Storage of volatile renewable energy in the gas grid applying 3-phase methanation

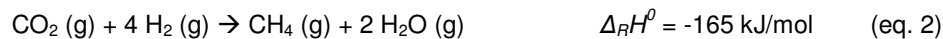***Main author***

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ABSTRACT

In order to face the enormous challenges of the climate change and of the rising energy demand of the world, an increased use of renewable energies is one viable solution. Thereby, wind power and photovoltaic offer big potentials. However, the production of electric energy from wind power and from photovoltaic is highly volatile. Therefore, in times of surplus electricity production a part of the electric energy has to be stored. By the year 2050 a storage capacity of about 16 TWh is needed for Germany, for example [2]. Unfortunately, for such high quantities of electric energy and for storage durations of several days or even weeks technologies are sparsely available. Pumped storage hydro power offers large power in and out but is limited in capacity in most countries, and the latterly discussed compressed air energy storage technology has the drawback of low energy density and poor efficiency. A reasonable alternative to store high amounts of electric energy is its conversion to chemical energy carriers like hydrogen or methane. While hydrogen can be added to the gas grid especially well in high pressure grids the generation of SNG (**s**ubstitute **n**atural **g**as) seems to be more viable for the storage of surplus energy in regional and rural distribution grids. In 2006 the world wide storage capacity for methane was about 3686 TWh of chemical energy in underground storages which was 24 % of the world's electricity demand in that year [5].

During times of surplus electricity production the excess energy could be used to produce hydrogen via PEM-electrolysis (**p**roton **e**xchange **m**embrane-electrolysis). The PEM-electrolyser copes better with fluctuating volatile electric power than the common alkaline electrolyser. Hydrogen from electrolysis and CO_2 from biogas or other CO/CO_2-sources can be used to synthesize CH_4 by the methanation reactions (eq. 1 and 2):

$$CO\ (g) + 3\ H_2\ (g) \rightarrow CH_4\ (g) + H_2O\ (g) \qquad \Delta_R H^0 = -206\ kJ/mol \qquad (eq.\ 1)$$

$$CO_2\ (g) + 4\ H_2\ (g) \rightarrow CH_4\ (g) + 2\ H_2O\ (g) \qquad \Delta_R H^0 = -165\ kJ/mol \qquad (eq.\ 2)$$

The conversion efficiency from electric energy to chemical stored in SNG is about 64 %. If the waste heat of the methanation process can be used additionally the efficiency increases. Due to the fluctuating surplus power from wind and sunlight and hence a fluctuating hydrogen production a new methanation process with increased flexibility to variable hydrogen feed flows than the established 2-phase reactor systems (mostly fixed-bed and fluidized-bed reactors) has to be developed. Due to the high heat capacity of its liquid phase, a 3-phase reactor is better suited to store the reaction heat of the methanation step in order to buffer the effect of fluctuating feed streams. Further the 3-phase methanation facilitates temperature control and heat removal.

In a joint research project of the DVGW research station at the Engler-Bunte-Institut (EBI) of the KIT (Karlsruhe Institute of Technology) and six other partners from science and industry (EnBW, Outotec, IoLiTec, h-tec, Fraunhofer ISE and Engler-Bunte-Institut) the conversion process from electric energy to SNG will be researched and developed. In this project the DVGW research station focuses on the improvement of the 3-phase methanation. This primarily means that liquid-side mass transfer limitations should be reduced and that suitable heat transfer liquids can be identified.

