CO-OPERATIVE RESEARCH PROJECT No. 018109

»GREASOLINE«

New Technology for the Conversion of Waste Fats to High-Quality Fuels

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Dr.-Ing. Volker Heil

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CO-OPERATIVE RESEARCH PROJECT No. 018109
»GREASOLINE«

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3 **R**= Report, **P**=Prototype, **D**=Demonstrator, **O**=Other.

4 **PU** = Public; **PP** = Restricted to other programme participants (including the Commission Services); **RE** = Restricted to a group specified by the consortium (including the Commission Services); **CO** = Confidential, only for members of the consortium (including the Commission Services).

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1 Preface

GREASOLINE is a process concept to convert waste fats to high-quality, fossil-fuel-like diesel and kerosene fuel. A consortium of 6 SMEs from 2 European countries is ready to build GREASOLINE plants, aiming at a commercial potential of 173 million € for SMEs during the first 5 years. However, still some technological barriers are to be overcome. To reach this aim, the SMEs and reputable RTD providers joined forces for advancing the process concept and for building a small technical scale plant for prototype operation.

Far more than 3 million tons of waste fats arise annually within the EU. Since 2001, their use for animal feed is prohibited due to BSE. So other ways of utilisation had to be developed. Now, 5 years later, technologies are available to use the high-quality segment for biodiesel production, but the mid- and low-quality waste fats still lack suitable utilisation pathways.

Being the most advanced low-quality fat-derived product, biodiesel fuel is chemically different to fossil fuel, it is sensitive against oxygen and it faces a limited acceptance in the car industry. Moreover, suitable outlets for the by-product glycerol are often a major bottleneck and an economical threat for smaller producers.

Based on a patent applied by one of the RTD performers, the GREASOLINE process generates hydrocarbons, which are known from fossil diesel fuel, from fatty acids resp. fats and oils.

Glycerol, naturally incorporated in the fat or glycerol co-fed is converted to acrolein (only as reactive intermediate), syngas and ethylene, and partially also to propene. The latter components can be further processed as chemical components in a separate plant as feedstock, or they can be used as process-internal autothermal heating. Both ways of utilisation for those by-products depend on the local infrastructure and intended process design.

Other processes have been developed to produce fossil-type diesel and gasoline fuels from waste fats, but did so far not manage to enter the market.

GREASOLINE introduces an improved process concept for a long-forgotten pyro-catalysic catalyst: activated carbon. The catalyst itself can be made from renewable resources like coconut shells and related materials.

GREASOLINE catalysts display major advantages concerning applicability, catalyst structure and regeneration possibilities. Typically activated carbon displays an inherent resistance against ageing effects. Furthermore, it is a well-proven support material for supporting expensive nobel metal catalysts.

Bottom-line GREASOLINE-derived fuel will not contribute to the greenhouse effect, but will help to reduce fossil CO_2 emissions as requested in the Kyoto protocol. Performing a green-house-gas balance was not part of the scope of this project. However there is meanwhile agreement in Europe, that processing waste streams to fuels is generally considered the preferred way for the future, displaying by far the best green-house gas balance compared to first generation fuels like biodiesel.

2 General project objectives and the project's relation to the state-of-the-art

The basic idea of GREASOLINE aims at developing a catalytic cracking process to convert biogenous waste fats into fossil-style fuel components, using activated carbon as catalyst.

A small technical scale (sts-) plant to convert up to 3 kg/h of waste fats will be engineered and built to test the GREASOLINE concept. Samples of gasoline and diesel products will be produced. By performing this project the fossil style fossil fuel market can be opened.

Older publications (1935) deal with comparable processes that also use activated carbon as catalyst[5]. More recent developments lead to processes incorporating other catalysts, most of them silica- or alumina-based[6]. However, all of these concepts seem to face applicability problems, since none of them affects the biofuels-technology market seriously by now.

Based on two patents owned by one of the RTD suppliers, the main targets of the project were to further investigate the catalytic cracking process in lab-scale, to engineer and build a small technical scale GREASOLINE plant, and to produce sample product for target clients.

The patents are:
DE000010327059B4 (has been granted) / EP000001489157A1 (The patent has been granted and is being nationalized in Austria, Switzerland, Germany, Denmark, Spain, France, United Kingdom, Ireland, Italy, The Netherlands, Poland and Slovenia): »Process for converting of raw materials and waste materials containing oil or fat into a mixture containing hydrocarbons«
DE102005023601B4 (has been granted) / EP000001724325A1(in written negotiation) »Process for continuous or partly continuous conversion of fat- or oil-containing raw and waste materials into mixtures having a high hydrocarbon content, products obtained by this process and their use«

The GREASOLINE process is outlined in Figure 1. Waste fats (alternatively, related materials like fatty acids, vegetable oil and so on) are evaporated and led – optionally together with steam – over an activated carbon bed at up to 500 °C. During this procedure, the waste fats are cracked to hydrocarbons also occurring in fossil fuel (diesel, kerosene, gasoline) and to light hydrocarbon gases. After cooling and product separation, the hydrocarbon gases and other gaseous by-products (mainly CO and CO_2) can be used as process-internal fuel for heating the reactor and can be partly recycled into the process, depending on the detailed process design. The main products, i.e. the heavier hydrocarbons, form the liquid product and are intended to be used as fuel for transportation but could also serve as a renewable chemical feedstock.

Concerning ethical issues the GREASOLINE technology is most advantageous, since it produces fuel from wastes and does not touch agricultural resources. Furthermore, no expensive and mostly rare resources like precious metals, or even toxic metals like nickel or chromium, amongst others are needed for producing of the catalyst. Despite, the GREASOLINE catalysts are made out of renewable materials like coconut shells and other similar left-over materials from processing natural materials.

Spent GREASOLINE catalyst can simply be incinerated like coal and thus serve as a source for renewable energy.

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5 Oppenheim, 1932, *US 1,960,951: Conversion of* Vegetable oils, 300-350 °C at *Activated carbon fibres*, liquid feed supply to the catalyst

6 Prasad et al. 1986, Can. J. Chem. Eng. 64: Conversion of Rapeseed oil, 340-400 °C, at HZSM-5-Zeolites; Hahn, Oberländer 1995, DE 10 049 377: conversion of vegetable pressed oil, 350-500 °C, at Perovskites with La-, Ce-, Co-Oxides; Demirbas, Kara 2006, Energy Sources 28, Conversion of Soy bean oil and sun flower oil, 340-420 °C at $ZnCl_2$; and others

