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CPERI/CERTH



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1 Summary

The main task of SP 1 in RENEW was to produce various qualities of BTL fuels using the Fischer-Tropsch process, test them and arrive at conclusions for creating BTL specifications with a view to reaching a harmonised standard.

Subproject 1 (SP1) has covered research, demonstration and innovation activities on the production of BTL (bio-mass to liquid) fuels primarily from ligno-cellulosic biomass through a combination of the proven CHOREN Carbo-V[®] gasification process with Fischer-Tropsch (FT) synthesis and subsequent refinery steps, with a focus on fuel quality and process optimisation. The SP has comprised essentially research and development on the entire production process chain both at process chain level (optimum adjustment of the different process steps) and at component level (development of process parts and related hard-ware).

A number of process variations were checked and improvements were achieved at the level of the biomass gasification allowing for a more efficient and cost-effective gasification of biomass to very clean synthetic gas suitable for Fischer-Tropsch synthesis. These improvements were accompanied by a thorough economic and safety analysis.

A Fischer-Tropsch synthesis unit was set-up and a suitable catalyst was selected. Process parameters were varied and optimised. The combined gasification/ Fischer-Tropsch process was run to produce BTL samples. In total, more than 5,000 liters of different fractions of BTL were produced within SP1. These were distilled and mixed for achieving different fuel qualities and then delivered to the other project partners for further processing or engine tests. An analysis of the different samples was made by ITN.

With part of the produced BTL (BTL Wax), CERTH did experiments on upgrading this feed by the mild hydro-cracking process. The major part of the fuels produced by UET was used by VW, DC and Renault/REGIENOV for extensive engine tests. These tests revealed that properly selected distillates of the produced BTL fit perfectly to existing automotive engines and allows achieving significant improvements of emission levels comparable with available GTL fuels, i.e. achieving EURO 4 emission levels straightforwardly without additives and without particulate filter.

Based on the results achieved with biomass gasification and BTL production, input was provided for the various assessments carried out in Subproject 5 (SP5) within RENEW.

In the field of knowledge protection, UET succeed in preparing a patent in the field of Fischer-Tropsch synthesis though this process step has seemed to be quite extensively developed already. The patent process is very much advanced at the time of writing of this report and the prospects for getting the patent request accepted are very promising.

The conclusions with regard to BTL specifications are that pure BTL should fit to the existing EN 590 Diesel standard, except the density, with improvements in sulphur content, aromatic components and Cetan No. For BTL used as blending component for a 20% BTL blend (BTL 20), the boiling range may be extended and cold flow properties and flashpoint of the BTL component may be better than required by EN590. The consequence of the latter is that a larger part of the raw BTL can be used directly in the fuel without further cracking or other treatment.

2 Objectives, methodology and main results achieved within SP1

The main task of SP 1 in RENEW was to produce various qualities of BTL fuels using the Fischer-Tropsch process, test them and arrive at conclusions for creating BTL specifications with a view to reaching a harmonised standard.

Subproject 1 (SP1) has covered research, demonstration and innovation activities on the production of BTL (bio-mass to liquid) fuels primarily from ligno-cellulosic biomass through a combination of the proven CHOREN Carbo-V® gasification process with Fischer-Tropsch (FT) synthesis and subsequent refinery steps, with a focus on fuel quality and process optimisation. The SP has comprised essentially research and development on the entire production process chain both at process chain level (optimum adjustment of the different process steps) and at component level (development of process parts and related hard-ware).

The objective was to develop an optimised BTL fuel and an accordingly adjusted production process for a range of different fuel specifications, fitting requirements of both marketable and future power trains (e.g. HCCI). For the latter, a link to the IP NICE was created).

The work followed a holistic approach comprising (1) development and optimisation of the process chain with regard to fuel quality, (2) pilot fuel production and (3) fuel specification and long-duration tests in motors. These three work phases were implemented in an iterative loop: results of the test entered in the respectively next round of process chain development and fuel production. (4) Patents arising from this research work were applied for. In order to prepare industrial-scale demonstration of the Carbo-V® based BTL production path and to create data for SP5 were all assessments took place, (5) a first process design for a 500 MW BTL unit was done.

The outcome of SP1 are (1) an optimum design and operation procedures for BTL production of fuels with different specifications at pilot scale; (2) several tons of BTL, representing the major part of the test fuels being effectively produced within the entire IP RENEW; (3) test fuels for the IP NICE; (4) recommendations for BTL fuel specifications as a first step towards future standards; (5) a patent application; (6) technical data for the environmental and economic assessment of a BTL plant in industrial scale (500 MW).

3 Process development and optimisation (WP 1.1)

The objective of WP1.1 was to develop and optimise the entire BTL production chain based on the proven CHOREN/ UET Carbo-V® biomass gasification process.

WP1.1 was repeated iteratively: starting from an initial definition of specific components (notably catalysts) and process parameters, the results of WP1.2 and WP1.3 were used for successively optimising both the process architecture and process parameters for the entire BTL production chain:

- Syn-gas production from biomass
- Raw product synthesis from syn-gas based on Fischer-Tropsch (FT) process
- Up-grading of the FT raw product

Furthermore, this workpackage included the protection of know-how and economic, environmental and safety analysis.

3.1 Task 1.1.1 Fine-tuning of syn-gas generation parameters

- 1) The syn-gas composition can be influenced by (1) varying the gasifying temperature, notably in the high-temperature reactor (HTV), (2) adjusting the composition of the gasifying reactant (oxygen, steam), and (3) adding residual long-chain hydro-carbons from the subsequent FT synthesis. In this subtask, the exact parameter values for the respective experimentation period were defined.
- 2) The existing gasifier produces a tar-free syn-gas which, after passing through a two-stage gas cleaner, has a quality that allow to keep within the emission limits of the German 17. BImSchV (ordonance regulating the emission limits of waste treatment plants.) when using the gas as fuel within a gas motor for instance. However, in order to avoid damage of the FT catalysts, several substances as sulphur need an additional reduction. For this purpose, a further fine cleaning stage was foreseen. The latter consisted of various cleaning materials which were arranged in a layered structure. The type and arrangement of the layers were varied in order to identify the best ratio of cleaning effectiveness to costs of cleaning materials. In order to facilitate research at this highly sensitive point from a protection of knowledge point of view, UET had concluded a co-operation agreement with one of the world's leading catalyst manufacturers, Südchemie AG, Munich.
- 3) The Carbo-V® process allows for achieving a top value of more than 80% cold gas efficiency for biomass gasification. Nevertheless, the potential for further increasing the efficiency was explored within this project by the following means:
 - Reduction of gasifying temperature within the high-temperature reactor (HTV) to the minimum value, thus reducing thermal losses.
 - Reduction of transport gases: for bringing bio-coke dust and coke/ash mixtures into the gasifier, transport gases are used. The latter are heated up within the gasifier, thus taking off part of the energy. Further gas is needed for batch-cleaning of the raw gas filter. Research was done on the potential to increase the transport capacity of the gas and on constructive measures to reduce dust particle flow out of the reactor.

- Minimising carbon-losses within the gasification process: the Carbo-V[®] reactor is a fluid mechanical device. Bio-coke is transported through pneumatic hauling into the gasification zone. Load variations and therefore changes in the gas stream pattern can lead to bio-coke surplus within short intervals. Notably, larger bio-coke particles can reach the water bath and are then lost for the rest of the process. Research was done on fine-tuning the regulation of the bio-coke powder injection for further reducing bio-coke losses of this kind. The conception and prototype production of this regulation was part of the work to be done.
- 4) Relevant operating costs result from the consumption of electric energy, oxygen and water. For allowing to reduce these costs, research was done on the following measures:
- Minimising gas stream for pneumatic hauling, thus reducing compressor power and electricity consumption.
 - Adjusting gasification temperature through reduction of oxygen input to minimum value.
 - Reuse of waste water from gas cleaning and clinker removal.
- 5) For the following components the constant work performance was optimised:
- Materials in the high-temperature parts such as the combustion chamber (in the upper part of HTV), gas cooling and coarse dust separation (cyclon), notably with regard to structure changes, carburization, aging and corrosion.
 - Materials with regard to changing gas composition (oxidising/ reducing).
 - Masonry of combustion chamber and reactor.
 - Orbital rod mill for coke-grinding.
 - Materials for fine cleaning.

6) Optimization of gas conditioning for large scale BTL production

This task was based on intermediate results from RENEW SP5 assessment. Its aim was to improve the overall BTL production process by mainly comparing Rectisol and Selexol technology. From the results summarized in Del. 1-30, it can be concluded that

- Both the Rectisol and Selexol washing processes can be recommended for removing H₂S and CO₂ on a large industrial scale.
- From an economical point of view, no clear preference can be recognized. The presumed cost advantage of Selexol could not be confirmed. Further analysis is required.

3.2 Task 1.1.2 Optimization of fuel synthesis

1. The FT raw liquid consists of a broad spectrum of hydro-carbons from methane (CH₄) to C₅₀H₁₀₂. Its composition is determined by the catalyst used in the FT synthesis, the process parameters (temperature, pressure and throughput) as well as the composition of the input syn-gas. Building on the experience in the first year of the project, a specific catalyst (catalyst B) which has been identified as the most appropriate one for achieving a high productivity of the process, was used for the fuel synthesis in the forthcoming production periods. The

requirements of gas motors for the combustion of the residual gas were taken into account.

2. Increasing the throughput through the FT reaction chamber reduces costs for catalysts, but – on the other hand – reduces the specific yield, i.e. leads to a higher fraction of residual gas. The work in this project consisted in searching the optimum throughput, taking into account the entire production chain.
3. The long-chain hydro-carbons coming out of the FT synthesis can be used as basic material for the production of synthetic lubricants. European lubricant producers import such basic materials at present from South Africa for instance. Within this project, contacts to these lubricant producers were searched for defining quality requirements, thus opening the option of non-energetic use which eventually could improve the overall cost-effectiveness.
4. The influence of load fluctuations and part load operation on FT raw product yield and its composition was studied.

Any future industrial implementation of BTL production will require a high degree of automation for ensuring low end-product costs. The potential for automation was explored within this project. A concept for start-up and systematic testing for preparing continuous operation was developed.

3.3 Task 1.1.3: Optimization of FT-upgrading I: catalyst evaluation and selection in a bench scale unit

CERTH has completely characterised the raw FT Wax feedstock using all standard fuel quality control techniques (density, distillation, viscosity, flash and pour point, CFPP, water content, sulfur, nitrogen). Moreover, the detailed hydrocarbon analysis has taken place with gas chromatography and mass spectroscopy.

Many commercial mild hydrocracking catalysts are available today, tailored to specific needs. These catalysts differ in metal features, which influence the cracking, hydrogenation and isomerisation and thus the spectrum of the final product. For the upgrading of FT Wax by mild hydrocracking process CERTH proceeded to the evaluation of 3 commercial catalysts. The target of this evaluation study was the selection of the best catalyst for maximum diesel production. The work was performed in an existing HYD pilot plant unit located in CERTH facilities after major modifications for wax handling. From this work the best catalyst (Catalyst B) and the best conditions for wax hydrocracking (towards diesel maximization) were selected in order to be used for pilot fuel production (WP1.2)

3.4 Task 1.1.4: Optimization of FT-upgrading II: optimization of process design and parameters

The UET pilot plant is designed to produce a FT raw product which is refined in a separate part of the BTL production chain. Essentially, the FT raw product is distilled. According to the specific requirement of the final product users, represented within this project by the automobile industry partners, the boiling points can be freely defined and even products from different boiling ranges can be mixed afterwards. The work consisted in the development of a concept and hardware for bringing out, stocking and transporting the bottom products. Existing solutions were tested for their suitability and were adapted and improved where necessary.

3.5 Task 1.1.5: Economic, environmental and safety analysis

Based on the results of UET's α -plant test operation and the design of the β -plant, the heat and mass balances for a 500 MW BTL facility was developed. UET/CHOREN's Carbo-V-gasifier was combined with a Cobald-based Fischer-Tropsch/Upgrading unit link with the required gas conditioning steps.

The following scenarios were balanced:

- Starting point (SP),
 - feedstock: willow
 - feedstock: straw
- Maximum biofuel, feedstock: willow

The results together with a process description were supplied as input data for the assesemts in SP 5. The process description is reported in detail in the deliverables of task 5.4.2 in SP5.

Table 3-1 shows the key figures for the scenarios.

Table 3-1: Key figures for UET BTL scenarios

		scenario		
		SP, willow	SP, straw	Max. BTL
feedstock	t_{ad}/h	103,4	107,95	103,4
	MW	499,0	499,9	499,0
auxiliary Energy	MW			489,0
BTL output	t/h	21,8	21,6	44,2
	MW	267,9	265,3	542,9
power	MW	6,0	7,4	0,0
conversion rate	t_{BTL}/t_{Feed}	21,1%	20,0%	42,8%
efficiency	BTL	53,7%	53,1%	54,9%
	BTL+ power	54,9%	54,5%	54,9%

For the use of the surplus steam either power prodction or the use as process heat/district heating is possible. The table indicates the max. power prodction and the max. steam supply as an alternative. For CHP-applications any figure between is feasible.

The results of the heat and mass balances together were feed into the questionnaire prepared by SP5 as base of the environmental analysis (life cycle analysis LCA).

Based on the process design and the heat and mass flows, a preliminary cost estimate was provided to SP5.3, were the all processes were economically assessed. Comments and hints on assesement methodology and commercial framework were provided to SP5.3 in various workshops and interviews.

Analysis of risks for accidents along the production chain and potential to avoid them or to reduce the impact in the case of an accident were completed. This includes a thorough analysis of the plant, the process, the materials and material flows as well as a detailed investigation of sources of potential hazards such as CO, explosive gas or dust/gas mixtures, high pressure vessels, etc. Further, different external sources of hazards were checked. The investigation followed largely the prescriptions of the relevant German regulatory framework because the first few industrial plants are expected to be set up in Germany. However, the principle of this investigation will be applicable for plants to be set up in other EU countries and the differences to be taken into account will be much more due to the further development of the regulatory framework in the forthcoming years and the development of the BTL production technology than due to the differences between different countries. Additional input for deeper specification and explanation and to support the following evaluation process was provided at the beginning of 2006.

In the 4th year of the project, the evaluation of high value market opportunities for BTL naphtha was amended. The objective of this task was to offer a clear perspective for naphtha as an important by-product of BTL Diesel and thus a success factor for BTL market implementation. Several opportunities were considered. It can be concluded, that

- A refinery offers several opportunities to blend a limited amount of BTL naphtha as a green component to gasoline.
- Several technical solutions to increase the naphtha quality, exp. the oktane No. are available.
- various future opportunities for both chemical and powertrain application are under development.
- BTL naphtha offers the opportunity to both greenhouse gas reduction and decrease dependency on imported oil.