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2.   BODY OF PAPER

2.1   STORAGE OF VOLATILE RENEWABALE ENERGY

2.1.1   Exploitation of renewable energies
Undoubtedly, the exploitation of renewable energies has to be extended. Especially wind power and photovoltaic offer big potential. Hence, the world total installed capacity of wind power has increased considerably over the recent years (see fig. 1). In 2010, the strongest increase was observed in China, but also in the USA and in Europe power production from wind has been growing year after year.
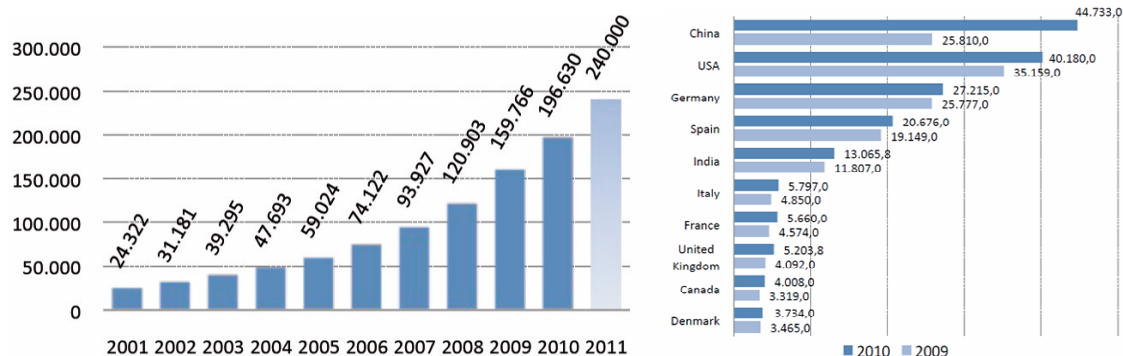

Fig. 1: Left: World total installed capacity of wind power in MW; right: Top 10 countries by total capacity in MW [1]

Unfortunately, wind power and photovoltaic produce electric energy unsteadyly and dependent on season, daytime and weather. Due to the fact that the power input and the power output of the power grid has to be in balance permanently, in times of surplus electricity production storage capacities for electricity are needed. By the year 2050 a storage capacity of about 16 TWh will be needed for Germany [2], e.g.

2.1.2   Storage technologies for electrical energy
For a storage capacity in the order of magnitude of TWh and for storage durations of several days or weeks only a few technologies are available. Some of these technologies will be described below:

Pumped storage hydro power
Electric energy is stored in the form of potential energy of water being pumped from a low level reservoir into a reservoir at a higher level and recovered by discharging the stored water through turbines. Therefore the energy density is limited by the altitude. Pumped storage hydro power stations have high energy efficiencies from 70 to 85 % (from electricity to electricity). Today, the pumped-storage plants are an important source of balance energy. However, the construction of a pumped storage plant is a massive intervention in the landscape and objections from the public are common. Furthermore, only a few countries have sufficient potential. As shown in tab. 1, the majority of the potential in Europe is restricted to a few countries (Scandinavia, Austria and Switzerland).

Tab. 1: Potential capacities for pumped storage hydro power in Europe [3]

Europe (total)	Scandinavia	Austria + Switzerland	Germany
170 TWh	120 TWh	30 TWh	0.04 TWh

Compressed air energy storage (CAES)
Surplus electric energy is used to compress air and to store it underground. Alas, the compressed air has to be cooled before storage. The stored energy is regained by routing the compressed air over a gas turbine after being preheated. If the heat is supplied by burning natural gas (= diabatic mode), the efficiency of a CAES is rather poor. By using the waste heat of a power plant the efficiency can be increased up to 55 %. Adiabatic CAES-plants which store the heat of compression in some way are

not yet state of the art. Due to the low energy density of compressed air (Fig. 2) large underground storages are necessary. This could cause a competitive situation with regard to the storage of natural gas and hydrogen or the use of the caverns or aquifers for future CCS technologies. Yet, there are two CAES plants worldwide, in Huntorf, Germany and in McIntosh, Alabama.