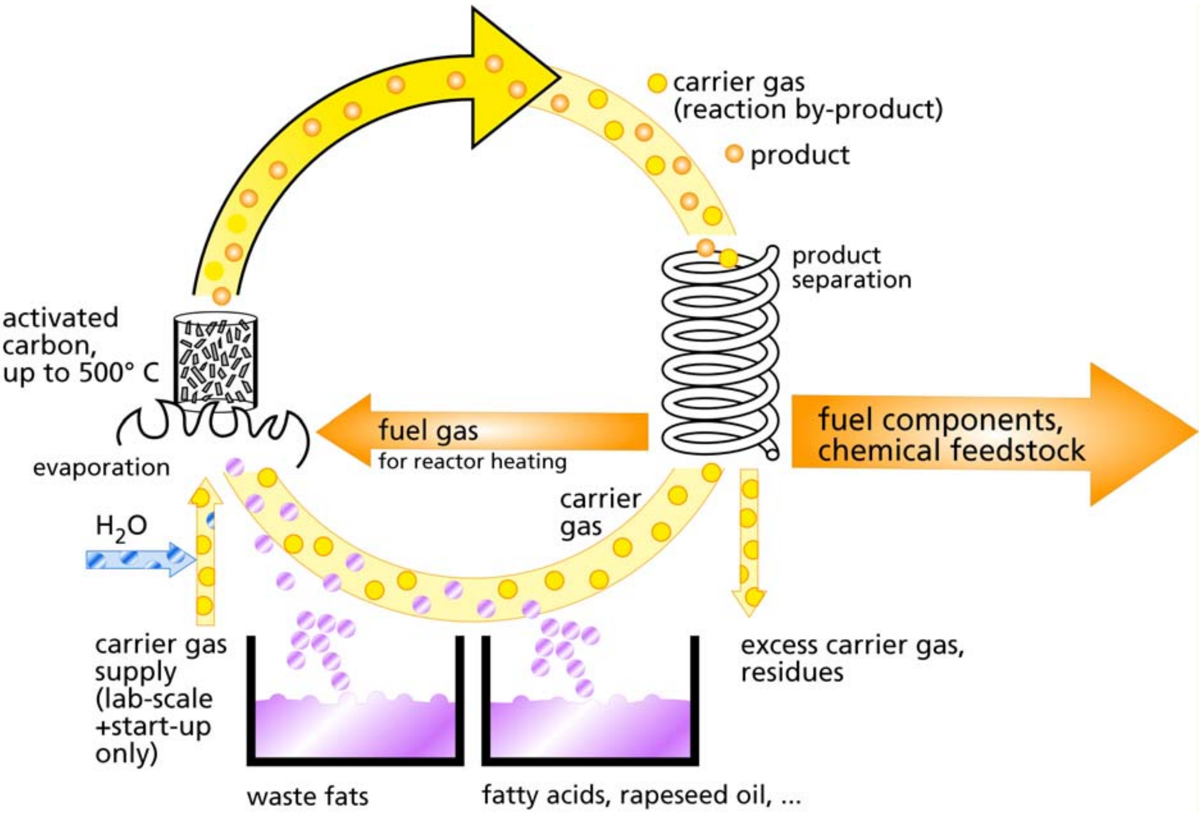

Figure 1: The concept of the GREASOLINE process

Recently planed pilot plants are operated to process quality fats and oils to hydrogenated vegetable oil (HVO) by BP and Neste Oil (tradename NextBTL). However, investment costs are quite high compared to biodiesel plants and considerable amounts of fossil hydrogen is required. Gycerol incorporated in the fat and oil is hydrogenated to propane. For chemical reasons, HVO is preferably produced from palm oil, since palm oil requires, relative to other fats and oils, the lowest amount of hydrogen and displays the lowest pour point.

GREASOLINE is, relative to HVO advantageous, since is requires neither fossil hydrogen nor expensive catalysts, which can only be manufactured by a few companies in the world.

Certain amounts of not chemically contaminated waste fats can be digested together with other food residues to bio-gas (methane). This is a way of utilization that is currently preferably performed in Germany, e.g. by Remondis.

3 Contractors

3.1 Contractors: industrial participants (SMEs)

WETEC Elektrotechnik GmbH, Moers, Germany, is specialised in process automation and central building control systems [HLK], low voltage main distribution frames and power distribution installations. WETEC develops and builds test stands, switch gear, programmable logic controllers [PLC] and conventional control cabinets. The main working fields are consulting, planning and execution of process control technology and electrical engineering.

The activities of *Soldesa Hydrogen B.V., The Hague, The Netherlands,* cover among others the design, engineering, purchasing and constructionof equipment and the execution of projects, which are related to thermal conversion of Hydro-Carbon feed stocks, directly or with the help of catalytic cracking of said feed stocks, into (synthesis) gas. Main objective is the production of hydrogen or hydrogen related products, like methanol etc.

Silcarbon Aktivkohle GmbH, Kirchhundem, Germany, supplies activated carbon, »white adsorbents« (silica gel) and adsorbers, and consults in the field of possible applications. The company operates a reactivation plant for activated carbon in Vierumäki, Finland. The activated carbon that was used during the invention of the GREASOLINE process concept has been a Silcarbon product.

Ralf Hacker Edelstahl, Hüllhorst, Germany, is a steel builder experienced in building up plants. Main focus is plant engineering, construction and mechanical engineering. Also custom made products made of stainless steel are produced. The company has considerable experiences in pipeline construction and boiler construction.

Established in 1991, *Ingenieurbüro r.efkes GbR, Willich, Germany,* mainly deals with engineering and research projects in the field of renewable energy. The company has a good experience in biogas, landfill for solid waste, coal mine methane (CMM) sewage gas, natural gas and solutions for gas pumping, flares and CHP technology with gas or oil (incl. plant oil).

Dinnissen BV, Sevenum, The Netherlands, is a leading producer of machines and installations for various branches of industry, specialising in powder and process technology. Dinnissen provides a full service for development and manufacturing of machines and systems from the design stage through to after-sales service in the dry solids handling and processing. Their most important market is in the feed compounding, aquafeed, pet food industry, chemical, food and pharmaceutical industries throughout the world.

3.2 Contractors: RTD performers

The *Fraunhofer Institute for Environmental, Safety and Energy Technology UMSICHT (Fraunhofer UMSICHT), Oberhausen, Germany,* is a non-profit scientific and technical institution. As one of 56 institutes of the Fraunhofer-Gesellschaft zur Förderung der angewandten Forschung e.V. (translation: Fraunhofer association for promotion of applied research), Fraunhofer UMSICHT develops, evaluates and optimises technical processes in the fields of environmental safety process and energy technologies. The institute strives for the introduction of new, intelligent technologies, which operate reliably in an economic, environmentally-friendly way. Fraunhofer UMSICHT acts as an interface between academic research and industrial practice. New scientific findings are transformed into sellable products and technologies.

Pera Innovation Ltd., Melton Mowbray, GB, has grown from the Production Engineering Research Association of Great Britain, and is a technology R&D organisation with high levels of expertise and facilities. Pera is a non-profit distributing company, limited by guarantee, and owned by its 1,000 SME Members. Operating for over 50 years as a non-IPR holding business, its constitution dictates that it should generate and transfer new technology to manufacturing industry. PERA provide industry support activities geared towards providing step change improvement in competitive advantage for industry as a whole, and these are undertaken in response to direct approaches by industrial companies themselves, or in many cases delivered on behalf of national governments seeking to support specific regions or national industrial sectors.

4 Project execution: work performed and results achieved

4.1 Analytics

As a basis for all practical examinations analytical methods especially for the characterization of gaseous and fluid products were developed. On one hand an online gas chromatographic procedure for the analysis of gaseous hydrocarbons in the product stream was set up. On the other hand an off-line gas chromatographic method coupled with mass spectroscopy for the analysis of organic compounds in the fluid products was elaborated. The hardware consisted of a GC/MS 6890/5973, Agilent Technologies, Column: Phenomenex ZB-5MS (30 m x 0.25 mm x 0.25 µm). Also dedicated procedures for analysis and characterization of activated carbon catalysts were established in particular BET, iodine value and mercury porosimetry. Furthermore, measurements like water content according to Karl Fischer, viscosity and iodine value for waste fat characterization were established at Fraunhofer UMSICHT.

4.2 Lab-scale investigations

In a first practical approach, a lab-scale plant was engineered and built to convert 55 g/h (60 ml/h) of waste fat. Mainly pre-homogenised and pre-filtrated waste fat, delivered by a professional waste fat collector, was used as feedstock. In order to avoid damage to the lab-scale gear pump, small solid particles were removed by centrifugation at 80 °C. Figure 2 shows the raw and centrifuged waste fat (the latter with sediment to be separated by decantation at the flask bottom).

Figure 2: Waste fat before (left) and after (right) centrifugation

It can be seen that after the centrifugation the particles resulting from, in this case, spent cooking oil, are concentrated at the bottom of the sample. Hence it is easy to separate the two phases by decantation. After this the fat is filled into the waste fat supply of the plant.

In a first approach, the lab-scale plant was built and constantly improved, solving problems like deviations in the input flow (by stirring the heated feedstock vessel and heating the pump), rapid catalyst ageing (by choosing the appropriate activated carbon catalysts and implementing steam supply[7]) or product loss in separation (by choosing a cooling concepts that ensures heat transfer even at the given, very small volume flow rates). The resulting lab-scale plant, shown in Figure 3, was equipped with a GC-TCD/FID on-line gas chromatograph by Agilent Technologies for on-line analytics of the gaseous products. Gaseous products are analysed before they enter the incinerator.

