# Processes for Advanced Fuel Production from Biomass

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Introduction

In view of a world-wide growing fuel demand coming along with a strong environmental impact, exploration of alternative resources, preferably renewable ones, is vastly stimulated. Regarding carbon-based fuels, biomass is the only renewable feedstock which can be converted efficiently to fuels employing either fermentative or chemical processes.[1] Naturally, the use of residues and wastes is preferred to circumvent competition with customary markets, especially the nutrition sector. Typical fermentative procedures, which are already employed on large scale, are the production of ethanol from sugars, starch or cellulose[2] as well as methane production via anaerobic digestion of biomass.[3]

Regarding the non-enzymatic pathways, one major strategy is the production of synthesis gas (carbon monoxide/hydrogen mixtures) by thermochemical conversion of biomass, employing pyrolysis and gasification methods (Scheme 1).[4] In principle, synthesis gas can also be obtained from CO_2, e.g. by dry-reforming (reaction of CO_2 with methane) or reduction with hydrogen (reverse water-gas shift reaction). Traditionally, synthesis gas is produced from fossil resources like coal and natural gas via gasification and reforming processes (Scheme 1) and the corresponding know-how can be transferred, at least partially, to biomass conversion.

Synthesis gas acts as a platform for the conversion of different feedstocks to a series of potential fuels, e.g. hydrogen via the water-gas shift reaction, methane via methanation reactions or hydrocarbons with variable chain lengths via Fischer-Tropsch synthesis (Scheme 1).[5] Further options are the synthesis of methanol and dimethyl ether (DME). The latter can be
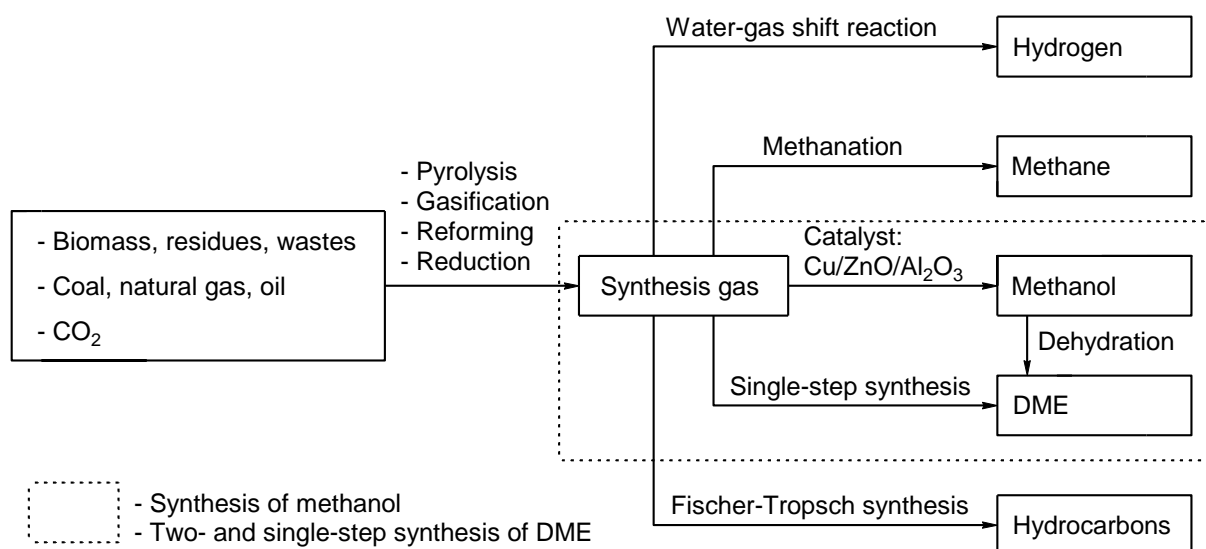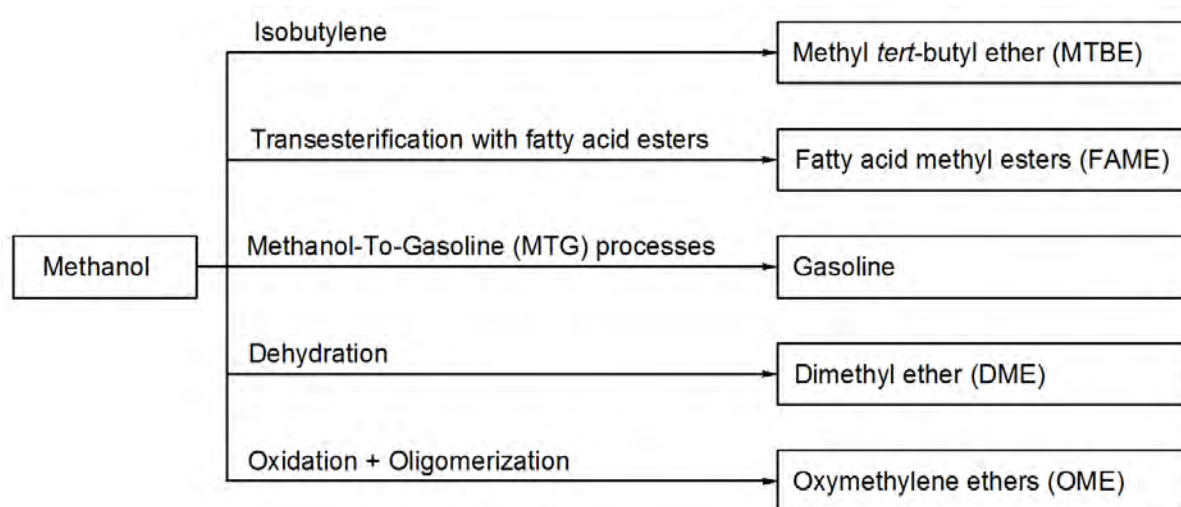

