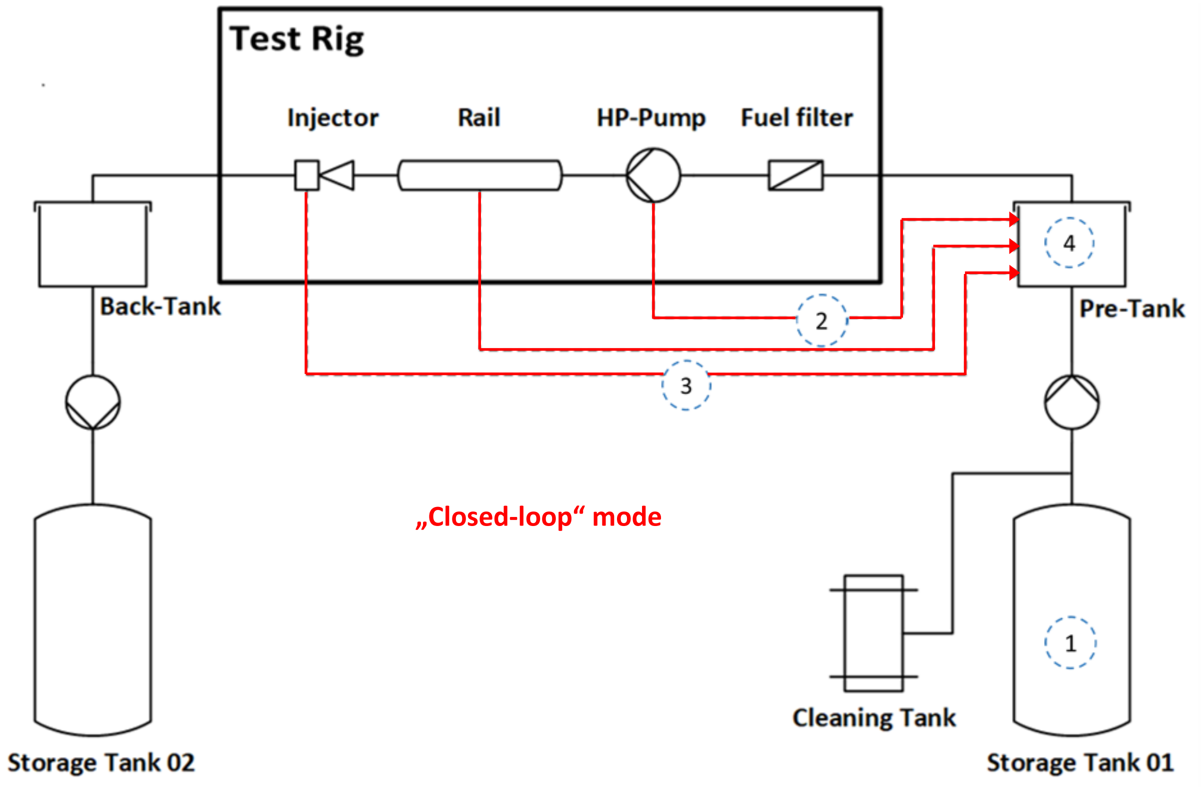


Figure 4-8: Test bench schematic design of an injection system test bench – closed-loop operations

Table 4-10: Bench test testing conditions

System pressure [bar]	2000
Leakage temperature injector [°C]	150
Injection quantity [mm³/stroke]	20
Speed [rpm]	2000
Running time [h]	192
Operating mode	Open loop, stationary
Fuel	Reference Fuel RF 79-07

The additive tests required the generation of a fuel that exhibits significant deposits in the measuring range of the ellipsometer so that the thickness of the deposit layers could be evaluated reliably and serve as a reference for the cleaning effects of additives and/or an undesired increase in deposits.

To be able to stop test bench runs once the thickness of the deposit layers reached a defined range, DDFT measurements were conducted after every 24 hours of operation, using the resulting fuel from the pre-tank. The test run was stopped after a duration of 50 hours of operation, as the DDFT conducted on the 48 h sample already exhibited a sufficiently thick layer in the high temperature area. The 40 litres of "pre-aged" RME (Test No. 404) thus generated in the pre-tank of the test bench after

50 h was used in subsequent additive tests. In addition, tests were run to determine whether filtering the fuel through a folded filter has an impact on DDFT results.