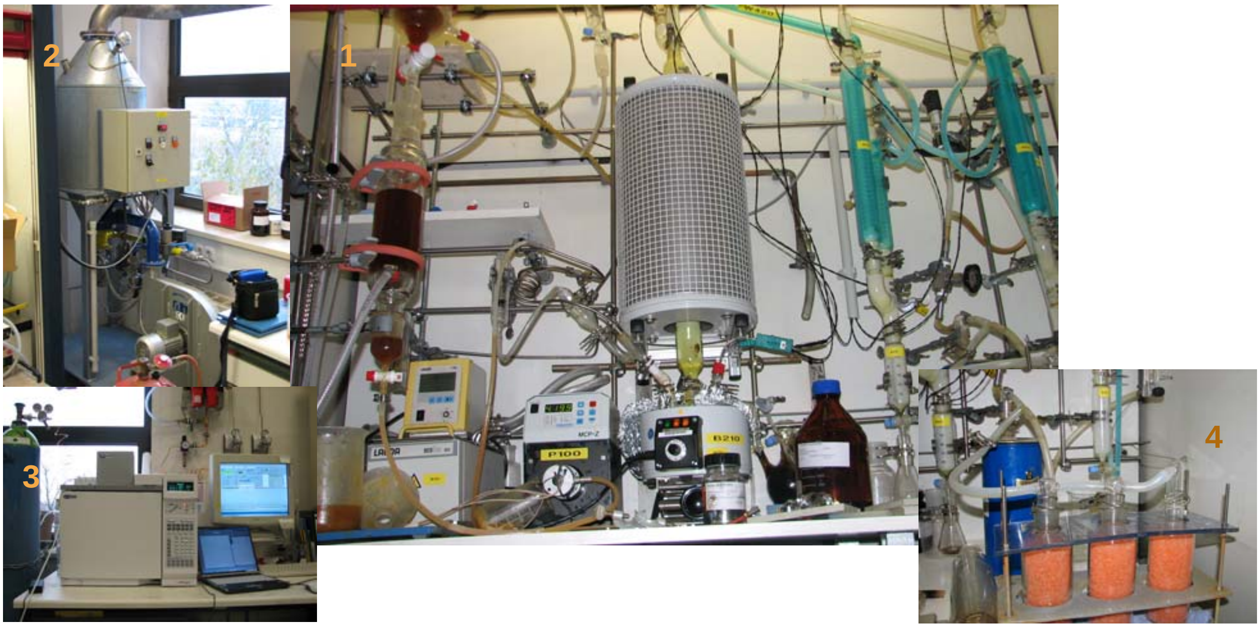

Figure 3: The lab-scale plant, first set-up, with core lab-scale plant (1), thermal afterburner (2), online-gas chromatograph (3) and gas samples pre-treatment (4)

In late summer 2007 the lab-scale plant was partially revamped. The desktop-based set-up was replaced by a metal framework setup, the superheated steam supply system was improved, and the waste fat evaporation and catalytic reactor performance was enhanced. This set-up was attractive enough to be chosen for TV broadcasting (see Figure 17) by regional TV reaching a few million of households in North-Rhine-Westfalia. The revamped set-up is shown in Figure 4.

7 Steam supply in a much higher steam-to-feed-ratio was proposed previously for other catalysts: Idem, R. O., S. P. R. Katikaneni, et al. (1996). "Thermal Cracking of Canola Oil: Reaction Products in the Presence and Absence of Steam." Energy & Fuels 10: 1150-1162.

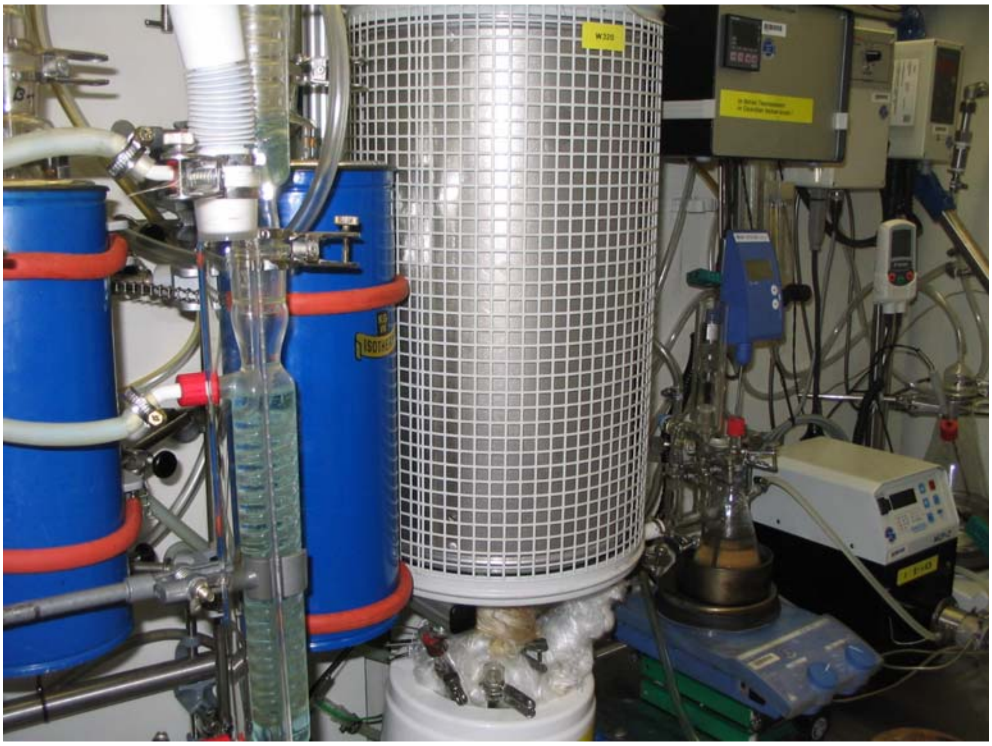

Figure 4: The lab-scale plant, improved set-up

Lab-scale trails revealed that an evaporation temperature of at least 450 °C is sufficient to get nearly all the introduced waste fat into the gas phase. For details see Figure 5.

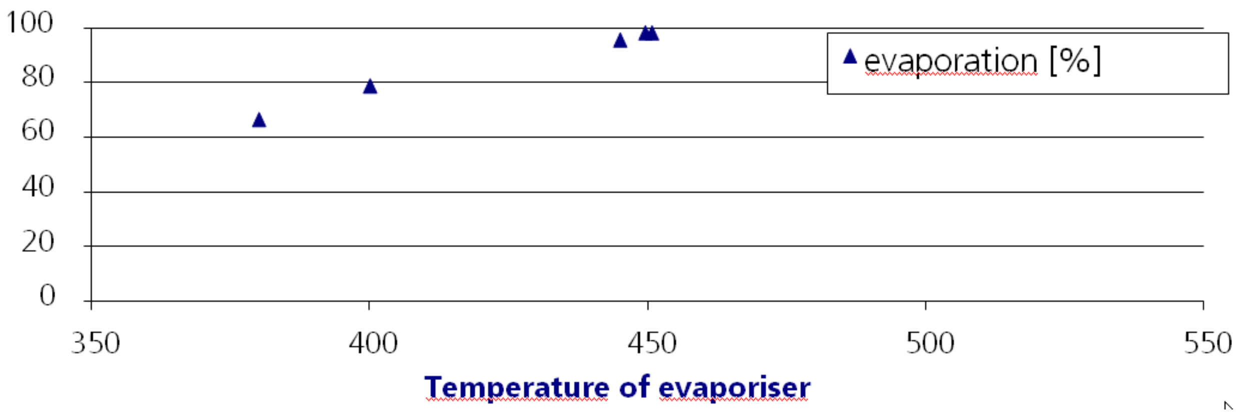

Figure 5: Yield of waste fat evaporation

Reaction temperatures were found to be at optimum in a range between 450 and 500 °C in order to convert all the waste fat, avoiding unnecessary product cracking at the same time.

Figure 6 shows the results of a test series for the conversion of a mixed waste fat with 5 wt.-% water content. At the diagonal between minimum residence time (proportional to bed height) / maximum temperature on one hand and maximum residence time / minimum temperature on the other hand, the liquid yield displays its optimum. Here, a product with 49 wt.-% of liquid phase yield and 60 wt.-% of components in the diesel boiling range (the rest being in the gasoline boiling range) was achieved. The 49 wt.-% of liquid phase yield (always calculated without water phase, in case it should occur) are 66 % of the theoretical maximum of 74 wt.-% that stands for sole primary decomposition of the waste fats without any coke formation or secondary cracking.

The »unidentified substances« shown in the figure arise from the methodology of analytics. The liquid product is analysed via gas chromatography enhanced by MS. Only the substances being part of the 30 highest peaks of each chromatogram were analysed. However, the remaining approximately 170 substances always showed the same distribution of gasoline and diesel boiling ranges as the identified ones did. This procedure is quite common for analysing diesel.