4 Pilot fuel production (WP1.2)

The objective of this WP was the production of large amounts high quality FT biofuels, sufficient for engine tests (see WP1.3; IP NICE), in a pilot plant. The workpackage covered pilot BTL fuel production according to the hardware and process design defined within WP1.1.

4.1 Task 1.2.1: Production of FT raw liquid

Each production period comprised the following procedures:

1. Starting and warming-up of plant (about 24 hours)
2. Pilot production according to specific experimental programme (24h/24h)
3. Continuous assessment of experimental data
4. Sampling of products and parameters as base of material and energy balances
5. Cooling down of pilot plant (about 24 hours)
6. Investigation of all plant components
7. Analysis and interpretation of measurement data
8. Analysis of samples in laboratory (external)
9. Formulation of research questions for next pilot production period

4.2 Task 1.2.2: Up-grading of FT raw liquid to refined fuels with specific characteristics by UET

The FT crude liquids produced within Task 1.2.1 were up-graded at the UET facilities in Freiberg to fuels that could be directly used or blended with conventional fuels for use in existing motors. For this purpose, the FT crude liquid were first distilled within pre-defined boiling limits. Through variation of the boiling limits and blending of different distilling fractions, the fuel composition and its quality was determined.

Apart from larger testing samples sent to the automobile industry partners, small samples of 10 litres were submitted to ITN for analysis pre-checking (see chap. 4.4 and 5 below).

4.3 Task 1.2.3: Up-grading of FT wax by hydro-cracking

With the best catalyst from Task 1.1.3, long duration experiments took place in the CERTH HYD pilot plant. The plant was operated in a one-through-mode. As a result, more than 900 lit of Wax upgrading liquids were produced contained a wide spectrum of products (naphta, kerosene, diesel and a rest of waxes).

Because CERTH does not operate a distillation unit of the required size, UET agreed to distille the product mixture produced by CERTH fullfilling the specification from VW.

4.4 Production of BTL for motor tests

During the programme of experiments (report as Deliverable (DEL) SP1 WP2 1-12-05-04-18 UET), which took place from 25 September – 14 December 2004, the BTL

fuels required for the experiments by the project partners, DC and VW, were first manufactured. As is evident from that report, the complete BTL process was operated, ranging from the gasification of wood-based biomass into synthesis gas up to the stage where the purified and compressed synthesis gas is converted into liquid hydrocarbons using the Fischer-Tropsch process.

The hydrocarbons, from which water and any residual gas have been removed, were then processed in the next stage by distillation in line with the specifications laid down by the project partners.

However, these fractions, which are generally called "straight-run diesel", still contain fairly small amounts of unsaturated hydrocarbons and oxo-components, such as alcohols and aldehydes. The experiments performed in August 2005 (DEL SP1 WP2 1-19-06-02-08 UET) examined to what extent these substances could be removed by hydrotreating (with specific catalysts) and how the ratio of n and iso paraffins could be improved to provide greater efficiency and advanced cold flow properties.

The following tables contain a list of all the fuel fractions manufactured within the confines of this project.

Table 4-1: Fractions produced for VW

Description	Boiling point in °C T5% - T95%	Sample	Amount in l	Del. no.	Test application/ engine/blending rate (to be added by VW)
BTL cerosine high	194 – 267	P7	600	1.6.1	Test as neat fuel
BTL cerosine	166 – 204	P10	600	1.6.2	Test as neat fuel
BTL diesel	220 – 314	P11	600	1.6.3	Test as neat fuel

Table 4-2: Fractions produced for DC

Description	Boiling point in °C T5% - T95%	Sample	Amount in l	Del. no.	Test application
BTL cerosine high	145 – 263	P3	300	1.7.1 – 1	Test as neat fuel
BTL diesel wide range	166 – 291	P4	300	1.7.1 – 2	Test as neat fuel
BTL cerosine high	194 – 267	P7	600	1.7.2	Blends with conventional fuel and oxygenates
BTL diesel wide range (HC)	149 – 285	P9	600	1.7.3	Blends with straight run UET-BTL

Tables 4-1 and 4-2 illustrate that the fuels largely differ in the boiling ranges that are required. Further analysis data is added to these in table 3. The parameters, such as the cetane number, the flash point, the density and Cold Filter Plugging Point, can be largely correlated using the selected boiling ranges. The remarkably good cold flow properties are the result of the oxo-components still present in the fuel. They

clearly slow down the formation of paraffin crystals at low temperatures. As was demonstrated in the DEL SP1 WP2 1-19-06-02-08 UET report, the catalysts that were tested to improve the fuel's behaviour in cold conditions were only marginally effective.

Table 4-3: Summary of important analysis figures

Parameters	Unit	P3	P4	P7	P9	P10	P11
5 % (V/V) recovered at	°C	145	166	194	149,5	166	220
95 % (V/V) recovered at	°C	263	291	267,5	285,5	204,5	314
Olefins content	% (V/V)	11,7	8,8	13,7	7,8	16,7	8,4
Aromatics content	% (V/V)	0,1	0	0	0	0	0
Sulphur content	mg/kg	4	4	4,1	2,7	2,8	3,3
Characteristics							
Cetane number	-	74	76	77	66,4	70,2	80,2
Cold Filter Plugging Point (CFPP)	°C	-24	-14	-18	-24	-44	0
Flash point	°C	31	35	69	5	47	64
Density at 15 °C	kg/m ³	747,5	756,5	758,1	738,8	740,8	769,2
H/C							
Calorific value	kJ/kg	42725	43655	43930	43765	43418	43933
Lubricity, corrected wear scar diameter (wsd 1.4, at 60°C)	µm	468	401	499	273	473	415
Others							

5 Motor test results of BtL and BtL/ diesel blends (WP1.3)

The objective was to characterise the produced BTL fuels and deduce recommendations for future biofuel (blends). This was done through (1) laboratory analysis (serving also to adjust the pilot production in WP1.2 and to select fuels to be tested) and (2) engine tests.

5.1 Task 1.3.1: Concept phase and laboratory tests

5.1.1 Concept Phase

In the concept phase the basic test procedures were defined. Special emphasis was put on the fuel specifications. The requirements for the engine and vehicle tests were fixed and discussed. As the tests were carried out with fuels having new compositions and properties, a wide range of analysis were necessary. The exact specifications of the to be produced BTL fuels were also be defined in the concept phase and the links between RENEW and the projects NOE and NICE were fine-tuned.

5.1.2 Detailing the experimentation programme

The arrangement and the content of tests were defined in detail. The exact schedule was fixed jointly with the project partners and the different research departments. The different fuels, pure and as well as blended with conventional fuels, were tested. These new fuels were also evaluated with regard to their properties and performances. The main objectives of this activity was the determination of the required specifications of the fuels and their quality standards. The link to the project NICE was also of great importance and has been defined.

5.1.3 Basic Tests

Before the tests could be carried out, it was essential to define the test procedures as well as to fix the design and assembly of the necessary test equipment. The non-standard test equipment had to be acquired and assembled. The special equipment for the application of the APCRS system had to be built up and tested as well. With this new equipment the first tests were carried out to check the complete system.

After these checks were successful, the tests on the engine test bench have started. During these basic tests the performance of the engine with six different fuels was tested. For the evaluation of the fuels, standard test procedures were applied. In this first step, the engine data of the standard production setting were measured. The results of these tests were documented and were the basis for all subsequent tests.

5.1.4 Fuels physic-chemical properties analysis and performance tests

The following properties have been determined for each of the tested fuels: (1) Composition (determination of hydrocarbons types and elements /aromatics content/, sulphur etc.), (2) volatility (distillation, flash point, residue, etc.), (3) fluidity (viscosity, low temperature properties), (4) combustion (specific energy, Cetane Number), (5) corrosion, (6) stability, (7) contaminants, (8) lubricity, (9) foaming characteristics, (10) water reaction, (11) elastomer compatibility, (12) other analysis and tests according to necessities.

Input: 10 l sample of each fuel, 17 different fuel samples in total.

5.1.5 Determination, working out and investigation of additives for new fuels

These activities comprised: (1) Selection of necessary components of additives packages to improve fuel properties, (2) examination of selected additives, (3) investigations of additive packages and additized fuels, (4) other analysis and tests according to necessities (e.g. compatibility of fuel package with engine oils selected to engine tests).

Based on a first 50 liter sample, tests were performed to improve the cold flow properties via additivation. Based on the results of the ongoing analysis, it was discussed between ITN and UET what kind of additional investigation are useful. Another 50 liter fuel sample was supplied to ITN.

5.2 Long-duration tests by automobile manufacturers

The objectives in SP 1 of the RENEW project were to develop optimised fuel specifications for future BtL-fuels. If available, these BtL-fuels will be used in conventional diesel as well as in forthcoming HCCI engines. Therefore, the behaviour of the fuels in an engine has to be determined in detail. The influence of certain fuel properties like cetane number, boiling range, density and viscosity on the engine behaviour and relevant parameters such as fuel consumption and emissions has to be assessed.

The general approach within this task was to assess BtL use in engines in terms of emissions and engine performance and to derive the potential for future use of BtL from that. The work was conducted with three different engines. The conventional diesel combustion was looked at by DaimlerChrysler using an engine with a common rail injection system and by Volkswagen using a unit injector injection system. The third part was conducted through by Renault/REGIENOV and consisted in looking at the potential in terms of new combustion concepts, i.e. homogeneous charge compression ignition (HCCI).

5.3 Task 1.3.2: Long-duration tests by VW

In this section, the results of engine tests by Volkswagen are presented. Three different BtL-samples were delivered by UET, differing mainly in terms of boiling range (Table 4-1), and were tested in a 2 liter diesel engine with a unit injector injection system. The nominal power of the engine was 103 kW. The tests were carried out mainly in the second and third project year and comprised the following investigations:

- ⇒ The impact of the BtL fuels on limited emissions was determined. Limited emissions are hydrocarbons (HC), carbon monoxide (CO), nitrogen oxides (NOx) and particulate matter (PM). For that purpose, different data sets for the motor software control were developed for determining the upper limit of the potential for emission reduction without changes in the engine hardware. Finally, two parameters have been adjusted: the start of injection and the exhaust gas recirculation (EGR) rate. The adjustment was solely by changing the values in the engine control unit (ECU), i.e. by a software modification.
- ⇒ The ignition delay was described carefully by conducting several tests in a combustion research unit (CRU).

- ⇒ The impact of BtL fuels on non-limited emissions was measured without change of the ECU nor the engine hardware, but with the conventional engine and software parameter data set. Non-limit emissions are among others ozone formation potential and polycyclic aromatic hydrocarbons (PAH).
- ⇒ Assessment of the influence of the ignition delay, described by the Cetan no., on the emissions.

The emission tests, including the development of special data sets, have been elaborated on an engine test bench and through chassis dynamometer tests with a Golf V vehicle. The New European Driving Cycle (NEDC) was simulated in all emission tests on the dynamometer test bench. Engine and vehicle tests were performed successfully as well as measurements concerning non-limited emissions and the determination of the ignition delay in dependence on temperature and pressure. A report of the results of two out of three fuels was prepared and submitted (D 1.14.1-1).

Then, all results including the third fuel were assessed carefully and a detailed report was made available (D 1.14.1-2). The essentials of that report are summarized hereafter.

Emission reduction potential of BtL fuels

Figure 5-1 shows the potential of NO_x and particle emission reduction of fuels samples labelled P 7, P 10, P 11 and the mineral oil based kerosene. The mineral oil based kerosene was taken as a reference to the rather kerosene like BtL fuel P10. For the diesel-like BtL fuels P7 and P10 CEC reference diesel was taken. The values are compared to the corresponding CEC diesel emissions and the ratio of both is shown in Figure 1.

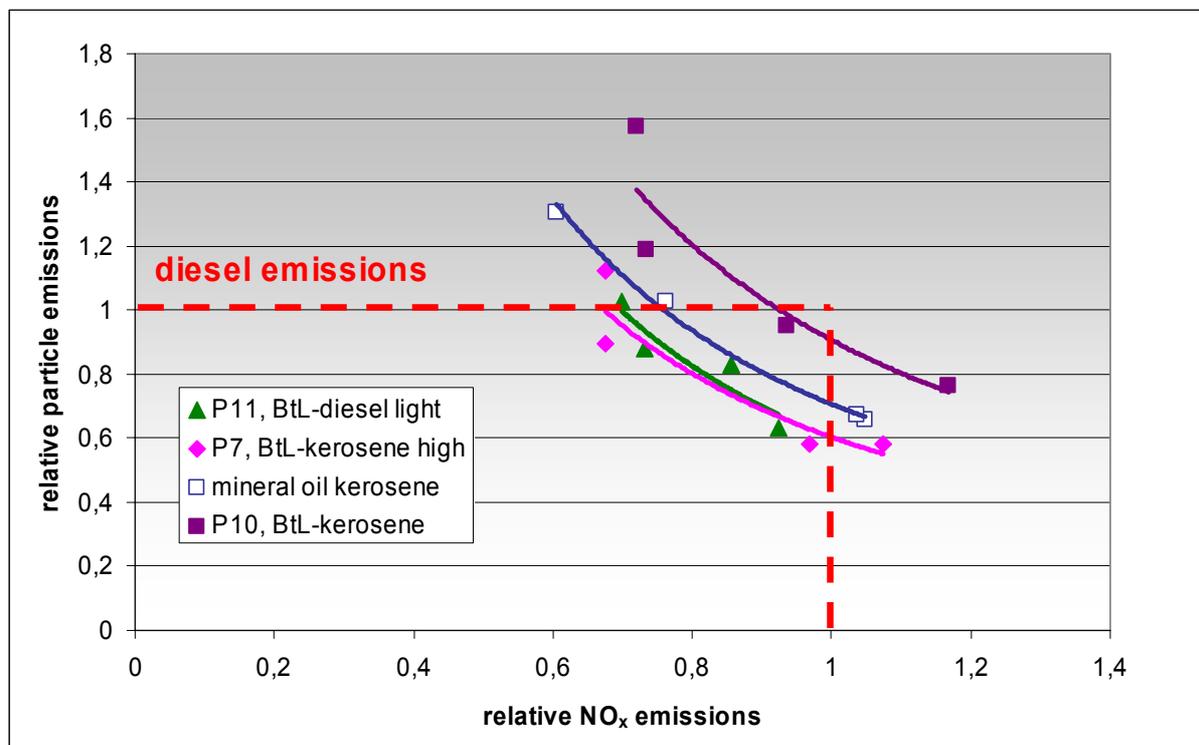


Figure 5-1: Particle-NO_x –trade-off

Values higher than one of either NO_x or particulate emissions are observed for some parameter values because the emissions of the fuels reach the EU IV limits very closely. The emissions of a standard diesel application have a certain distance to the EU IV limits.