Findings showed that the LTA and HTA MDT parameters did not change substantially (only the mean values of the six highest layer thickness measurement points were included in the calculation, see Figure 3-2). The thickness map, however, shows substantial change. In total, the thickness of the deposit layers decreases in the HTA, meaning that the sample contains filterable particles (precursors of deposit, see Figure 4-9). This result was not reflected in a change in differential pressure (see Table 4-11).

Table 4-11: Fuel parameters and differential pressure in the DTOT – RME ageing studies on the test bench

Test No.	Fuel	Test duration and temperature	Filtration	Acid number ASTM D664 [mg KOH/g]	Oxidation stability at 110°C [h]
335	RME fresh	0 h	No	0,41	6,16
407	RME „closed loop“ test	24 h, 150°C	No	0,44	2,73
408	RME „closed loop“ test	48 h, 150°C	No	0,46	0,1
404	RME „closed loop“ test	50 h, 150°C sample from tank	No	0,47	<0,1
422	RME „closed loop“ test	50 h	Yes	0,47	<0,1

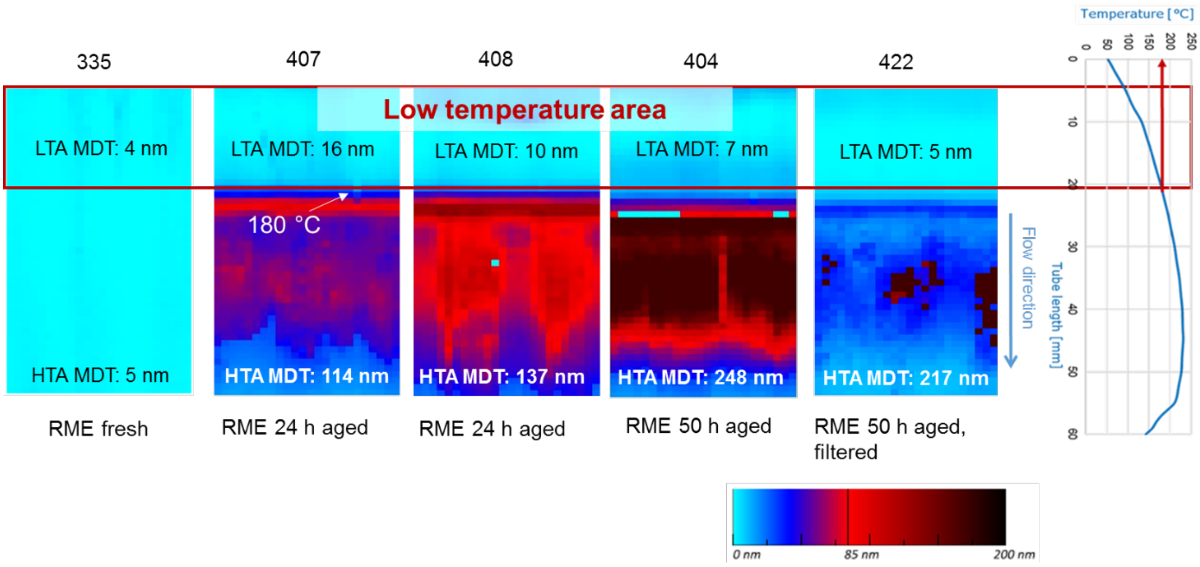


Figure 4-9: Thickness maps and DDFT results – RME ageing on the test bench

4.6 Testing the Deposit Formation Tendency of B10 With Various Concentrations of Antioxidants

A B10 diesel fuel was created using the RME pre-aged on the test bench (see section 4.5) and CEC reference fuel RF 79-07. Prior to blending, both fuel components were filtered separately using a folded filter model MN 615 (Ø retention 4-12 µm; total retention 12 µm) to exclude the impact of filterable particles on results. Bisphenol was used as an antioxidant (AO), various concentrations of which were added to the B10 (see Table 4-12). This is an antioxidant from the group of sterically hindered phenols with two active centres (see Figure 4-10) that successfully passed the AGQM No-Harm test for diesel fuels.