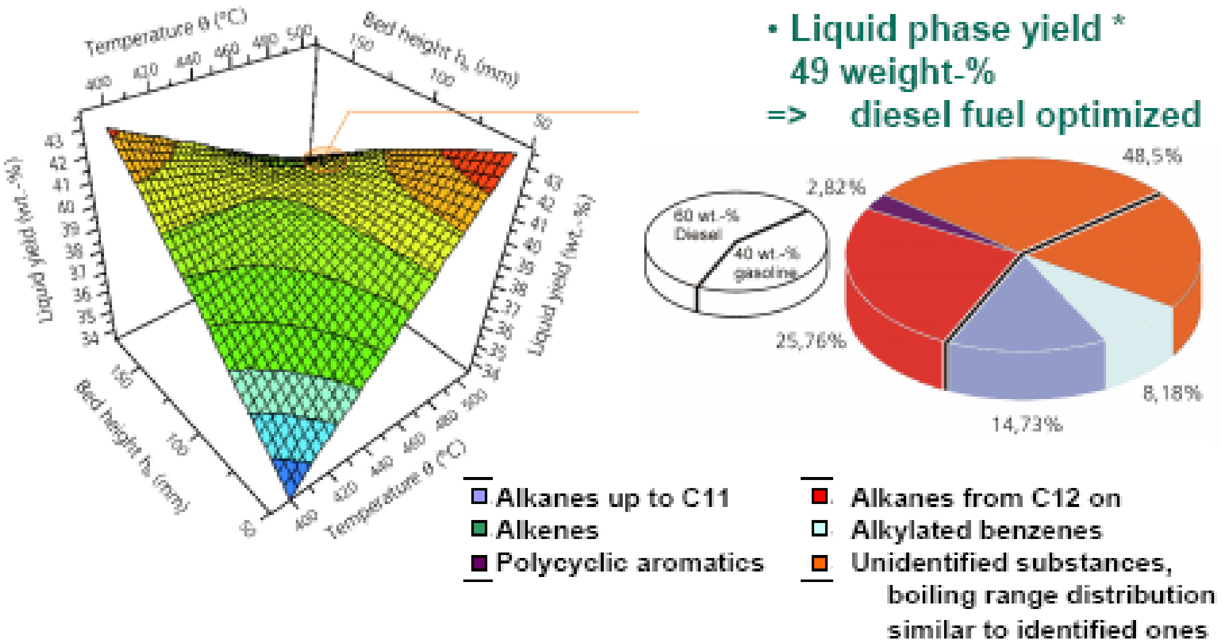

Figure 6: Diesel-fuel-optimised product

The proper choice of an alternative activated carbon catalyst can relocate the said maximum diagonal. In Figure 7, a set of experiments is reported where the maximum diagonal is already passed when entering the investigated parameter (please note the different directions of the axis in Figure 6 and Figure 7). Here, a gasoline-optimised product with a proportion of 76 wt.-% of the components inside the gasoline boiling range was achieved at an overall liquid product yield of 41 wt.-%.

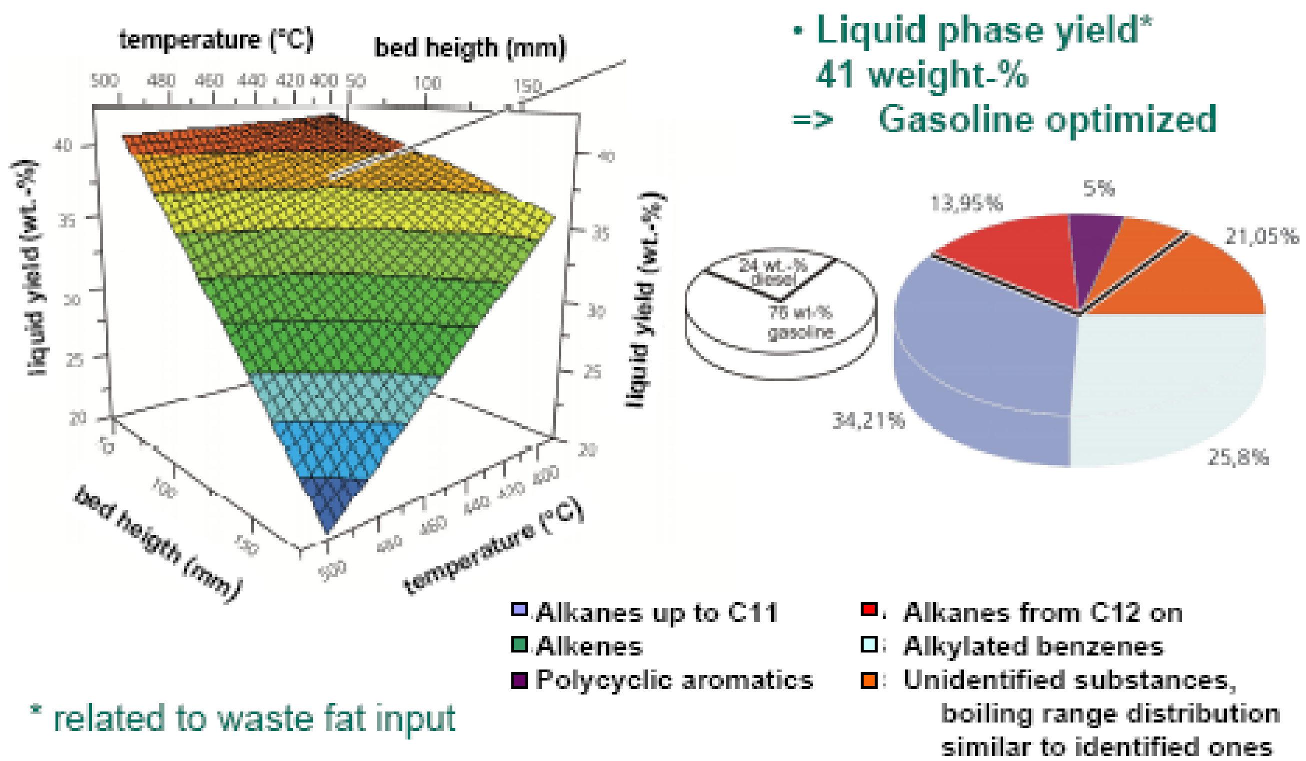

Figure 7: Gasoline-optimised product

Vegetable oils and fats contain fatty acids of nearly exclusively even-numbered numbers of C-atoms – in European waste fats, mostly C16 and C18[8]. During the GREASOLINE process, fats are decarboxylated, i.e. one CO_2-molecule is separated from the molecule, to C15 and C17 in primary cracking. Secondary cracking breaks the decarboxylated molecules down to shorter hydrocarbons. Side-reactions lead to aromatic molecules and coke formation. Reaction pathways have been proposed for pyrolytic cracking over HZSM-5 zeolites by Katikaneni et al.[9]

Figure 8 compares a lab-scale-derived primary product with EN 590[10] diesel fuel from a German fuel station. The GREASOLINE-derived product was not refined, therefore the lower boiling (shorter chain) components C8-C10 are present, too – in contrast to the shown refined diesel fuel according to EN590.

The vast majority of product is C15 and C17 – the primary cracking products, as explained above. Apart from the low-peak-ground-level of »other components« which even is distinctly lower than for diesel fuel, the main peaks in the diesel boiling range are the ones of C11-C17 hydrocarbons. The C18-peak in the GREASOLINE product is the result of a special side-reaction.

Even without having performed motor tests by now, this result can be regarded as a very strong indication that GREASOLINE can produce a fuel that should be well motor-applicable.

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8 C16: a hydrocarbon with 16 C-atoms

9 Katikaneni et al.: »Catalytic Conversion of Canola Oil to Fuel and Chemicals over Various Cracking Catalysts«. Can.J.Chem.Eng. 73(1995), p. 484-497