Scheme 1. *Synthesis of fuels from various resources via synthesis gas.*

obtained either by dehydration of methanol or in a single-step procedure directly from synthesis gas (Synthesis gas-To-DME process, STD).[6] Fischer-Tropsch or methanol/DME syntheses are integral technologies of so-called Biomass-To-Liquid (BTL) processes. Major

advantages of the resulting BTL fuels are the broad feedstock variability, high fuel quality and their similarity to customary fuels. Thus, elaborate changes of the fuel supply infrastructure and modification of engines are not necessary. Compared to Fischer-Tropsch synthesis, Methanol/DME-producing and -converting processes offer several advantages such as a high product flexibility and also selectivity. Thus, complex and costly product separation technologies can become unnecessary.

The use of methanol as a fuel has been investigated extensively in the past and, according to literature, methanol/gasoline blends containing up to 20% of methanol can be used without considerable engine modification.[7] However, obstacles such as the increased vapour pressure, increased corrosion, lower energy density and safety concerns have restricted large scale applications.[8] Nonetheless, methanol is needed for the production of methyl *tert*-butyl ether (MTBE) from isobutylene[9] and for the production of fatty acid methyl esters (FAME, biodiesel) by the transesterification of fats (Scheme 2).[10] Both procedures are well-established and MTBE as well as FAME are largely employed in the gasoline and diesel sector, respectively. Methanol can also be converted to hydrocarbons in so-called Methanol-To-Gasoline (MTG) processes, which are, compared to Fischer-Tropsch processes, highly selective and usually yield only hydrocarbons with up to ten carbon atoms.[11] Methanol can also serve as starting material for the production of DME[12] and the related oxymethylene ethers (OMEs, Scheme 2).[13] Both can be described by the formula $CH_3O\text{-}(CH_2O)_n\text{-}CH_3$ (DME: $n = 0$, OME: $n = 1\text{-}7$) and offer promising characteristics such as high cetane numbers and soot-free combustion.[14] Thus, DME as well as OMEs are considered as promising substitutes or additives for diesel fuels.

| Methanol		
	Isobutylene	Methyl *tert*-butyl ether (MTBE)
	Transesterification with fatty acid esters	Fatty acid methyl esters (FAME)
	Methanol-To-Gasoline (MTG) processes	Gasoline
	Dehydration	Dimethyl ether (DME)
	Oxidation + Oligomerization	Oxymethylene ethers (OME)

Scheme 2. *Synthesis of fuels and fuel additives from methanol.*

Another promising pathway for the production of hydrocarbon fuels is the conversion of methanol or higher alcohols to olefins followed by olefin oligomerization and hydrogenation of the resulting products. Compared to conventional MTG processes, the product spectrum can be extended to hydrocarbons with more than ten carbon atoms and thus, kerosene and diesel are also accessible. Methanol-To-Olefin (MTO) processes have been investigated widely in the past and coupling with so called Conversion-of-Olefins-to-Distillate (COD) processes has been realized for instance in the Methanol-To-Synfuel (MTS) process developed by Lurgi[15] or the Mobil-Olefin-To-Gasoline/Distillate (MOGD) process.[16]
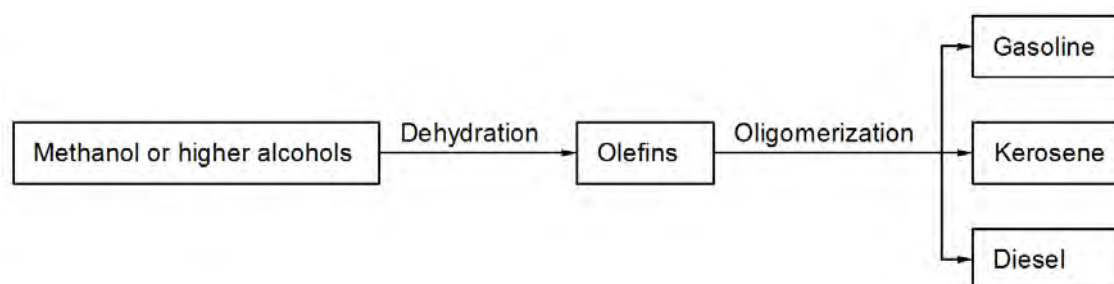
Scheme 3. *Synthesis of hydrocarbon fuels from alcohols via olefin oligomerization.*