Table 4-12: Concentration variations of AO additives in B10

Fuel blend	AO Addition
B10 from aged RME and RF 79-07, filtered	-
B10 from aged RME and RF 79-07, filtered	200 ppm
B10 from aged RME and RF 79-07, filtered	750 ppm
B10 from aged RME and RF 79-07, filtered	1000 ppm
B10 from aged RME and RF 79-07, filtered	1500 ppm

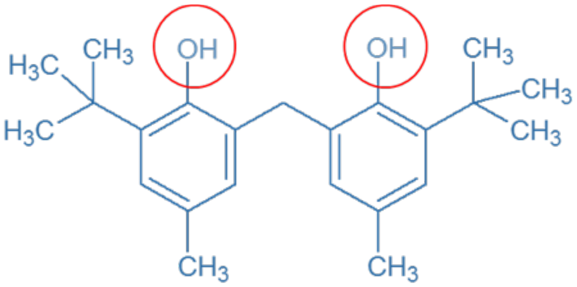
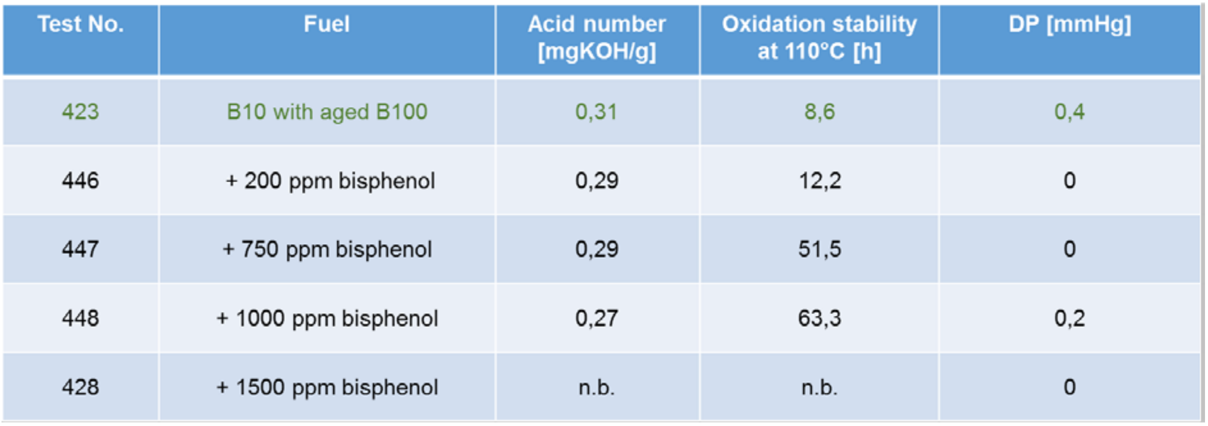


Figure 4-10: 6,6’-di-tert.-butyl-2,2’-methylene-bis-p-hydroxytoluene

The results in Table 4-13 and Figure 4-11 show that the antioxidant bisphenol significantly reduces the deposit formation tendency of the (pre-aged) B10 fuel at concentrations of 200 mg/kg and above.

Table 4-13: Fuel parameters for B10 made using aged RME with various concentrations of AO and differential pressure in the DTOT – Measurement series 6



Test No.	Fuel	Acid number [mgKOH/g]	Oxidation stability at 110°C [h]	DP [mmHg]
423	B10 with aged B100	0,31	8,6	0,4
446	+ 200 ppm bisphenol	0,29	12,2	0
447	+ 750 ppm bisphenol	0,29	51,5	0
448	+ 1000 ppm bisphenol	0,27	63,3	0,2
428	+ 1500 ppm bisphenol	n.b.	n.b.	0