10 European standard EN 590:2004: Automotive fuels - Diesel - Requirements and test methods

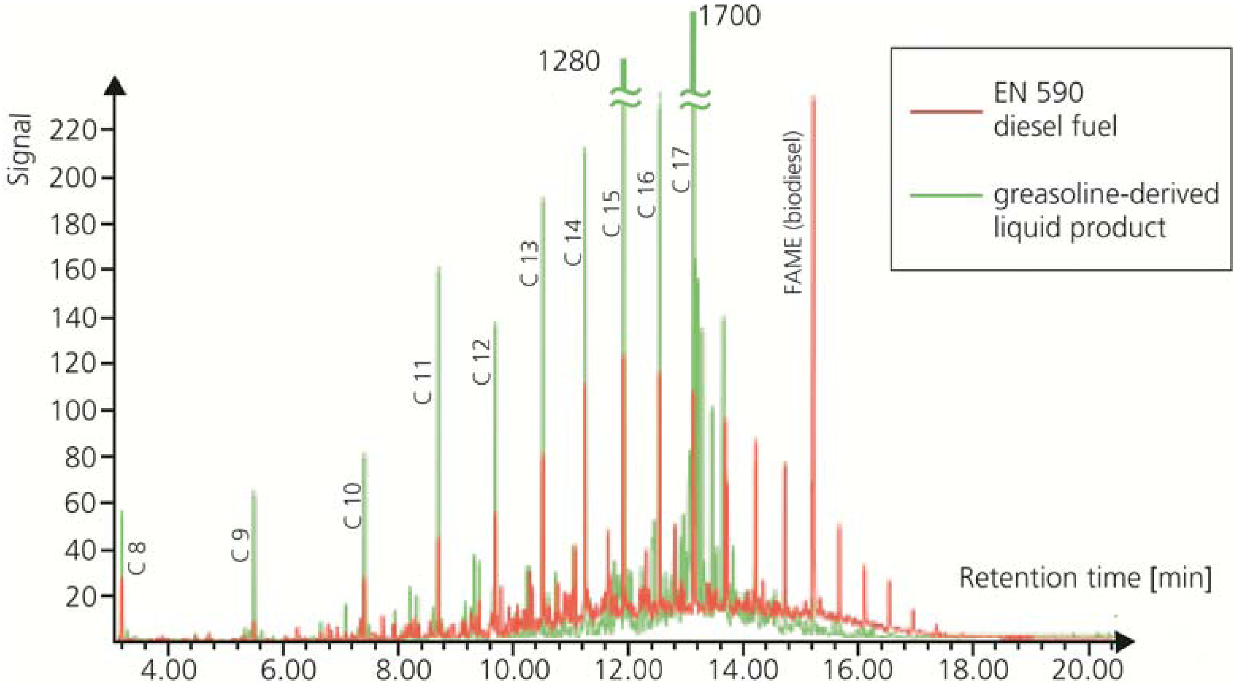


Figure 8: Gas chromatography (GC-MS) plot of GREASOLINE-derived product and Biodiesel-blended diesel fuel according to EN 590 (peak heights are comparable within the individual plots only)

Apart from preparing work of the RTD-partner UMSICHT, these results are the first ones ever reported for catalytic cracking of waste fats at activated carbon with evaporation prior to catalytic reaction or with addition of steam. The similarity of the derived product to standard diesel fuel is outstanding and clearly goes far beyond the state of the art.

Pera built an additional experimental rig for the product separation unit. Experiments were carried out by both Pera and UMSICHT to reveal and to obtain important practical information for the product separation concept.

4.3 Small technical scale-experiments and product samples production

4.3.1 Preface

In the course of the laboratory experiments several parameters were considered. The most important of them were temperature of reactor and evaporator, ratio of steam addition, overall residence time, type and chemical and physical nature of the activated carbon catalyst.

Specific set-ups for two different conversion tasks were established: for gasoline-style and diesel fuel-style optimised product. However, the production of gasoline-style product goes along with poor overall liquid product yields and is therefore more of academic than economic interest.

Furthermore, the lab-scale results revealed that the chemical composition of the feedstock has a minor effect on the rate of conversion and product composition, apart from the fact that the maximum length of product hydrocarbons is limited by the length of fatty acid chains in the feedstock. However, strongly diminishing or increasing the content of free fatty acids in the feedstock will lead to a higher or respectively a lower consumption of added steam, eventually different catalyst life-time and will have an impact on corrosion related requirements for equipment. However, exploration of the full parameter space of all raw material related questions was beyond the scope of this project.

Only the conversion of diesel fuel-style product from mixed waste fat feedstocks was transferred from small-scale to pilot-scale. For production scale plant of course the impact of major raw material related variations would have to be critically scrutinized beforehand. A possible positive or negative impact should also be considered when performing detailed cost estimations and cash-flow projections for investors.

While engineering and building the envisioned sts-plant in Oberhausen, a smaller and somewhat simpler GREASOLINE sts-plant located in Teterow (a German subsidiary of Fraunhofer UMSICHT in eastern Germany) was revamped. This so-called Teterow sts-plant was employed for performing part of the process development. It was also utilized for producing larger quantities of product samples for project internal use and as reference for external clients.

4.3.2 The Teterow sts-plant

Results of lab-scale experiments were checked with respect to specific parameters on larger scale in the Teterow sts-plant. Process specific parameters like contact time, amount of activated carbon per amount of feed, temperature and process control settings, mixing and preheating evaporated feedstock, steam and inert gas were adjusted and fine-tuned to fit the larger scale. The same batch of waste fat as already employed in lab-scale experiments described was used.

4.3.2.1 Configuration of the Teterow sts-plant

The waste fat is preheated then conveyed into a second preheater. Steam and nitrogen are added, preheated and mixed with the waste fat subsequently. Then fat, nitrogen and steam reach the evaporator where the fat is evaporated and the mixture flows into the reactor. Products leaving the reactor are cooled down immediately and are collected and analysed. No online product gas analysis was performed. The set-up is depicted in Figure 9.

Figure 9: Evaporator and reactor of Teterow sts-plant

With steam addition, liquid yields (organic liquid product, referred to waste fat feed) of up to 57 % were reached, which is 77 % of the theoretical maximum. The water content in the waste fat is around 5 wt-%. Purification by batch distillation resulted in a clear green, photo-insensitive product shown in Figure 13.

4.3.2.2 Production of greater amounts of liquid product

As both the Teterow and the Oberhausen plant are equipped with single fixed-bed reactors without regeneration, no long-term operation was possible. Thus, the long-term operation experiments were conducted as simulated semi-continuous experiments using the Teterow sts-plant. That meant, that the activated carbon catalyst was replaced after it was spent. In between the plant was cooled down and fresh catalyst was introduced into the reactor. The operation parameters were not changed, and each experiment continued like the one before ended. This way, it was possible to run a simulated semi-continuous operation of more than 25 hours relevant catalytic conversion.

For economic reasons, i.e. the current high diesel demand in Europe, a high liquid yield and preferably a high diesel content in the product are favoured. During the lab scale experiments, the parameters temperature, activated carbon, steam addition and residence time were identified as most important parameters impacting conversion and liquid yield. The first experiment was done according to optimized conditions found in lab-scale experiments. Those experimental conditions offered already a high yield of liquid product. However, the identification of the product composition showed that there was a noticeable amount of unconverted waste fat and acid components. Based on these results the specific parameters were adapted in the following experiments for the production of product samples for target clients.

4.3.2.3 Results of experiments for specific conversion task

During an experiment a product sample was collected each 30 minutes. Typical samples with and without condensed water are shown in Figure 10. The water results mainly from steam addition but can be formed in small amounts during the process. As mentioned before the waste fat itself contains small amounts of water.

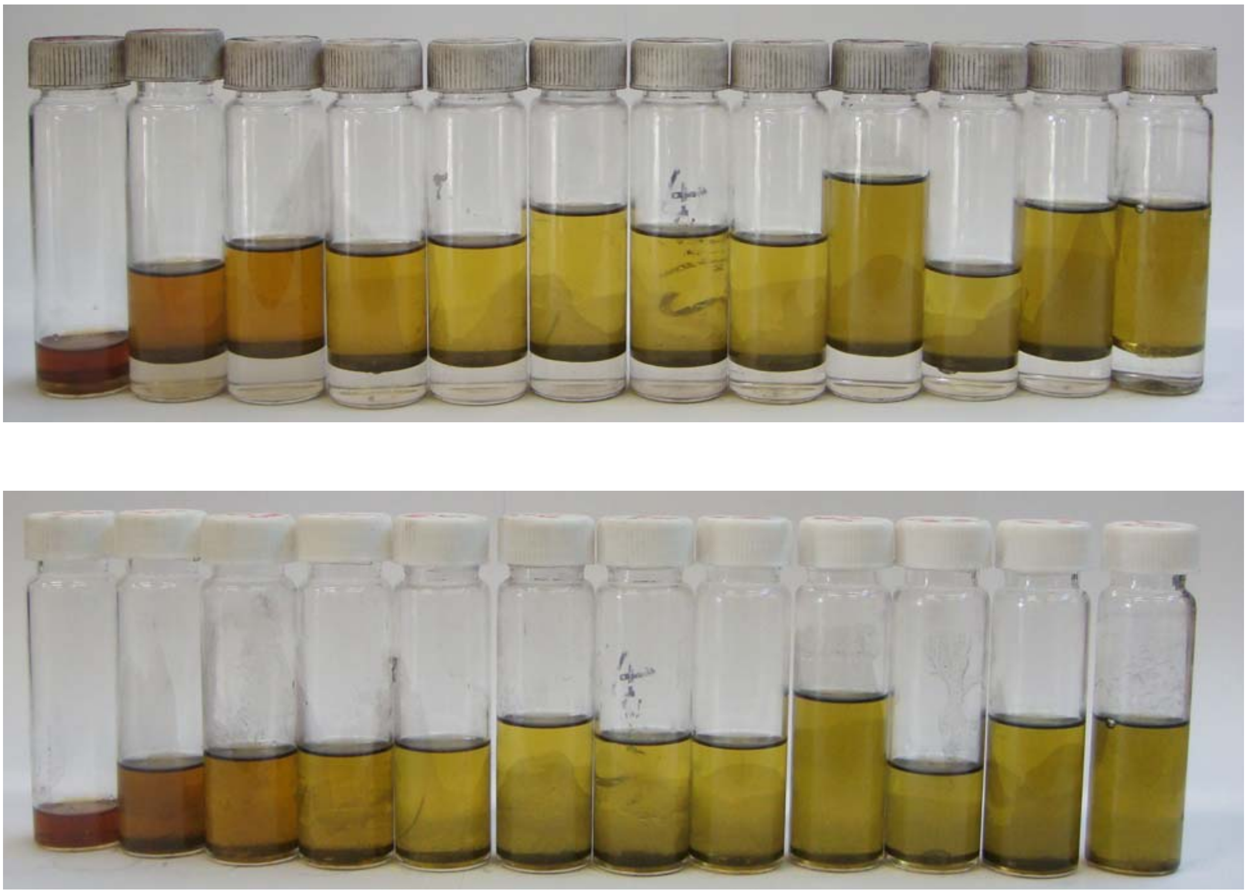


Figure 10: Product samples with and without water (lower part of figure)