This work highlights recent activities at KIT-IKFT in the field of biomass conversion to fuels, focussing on the methanol/DME pathway. These include the bioliq® process, which represents the entire process chain from biomass conversion to synthesis gas up to gasoline production via DME. Thus, single-step synthesis of DME from synthesis gas via STD technology is described as well as DME conversion to gasoline (DME-To-Gasoline process, DTG), analogous to classical MTG reactions. A related topic, which is also considered, is the synthesis of OMEs from methanol and methanol-derived products such as formaldehyde, dimethoxymethane (DMM) or trioxane. The latter topic is rapidly developing due to the similarity and compatibility of OMEs with diesel fuel and due to their favourable combustion properties. In all processes catalysts play a key role and thus, recent progress in the field of catalyst development is also reported.
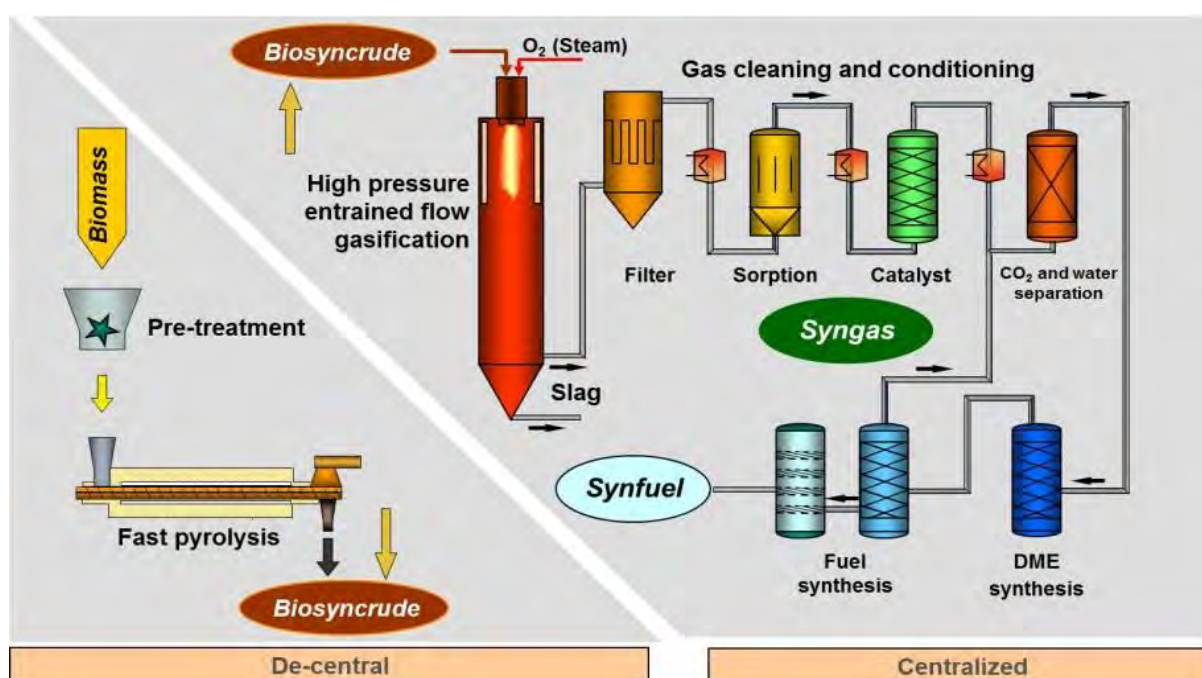
Scheme 4. The bioliq® process.

The bioliq process

Installation of the bioliq® process (Scheme 4)[17] was finalized and the entire process chain was launched in 2014. It comprises fast pyrolysis of biomass in the initial step (bioliq® I) followed by gasification of the thus obtained pyrolyzate (bioliq® II) and, after cleaning of the resulting syngas, direct conversion to DME (bioliq® III) without isolation of intermediately formed methanol. The process is dimensioned for a 700 Nm^3h^{-1} syngas flow and DME is

subsequently converted to gasoline in a DTG step (bioliq® IV). Fundamentals of fuel synthesis in the bioliq® steps III and IV are outlined in the following chapters.

The balances for mass and energy and the cost situation of the conversion of biomass into fuels has been investigated in detail by Trippe et al..[18] From the primary energy in the feedstock 34% are converted into energy content of the fuel which is produced. According to 1,0 and 1,8 €/kg fuel depending on the scale of the installation and the feedstock costs.[19]

Single-step synthesis of DME from synthesis gas

Due to its physico-chemical properties, DME is an appropriate substitute for liquefied petrol gas (LPG) and, due to its high cetane number, it is also considered as a promising diesel fuel.[20] In contrast to common diesel fuel, combustion of DME is soot free, its ozone depletion potential is zero and its global warming potential is significantly lower compared to other greenhouse gases. Furthermore, replacing diesel by DME does not require any elaborate modification of diesel engines. DME can easily be liquefied and used in household as well as industrial applications. Favorably, the established LPG infrastructure for storage and transportation can also be used in the case of DME, requiring only slight modifications.[21]

Formerly, applications of DME concentrated on its use as propellant and for the preparation of dimethyl sulfate but the markets have shifted and today DME is predominantly used as a LPG substitute. The variety of applications caused an increased interest in DME, which is reflected in the growth of the global DME market. Global DME capacity was approximately 5 Mio t/a in 2009 and, as an example, Chinese capacities for DME production are expected to reach 13 Mio t/a in 2018.[22] The use of DME as transportation fuel has been investigated intensely within the last years and fleet tests, employing especially trucks and buses, have already been conducted.[23]

As already mentioned above, DME can be synthesized either by catalytic dehydration of methanol or in a single-step directly from synthesis gas (Scheme 1). The one-step STD procedure comprises three reactions, according to equations (1) to (3). Synthesis gas is first converted to methanol (1), which is dehydrated in the following etherification step to yield DME (2). The released water is consumed in a simultaneously proceeding water-gas shift reaction, which produces hydrogen and CO_2 (3). Thus, equation (4) applies for the overall process.[24]

$$2\,CO \;+\; 4\,H_2 \qquad \rightarrow \quad 2\,CH_3OH \qquad\qquad\qquad (1)$$

$$2\,CH_3OH \qquad\qquad \rightarrow \quad CH_3OCH_3 \;+\; H_2O \qquad\quad (2)$$

$$CO \;+\; H_2O \qquad\quad \rightarrow \quad H_2 \;+\; CO_2 \qquad\qquad\qquad (3)$$