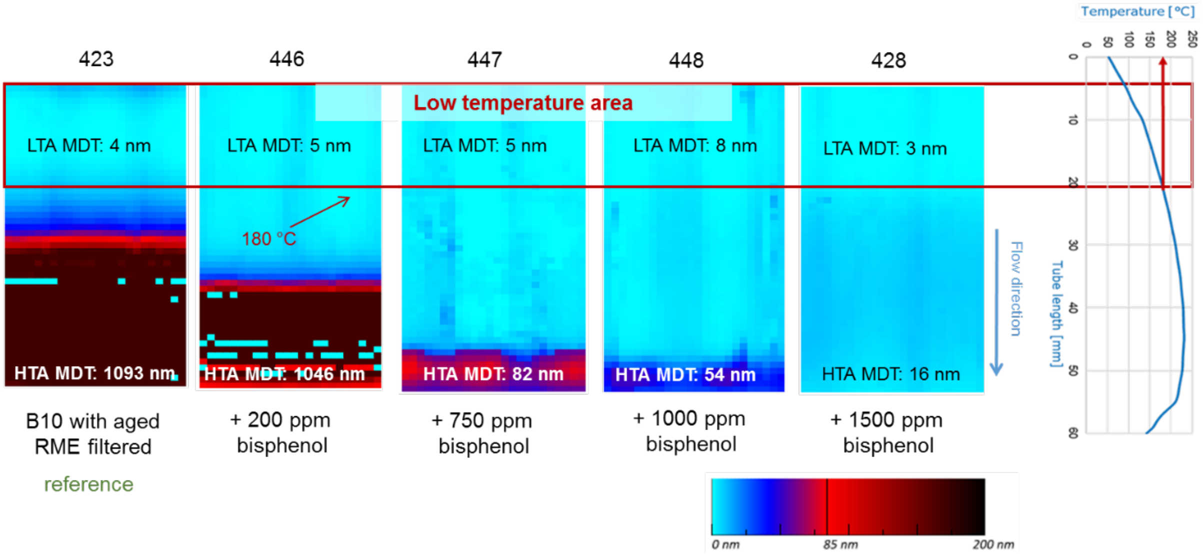


Figure 4-11: Thickness maps and DDFT results – Impact of various concentrations of AO on the deposit formation of B10

It should also be noted that the deposit formation tendency of the aged RME increases significantly when blended with the reference fuel (see Figure 4-9 (Test No. 422) and Figure 4-11 (Test No. 423)).

4.7 Deposit Tests Using B10 with the Addition of Various Additives and Deposit Forming Materials

The components listed in Table 4-14 were used to test additives and additive combinations. In detail, these include a middle distillate flow improver (MDFI) on the basis of an ehtylene-vinyl acetate, a wax anti-settling additive (WASA) on the basis of a carboxylic acid amide, a detergent developed for common rail engines (on a PIBSI basis) and a new generation of deposit control additives optimised specifically to remove internal deposits.

Table 4-14: Selected additives and deposit forming materials for the DDFT

Function	Designation	Recommended dosage [mg/kg]	Maximum Dosage [mg/kg]
Middle Distillate Flow Improver	MDFI	250	500
Wax Anti Settling Additive	WASA	150	250
Detergence	DW10	50	150
Deposit Control Additive	DCA1	100	300

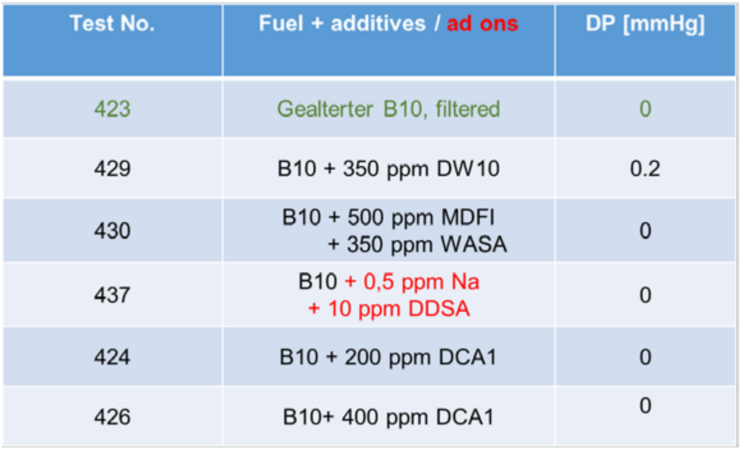
The DW10 additive is usually added to B10 to reduce nozzle fouling (deposit formation on the nozzle). DCA1 is a state-of-the-art additive that prevents and removes deposits in the combustion chamber of indirect and direct injection diesel engines.

Dodecenyl succinic acid (DDSA), a saponification agent, and an organic sodium component acted as deposit forming materials. Table 4-15 shows an overview of the concentrations of additives and deposit forming materials used.

Table 4-15: B10 test fuel with various additives and one saponification agent

Fuel blend	Additive /deposit forming agent
B10 from aged RME and RF 79-07, filtered	-
B10 from aged RME and RF 79-07, filtered	350 ppm DW10
B10 from aged RME and RF 79-07, filtered	500 ppm MDFI und 350 ppm WASA
B10 from aged RME and RF 79-07, filtered	0,5 ppm Na und 10 ppm DDSA
B10 from aged RME and RF 79-07, filtered	200 ppm DCA1
B10 from aged RME and RF 79-07, filtered	400 ppm DCA1

Table 4-16: Differential pressure in the DTOT for B10 using aged RME with various additives and one added saponification agent – Measurement series 7