However, figure 5-1 shows a possible reduction of particle emissions by almost 45 % and NO_x emissions by 30 %. These values can be achieved for the optimised engine application for particles or NO_x respectively. A suitable data set for a series application would be between these values. Furthermore, BtL-diesel (P 11) and BtL-kerosene high (P 7) have obviously not a significant difference in terms of emissions. In contrast to this, the mineral oil-based kerosene has a smaller potential referring to the emission reduction in a conventional diesel engine.

But the most astonishing finding is that the BtL kerosene (P 10) is much worse in terms of the particle and NO_x emissions than the mineral oil based kerosene. This difference is surprising because the fuels differ only significantly in terms of cetane number (i.e. n-paraffin content) and olefin content. One reason for the difference in terms of these emissions could be the different n-paraffin content. The high n-paraffin content of P 10 leads to a slow and delayed combustion, as the combustion analysis has shown. The delayed combustion implies an increase in particles as well as HC- and CO-emissions. However, it is not finally clear, whether the combustion differs in fact from P 10. At least the n-paraffin content is much lower, so that it can be assumed that the combustion is not as slow and delayed as the combustion of P 10. Further, for the assessment of these results it has to be taken into account that both fuels (P 10 and the mineral oil kerosene) lead to a severe nozzle coking. Nozzle coking has an impact on the injection rate and even more important on the spray behaviour. Hence, it is not clear whether the measured values are really comparable. The following assessments of P 10 and the mineral oil based kerosene have therefore to be taken with some cautiousness.

In figure 5-2 to 5-5 the results are depicted, which were achieved with different data sets in the engine control unit. The different data sets are marked as follows:

- A: data set of series, no adjustment
- B: data set with adjusted injection rate
- C: data set optimised for NO_x-reduction
- D: data set optimised for particle-reduction

For all fuels, a reduction of particle emissions occurs even without any engine optimisations (A). This goes along with the well known particle-NO_x-Trade-Off relationship since for case (A) the NO_x emissions increase and exceed even the emissions that occur with a conventional diesel fuel. This is due to the lower density due to which more fuel has to be injected for achieving the same power. Hence, a higher load point is chosen in the engine map which is characterised by a lower EGR. The lower EGR leads to higher temperatures and with it to higher NO_x emissions.

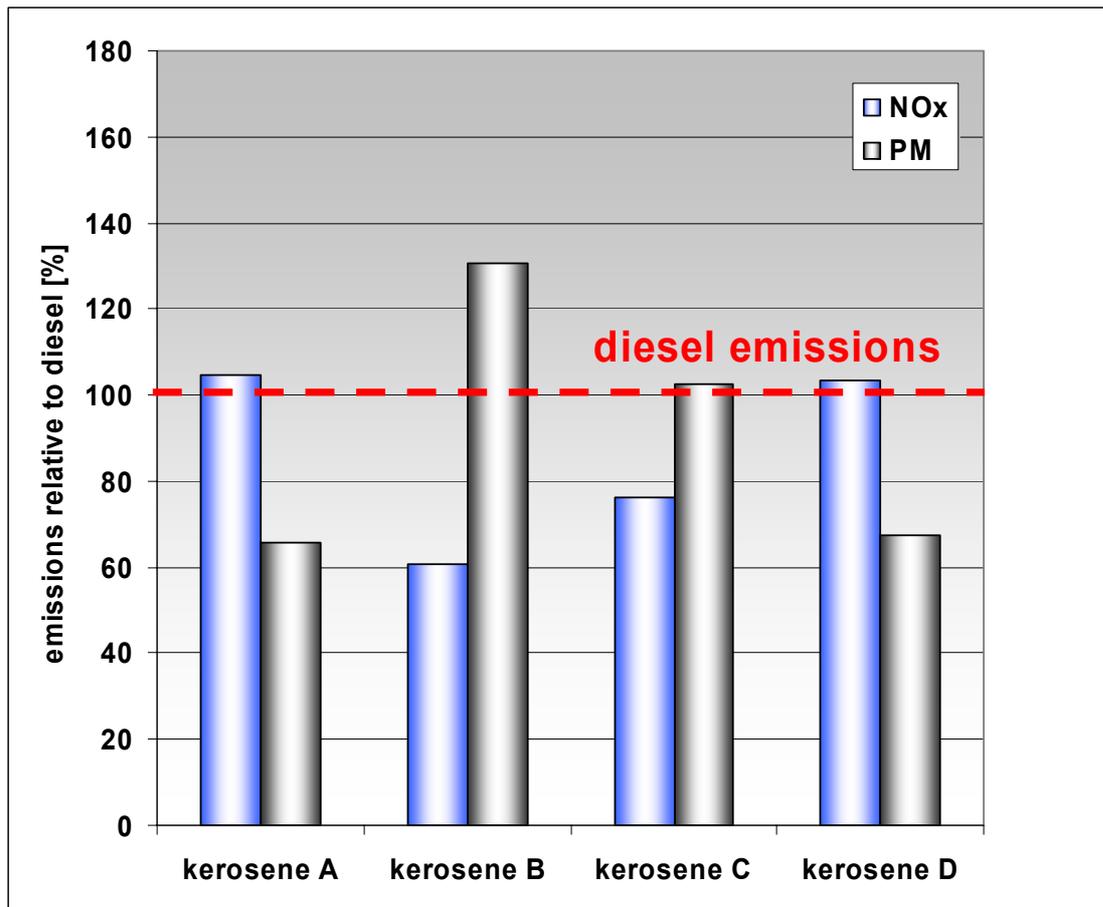


Figure 5-2: Relative emissions of *mineral oil based kerosene* for different data sets in the engine control unit

The high particle emissions of the mineral oil-based kerosene for case B can be ascribed most probably to the small cetane number of 43. In a conventional diesel engine, this leads to a late ignition and a delayed centre of combustion. This explanation is supported by figure 5-6 where the relative HC and CO emissions are depicted. The relative HC and CO emissions are comparably high for the mineral oil based kerosene. High HC and CO emissions indicate a low cetane no. Hence, the combustion is delayed.

However, the mineral oil-based kerosene has still a potential for emission reduction in conventional diesel engine. This is most probably due to the higher volatility which goes along with an improved preparation of the fuel. Additionally the lack of sulphur and the small aromatic content has a positive effect on the particle emissions, as well.

If a high volatility occurs in connection with a high cetane number as it is the case for P10, this does not yield any further improvements. It is even worse as figure 5-1 shows. This can also be seen from figure 5-3 where the results for the different data sets are depicted for P10. The behaviour is actually the same as for the mineral oil-based kerosene. The fuel does not have a potential in terms of particles due to engine optimisation, since the base case is also the best. But additionally, the particle-NO_x-Trade-Off is more extreme leading to a narrower range for optimisation and hence higher emissions. Obviously, the benefits through a high volatility are com-

compensated with the short ignition delay which could lead finally to a worse fuel preparation than with the mineral oil based kerosene.

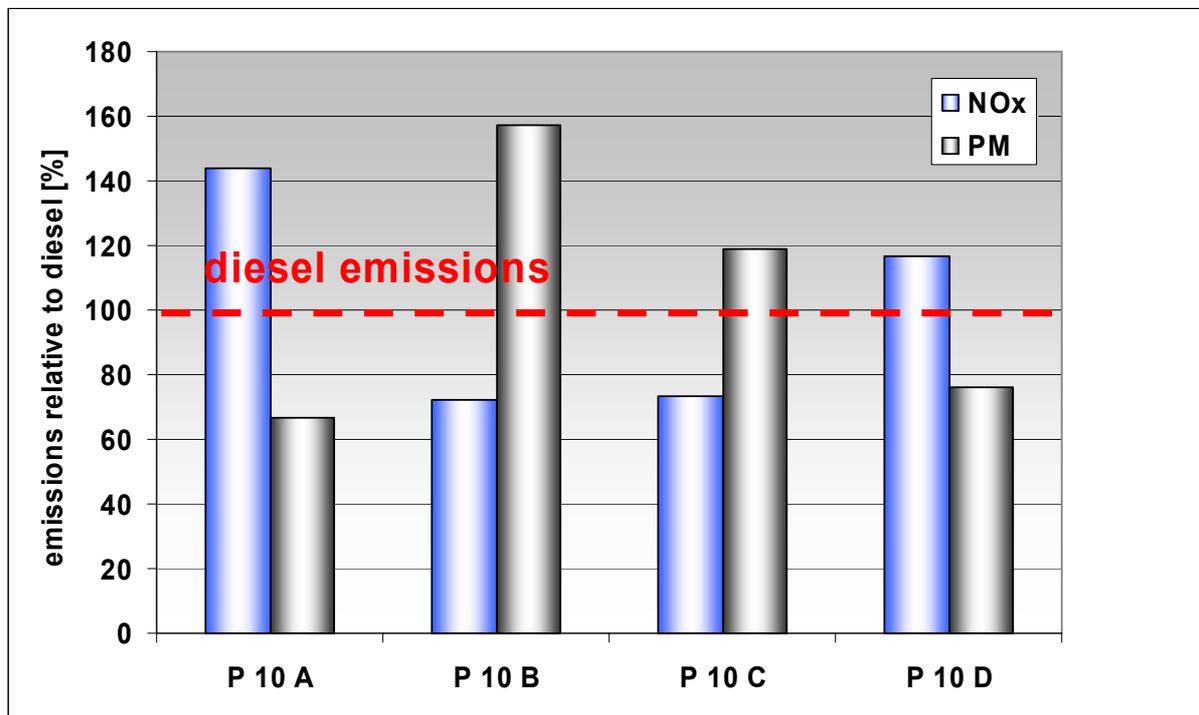


Figure 5-3: Relative emissions of *BtL-kerosene, P 10* for different data sets in the engine control unit

The diesel-like fuels P 7 and P 11 are characterised by the potential of further reductions in particle emissions through engine optimisations. Obviously the adjustment of the injection rate is sufficient to achieve a considerable reduction in the particle and NO_x emissions. Figures 5-4 and 5-5 show only small differences in the reduction of particle and NO_x emissions for the corresponding optimised data sets (C and D). Taking the physical properties of P 7 and P 11 into account, it can be concluded, that a further increase of the cetane number beyond 79 does not lead to a significant improvement in terms of emissions. The higher final boiling point of P 11, which goes along with a worse volatility and even longer n-paraffin, seems to compensate the better ignition behaviour, which is described by the cetane number. The longer n-paraffin of P 11 leads to an even more delayed centre of combustion than for P 7. This can be derived from the combustion analysis.

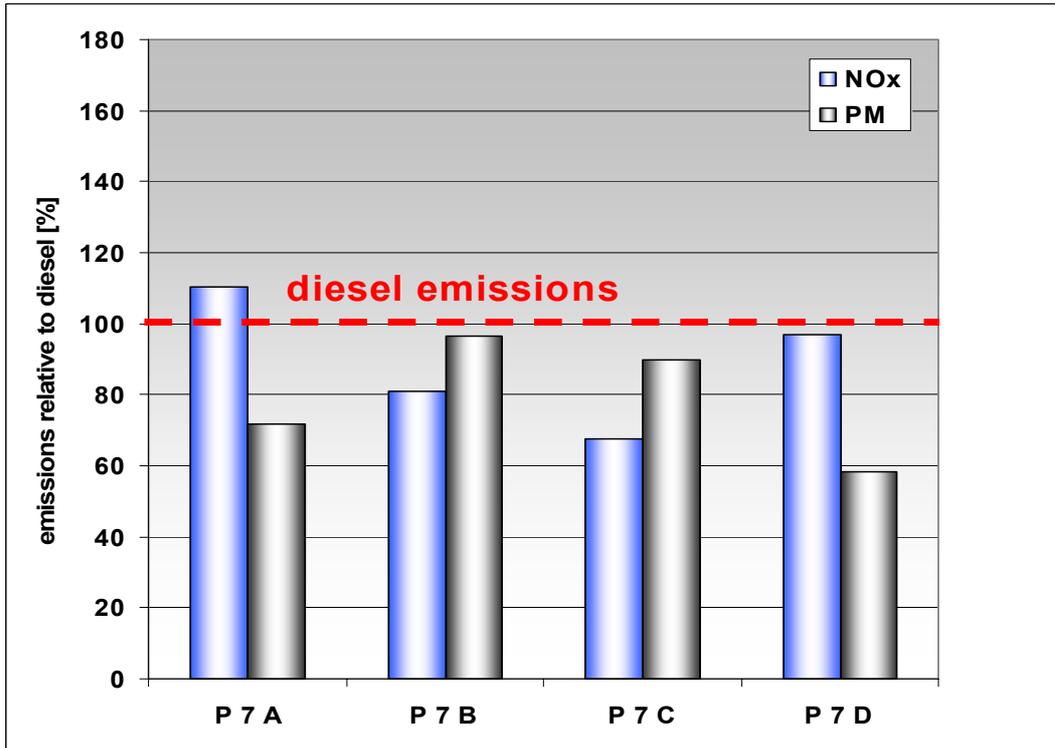


Figure 5-4: Relative emissions of BtL-kerosene high, P 7 for different data sets in the engine control unit