Overall:

$$3\,CO \;+\; 3\,H_2 \qquad \rightarrow \quad CH_3OCH_3 \;+\; CO_2 \qquad\quad (4)$$

Slurry phase as well as fixed bed technologies were developed for this process and the latter technology was chosen for the STD process elaborated at KIT. Investigations concentrated on the use of biomass-derived syngas, i.e. carbon monoxide-rich syngas, as starting material with H_2/CO ratios around 1 and variable further components. Regarding reaction conditions, best results were obtained in the temperature range from 200 to 300 °C under pressures ranging from 3 to 8 MPa.[25] Regarding catalyst systems for the STD process, ternary mixture of copper-, zinc- and aluminum oxides ($Cu/ZnO/Al_2O_3$, CZA) are among the most efficient for methanol synthesis via hydrogenation of carbon monoxide. The dehydration step requires acidic catalysts, such as zeolites or γ-Al_2O_3. Thus, mixtures of CZA and γ-Al_2O_3 are frequently applied. In this context, new bifunctional catalysts were developed which contain a

component for methanol formation as well as an acidic component for methanol dehydration in one and the same catalyst system.[26] Furthermore, highly active catalysts for methanol synthesis were developed employing innovative preparation concepts, e.g. flame-spray synthesis.[27]

Most of the currently operating DME plants use methanol as starting material.[28] The good availability of methanol and the simple processing favors the two-step process. Furthermore, water is the only by-product, whereas the STD reaction also produces CO_2 as by-product. However, several benefits of the single-step process lead to the installation of pilot plants that produce DME directly from synthesis gas. The combination of two processes in one plant can reduce not only operational costs but also energy consumption. The direct synthesis requires lower activation energies than the two-step process. Furthermore, methanol synthesis is an equilibrium reaction that is thermodynamically limited. A subsequent removal of methanol by dehydration leads to improved conversion rates.[29] If biomass-derived synthesis gas is applied, the direct synthesis of DME is favorable since an equimolar H_2/CO ratio can be employed according to equation (4) for the overall reaction.

Conversion of DME to gasoline

In the 1970ies, development of several MTG processes for the conversion of methanol to gasoline started in consequence of the oil crises.[30] Some of the most important processes were the fixed-bed process from Mobil in Motunui/New Zealand, the fluidized-bed process from Mobil/Uhde/RBK in Wesseling/Germany and the Topsøe Integrated GAsoline Synthesis (TIGAS) in Houston/Texas. Some processes such as the Mobil-Olefin-To-Gasoline/Distillate (MOGD) process and the Methanol-To-Synfuel (MTS) process developed by Lurgi also allowed for the production of diesel besides gasoline.[31] Precondition for these process developments was the discovery of ZSM-5 zeolites by co-workers of Mobil, which exhibited high catalytic activity and selectivity in the conversion of methanol to hydrocarbons.[32] Instead of methanol, DME can also be employed as starting material for the synthesis of gasoline. The so-called DME-To-Gasoline (DTG) process has been investigated in detail and several advantages regarding reaction heat, yield, selectivity, reactor design and process conditions were reported.[33]

Major benefits are the high overall energy efficiency of MTG/DTG processes with almost quantitative methanol conversion and high gasoline yield as well as the high fuel quality, which can also exceed current gasoline specifications. The resulting gasoline typically contains highly branched alkanes and alkenes (53 and 12 wt.%, respectively), cycloalkanes (7 wt.%) and aromatics (28 wt.%),[34] which is in good agreement with common gasoline.

Major constraints of MTG and DTG processes are predominantly related to the catalysts. These are prone to deactivation either by coking or dealumination. To improve catalyst performance, hierarchical zeolites containing both micropores and mesopores were synthesized at KIT-IKFT. Top-down methods, e.g. modification of already existing ZSM-5 zeolites by desilication, as well as bottom-up methods such as hydrothermal zeolite synthesis in the presence of templates have been employed. Organosilanes proved to be suitable templates and the resulting catalyst systems exhibited enhanced stability and selectivity in the DTG reaction. This was attributed to an optimized interplay of acid sites, micropores and mesopores.[35]

### OME synthesis from different starting materials

As can be seen from the world-wide increasing number of publications and patent applications, synthesis and application of OMEs is a rapidly developing research field (Figure 1). Especially Chinese OME activities grew enormously in recent years, which can be