Test No.	Fuel + additives / ad ons	DP [mmHg]
423	Gealterter B10, filtered	0
429	B10 + 350 ppm DW10	0.2
430	B10 + 500 ppm MDFI + 350 ppm WASA	0
437	B10 + 0,5 ppm Na + 10 ppm DDSA	0
424	B10 + 200 ppm DCA1	0
426	B10+ 400 ppm DCA1	0

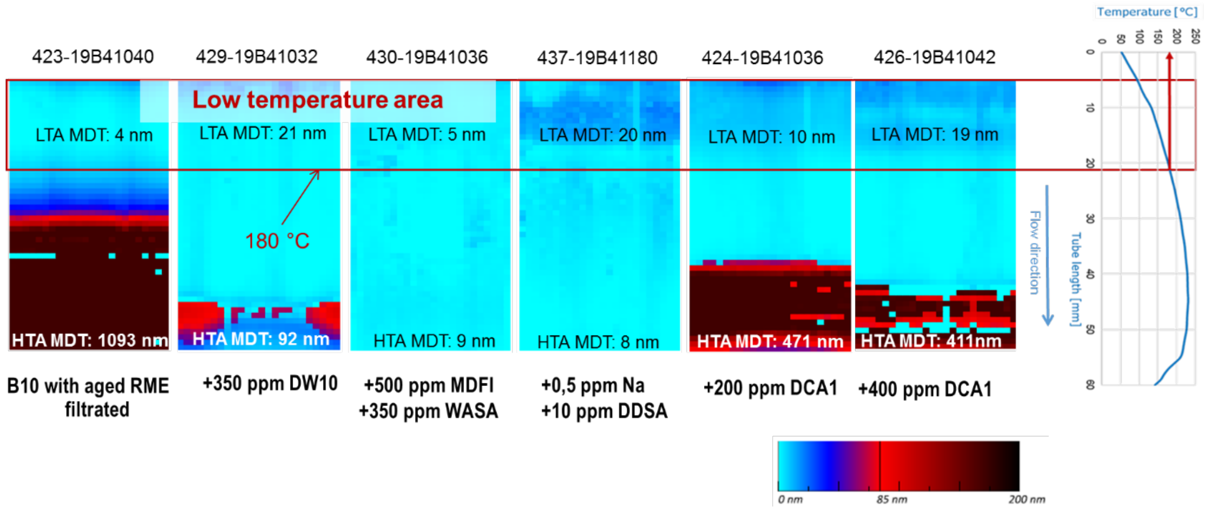


Figure 4-12: Thickness maps and DDFT results – Impact of various additives and saponification agents on the deposit formation of B10

The results presented in Table 4-16 and Figure 4-12 show that the DDFT can be used to examine the effects of additives. The addition of additives DW10 and DCA1 resulted in a substantial reduction of layer thickness in deposits in the high temperature area. However, both additives exhibited an increase in deposits in the injector in the relevant low temperature area (DCA1 exhibits said increase at a dosage of 400 ppm, meaning a dosage that is higher than the recommended maximum dosage). The same applies to the addition of saponification agents. The addition of soap results in a cleaning effect in the HTA but in an increased deposit formation in the LTA. Findings indicate that an increase in layer thickness to >15 nm in the low temperature area is to be classified as critical in injectors. Due to the low layer thickness of the deposit, it was unfortunately not possible to conduct a chemical characterisation of the deposits using a FTIR microscope.

In both areas, the addition of MDFI and WASA completely reduced deposits. These components appear to prevent deposits caused by fuel degradation products. They disrupt crystallisation processes and form smaller yet still soluble agglomerates.

The effects demonstrated here must be examined on injection system test benches or in engine tests.

4.8 Deposit Formation Tendencies of Fuel Samples Used in the No-Harm Test and Correlation of These Results to Those Gained in the XUD9 Engine Test

The ability of diesel fuels to prevent the formation of deposits on injector nozzles is to be evaluated using the XUD9 test. In the No-Harm test programme, the XUD9 test is used in accordance with CEC F-23-01. This test provides a general basis of information on the behaviour of fuels in regard to the formation of deposits on injector nozzles in indirect injection diesel engines. The results are expressed as a percentage of the air flow loss at the defined injector needle lift points [18].

When used as a No-Harm criterion, the nozzle contamination of the untreated B10 reference mixture must be lower than 70% and must be defined for each test cycle. The general level of nozzle contamination of all test fuels is set by adding a commercially available detergent (polyisobutylene succinimide, PIBSI). All fuels are made using the same concentration of PIBSI; as a result, only the oxidation stabiliser's contribution to nozzle contamination is measured. Test results are expressed as the percentage of average nozzle contamination (of four cylinders) with a needle lift of 0.1 mm [18]. Like all engine tests, this test costs a lot of time and money. Comparative DDFT measurements are to verify whether the results of XUD9 engine tests (EDID) correlate to the DDFT results (deposits in the HTA) and whether the XUD9 test can be replaced.