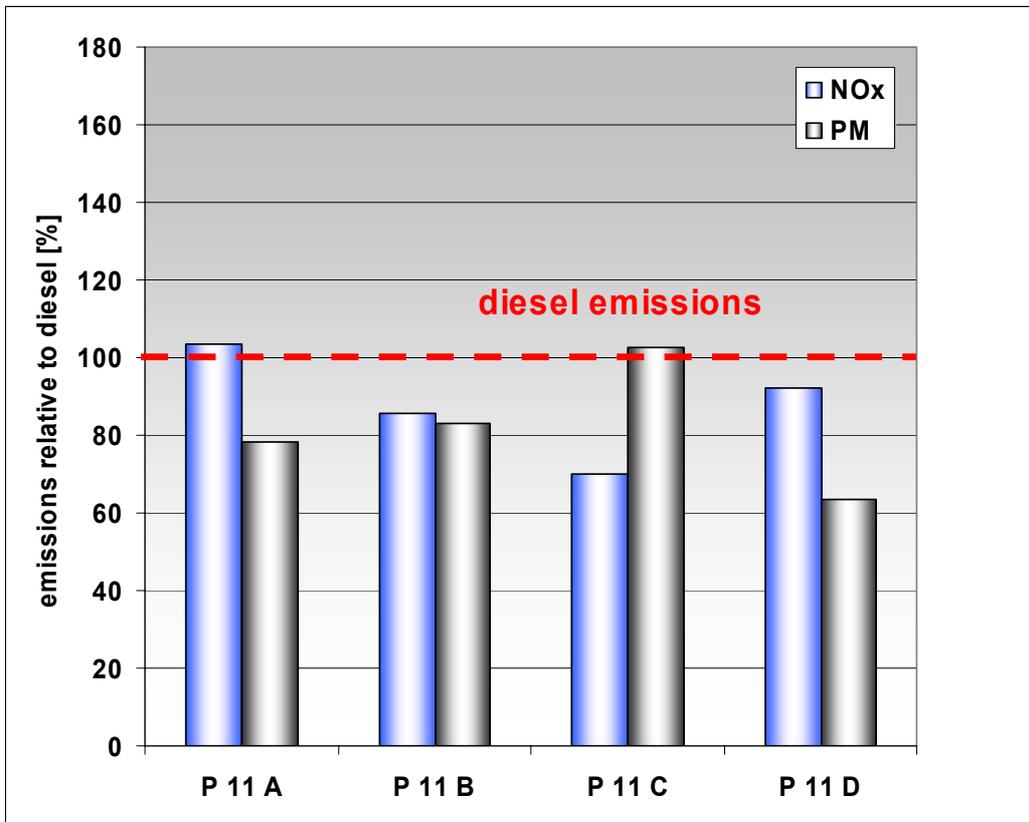


Figure 5-5: Relative emissions of BtL-diesel, P 11 for different data sets in the engine control unit

As last point in terms of emissions, figure 5-6 shows the HC and CO emissions relative to the respective diesel emissions. All values are measured behind the catalytic converter. Either the combustion is more complete with BtL-fuels or the conversion rate of the catalytic converter is higher for these fuels, since all BtL fuels have a huge reduction. Even the mineral oil-based kerosene has less HC and CO emissions than diesel, although they are not as small as for the BtL fuels.

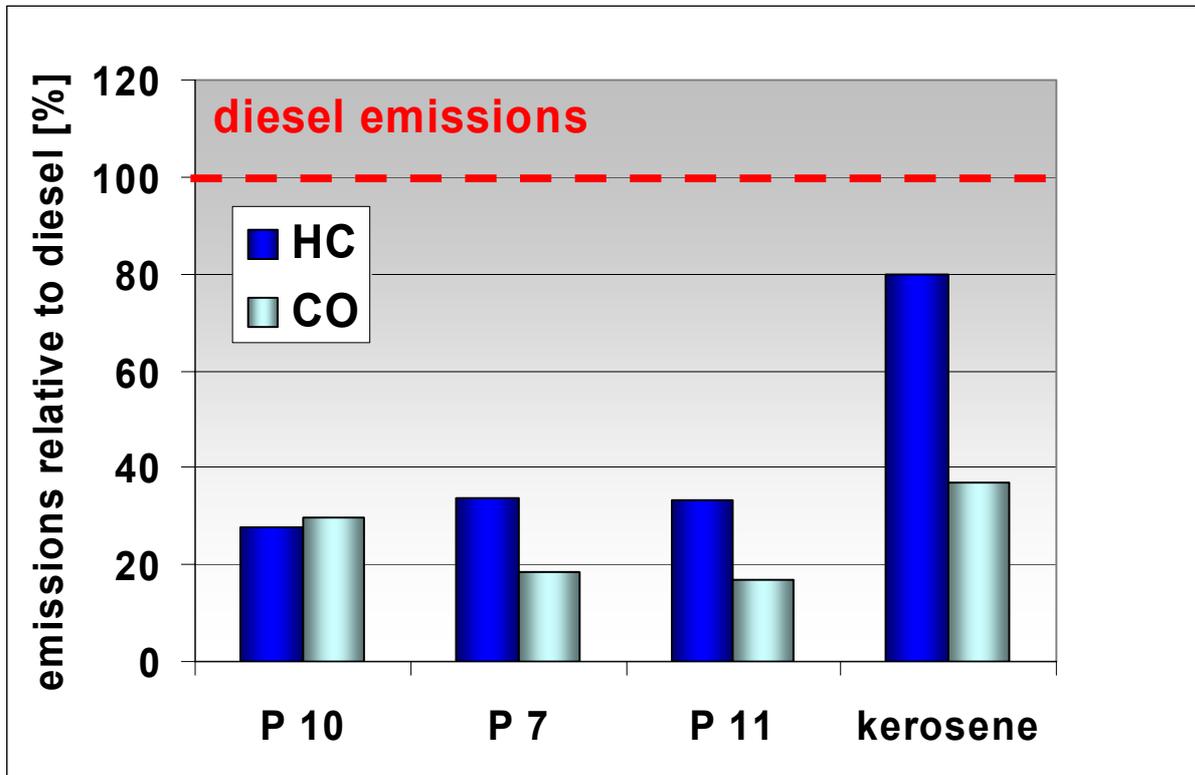


Figure 5-6: HC and CO emissions for different fuels

Finally, the fuel consumption has been looked at for the different applications. Generally speaking, the volumetric fuel consumption for the BtL-fuels is expected to be higher than for mineral oil-based fuels. This is due to the lower volumetric heating value of the fuels. On the other hand the lower heating value on mass bases is higher (P 7: +2.6 %; P 11: +2.8 %) for BtL-fuels than reference diesel due to the higher H/C-ratio. This partly compensates the lower densities of BtL-fuels (P 7: -8.98 %; P 11: -7.78 %), so that the corresponding reductions in volumetric heating values of P 7 and P 11 are 6.6 % and 5.2 %.

In Figure 5-7, the fuel consumption on volume bases is related to the diesel consumption in the NEDC. The increased fuel consumption on volume bases for P 7 is between +5 % and +7 %. Considering the BtL-diesel P11, the results are similar. Here the increase in fuel consumption is between 2.2 % and 5.3 %. For P 10, the fuel consumption is about 5.8 % and 9.6 % higher. Astonishingly, the increase in volumetric fuel consumption of the mineral oil-based kerosene can be as small as 1.6 %.

Hence, generally speaking, the fuel consumption behaves as expected, i.e. due to the lower volumetric heating value the volumetric fuel consumption increases re-

spectively. But on the other hand, it could be shown, that with a proper engine adjustment the increase of volumetric fuel consumption can be limited, as the results of P 11 reveal. Due to the coking behaviour of the kerosenes, the minor fuel increase of 1.6 % is taken as an exception that does not show the truth.

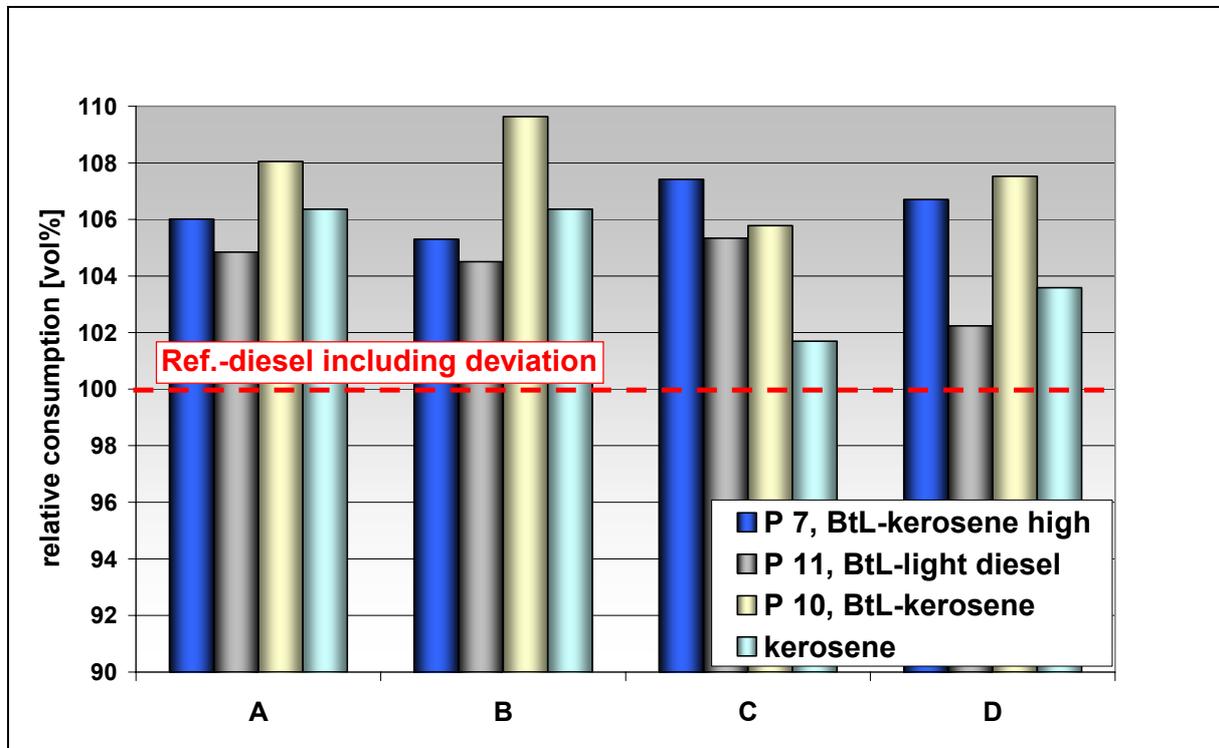


Figure 5-7: Relative fuel consumption (volume based) of different applications in the NEDC

However, the relevant parameter is not the fuel consumption, but the consumption of the energy contained in the fuel. Figure 5-8 shows the respective values. Taking into account the deviations of the measurements the energy consumption is the same for the BtL fuels as for the conventional diesel. That means that the combustion efficiency of the BtL fuels is not significantly different from conventional diesel. It also confirms the above-mentioned conclusion that the increased volumetric fuel consumption is caused by the lower volumetric heating value of the BtL fuels.

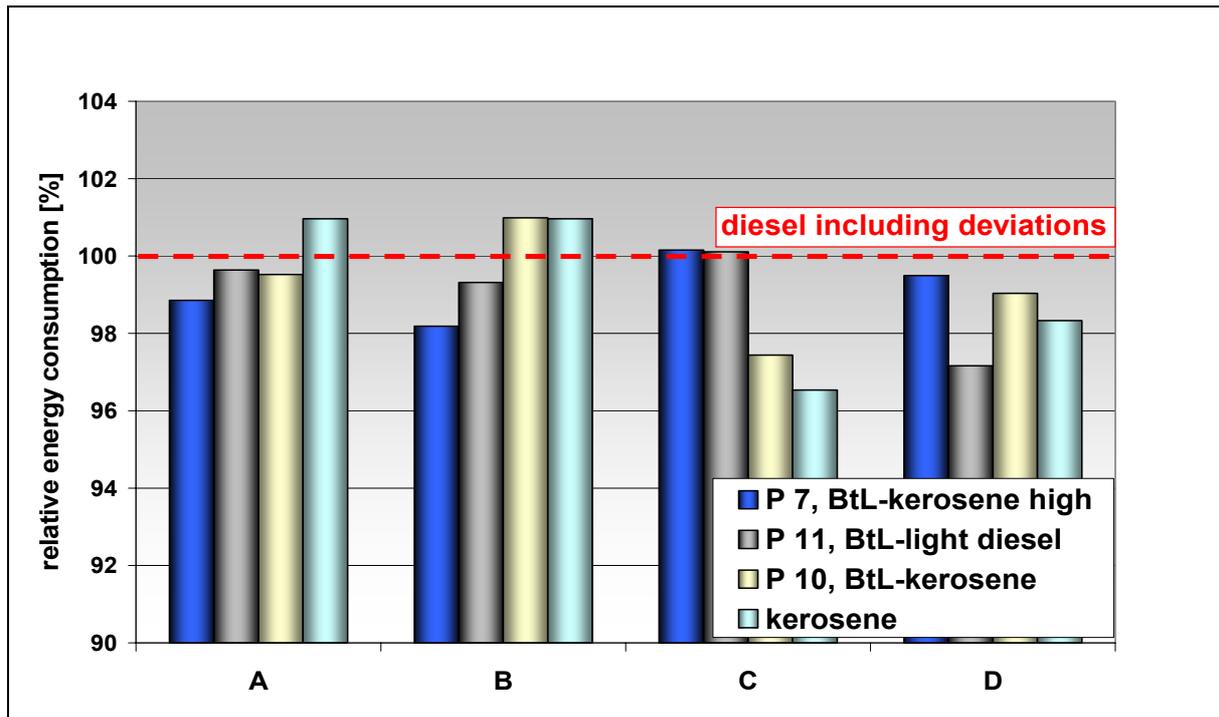


Figure 5-8: Relative fuel consumption (energy based) of different applications in the NEDC

Assessment of ignition delay

The ignition delay was determined with the above mentioned combustion research unit. Unfortunately, the maximum temperature, which is within reach, is 590°C. Therefore, only a low load point (1460 rpm, 20Nm) can be taken for an assessment of the results in terms of the real conditions prevailing in an engine. Figure 15 shows the results of the ignition delay as a function of temperature for a chamber pressure of 40 bar and a pressure in the injection nozzle of 800 bar.

Bearing in mind that a difference in ignition delay of only a few degree crank angle may have a considerable influence on the combustion, the resolution is depicted in degree crank angle (°CA) for an engine speed of 1460 rpm. In Figure 15, only the part close to the actual engine conditions is depicted, which is about 500°C to 600°C. It shows the difference between the ignition delay of diesel and the BtL fuels, i.e. ignition delay (Diesel) minus ignition delay (BtL). Hence, positive values mean a smaller ignition delay than diesel.