attributed to the high capacity for methanol production and the search for alternative applications of methanol, including new strategies for methanol processing. Additionally, physico-chemical as well as combustion characteristics indicate a very high potential of OMEs for fuel applications. Oligomeric OMEs bearing five to seven carbon atoms (CH_3O-$(CH_2O)_n$-CH_3 with n = 3-5) exhibit physico-chemical properties similar to diesel fuel, exceptional high cetane numbers far above 100 and clean combustion without soot-formation.[36]
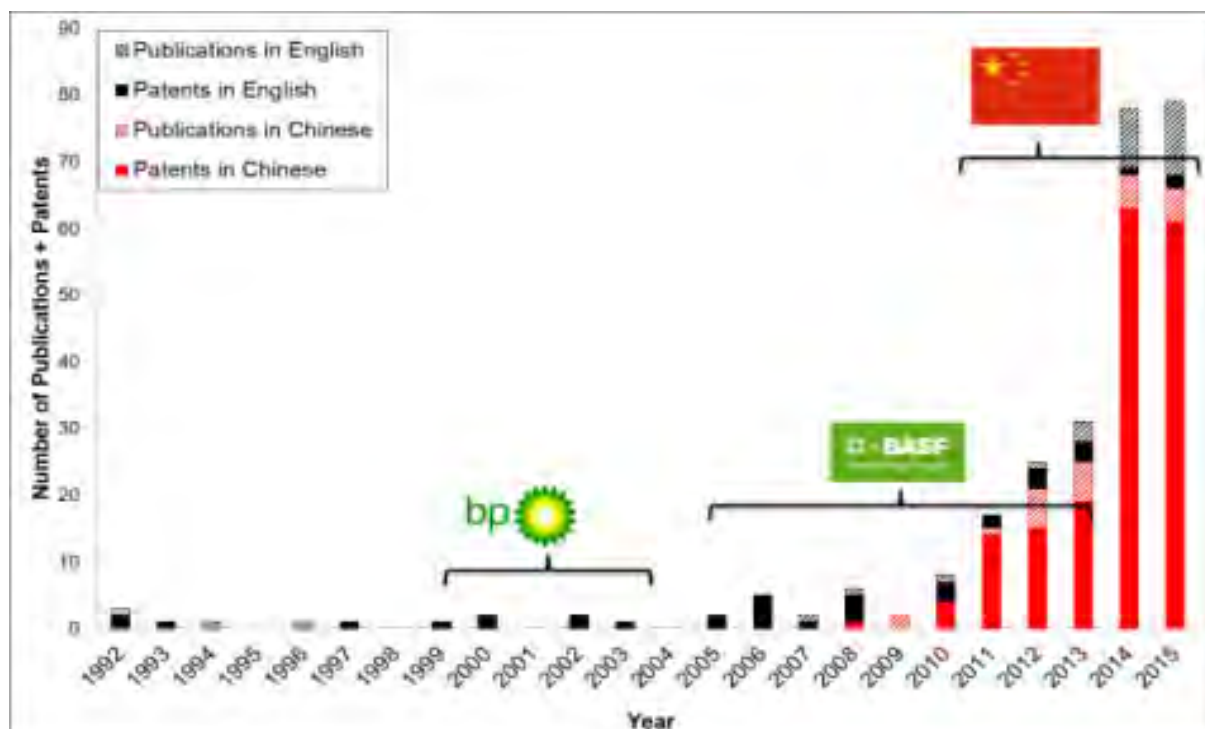
Figure 1. Publications and patents on oxymethylene ethers (OME) according to SciFinder.

Several pathways for the synthesis of OMEs are described (Scheme 5). Comparatively well-known are the synthesis from dimethoxymethane (DMM = OME-1) and trioxane (Scheme 5, pathway A)[37] and synthesis from methanol and formaldehyde (Scheme 5, pathway B).[38] The latter strategy is certainly the most desirable since the low cost educts methanol and its oxidation product formaldehyde can be employed. Further process variants, which are not displayed in Scheme 5, are OME synthesis from DMM and formaldehyde and the reaction of DME with formaldehyde sources.
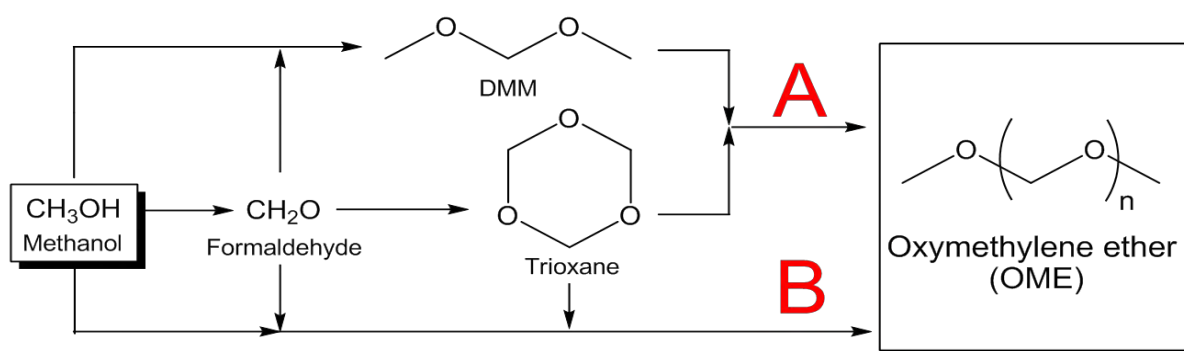

*Scheme 5. Synthesis pathways for oxymethylene dimethyl ethers (OMEs).*

At KIT, the classical synthesis of OMEs from DMM and trioxane (pathway A) has been investigated in detail. As a result, the reaction could be further optimized. The resulting OME mixtures have been separated, purified and physico-chemical as well as fuel properties of each oligomer have been determined.[39] Furthermore, synthesis of OMEs from methanol and formaldehyde (pathway B) has been investigated and recent results regarding both pathways are described in the following.