The feed rate was 1.8 g/min. In Figure 11, the overall yield is shown [weight/weight]. It can be seen that a liquid yield of 56,3 % with respect to crude fuel, i.e. gasoline and diesel, could be reached. Also important to notice that about 3,5 % of the feed was lost as solid carbon-like material ("coke") on the catalyst.

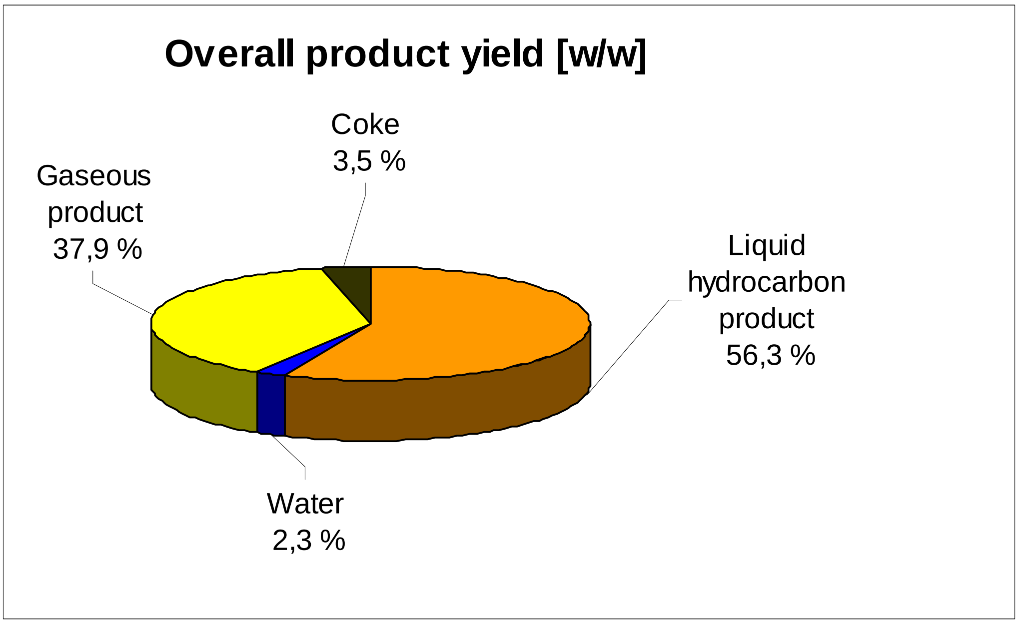


Figure 11: Overall product yield achieved under optimized conditions

In the liquid product a high amount of substances belonging to the diesel fraction was found. Figure 12 shows the composition of the liquid product. The 30 highest peaks from each experiment were determined by GC-MS analysis. For the determined components it was found, that the magnitude of boiling point is directly proportional to the residence time in the GC-MS. Experience tells, that this behaviour can be anticipated also for the not specified (»rare«) substances, i.e. low retention time in the GC-MS indicates gasoline fraction and high residence time indicates diesel fraction. The cut between diesel and gasoline fraction starts roughly at about the retention time were C12 shows up in the GC-MS (see figure 8 for details of the GC-MS diagram).

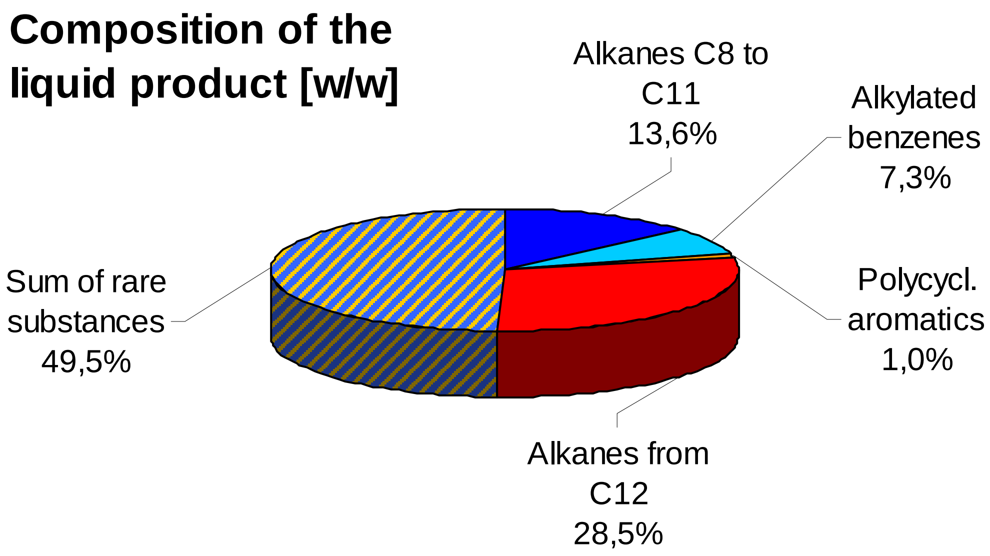


Figure 12: Composition of liquid product

The fraction of alkanes C8 to C11, and the major part of the alkylated benzenes belong to the gasoline fraction. Around two third of the “rare substances” can be allocated to diesel. Overall around 60 % of all measured GC-MS substances belong to the diesel fraction. All polycyclic material, mainly naphthalene and several types of alkylated naphthalene, belong to the diesel fraction.

Figure 13 shows the crude product after condensation, the distillation residue and a purified photo-insensitive greenish product sample containing the diesel and the gasoline fraction, for use in transportation fuels.

Figure 13: Product sample: crude product (l), purified sample (m) and distillation residue (r)

4.3.2.4 Conclusions

The Teterow sts-plant confirmed experimental results from lab-scale. Small corrections were needed for scaling up the recipe from lab scale. The liquid product yield and the high amount of substances belonging to the diesel fraction were confirmed. It can be assumed that when using very similar parameters for the Oberhausen sts-plant, a similar product composition can be expected. Furthermore, with the extensive experience and know-how gathered, it should be possible to adapt the operating window and operation conditions to varying feedstock in a way that always rather high liquid yields and a high diesel fraction are obtained.

4.3.3 Planning and Construction of an sts-plant at Fraunhofer UMSICHT (»Oberhausen sts-plant«)

Based on the lab-scale results and aided by process modelling calculations, engineering for a further plant on small technical scale to convert 3 kg/h of waste fat for the production of about 1,6 l/h of high-grade fossil-style fuel (1,8 l/h raw product) began. Waste fat supply, feedstock evaporization, catalytic reaction and product separation were completely revised compared to the original engineering concept. The plant was engineered, components were manufactured, facilities prepared, a process control concept was developed and implemented and the components were assembled. This plant is called the »Oberhausen sts-plant«.

To house this Oberhausen sts-plant, a complete building (built and previously used for another thermal bio-energy process) of 12 m height and about 31m^2 base area at Fraunhofer UMSICHT's headquarters was emptied. The internal construction frame was adapted and the whole building was dedicated to potentially hazardous operations like fuel processing.

Conceptual flow sheets for the Oberhausen sts-plant including Piping & Instrumentation Diagrams (P&IDs) for for all unit operations and the process control system were discussed and reviewed internally and with the SME-partners. Safety concepts were developed and put into practice. Repeated auditing by a well trained safety engineer helped to ensure and to reinforce a safe operation of the complete plant.

An Aspen Plus[11] flowsheet model for the whole plant helped to perform mass and energy balance calculations as accurate as possible. Thus a realistic and consistent concept for waste fat supply, waste fat vaporization, catalytic reaction and product separation was developed.