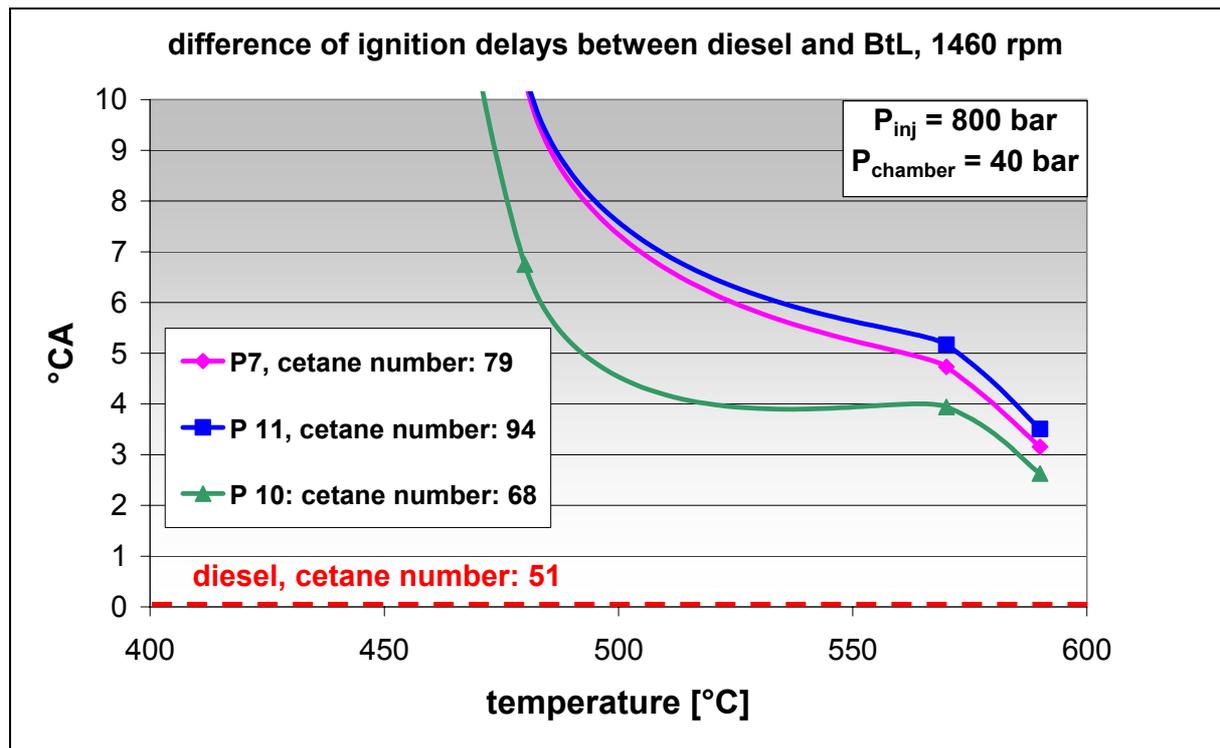


Figure 5-9: Ignition delay versus temperature

Although the differences in the ignition delay are not as big as expected from the huge differences in cetane number (from 55 to 93), all fuels principally behave as it is described by the cetane number. That means that the light diesel (P 11, cetane number of 94) is characterised by the shortest ignition delay followed by the kerosene high (P 7, cetane number of 79) and finally the kerosene (P 10, cetane number of 68). Furthermore, the differences decrease with an increasing temperature, as expected. Close to the actual engine conditions (590°C) at 1460 rpm and 20 Nm, the differences between the BtL fuels are rather small compared to the differences relative to diesel of about 2.5°CA and 3.5°CA. It seems to be the case that the potential benefit in terms of the combustion in the engine due to higher cetane numbers becomes smaller with increasing cetane number.

Here, further investigations are necessary for checking out whether cetane numbers above 70 are really helpful for a pure fuel. However, using fuels with cetane numbers above 70 as a blending component may have positive effects on the base fuel.

Impact on non-limited emissions

All emissions were measured behind the catalytic converter.

As it is known from the results from the limited emissions the hydrocarbon emission (HC) are considerably reduced, if a BtL fuel is used. In addition, it becomes clear that the HC emissions of the BtL fuel consist of the same compounds as the emissions from diesel. Figure 5-10 shows that some aromatics are also detected, although the aromatic content of the BtL fuel is close to zero. These aromatics are created during the combustion reactions and do not stem originally from the fuel.

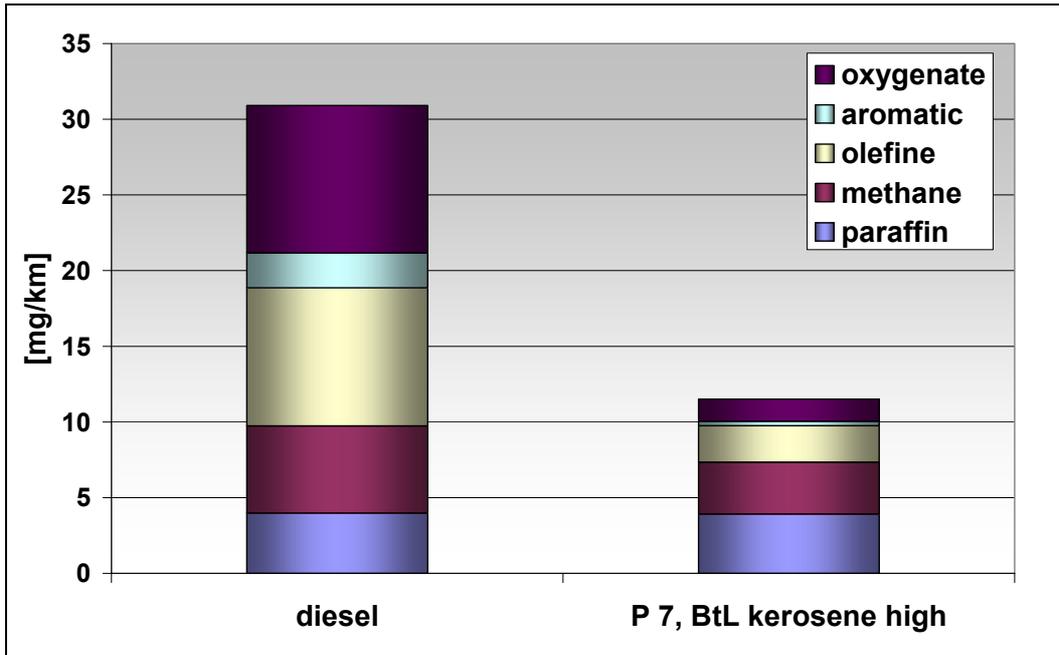


Figure 5-10: HC emissions in the NEDC

Since the ozone formation potential is determined by some of the HC emissions the results depicted in figure 5-11 are not astonishing. BtL fuels lead to considerable reductions in the ozone formation potential even if used as a blending feedstock for fuels.

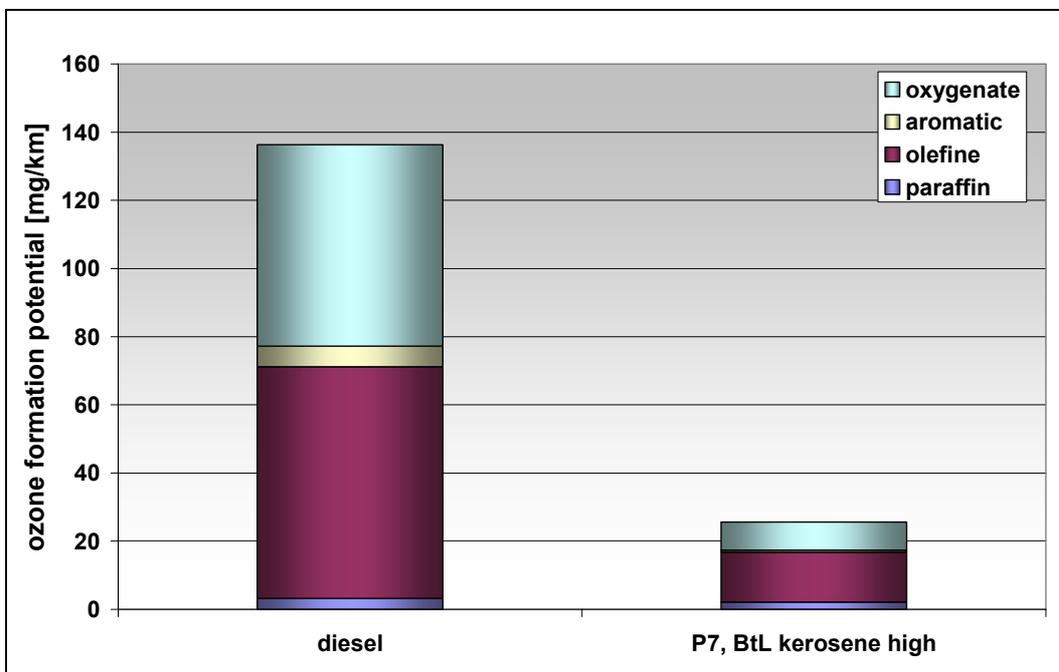


Figure 5-11: Ozone formation potential in the NEDC

Figure 5-12 shows the sum of polycyclic aromatic hydrocarbons (PAH) and two components as examples. As for the other emissions, the BtL fuel lead to a decrease in the sum of cancerous PAH.

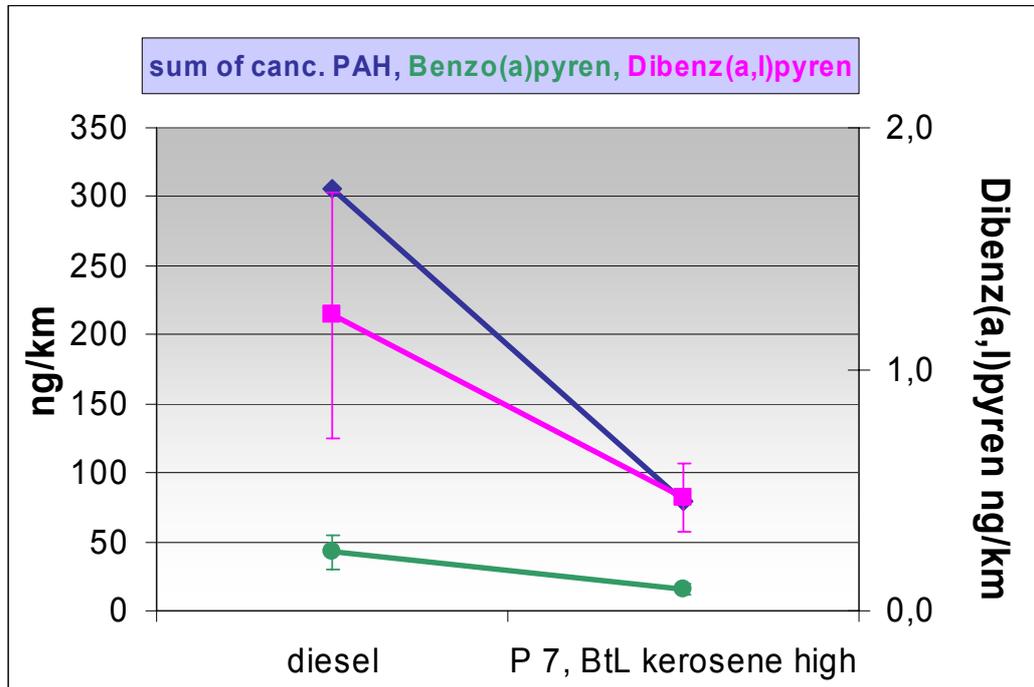


Figure 5-12: Cancerous polycyclic aromatic hydrocarbons (PAH) in the NEDC

In conclusion, it can be said that in terms of non-limited emissions the same is true as for the limited emissions, i.e. that the use of BtL fuel leads to considerable and partly tremendous reductions in emissions. It could be shown, that BtL fuel has a positive effect.

Influence of cetane number

In the third project year (2006), a new test campaign was started which had the assessment of the influence of the cetane number as goal. For it, a BtL fuel from UET (P7) was blended with a low component featuring a low cetane number. With it, fuels with different cetane numbers were mixed. These fuels differed only in the cetane number, since the blending component had a boiling point that was in the boiling range of the base fuel P7. These tests were conducted solely on an engine test bench with the before used engine: 2 liter 4V PD 103kW. The results have been put down in deliverable D 1.14.1-3, detailed report of experiments assessing the influence of the cetane number in a conventional diesel engine. Here, the main results of D 1.14.1-3 are summarized.

For these investigations, BtL fuel (P7) was blended with a component having a very low cetane number, so that the cetane number of the fuel is reduced. By choosing tetra-isobutane as a component only the density is affected besides the cetane no. since the boiling point is within the boiling range of the BtL fuel. In that way, three fuels with different cetane numbers were prepared.

From the investigations of the influence of the Cetan no. in a unit injector engine, the following points can be derived:

- the ignition delay is described by the Cetan no.;
- a high cetan no. (high n-paraffin-content) seems to implicate a longer combustion;
- a longer combustion is rather caused by a retarded injection than by slower combustion of n-paraffin. Therefore and with regard to combustion behaviour, the hydraulic behaviour of the fuel is obviously more decisive than the chemical reactivity of the components for a unit injector engine;
- a comparison between diesel and BtL56 shows again very clearly the potential of BtL fuels in terms of particle and NOx reduction;
- with respect of the influence of Cetan no. on emissions, no clear trend could be shown; hence, a high Cetan no. seems to be not particular beneficial for unit injector engines.

In particular, the last results are surprising because it is generally well known from literature, that a high cetane no. is reducing HC and CO emissions (World Wide Fuel Charter 2006, ACEA, JAMA, EMA). Since this was not found in these engine tests, some vehicle tests were conducted on the roller test bench. Therefore, three fuels of the Cetane no. 65, 60 and 39 had been prepared in the same way as described above. These fuels were then tested sequentially in the vehicle (Golf V) by measuring HC, CO, NOx and particulate matter in NEDC tests without any modification in the engine software or hardware. Figure 13 shows the emission results relative to the diesel emissions.