Deposit formation in injection components is a dynamic and temperature-dependent process. An overview of the areas in which deposits form in vehicle components and the respective temperature areas can be found in Lacey et al. [19]. According to this, typical deposits on the nozzles (e.g. deposits caused by zinc-neodecanoate (DW10 engine test)) occur between 220 °C and 280 °C. For deposits caused by fuel oxidation, the temperature area is even higher (270 °C – 380 °C or > 380 °C). The test temperature in the DDFT should accordingly be increased to 270 °C to 280 °C when testing for deposits on the nozzle. Unfortunately, no suitable notable samples obtained in No-Harm tests could be provided over the course of the project.

4.9 The impact of diesel as a blend component on deposit formation

Over the course of the project, two different batches of CEC reference fuel RF 79-07 were used (see Table 4-17).

Table 4-17: Fuel characteristics of blend components and differential pressure in the DTOT

Test No.	Fuel	Delivery Date	Test temperature	Filtration	Acid Number [mg KOH/g]	Oxidation stability at 110°C [h]	DP [mmHg]
335	RME	29.06.2018	240°C	no	0,64	8,6	0
306	DK B0 RF 79-07-1	30.01.2018	240°C	no	0,26	44	0
Ø 386, 390, 391, 394, 395	DK B0 RF 79-07-2	18.12.2018	240°C	no	0,25	27	0,6 - 3,8
Ø 387, 388, 389, 392, 393	DK B0 RF 79-07-2	18.12.2018	240°C	yes	-	-	0

Whilst the first batch, dated 29 June 2018, was completely unremarkable in the DDFT, the second batch showed an increase in differential pressure, as well as high deposits, in the high temperature area and extreme fluctuations in layer thickness measurements (MDT) in the DDFT (see Table 4-17 and Table 4-18). The sample was not homogeneous and contained filterable particles, despite this fuel complying with the limit value for total contamination in accordance with EN 590. Therefore, this batch had to be filtered prior to its use as a blend component, for which a filter model MN 615 was used (see Table 4-18).

Table 4-18: DDFT results for batch 2 of the CEC reference fuel

Test No.	Date of measurement	Filtration	DP [mmHg]	LTA MDT [nm]	HTA MDT [nm]
386	15.03.2019	no	3,8	4	903
390	19.03.2019	no	0,6	3	117
391	19.03.2019	no	1,2	4	309
394	20.03.2019	no	1,9	2	885
395	21.03.2019	no	2,4	3	268
387*	15.03.2019	yes	0	3	58
388*	18.03.2019	yes	0	5	21
389*	18.03.2019	yes	0	2	37
392*	19.03.2019	yes	0	3	65
393*	20.03.2019	yes	0	3	55
438**	19.06.2019	yes	0	10	9

* Filter MN 615, retention capacity Ø 4 - 12 µm – (Filter type from JFTOT method)
**Filter MN 619, retention capacity Ø 2 - 4 µm

Section 4.6 already determined that the deposit formation tendency of aged B100 fuel increases significantly when mixed with diesel. The impact of diesel as a blend component was therefore examined more closely. The Fuel Changes II project [Kraftstoffveränderungen II, 3] already proved that polycyclic aromates contained in diesel are considerably more thermally and oxidatively unstable than biodiesel and that these components are to be classified as precursor substances of deposit formation.

Initially, the first batch, RF 79-07-1, was re-tested at 240 °C to verify whether the deposit formation tendency of this fuel had changed after one year in storage; this was not the case. When evaluating the examined blends, it must be taken into account that the RME component had also been stored for one year at the time of testing. Even though the fresh RME and the 1-year-old RME both came from the same batch, the oxidation stability had already declined from 8.25 h to 3.35 h during this time. The acid number of 0.64 mg KOH/g had not changed. A blend of RF 79-07-2 and RME (fresh) could therefore not be used for comparative purposes.

The DDFT measurements at 240 °C show that, when added to the two different diesel batches (RF 79-07-1 and RF 79-07-2), the RME (fresh and 1-year-old) did not cause an increase in the deposit formation tendency (see Table 4-19 and Figure 4-13 – Test Nos. 338 and 444). Even the deposits formed by pure diesel components (see Table 4-18; Test Nos. 387, 388, 389, 392 and 393) are now evidently no longer demonstrable due to the excellent solubility of RME on oxidative deposits.