An operating manual was generated covering regular operation as well as start-up and shut-down operation, also covering a rapid shut-down, in case of emergency or other unforeseen situations might occur.

By the time the official project termination was reached, the installation of the main parts of Oberhausen sts-plant were finished. Most piping and assembly tasks had started. Subsequent to the project end the installation including final piping, electrical installation and implementation of process control is being finished up. Efforts towards delivering a fully tested and operational sts-plant in Oberhausen made good progress. This will be the major deliverable and success of all the efforts put into this European project.

Figure 14 shows a part of the (not fully insulated) pilot plant. At the left side, the waste fat supply unit comprising a stirred tank, a feed pump and a preheating unit are situated. On the right side of the picture, elements of the waste fat vaporisation, the product separation unit - quench – are depicted. The catalytic reactor is located on the second floor and not visible here.

11 Aspen Plus™ is a process modeling tool for conceptual design, optimization, and performance monitoring for the chemical and related industries by Aspen Technology, Inc.

Figure 14: Part of the Oberhausen sts-plant. The remaining part is located in the upper floor.

4.3.3.1 Detailed design of unit operations of the sts-plant

In order to cope with chemical variation in feedstocks and to be able to optimize the ratio of water, waste fat and inert gas, most components of the sts-plant had to be designed larger than optimum. Modelling was performed to be able to asses the limits and the flexibility of operation of all unit operations (operation window). This is in particular important for unit operations processing gas, where not only heat transfer, but also pressure drop is a crucial success factor. Mathematical models and predictive parameter estimation models, mainly taken from VDI-Wärmeatlas, have been employed to predict individual operation windows of all unit operations.

For illustration purposes the following picture (Figure 15) depicts a calculated temperature profile of the so-called super-heater, used to preheat the mixture of water, inert gas and fat to reactor inlet temperature.

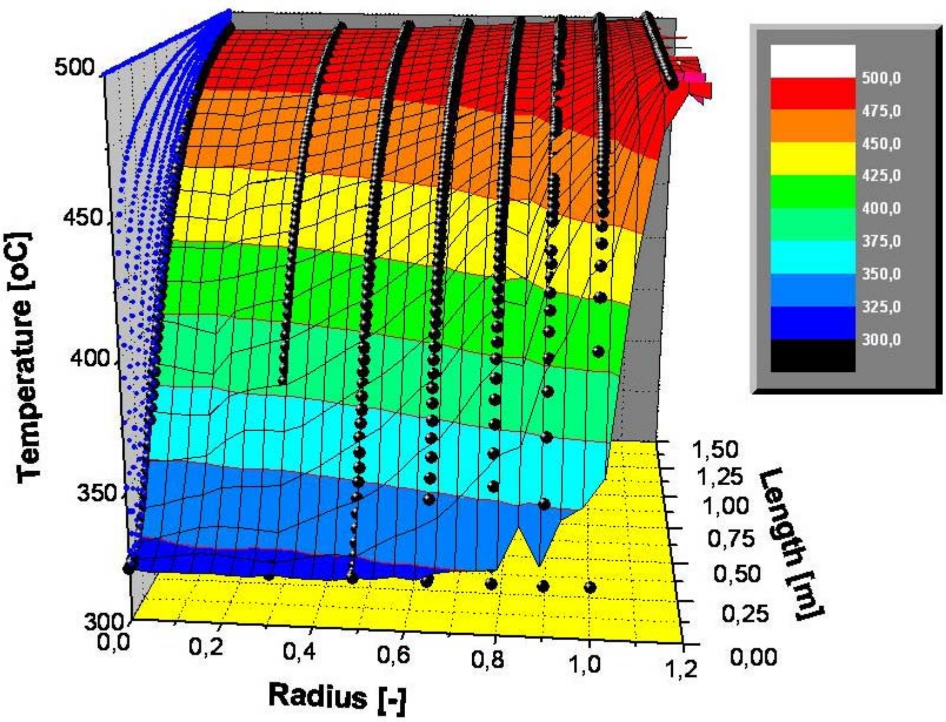


Figure 15*:* Calculated steady-state temperature profiles (cylindrical coordinates, including reactor wall temperature) of super-heater. Interpolation used to create a surface.

4.3.4 Project management

A quality management procedure was prepared by Bioxy/Soldesa and UMSICHT. Solarpunkt presented a first exploitation plan, and UMSICHT provided and maintained a server-based groupware that all partners use for internal communication. The homepage can be found under: www.greasoline.com

5 End results, intentions for use and impact

Within the GREASOLINE project, catalytic pyrolysis of waste fats and oils into high-quality fossil-style gasoline- and diesel-type fuel was developed from a concept towards a small technical scale-process by theoretical and via experimental work on lab-scale and pilot-scale.

A small technical scale (»sts«-) plant was improved and used to produce fuel samples for target clients. Liquid yields of up to 57 wt.-% (crude product) – this is 77% of the theoretical maximum – were achieved. Refining of the crude product via batch distillation led to a clear green, photo-insensitive product.

A larger sts-plant to convert up to 3 kg/h of waste fats was engineered and components were built and assembled. It is ready to be operated after preliminary testing of the control and emergency systems and other minor fine-tuning activities are finished up.

Preliminary cost assessment indicates that GREASOLINE plants can be operated economically at a scale of some 1.000 - 10.000 tons per year. No risky scale up of unknown unit operations, not yet known to those skilled in the art, is required to be able to operate at larger scale. However, catalyst lifetime and cost, steam supply ratio and raw material feed ratio, in terms of kg feed per kg catalyst, may need adaptation and fine tuning to maintain an economical optimum or to even reach an economical viable operation.

Suitable locations for a grass-root GREASOLINE plant or preferentially a retrofit into an existing refinery infrastructure as well as the local raw material supply situation, in terms of quantity, quality and price needs to be critically scrutinized to make GREASOLINE an economically viable process.

Needless to say that a company, which wants to operate a GREASOLINE plant, requires access to a suitable fuel distribution and storage or fuel blending system.

The GREASOLINE technology can open the fossil-style fuel market for SMEs. The main technological barriers have been overcome. Operation of the erected small technical scale plant will prepare and facilitate fund-raising for the next stage, which implies engineering and construction a larger pilot plant to process about 50 kg/h of waste fat or more. This means a rather low risk scale-up of the 3 kg/h »Oberhausen sts-plant«.

Theory and experiments clearly showed that especially the waste fats and oils of poor quality, i.e. those displaying a high content of free fatty acids, are an ideal low-cost feedstock for GREASOLINE plants. Thus, the technology contributes to bio-based fuel supply without touching food resources. Apart from this ethics- and sustainability-impact, European SMEs will be enabled to engage themselves in the production of plants for a world-wide biofuels market as well as in advanced fuel production. A strong impact on Europe's ecology and SME competitiveness will be ensured, resulting even in an export of the technology beyond the EU's borders.

The GREASOLINE technology will also allow to participate in technology developments of the future, like employing low-quality fats and oil containing left-overs from the processing of Jatropha and algae derived fats and oils. Also not refined crude natural oils from those or other sources, even when contaminated with fossil fuels, e.g. vegetable oil based lubricants, can be processed.

6 Dissemination and use

6.1 Project logo and project website

While »greasoline®« was registered as a trademark in the project's run-up already, also a project logo was created, and a project website went online (**www.greasoline.com**).

Figure 16: The GREASOLINE logo

6.2 Exploitable knowledge and its use

An exploitation plan was developed, and exploitation agreements between the partners were made. Target clients were contacted early.

Exploitable Knowledge (description)	**Exploitable product(s) or measure(s)**	**Sector(s)** of application	Timetable for commercial use	Patents or other IPR protection	**Owner** & Other Partner(s) involved
1. Technology for production-scale-applicable GREASOLINE plants	*Technology to build and operate GREASOLINE plants*	*1. Biofuels production* *2. Waste fast disposal*	*2009* *2009*	*A process and materials patent is planned for 2008*	*Not defined yet*

6.3 Dissemination of knowledge

A lot of trade fairs, partnering events and scientific symposia, lecturing on GREASOLINE and at the same time gathering GREASOLINE-related information were attended.

Two GREASOLINE patents, submitted before the starting date of the project, turned out to be so comprehensive that in the course of the project nothing sufficiently innovative and new for an additional registration could be developed. This means that the process is protected by patents held by the Fraunhofer-Gesellschaft and the project partners are able to exploit the knowledge gathered within the project under the coverage of these patents. Subsequently to the project, it is planned to continue the experimental phase and market exploration activities together with the consortium. This includes amongst other things, preparation of a business plan, and the initiation of activities geared towards construction of a larger pilot plant. By then, more patent-worthy developments are expected.