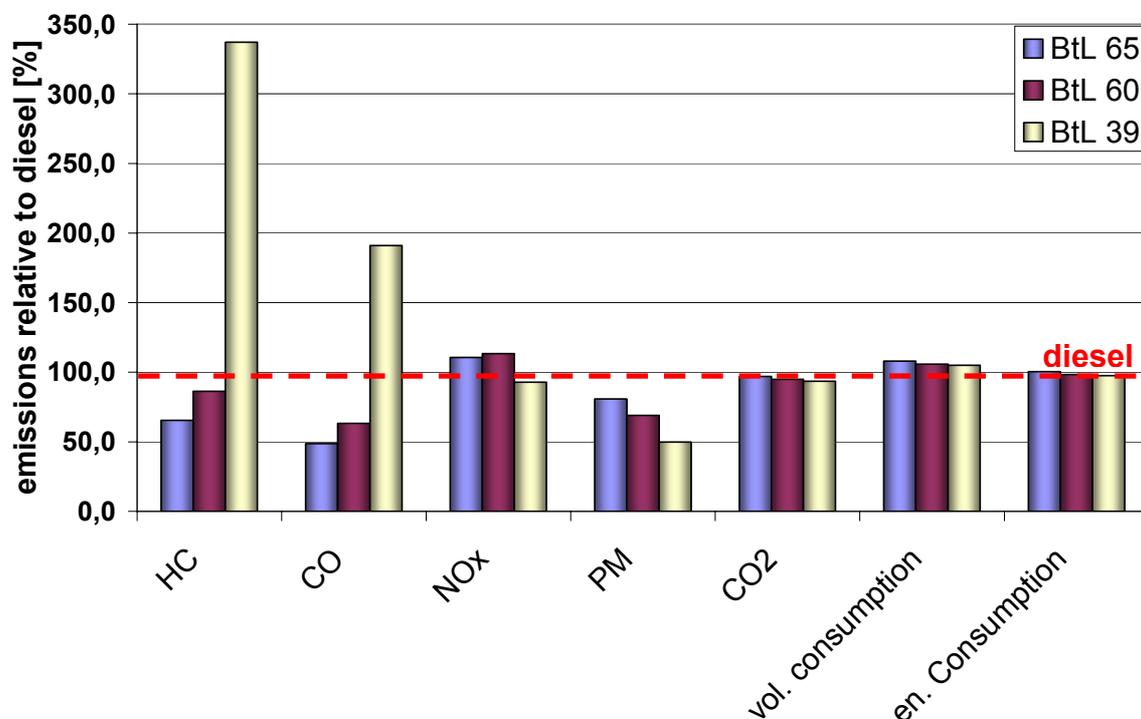


Figure 5-13: Influence of Cetane no. on vehicle emissions in NEDC

It becomes clear that with decreasing Cetane no. the HC and CO emissions increase and the NOx and particulate matter (PM) emissions decrease. A high Cetane

no. implicates a short ignition delay, hence less time for homogenisation of the fuel-air mixture. This leads to a more pronounced diffusive combustion which finally tends to decrease HC and CO but increases NO_x and particulate matter. On the other hand, a low Cetane no. implicates a longer ignition delay, hence more time for homogenisation of the fuel air-mixture. This involves less particles, less NO_x, but more HC and CO emissions. Figure 5 shows exactly that dependency on the Cetane no.

The following conclusions solve the contradiction between the engine test and the vehicle test results.

It seems to be that, in the engine test results the hydraulic behaviour dominates the combustion behaviour, so that the influence of Cetane no. is not obvious anymore. The retarded and longer combustion is due to the fact that in those measurements the same energy input was adjusted for BtL and diesel fuel. In the vehicle tests, however, the unmodified data set was used. Not even the lower volumetric energy density was considered. In such a case, more BtL fuel has to be injected, in order to reach the same power as with conventional diesel fuel, as it was described in Del. 1.14.1-2. In the vehicle the unmodified engine control unit reads this as if a higher power had to be reached. Hence, it alters the start of injection to an earlier point and increases the air flow rate. With the earlier start of injection the retard due the hydraulic behaviour is shaken out and the influence of the Cetane no. becomes obvious.

Finally based on these reports and the results from the partners in WP 1.3, recommendations in terms of most suitable BtL fuels for common and future engines are derived and included in deliverable D1.15.

5.4 Task 1.3.3: Long-duration tests (DC AG)

The objectives in SP1 of the RENEW project were to develop design criteria for optimized BTL fuel **and Diesel engines** running with this BTL fuel to enable improved designs for reaching lowest levels of pollutants in a holistic system approach of fuel and engine development. To this end, detailed experimental test-bed studies on DC passenger car diesel engine with different BTL formulation from CHOREN were performed.

- In the first project year, the work was focused on physical properties of the CHOREN BTL, especially on the influence of boiling range on emissions.
- In the second project year, the emission benefits of blends made BTL and conventional Diesel fuel were evaluated.
- In the third project year, the different behavior of straight run BTL and hydro-cracked BTL were examined. Also blends with biodiesel were tested.

A detailed test program was carried out by DaimlerChrysler in the Research Division, Research Body and Powertrain, Research Lab: Internal Combustion Engines using the most advanced Euro4 Diesel technology. Engine parameters which influence the combustion process and thus emissions and fuel consumption were varied systematically. These parameters were:

- Exhaust gas recirculation rate (EGR)
- Start of main injection (SOMI)

- Start of pilot injection (SOPI)
- Rail pressure (RP)

These engine parameters can be controlled by the Electronic Controlling Unit (ECU) and optimized for any load point (optimized engine map) according to the fuel properties. A detailed description of the engine and the test procedure can be found in the reports (deliverables D1.14.02.01 D1.14.02.02, D1.14.02.03).

Additionally to these bench tests, an optimized vehicle was tested on chassis dynamometer. Vehicle dynamometer test characterize the behaviour of the complete vehicle: vehicle size and weight, engine, gear ratio, after-treatment system. Detailed test procedures are prescribed which makes the test results comparable. The New European Driving Cycle (NEDC) takes into account cold start behaviour, representative driving speed and acceleration.

5.4.1 Tests with straight run fuels 1 and 2, influence of final boiling point

Both straight run UET-BTL fuels have shown significant reduction of CO, HC and Soot (FSN) emissions, compared to EU standard Diesel. The emissions benefits are similar to a state of the art GTL fuel (IFP-GTL-fuel).

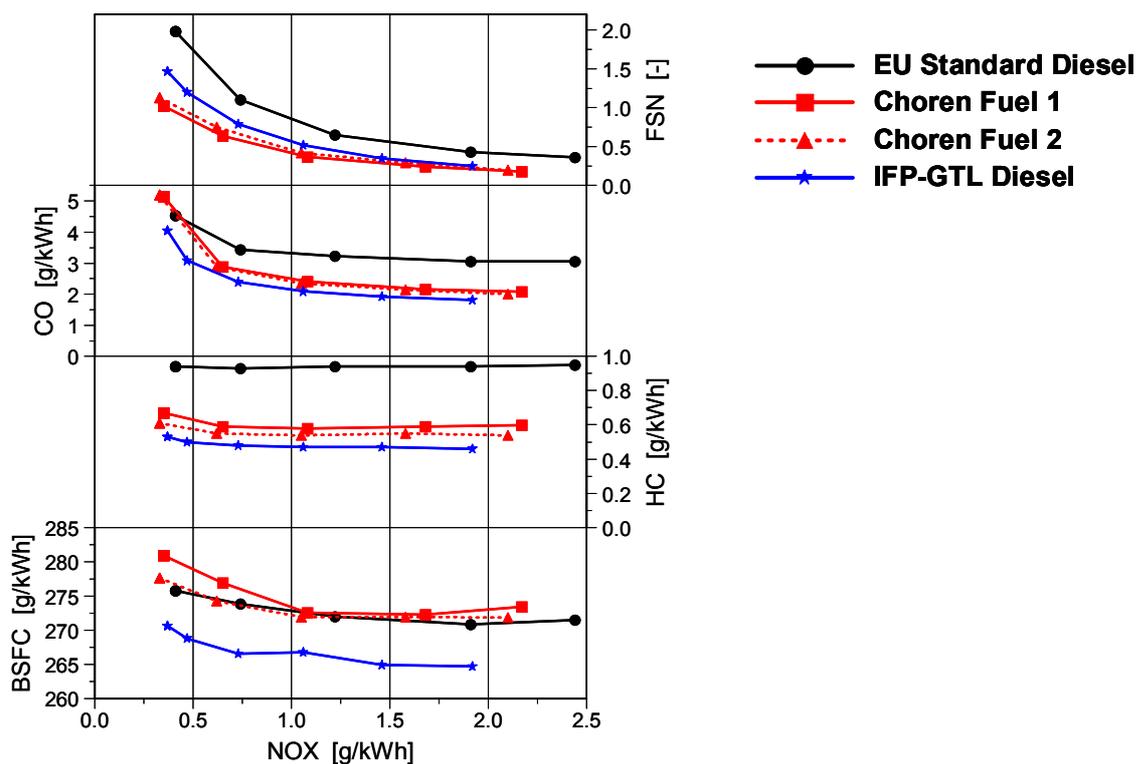


Figure 5-14: Trade-off curves as function of NOx emissions obtained by varying the EGR rate. Operating point: 1600 rpm, 3.3 bar

No significant differences could however be stated between the cerosine high fuel and the wide range BTL. The T95 difference of only 30°C is obviously too small to see increased particulate emissions of the wide range BTL.

5.4.2 Test with different blends with conventional diesel

From earlier tests with GTL fuels there were some hints that the effect of blending synthetic Fischer-Tropsch-Diesel to conventional diesel might be non-linear. The engines in these early tests complied with the former EU 3 emission levels. The GTL fuels were hydro-cracked industrial FT-fuel, whereas the CHOREN fuel is a straight run FT-fuel. So it was of interest to check if more then proportional benefits could also be observed with EU 4 engines and CHOREN BTL.

Blends of 50% BTL, 30%BTL, 15% BTL and 5% BTL were compared to neat BTL and conventional diesel. The main criteria for the comparison were the soot emission reduction at constant NOx level. As observed before, the soot reduction for neat BTL at high load is about 50%. The particulate emission reduction of blends is lower and correlates linearly with the BTL content.

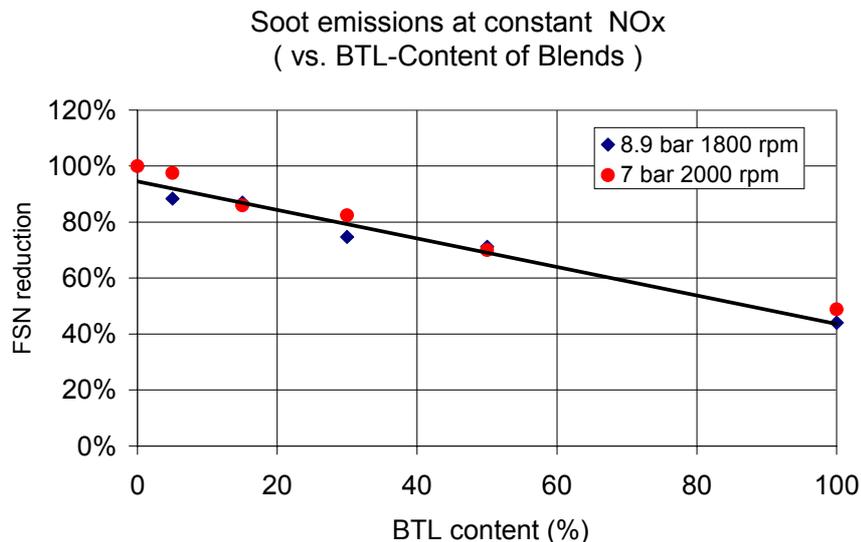


Figure 5-15: Relative soot emission at constant NOx for test points 8,9 bar, 1800 rpm and 7 bar 2000 rpm

5.4.3 Test with hydrocracked BTL

At the end of 2005 UET delivered cracked BTL wax from a small experimental cracking device to DaimlerChrysler. Two blends (50% HC BTL and 20% HC BTL) with straight run BTL were tested in the RENEW project. Analysis of the hydro-cracked BTL showed that the properties were very similar to the straight run fuel. The HC BTL consists mainly of unbranched, saturated alkanes. So no difference in the combustion behaviour could be expected (see figure 14-3).

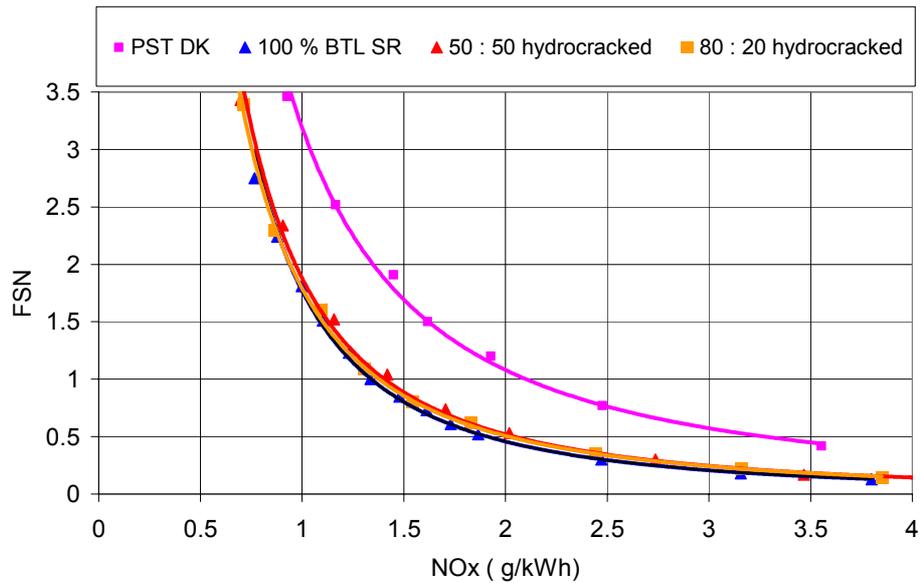


Figure 5-16: NO_x-particulate trade-off at 7 bar BMEP and 2000 rpm

5.4.4 Test with biodiesel – BTL – conventional diesel blends

From an earlier EU project - Optimum Diesel Fuel for Clean Diesel Engines, CLEAN - it is known that oxygen containing fuel components can reduce the soot emission of diesel combustion. FT-diesel fuels show a very low tendency for soot production. It is however interesting whether this inherent benefit of BTL can be improved by addition of oxygenates. As test compound biodiesel has been selected. The main reasons for this choice were:

- biodiesel is now frequently added into conventional diesel fuels
- biodiesel can act as a simple lubricity improver for GTL. Straight run GTL has an insufficient lubricity, which causes excessive wear in the high pressure fuel pumps. Biodiesel could substitute more expensive synthetic lubricant additives.

Two blends were tested:

- 10 % biodiesel + 90 % straight run BTL
- 20 % biodiesel + 80 % straight run BTL

The test results show a small improvement of the FSN (particulate emission) with both biodiesel blends. The 10% blend and the 20% blend behave very similar (see figure 14-4).