For the reaction of DMM with trioxane, a series of solid acid catalysts was investigated. Several ion exchange resins as well as zeolites exhibited high activity and the course of reaction employing the ion exchange resin Amberlyst 36 is illustrated in Figure 2. Reaction equilibrium is reached after about 30 min and the reaction mixture comprises residual educts and oligomeric OMEs with four to seven carbon atoms. Low amounts of OME-6 and higher oligomers are also present. Product mixtures can easily be separated by distillation and the remaining educts can be recycled. Formation of unwanted byproducts, especially formaldehyde, can be efficiently suppressed if dry educts are employed.
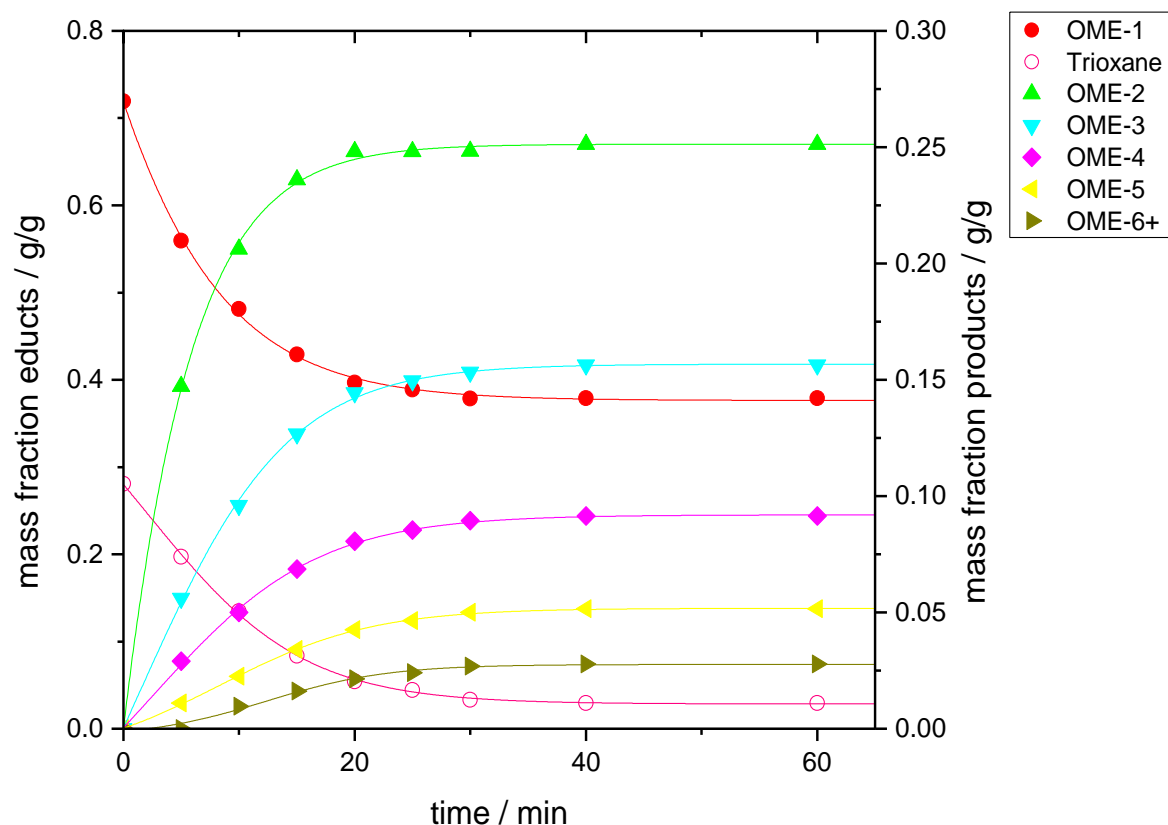

Figure 2. *Synthesis of OMEs from dimethoxymethane (= OME-1) and trioxane (reaction conditions: 19 g OME-1, 7.5 g trioxane, 0.075 g Amberlyst 36, T = 60 °C).*