Electrochemical energy storage
Batteries are widely used for small and mobile applications needing stored energy. But for large storage capacities the specific costs are too high. If the storage duration is long self-discharge of the batteries is a common problem.
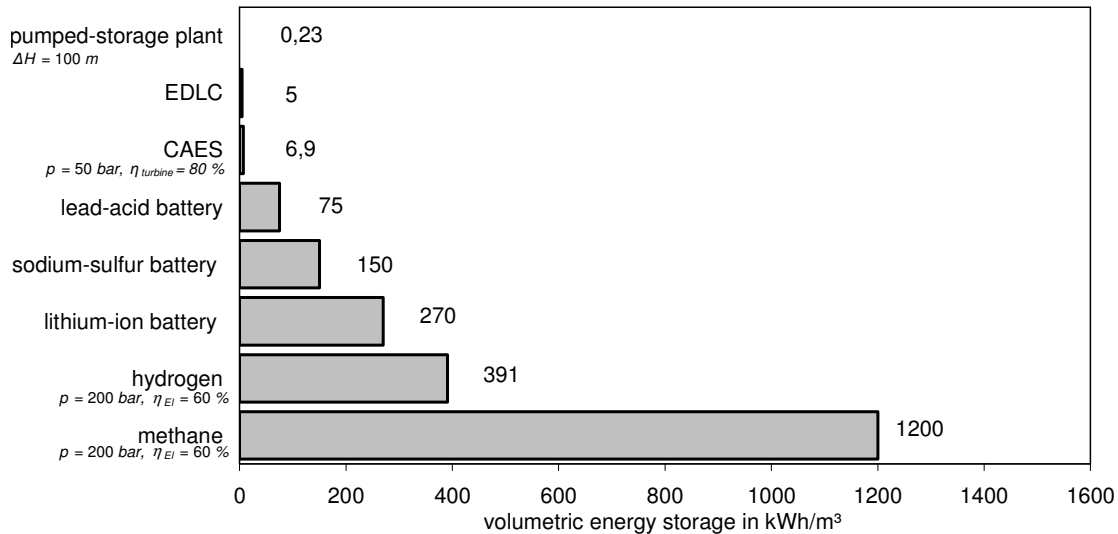

Fig. 2: Comparison of energy densities of different storage technologies (EDLC = electric double-layer capacitor) [4]

Power to gas (PtG)
As shown in Fig. 2, the highest energy density can be achieved by using chemical energy carriers like hydrogen or methane as storage medium. In 2006 the world wide underground gas storage capacity for CH_4 was about 3686 TWh of chemical energy [5]. A further advantage is that the storage time under technical conditions is not limited. Losses caused by the conversion steps are high compared to the other energy storage systems discussed earlier (fig. 3).
Surplus electric energy is used to produce hydrogen via electrolysis of water. The produced hydrogen is than methanized. Approximately 64 % of the electric energy which was used for the electrolysis can be stored as chemical energy in the form of methane and than being used as efficiently as natural gas. By using the waste heat of the methanation reaction a higher efficiency is achievable. The temperature level of this waste heat is 200 – 300 °C which offers different opportunities of application.
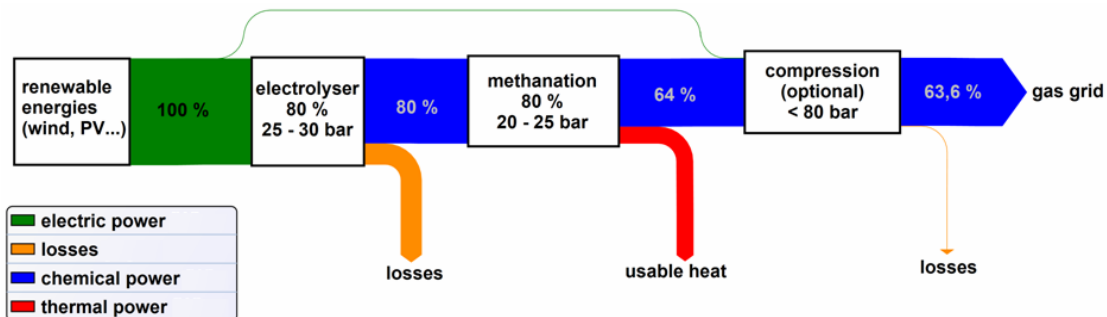