It should however pointed out, that due to the high boiling point of the biodiesel components blends with more than 10% will not be suitable for cars with particulate filters

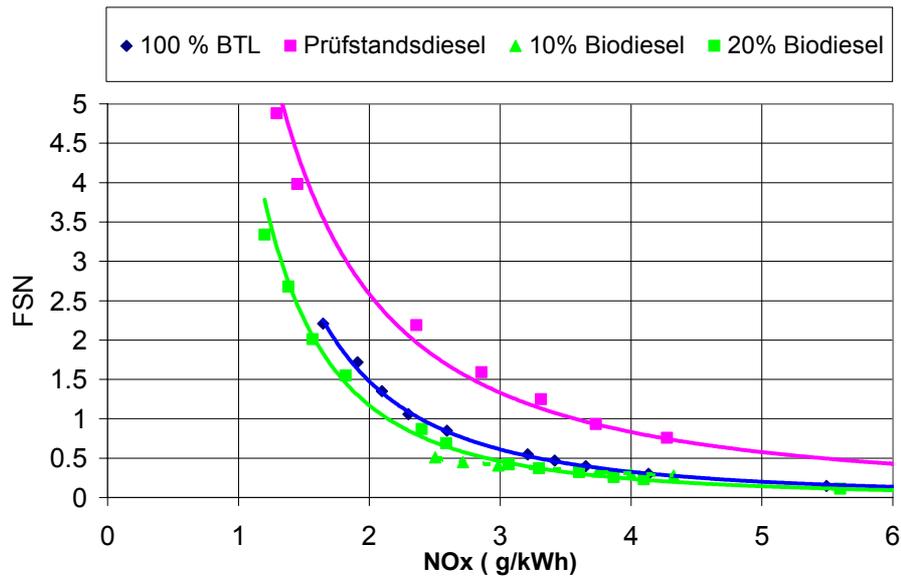


Figure 5-17: NOx-particulate trade-off at 8,9 bar BMEP and 1800 rpm

5.4.5 Vehicle dynamometer tests

Engine test benches are normally used to optimize or to gain a deeper insight of the combustion process whereas the vehicle dynamometer test characterise the behaviour of the complete vehicle: vehicle size and weight, engine, gear ratio, after-treatment system. Vehicle dynamometer test are usually done at the end of an optimization procedure to prove the success.

A Mercedes E class car equipped with a modern 3 liter V6 diesel engine (OM 642) was used for the dynamometer test. The engine uses a common rail injector. Maximum power output is 165 kW. The vehicle complies with the EURO IV exhaust emission standards. An oxidation catalytic converter is used as well as a particulate filter. As DaimlerChrysler now uses particulate filter technology in all new diesel passenger cars, the standard application uses this additional degree of freedom to reduce NOx emissions. The limits of a reasonable NOx reduction are confined by the increase of fuel consumption.

Figure 5-5 shows the relative emissions in the NEDC for the car with particulate filter. BTL shows emission benefits for NOx, CO and HC compared to the conventional fuel. Particulates seem to be slightly enhanced. But this apparent increase is probably an effect of measurement precision. The filter reduces the particulates both for conventional fuel and for BTL fuel to such a low level (0,002 g/km compared to the EU4 limit of 0,025 g/km) that precise measurements are difficult.

The red bars show the emissions relative to the EU4 limits. The impressive reduction of PM, CO and HC is of course assisted by the after-treatment components (oxycat and filter). The comparison to the conventional fuel (blue bars) however shows a significant influence of the BTL fuel. The reduction of NOx is only caused by the fuel.

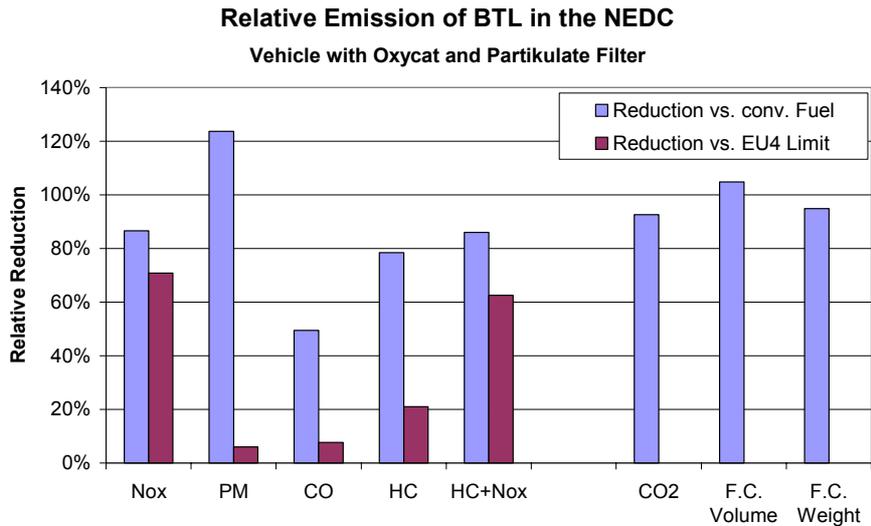


Figure 5-18: Relative Emissions of UET – straight run BTL compared to conventional EN 590 diesel and to EU4 limits for a vehicle with oxidation catalyst and with particulate filter.

To demonstrate the benefits of PM reduction of BTL in the raw gas the particulate filter was removed. Figure 5-6 shows the results. NOx, CO and HC emissions are similar to the results with filter. The PM reduction from the BTL fuel (blue bar) is 28%, which is not as high as the PM – optimized application of Volkswagen. The PM emissions of the car without the particulate filter would not be in the limits of EU4. The UET-BTL fuels show a slight reduction in CO2 – emissions and gravimetric fuel consumption, and an increase of the volumetric fuel consumption.

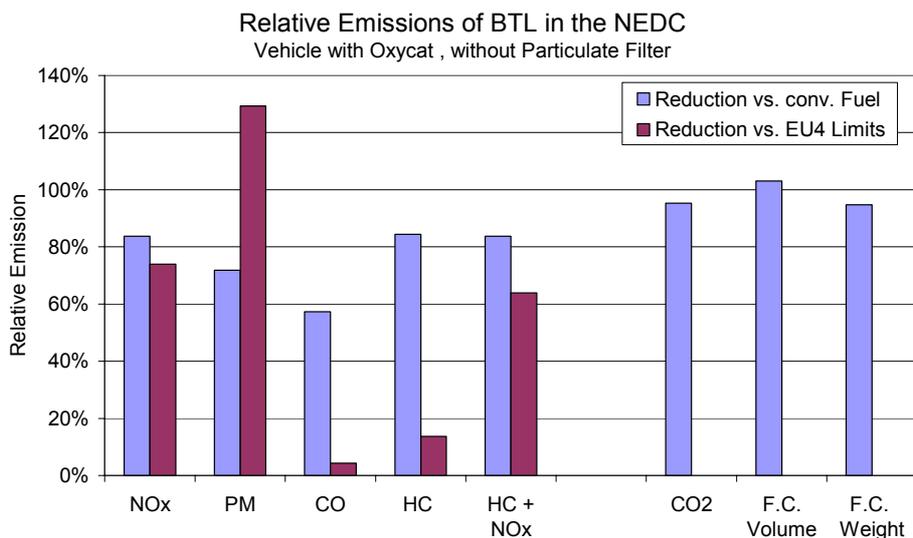


Figure 5-19: Relative Emissions of UET – straight run BTL compared to conventional EN 590 diesel and to EU4 limits for vehicle with oxidation catalyst, without particulate filter.

5.5 Task 1.3.4: Specific experimentation program on HCCI combustion

The objective of RENAULT / REGIENOV's engine tests was to identify the key fuel parameters to be promoted in order to achieve wide range HCCI operating conditions on a research single cylinder engine. For this purpose, in the first year, three different fuels were tested (conventional diesel fuel (EN 590), low cetane synthetic GtL (GtL 10), and high cetane synthetic GtL (GtL 20) under a wide range of operating conditions. The fuel characteristics were as follows:

- conventional Diesel Fuel (EN 590) : Ce = 54 ; IBP = 162 °C and FBP = 359°C
- synthetic GtL Fuel (GtL 10) : Ce = 46,2 ; IBP = 43 °C and FBP = 166 °C
- synthetic GtL Fuel (GtL 20) : Ce = 64,5 ; IBP = 152 °C and FBP = 208 °C

Despite still high combustion noise levels, the synthetic fuel (GtL 10, low Cetane and high volatility) presented the higher potential: therefore, the decision was made to produce a synthetic BtL (BtL 10) fuel with very similar characteristics (Cetane and boiling range).

In 2005, the BtL fuel (P6 sample) was produced by UET and analysed (for composition and properties). Unfortunately, the first analysis measurements detected an unexpectedly high cetane number (60). The results were uncertain and the test methodology probably not suitable for such type of unusual fuel. The sample was rejected. However, the P6 sample was further analysed in a different laboratory with an adequate methodology. The cetane number finally resulted at 46,2 with a suitable boiling range (IBP = 51,2 °C and FBP = 175, 8 °C). Hence, the P6 sample was considered as compliant and accepted for engine testing under HCCI conditions.

The fuel P6 sample was made available for REGIENOV by UET in mid-July 2005 and the engine test sequence scheduled for early September of the same year. Unfortunately, as from the beginning of the test campaign, the single cylinder engine failed and was fully deteriorated. Decision was made to urgently rebuilt it. The restart of the test program was re-scheduled for 2006.

In June 2006, REGIENOV asked for the sample being supplied to their test bench as soon as possible. After arrival a few days later, REGIENOV send part of this sample out for analysis and found an unexpected high water content. The reasons were impurities through long term storage on the UET site and mistakes while (rapidly) preparing the sample for transport. It was decided to send the sample back to UET and to prepare another sample with similar quality. This was send to REGIENOV in August 2006. A deviation of the boiling range of this sample compared to the first one was found. The reason probably was the undesired blending of small amounts of a different quality during long term storage on the UET site. As additional adaption of the fuel quality were no more possible in the timeframe and budget of RENEW, it was up to REGIENOV to decide if test with this modified specification should be done to show e.g. the influence of such a heavier fraction. Despite the non compliance of the supplied sample, the final decision was taken to run a limited number of investigation tests under HCCI combustion conditions to evaluate the potential of

such a fuel. Unfortunately, after only 15 hours, as a result of strong injector fouling¹ leading to a drastically reduced injection flow, the test campaign had finally to be stopped.

5.6 Relevant results from project NICE considered in this project

Within SP1 UET delivered 1000l BtL wax to CERTH. CERTH produced after preliminary research in catalysts liquid fuel out of this wax via hydro-treatment which was distilled on the UET facility. Different cuts with respect to the boiling range have been delivered to the NICE-project. The colleagues in NICE conducted engine tests and provided the results to the RENEW project.

A fuel sample being in the diesel boiling range (Renew_CN75) and a sample characterised with a naphtha boiling range (Renew_CN36) have been tested by Renault in single cylinder tests. The main properties are listed in the table below.

	Method	EN590 (Ref. fuel)	Renew_CN36	Renew_CN75
Density at 15°C [kg/m ³]	ASTM D4052	836.0	699.3	785.2
Cetan number	ASTM D613	51	36.3	74.8
Viscosity at 40°C [mm ² /s]	ASTM D445	2.557	0.5571	3.266
Flash point [°C]	NF EN ISO 13736	66.5	-	127
Sulfur [mg/kg]	PLASMA	44	<10	<2
Carbon [% wt]	ASTM D5291	86.42	84.18	84.91
Hydrogen [% wt]	ASTM D5291	13.51	15.73	14.85
Oxygen [% wt]	ASTM D5622	<0.20	<0.20	<0.20
LHV [kJ/kg]	ASTM D240	43243	46439	44669
AFRS ¹		14.59	15.10	14.90

Table 1: Main properties of the tested fuels

Figures 5-20-1 and 5-20-2 show the boiling range and the main components of the fuels.

¹ The reason was the high amount of oxygenates and acids in the naphtha fraction. In contrast to industrial BTL production units, the simple UET upgrading facilities were not designed for further treatment to reduce these components.

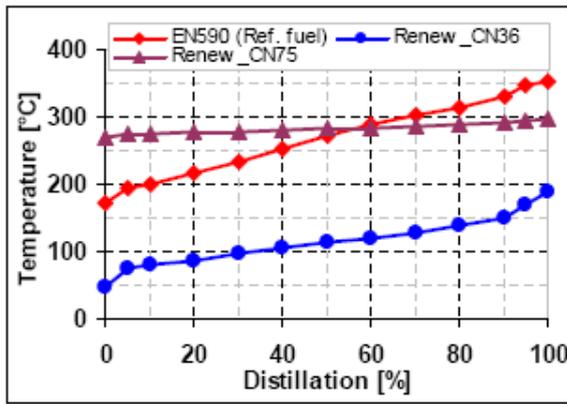


Figure 1: Distillation of the fuels

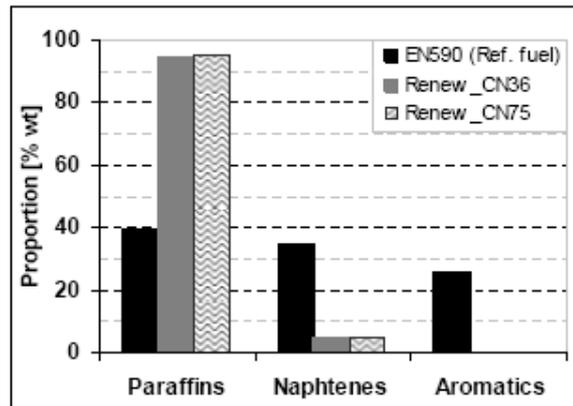


Figure 2: Composition of the fuels

Figure 5-20: Boiling range and main components of the fuels..

With these fuels tests on HCCI combustion were carried out on a single cylinder test bench. Different operating points were chosen, part load as well as one full load point. The latter present actually conventional diesel combustion, since it is not possible to adjust HCCI conditions in full load regime.

However, the main result is that with Renew_CN36, i.e. a naphtha type fuel, a considerable enlargement of the operation range under HCCI-combustion was achieved. The limits for reaching maximum HCCI-load are NO_x-emissions below 0.1 g/kWh and noise below 83 db. The criteria were defined for maximum HCCI-load as 230 g/kWh. The enlargement of the HCCI-range is shown in figures 3 and 4, which depict the max HCCI load for 1500rpm and the maximum HCCI load with supercharging. The latter being a suitable “engine” tool for enlargement of the HCCI-operating range.