Far more than 60 different dissemination activities were performed, ranging from scientific journal articles to a TV-edutainment-sequence in national-wide broadcasted TV.

Lectures ranged from practical-orientated experience reports *(Kraft, Axel: »Experiences with the program module cooperative research in FP6 from the view of a research provider«,)* to scientific congress lectures *(Heil, Volker: »Biogenous waste fats as raw material for diesel fuel- and gasoline-components via pyrocatalysis at activated carbons«)*. Posters were presented at trade fairs *(ENTSORGA-ENTECO - The International Trade Fair for Waste Management and Environmental Technology; Cologne/Germany, October 24th - 27th, 2006)* and international scientific congresses *(»5th Euro Fed Lipid Congress and 24th Nordic Lipid Symposium«, Gothenburg/Sweden; September 16–19th, 2007)*. GREASOLINE was presented at meetings of policy-makers and industrial representatives *(»NRW: Ideas and Innovation. Fuels of the future«, June 13th, 2007, Bibliothèque Solvay, Brussels/Belgium, DA 35)* and to the public at large with contributions to news- and edutainment-TV-shows *(Quarks & Co [a German scientific television show]: »Alternative ideas to crude oil application«, broadcasted March 11th, 2008)*. Printed press publications on GREASOLINE included papers both in practical-orientated (*Forum NewPower Vol. 1, 2007, Issue 1, pp. 46-47, DA 33)* and scientific-orientated *(Chemie Ingenieur Technik 79 (9), p. 1330, 2007)* journals. Moreover, the project team supported a team of secondary school pupils and enabled them to report about GREASOLINE in a daily newspaper, contributing to a federal-state-wide, unicef-supported project »ZEUS-Zeitung und Schule« (»Newspaper and School - http://www.learn-line.nrw.de/angebote/zeus/).

Figure 17, Figure 18, Figure 19 and Figure 20 provide impressions of selected dissemination activities.

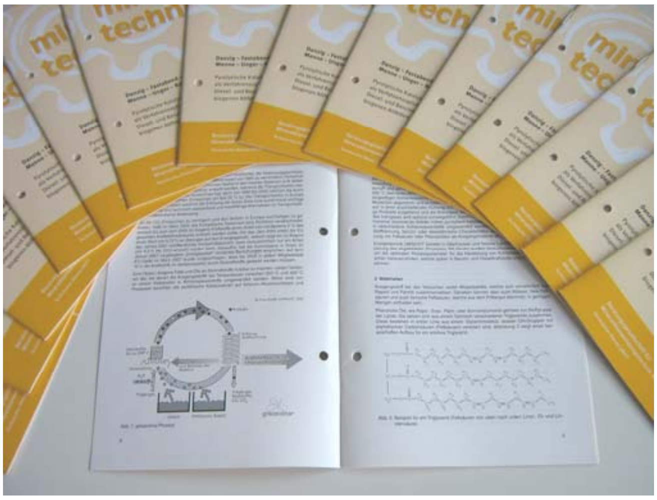


Figure 17: The GREASOLINE-issue of the scientific journal »Mineralöltechnik«; Screenshot of »WDR Aktuell«, broadcasted on November 21st, 2007

Figure 18: Senior classes' pupils, listening to a GREASOLINE school lecture

Figure 19: Interested visitors read the GREASOLINE poster and take hand-outs at the »oils+fats 2007«

greasoline-News - Windows Internet Explorer

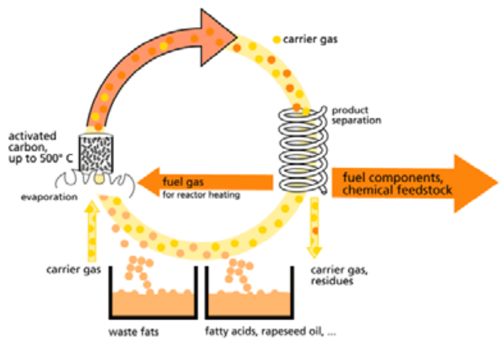
greasoline-News, last revision: 29th April 2008

Upcoming Events / Latest News / Work in Progress / Read More / Acknowledgements

Keep me posted: the e-mail newsletter

The greasoline homepage

Publishing Notes, Usage Rights and Disclaimer



The greasoline process concept

Upcoming Events: We will lecture on »Producing Petrol-quality Fuels from Biogenous Fat and Oil Residues and Wastes by Catalytic Pyrolysis at Activated Carbon« at the 4th International Conference on Renewable Resources & Biorefineries RRB4, 1st-4th June 2008, Rotterdam, The Netherlands.

Furthermore, we intend to present a paper on »Food-neutral, Petrol-quality Fuels from Biogenous Fat and Oil Waste Fats by Catalytic Cracking at Activated Carbon« at the European Biofuel Congress 2008, 24th-25th June 2008, Essen/Germany.

Latest News: Volume 10/2007 of the Journal »Mineralöltechnik« presents the greasoline technology in a detailed article »Pyrolytische Katalyse an Aktivkohlen als Verfahrensoption zur Herstellung von Diesel- und Benzinkomponenten aus biogenen Altfetten« (available only in German languauage)

The EnergieAgentur.NRW (EnergyAgency.NRW) published a booklet »Erfolgreich mit Energieprogrammen der EU - Good Practice-Beispiele aus Nordrhein-Westfalen« (Successful with energy programs, funded by the EU - Good Practice-Examples from North Rhine-Westphalia) in March 2008, covering GREASOLINE. (please choose: complete booklet / overall internet version / greasoline internet version)

On 11th March 2008, the German science TV show »Quarks & Co.« spent a complete show on biofuels - among others, on greasoline.

We addressed a scientific audience with a lecture on greasoline technology at the annual meeting of ProcessNet, the main event of Germany's chemical engineering scientific community, in Aachen, Germany, on 18th October 2007, and representatives of SMEs with an experience report concerning the greasoline FP6-project on a meeting »Successful with energy programs, funded by the EU« in Essen, Germany, on 9th April 2008.

Work in Progress: Current investigations in the **lab-scale plant** focus on the effect of additives that are led to the fat vapour stream and on the extension of the raw material base.

The **small technical scale plant** in Oberhausen is being assembled. We schedule initial operation for early summer 2008.

Read more: Please download additional information:

The latest greasoline hand-out (April 2008)

The sheets of the experience report concerning the greasoline FP6-project presented in Essen

NRZ

Freitag, 14. Dezember 2007
NBOB_35

ZEUS Zeitung und Schule
ein Projekt der NRZ
unicef

TECHNIK & UMWELT

Sprit aus Frittenfett

„Greasoline" ist ein Projekt, das vom Frauenhofer-Institut für Umwelt-, Sicherheits- und Energietechnik in Oberhausen durchgeführt wird. Ziel ist es, aus Altfetten und pflanzlichen Ölen Produkte herzustellen, deren Zusammensetzung Diesel beziehungsweise Benzin sehr ähnlich ist.

Diese Produktion ist sehr umweltfreundlich, da alles auf nachwachsenden Rohstoffen basiert. Die Fette stammen hauptsächlich aus minderwertigen Fetten (zum Beispiel Tierfette aus der Schlachterei, abgeschiedene Fette aus Kläranlagen). Aber auch Frittenfett wird gern genommen. Der aus Altfetten gewonnene Sprit ist geeignet für alle Diesel- und Benzinmotoren.

Allerdings kann dieser Diesel aus nachwachsenden Rohstoffen nur preislich mit den anderen Treibstoffen mithalten, wenn auf ihn eine Steuerbefreiung gegeben wird.

Vermutlich wird der Verbraucher diesen Sprit in circa drei Jahren nutzen können.

Text und Foto: Marius Diefenbach, Jonathan Heil, 7c, Sophie-Scholl-Gymnasium

Figure 20: The start-up view of the GREASOLINE newspage, retrieved on 29th April, 2008, and the GREASOLINE-article of the federal-state-wide, UNICEF-supported project »ZEUS-Zeitung und Schule« (»Newspaper and School«)

7 Co-ordinator contact details

Dr.-Ing. Volker Heil

Fraunhofer Institute for Environmental, Safety and Energy Technology UMSICHT

Osterfelder Str. 3

46047 Oberhausen

Germany

phone: ++49 208 8598 11 63

fax: ++49 208 8598 14 24

e-mail: volker.heil@umsicht.fraunhofer.de (personal); team@greasoline.com (team)

web: http://www.umsicht.fraunhofer.de/; http://www.greasoline.com