Corresponding reactions with methanol and formaldehyde were carried out and, besides a series of other catalysts, Amberlyst 36 was also tested (Figure 3).[40] Compared to the reaction of DMM with trioxane, a similar product spectrum was obtained. However, efficiency suffers from the formation of several byproducts, e.g. hemiformals and glycols. Another constraint is the formation of water during reaction, which would accumulate in a continuously operating process. Therefore, efficient separation and recycling strategies for the complex reaction mixtures are needed.
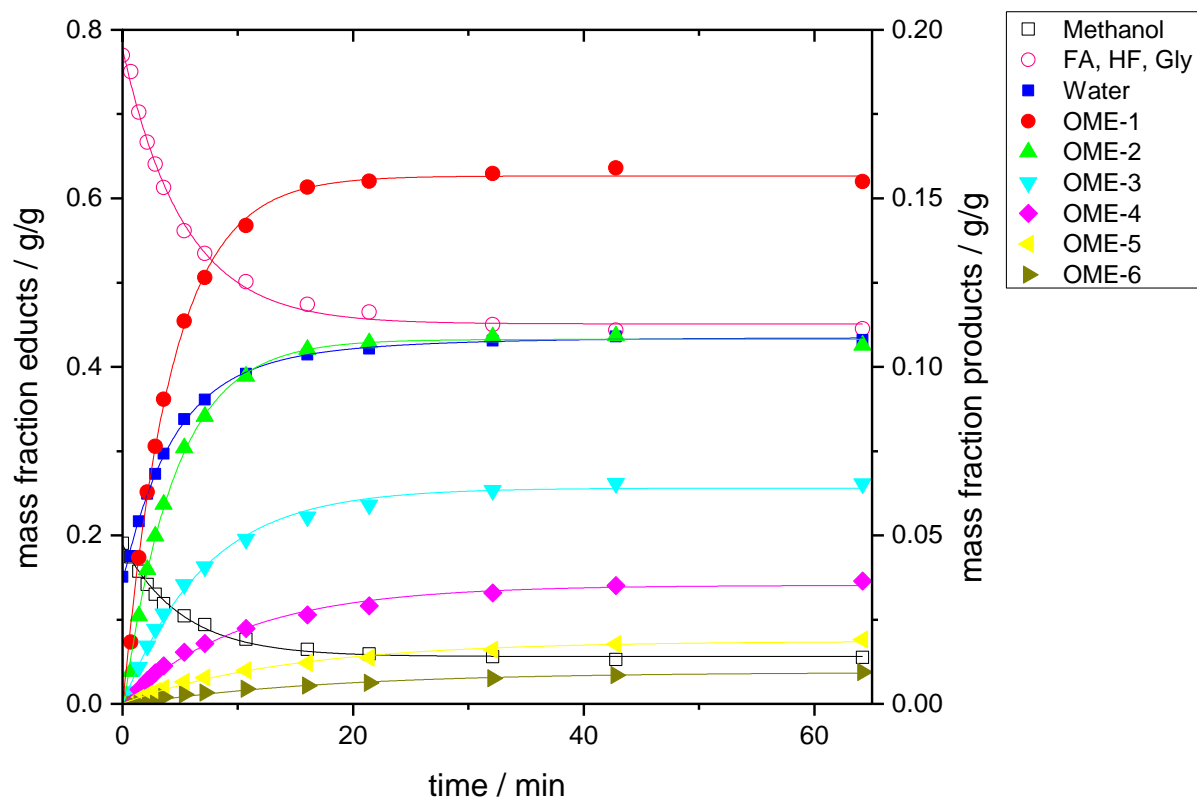
Figure 3. *Synthesis of OMEs from methanol and paraformaldehyde (reaction conditions: 40 g methanol, 60 g paraformaldehyde (5% water), 1.0 g Amberlyst 36, T = 80 °C); FA = formaldehyde, HF = hemiformals, Gly = glycols.*

Currently, important parameters for OME synthesis from methanol and formaldehyde are optimized, focussing on stoichiometry, temperature and catalysts. One promising option is a two-phase process comprising OME synthesis in aqueous phase and selective OME extraction with a tailored extracting agent. Such a concept is outlined in Scheme 6.
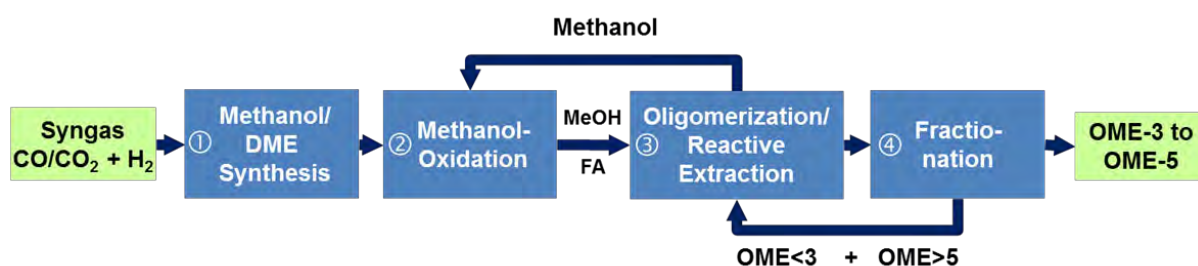
Scheme 6. *Synthesis of OMEs from synthesis gas via methanol and formaldehyde.*

Conclusion

Driven by the bioliq[®] process, which covers the complete process chain from biomass supply to gasoline production, several strategies for the production of fuels from biomass have been investigated in detail. Predominantly, technologies for the conversion of synthesis gas to methanol or DME and several downstream options have been considered with a focus on DME conversion to gasoline and OME synthesis from methanol and methanol-derived compounds.

At present, coal and natural gas are still the main sources for synthesis gas generation. Production and use of biomass-derived synthesis gas is on an advanced stage of development but not yet established on large scale. Regarding DME production, all commercial plants use methanol as starting material, i.e. the two-step synthesis via methanol dehydration is applied. Whether the single-step synthesis will become established depends on how synthesis gas will be produced in the future and on the optimization of the underlying processes. DME is mainly used as LPG-substitute and its importance is steadily increasing in this area. New DME production plants are under construction and global capacity will continue to rise. The use of DME as diesel substitute reached an advanced testing phase, including fleet tests with trucks and buses, and its application in future fuel markets is probable.

Regarding gasoline production from methanol, several activities are currently noticeable and these are based on experiences from the 1980ies. At that time, enormous efforts were made to minimize the dependence of fuel supply on crude oil. However, the employed synthesis gas in former and current developments is mainly stemming from natural gas and coal. Regarding biomass-derived synthesis gas, the following challenges are decisive: (i) Efficiency and heat integration of the overall process ranging from the generation of synthesis gas to fuel production have to be optimized. (ii) Due to a variety of biogenic raw materials, gasification of biomass leads to different synthesis gases with varying H_2/CO ratios, by-products and impurities. Thus, processes need to be highly flexible with regard to the gas feed. (iii) Catalyst systems have to be improved, particularly concerning resistance to poisoning, lifetime and product selectivity.