However, there is a significant difference if both samples are tested at 280 °C. The blend of RME (fresh) and RF 79-07 Batch 1 still does not exhibit deposit formation at 280 °C, whilst the blend of RME (1-year-old) and RF 79-07 Batch 2 exhibits extreme deposit formation in the high temperature area (> 180 °C) (Table 4-19 and Figure 4-13 – Test Nos. 352 and 425). If, however, the blend of RME (1-year-old) and RF 79-07 Batch 1 is tested at 280°C, the result is a low deposit formation in the HTA (see Table 4-19 and Figure 4-13 – Test No. 443). This clearly demonstrates the great impact the diesel admixture component has on deposit formation.

Table 4-19: Deposit formation tendency of B10 blends in dependency of diesel components and set-point temperature

Test No.	Blend	Batch No. RF 79-07	Test temperature	Filtration	Acid number [mg KOH/g]	Oxidation stability at 110°C [h]	LTA MDT	HTA MDT
338	B10 mit RME (fresh)	1	240°C	no	0,32	17,5	3	7
444	B10 mit RME (1 year old)	2	240°C	yes (only diesel fuel)	0,31	8,6	5	7
352	B10 mit RME (fresh)	1	**280°C**	no	0,32	17,5	5	16
425	B10 mit RME (1 year old)	2	**280°C**	yes (only diesel fuel)	0,31	8,6	Measurement not possible	>1100
443	B10 mit RME (on year old)	1	**280°C**	no	-	-	4	68

Oxidation stability of RME fresh: 8,25 h; RME (1 year old): 3,35 h; Acid number of RME fresh: 0,64 mg KOH/g; RME (1 year old): 0,64 mg KOH/g

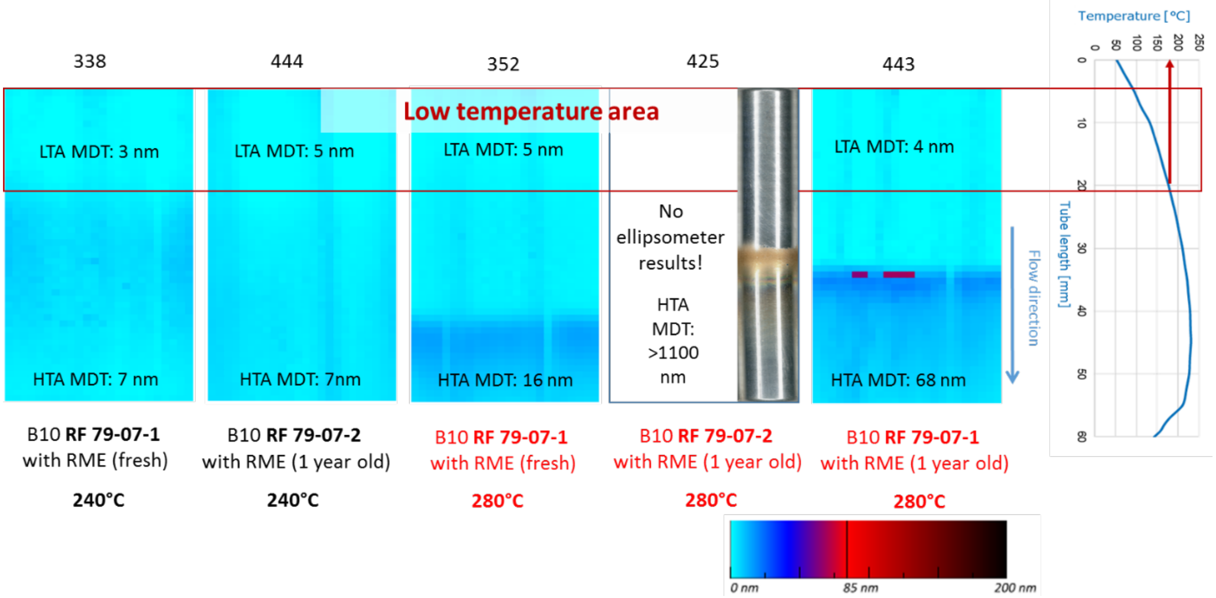


Figure 4-13: Thickness maps and DDFT results – Impact of diesel fuel admixture component and temperature