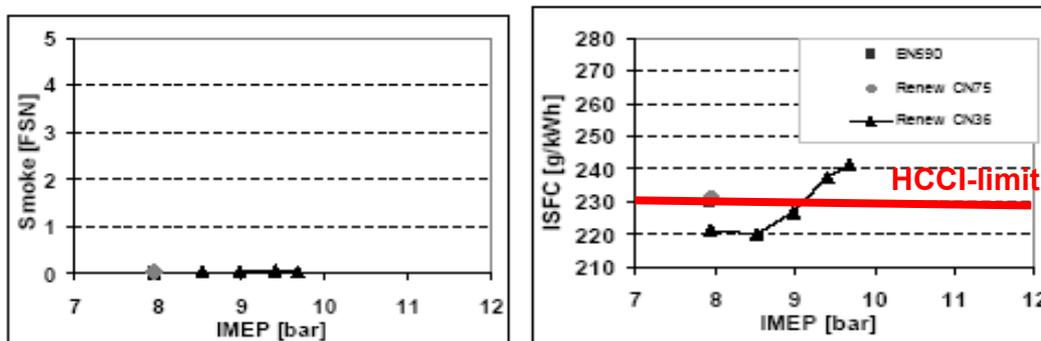


Figure 5-21: 1500rpm, smoke and specific fuel consumption versus mean effective pressure

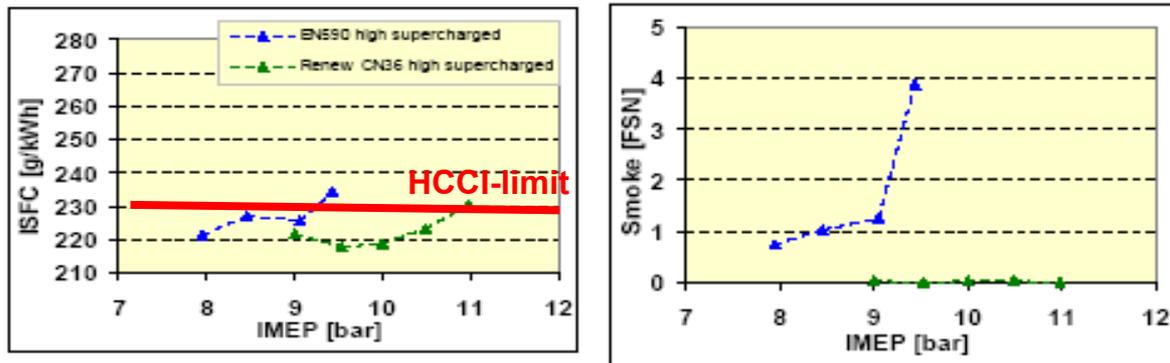


Figure 5-22: 1500rpm supercharging, smoke and specific fuel consumption versus mean effective pressure

The enlargement is about 12% to 18% which are considerable and interesting results, especially, since no smoke was measured whatever the load.

At full load Renew_CN36 burns a little more slowly than the other fuels. As a consequence, the global efficiency is slightly lower. Nevertheless, the best IMEP is reached due to its good limit equivalence ratio at the smoke number limit.

Concluding the hydro-cracked naphtha type fuel, Renew_CN36, has a very good potential to enlarge HCCI-combustion range. In fact, by combining this fuel with high supercharging, which is a further technology studied in NICE, a solution is presented to overcome the current HCCI barriers.

5.7 Task 1.3.5: Defining the characteristics for future fuels

Based on the results of the experiments, all partners have made proposals for specifications for various automotive fuels. From that a common proposal for specifications of future fuels were derived. This represents a major outcome of Subproject 1 within RENEW and is presented in more detail in Chap. 7.

6 Knowledge protection

In the course of the RENEW project, UET had research activities in several fields in which intellectual property could be generated. The main work areas and the patent activities are summarised in a brief description.

- **Gasification:** The syngas production via gasification of biomass is a core competence of UET, hence the gasification process is protected by several national and international patents. Based on the results of the optimization activities in RENEW, UET put effort into the research of patent opportunities. As the “innovation step” of most of the results in RENEW though being quite important for the CARBO-V gasification but are too small to be worth a separate patent, finally no new patents were applied in this field as a direct result of the RENEW activities.
- **Fischer Tropsch Process:** Even if the global players (Exxon, Shell, ConocoPhillips, Sasol, BP, etc.) have agglomerated a notable amount of patents on the Fischer-Tropsch Process over the last decades, UET has managed to claim its own patent on the operation of the FT-unit in this very active field of invention. The trial is still pending but due to the IPC research report we have in prospect to get through the patent granting procedure soon. This patent will find technical implementation in the beta plant in Freiberg.

7 Recommendations for BTL specification

7.1 Approach

7.1.1 Engines

It is obvious that the fields of application must first be restricted in order to provide meaningful specifications. In principle, the use of BTL is conceivable in the following fields:

- SI engines
- diesel engines
- combined processes (HCCI, CCS)

In the case of diesel and SI engines, a clear distinction must be made between the current and the future state of developments. And in the case of diesel, a further distinction between cars and trucks is also conceivable.

The following priorities were set by the group of partners involved in the discussions:

1) To concentrate on passenger cars, because:

- all the tests were carried out on passenger car applications;
- trucks are already fully equipped with secondary exhaust gas treatment facilities, which means that the potential benefits of BTL play a subordinate role in the case of engine technology;
- BtL will most likely be first introduced as a premium fuel (neat or blend) with an extra-on price. As it is not essential for the performance of the heavy duty engine to use such an improved fuel and, because fuel cost are the main cost factor for carriers, truck driver will unlikely go on premium fuel whatsoever.

2) Not to consider Otto engines, as fuels manufactured from cobalt-based FT plants primarily provide diesel or components that are similar to diesel. As a result, no Otto engines were tested in the RENEW programme.

Consequently the requirements for current and future car diesel engines and future combined processes were considered as far as engine design is concerned.

As far as the fuel is concerned, a distinction must be made in the specifications, depending on whether BTL fuels are used as a blend or as the pure component.

Specifications are being drawn up for current car diesel engines in two cases: when BTL fuels are used as the pure component and when 20% of the fuel comes from BTL. Reasons for the evaluation of BTL20 were, that

- the density of the mixture still fits to EN 590 requirements
- a 20 % BTL share at the Diesel market esp. in the passenger car sector is visible

The idea of investigating BTL 5 was also discussed. But at this level, the effect of the BTL component can barely be measured.

At this stage, it is assumed that a pure BTL fuel will be used in future diesel engines and combined combustion systems.

7.1.2 Specifications

The factors, which can be directly affected by the manufacturing process, have been initially considered for the characterisation/specification. These are marked as parameters and involve:

- the boiling range
- the n-paraffin/iso paraffin ratio
- the amount of olefins, aromates and sulphur
- the oxygen content

If a fuel is manufactured with particular parameters in mind, particular properties are created, which may be affected by using additives, but cannot be changed directly by the manufacturing process. A selection of the most important factors marked as characteristics is first made:

- cetane no.
- cold filter plugging point (CFPP)
- flash point
- density
- H/C ratio
- lower heating value
- lubricity

The European standard for diesel fuel, EN 590, has been viewed as the minimum quality level for all the other factors.

7.2 BTL specification matrix

The specifications in chapter 4.1 have been summarised in a matrix. This was filled in during discussions between the users and manufacturers. Figure 7-1 shows the results.

		conventional Diesel engines		future powertrains (homogenous combustion) ⁵⁾	
		BTL 100	BTL as 20 % blending component	future BTL 100	BTL Naphta 100
Parameters					
5% recovered at	°C	170	170	160	50
95% recovered at	°C	320	350 ³⁾	250	160
% n-paraffin/iso-paraffins		cannot be derived from RENEW results			
Olefin	mass %	< 1	< 1	< 1	< 1
Aromates	mass %	< 1	< 1	< 1	< 1
Sulphur	ppm	< 5	< 5	< 5	< 5
Oxygen content	mass %	see comment in text			
Characteristics					
Cetan No.		> 60	> 60	< 50 / > 65 ¹⁾	< 45
CFPP	°C	< -22	< -17 ⁴⁾	< -22	< -22
Flash point	°C	> 55	> 50	> 55	-
density	g/ml	0,76	> 0,76	0,74	< 0,70
H/C	mol/mol	> 2	> 2	>2	>2
lower heating value	MJ/kg	44,8	44,8	44,6	44,3
lubricity	µm	< 460 ²⁾	< 460	< 460 ²⁾	< 460 ²⁾
Others		+ EN 590	+ EN 590	+ EN 590	

1) from engine demands < 50 is required, modern Co-based low temperature FT units do offer > 65 as a standard product; see further comments in text

2) can only be achieved with additives

3) influence on engine emission have not been proven and might change the recommendation

4) probably lower

5) very preliminary because of early stage of engine development

Figure 7-1: BTL specification matrix

It is obvious that the content of olefins and aromatics should lie below 1% from the users' point of view in all the fuel/engine combinations. Even if from a production point of view, a sulphur level below 1 ppm is feasible, the partners agreed on a specification limit < 5 ppm suggesting the contaminations in transport and storage facilities and the limited measurement methods. Compared to the existing standard, this means a reduction of 50 %

Even if from a boiling range respective and from the combustion behaviour, a lower flashpoint for future powertrains seems appropriate, the minimum flash point shall be fixed at 55°C, mainly for safety reasons and compliance with usual standards as all transport and storage systems use to be designed for that value.

Particularly by restricting the ratio of aromates, the upper density level is limited and it will in any case lie below the current standard for diesel fuel, EN 590.

Clear differences can be seen in the boiling ranges:

- The upper boiling range of BTL 100 is clearly restricted by the cold flow properties. Even if the adverse performance of the paraffin components in cold conditions can be moderated by a high degree of isomerisation, there are of course limits to this, which cannot be significantly altered, even with standard additive packages. Initially it was decided that the final boiling point (95%) should be 320 °C. It is possible that the necessary cold flow properties can be achieved by having a higher final boiling point. But if the vaporisation or atomisation of fuels with a high final boiling point is inadequate, it is possible that their suitability for combustion engines may deteriorate. Further tests with the relevant fractions are required so that the engine manufacturers can agree to any increase upwards.
- In UET's view, BTL 20 may permit a higher final boiling point (95%) when it is blended with conventional fuel. But further analysis work is needed to confirm this. It was decided to set the final boiling point at 350 °C; but no information on the emissions created by using such high boiling substances in combustion engines has been provided yet.
- Analysis on the cold flow properties of low BTL blends (CFPP -17°C) to reference Diesel showed, that BTL blends below 30 % do not lower the CFPP of the reference Diesel but in some cases an improvement could be shown (see Del. 1-29). It can be expected, that even lower CFPP may be suitable. As no BTL samples with such lower CFPP were available, the CFPP of the current sample was fixed as a preliminary proposal
- The initial boiling point (5%) of BTL 100 was set at a relatively low figure of 170 °C. This is not a critical factor from a combustion point of view, but there is a risk of steam formation / cavitation in common rail systems; so this threshold needs to be observed carefully.
- For BTL20 a flashpoint of > 50°C compared to 55°C in the EN590 can be accepted. The analysis reported in Del 1-29 showed, that for BTL blends (flashpoint in that sample of just 33°C) below 30 %, the flashpoint of the mixture will stay above the existing EN590 standard.
- The automobile industry envisages that the final boiling point will be lowered for future diesel engines (Future BTL 100). Manufacturers initially need to register this as a request. Tests now have to be carried out to determine:
 - what use can be found for the high boiling fraction,
 - whether there are cheaper ways of converting this into low boiling components or
 - whether the selectivity of the FT/upgrading complex can be adjusted to match this boiling point range with a justifiable increase in costs,
 - whether a sensible application for the lower boiling fractions can be found in HCCI/CCS systems. The boiling point of 160 °C for both fractions is certain to be helpful.

In other respects, the specifications currently suggested are extremely unfavourable and impracticable from the manufacturers' point of view.

- It is important to note that the use of naphtha (BTL naphtha 100) for combined processes is an interesting option for car manufactures and it needs to be examined in greater detail. This opens up an interesting application for the components in the 50 - 160 °C boiling point range, which are less interesting for classical gasoline engines on account of their low octane number. This fraction appears to be a perfect combination - available for use in combined combustion processes and from a manufacturing point of view.
- Cetane no: for engine needs, a figure of < 50 is required; modern cobalt-based low temperature FT units do offer a CN of > 65 as a standard product; further refining, blending or isomerisation may help; an IBP of 120 would lower the cetane no., but may lead to by-products with a completely useless boiling range; further research is required
- Lubricity: for all fuels with a 95 % boiling point below 320 °C, additives are required to reach the < 460 µm level.

It is well known from earlier research projects that small amounts of oxo-components do have a positive effect on the engine's combustion performance. As oxo-components are also produced in the FT synthesis process, it stands to reason that manufacturing costs can be reduced by waiving the equipment needed to separate the oxo-components and the quality of the fuel could also be increased.

The experiments carried out in RENEW have shown that the oxo-components largely consist of alcohols and organic acids. While the alcohols do encourage the combustion process in an engine, the acids are not desired. As organic acids (<C5 carboxylic acids – no others were observed) occur primarily in the naphtha range, distilling naphtha and diesel out of the raw product could be a viable option in the upgrading process. While the diesel fraction, which contains alcohols (evidence of >C6 alcohols was found), could be used as the final product, the naphtha fraction, which contains both acids like acetic acid and low levels of alcohols, would need to be cracked. Because of the selectivity of the cracking catalyst, the oxo-compounds would be removed, but no significant cracking of the naphtha would take place. Current technology does not permit a situation where the acids alone can be separated by distillation.

7.3 Further research requirements

- In principle, it is well known that the n/iso paraffin ratio affects the properties of fuels². For this reason, this figure was selected as a parameter. During the course of the RENEW experiments, it became clear that it is only possible to alter this ratio to a very limited degree in connection with the catalysts and processes used. As a result, there are no test results available, which catalogue the effect of the n/iso paraffin ratios on the engine's combustion performance. This remains an unanswered question for further research projects.

² Chemistry of Diesel Fuels, edited by Chunshan Song, Department of Energy&Geo-Environmental Engineering and Applied Catalysis in Energy, Pennsylvania State University, published in 2000 by Taylor & Francis New York; or VDI-Wärmeatlas Teil Dc et. al.

- Extending the final boiling point (95%) for BTL 100 to beyond 320 °C; deliberate tests with high boiling fractions on the combustion performance in the engine.
- As the defined BTL Naphtha 100 fraction seems to be a promising solution for combined combustion systems and BTL manufacturing processes, further experiments should be carried out on this specific application. Tests were simply carried out on single cylinders in the RENEW programme. Engine tests in the various developments of different systems still need to be carried out.
- For combined processes (Future BTL 100), a cetane number below 50 is desirable from the engine manufacturers' point of view. But current cobalt-catalyst-based FT processes do not permit its production without some difficulty. Methods for reducing the cetane number are further process stages and mixing or reducing the lower boiling point e.g. to below 120 °C. Further research work is also required here.