With respect to OMEs, physico-chemical and combustion characteristics indicate an enormous potential as alternative diesel fuels. Nevertheless, availability is still very limited. Activities in this field are tremendously increasing and developments in China are certainly outstanding. Production on commercial scale is feasible, in principle, and methanol as well as methanol-derived products can be used as starting materials. However, production processes have to be explored much more in detail and offer a vast potential for optimization.

In summary, a variety of methods is available for the generation of synthesis gas from renewable resources and its conversion to fuels. Several processes, e.g. methanol production and conversion to DME or gasoline, are already on an advanced stage and applicable from a technological point of view. Furthermore, OMEs exhibit a promising potential, which has to be explored in the near future. However, sustainability, biomass availability and economic criteria must also be taken into account and especially the latter have restricted large scale applications so far. Some of the strategies will become important and competitive inasmuch as fossil resources will run short.

Future energy supply will certainly not depend on one but on several energy sources, e.g. solar energy, hydrogen and biofuels. A combination of biomass, acting as a carbon source, and hydrogen, which is produced through water-electrolysis with electricity from solar and wind energy, will help to tremendously increase the carbon yield of biomass and will help to utilize electricity which is available only intermittingly.

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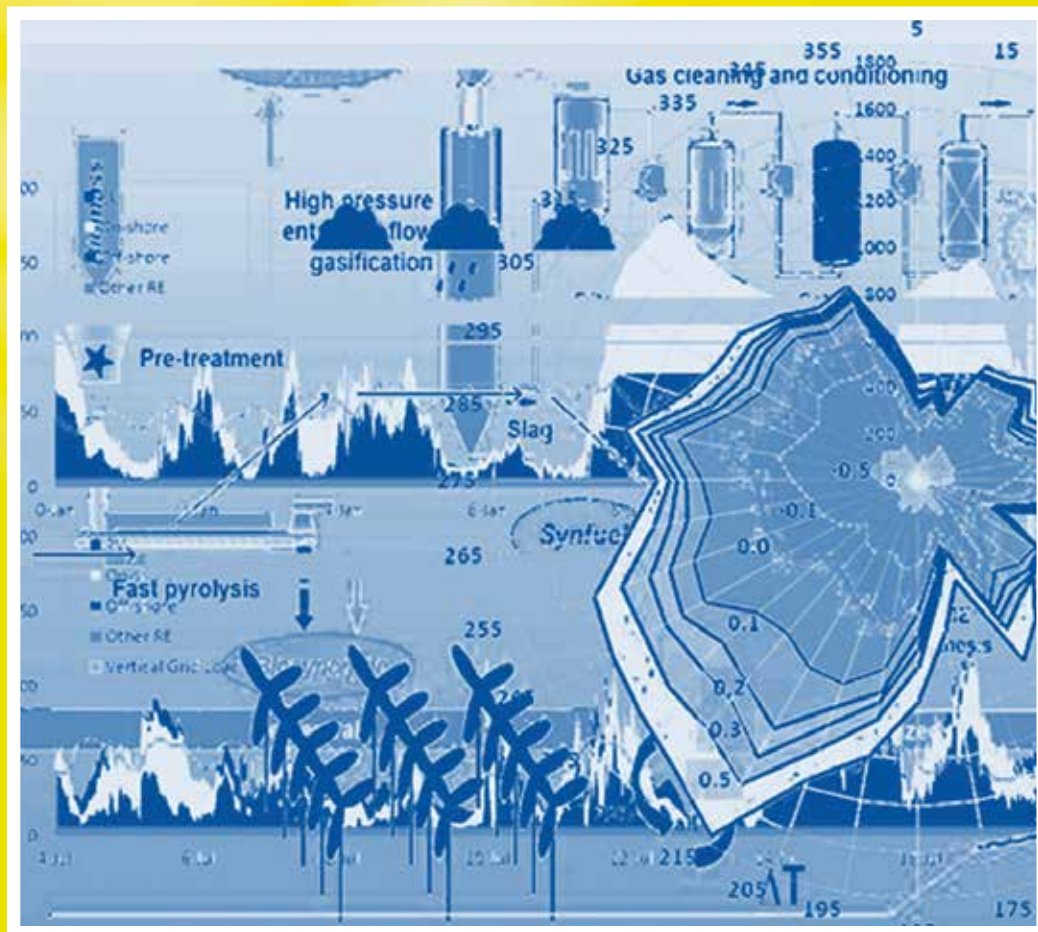
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Haupt- und Fachvorträge

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Der vorliegende Band versammelt schriftliche Ausarbeitungen von Vorträgen auf der Tagung des Arbeitskreises Energie in der Deutschen Physikalischen Gesellschaft des Jahres 2016 in den Räumen der Universität Regensburg. Leider ist es nicht gelungen, von allen Vortragenden Manuskripte zu erhalten. Die Präsentationsfolien der meisten Hauptvorträge können auf der Webseite des Arbeitskreises über:

http://www.dpg-physik.de/dpg/organisation/fachlich/ake.html

(von dort gelangt man zum Archiv des AKE) eingesehen werden. Allen, die zu diesem Sammelband beigetragen haben, sei an dieser Stelle sehr herzlich gedankt.

Düsseldorf, im August 2016   Hardo Bruhns