5 Conclusion

The Diesel Deposit Formation Test (DDFT) can be used to test biodiesel (B100) and diesel-biodiesel blends. Both the Diesel Thermal Oxidation Tester (DTOT) and the ellipsometer used in this project are suitable for use by the manufacturer with biodiesel and for the evaluation of diesel fuel/biodiesel deposits. When testing FAME and FAME blends, the test method requires additional adjustments:

1) The fuel-conducting components of the DTOT must be cleaned daily with solvents before the device is turned off
2) Additional cleaning steps are required when rinsing the heater tubes if deposits are flowable

In the DDFT, biodiesel in accordance with EN 14214 does not form deposits on the heater tubes at 240 °C (set-point temperature for diesel fuel measurements (B0)). The same applies to various FAME blends.

It has become evident that the test conditions for B0 diesel fuels are not conclusive for measurements taken to compare different FAME types or blends. Tripling the test period also did not result in deposit formation; increasing the set-point temperature to 280 °C seems to be the only possibility to determine differences in the thermal-oxidative stability of FAME and FAME blends.

In only one case (B10 fuel using RF 79-07 and UCOME) did diesel-FAME blends exhibit slightly increased deposits in the high temperature area. In the injector-critical temperature area of up to 180°C (low temperature area), no deposits were formed in general when using FAME or FAME-blend fuels.

For blended fuels made out of diesel and RME, tests showed that deposit formation was caused by the diesel fuel blend component used. FAME and FAME blends furthermore exhibited a deposit-reducing effect, both in the DDFT and on the injection system test bench in open-loop operation. The deposit formation tendency falls with an increasing share of FAME.

In closed-loop operation on the injection component test bench, biodiesel and blends thereof are subject to extreme thermal-oxidative stresses at leakage temperatures of as low as 150°C; when this occurs, both show a strong tendency to form deposits. Test benches that conduct the injected quantity of fuel into a loop are therefore only partially suitable for evaluating the deposit formation tendencies of fossil diesel fuels/biodiesel and/or blends thereof in the interior of injectors.

However, the pre-ageing of RME in closed-loop test bench operations provided the opportunity to generate test fuels with a defined deposit formation tendency in the high temperature area under practical conditions, and to use these fuels as reference fuels in additive tests (cleaning effects/testing of increased deposit formation tendencies of disadvantageous additive combinations).

Thus, the project was able to demonstrate the impact of additives and/or saponification agents on deposit formation as well as the deposit-reducing effect of antioxidants and MDFI/WASA. These results need to be verified by tests on injection system test benches.

In general, the achieved results correlate excellently with the results of the bench tests conducted in the Fuel Changes II and III and JFTOT Diesel I and II projects.

The DDFT method is excellently suited for preventive screening of fuels and additives and contributes significantly to the reduction of laborious bench tests.

6 Literature

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7 List of Abbreviations

AME	Animal fat methyl ester
AO	Antioxidant
ASTM	American Society for Testing and Materials
B10	Blend from (90 %(V/V) DF und 10 %(V/V) FAME)
B100	Biodiesel
CFD	Computational Fluid Dynamics
CR	Common Rail
DDSA	Dodecenylsuccinic acid
DIN	German institute for standardization
DCA	Deposit control additive

DDFT	Diesel Deposit Formation Test
DF	Diesel fuel
DP	Differetial pressure
DTOT	Diesel Thermal Oxidation Tester
EDID	External Diesel Injector Deposits
EN	European Standard
FAME	Fatty Acid Methyl Ester
HTA	High Temperature Area
IDID	Internal Diesel Injector Deposits
JFTOT	Jet Fuel Thermo Oxidation Test
KVÄ	Fuel Alterations
LTA	Low Temperature Area
MDFI	Middle Distillate Flow Improver
MDT	Maximum deposit thickness
NKW	Commercial vehicle
PIBSI	Polyisobutylensuccinimid
PKW	Passenger car
RF	Reference Fuel
RME	Rape seed oil methylester
SME	Soy bean methylester
AME	animal fat methylester
UCOME	Used coocing oil methylester
WASA	Wax anti settling additive
WWFC	World Wide Fuel Charter

Contact:

Dr. Ulrike Schümann

University of Rostock

Chair for Piston Engines and Combustion Engines

Albert-Einstein-Str. 2

D-18059 Rostock

Tel: + 49 381 498 9156

Fax: + 49 381 498 9152

E-mail: ulrike.schuemann@uni-rostock.de

web: www.lkv-rostock.de