Fig. 3: Sankey diagram for the synthesis of SNG based on renewable electricity

2.1.3   Process chain Power to gas (PtG)

For water electrolysis systems with alkaline and with PEM (**p**roton **e**xchange **m**embrane) electrolytes are available. The first one has the advantage of being a proven technology with comparatively low specific costs. The PEM-electrolyser by contrast copes better with fluctuating volatile electric power. Hydrogen from electrolysis and CO or CO_2 from foreign sources are used to synthesise CH_4 by methanation (fig. 4). Operation under elevated pressure would be advantageous for the methane synthesis, especially if injection into a transportation gas grid is envisaged. In this project the operating pressure of the electrolysis is 25 – 30 bars whereas the methanation is operated at 20 – 25 bar.
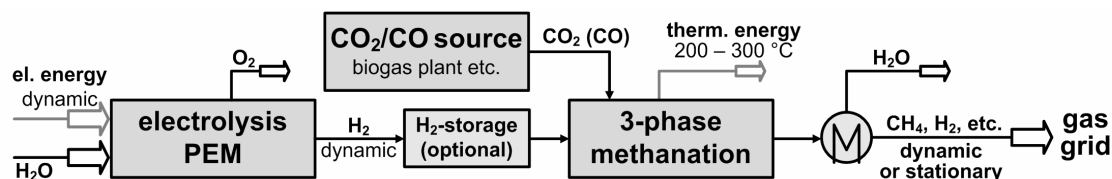

Fig. 4: Process chain for application of surplus electric energy to synthesize SNG

The carbon oxides necessary for methanation may stem from gasification or combustion processes as well as from many other forms of gas upgrading processes producing a CO or CO_2 rich off gas. Obviously, renewable carbon sources are favourable. CO or CO_2 sources are available in different sizes. Small CO_2 flows (≈ 250 – 1000 m³/h) are accessible from biogas plants or from combined heat and power units. Figure 5 shows the combination of a conventional biogas plant with the PtG process chain. Biomass gasification plants could provide CO and CO_2 flows of 10 000 m³/h or more. In this case there would be no need to shift the raw gas CO into CO_2 and consequently no need for CO_2 removal; both carbon sources could be converted completely into methane with H_2 from electrolysis. Very large CO_2 streams could be recovered from coal fired power plants' flue gases.
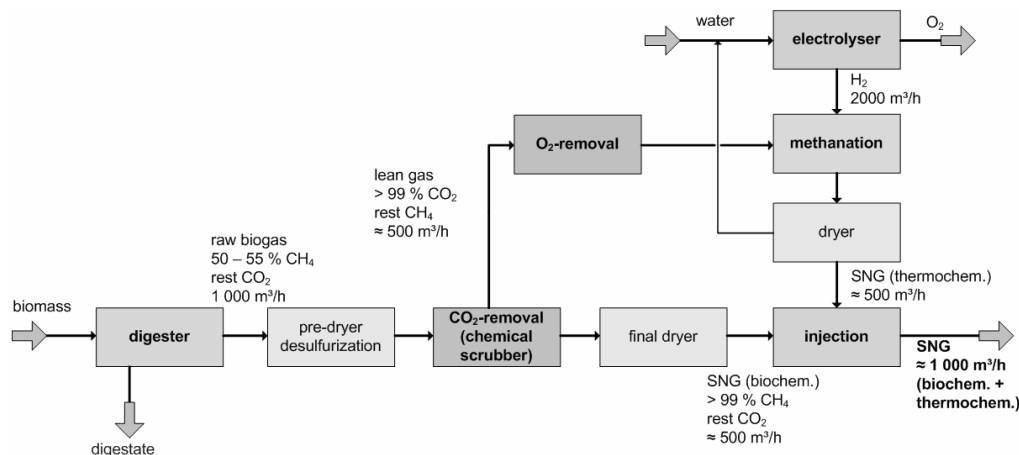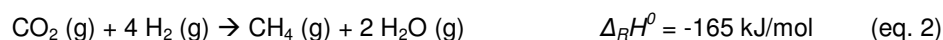

Fig. 5: Combination of a biogas plant with the PtG process chain

2.2   3-PHASE METHANATION

2.2.1   Fundamentals of methanation

The exothermic methanation reaction (eq. 1 and 2) is typically operated at temperatures between 200 °C and 500 °C and at reaction pressures ranging from 1 bar to some tens of bars. To avoid thermodynamic limitations the temperature should be low and the pressure high.

Usually Ni based catalysts are applied for methanation because of their high activity and their comparatively low price.

$$CO \; (g) + 3\,H_2 \; (g) \rightarrow CH_4 \; (g) + H_2O \; (g) \qquad \Delta_R H^0 = \text{-206 kJ/mol} \qquad (eq. \ 1)$$

$$CO_2 \; (g) + 4\,H_2 \; (g) \rightarrow CH_4 \; (g) + 2\,H_2O \; (g) \qquad \Delta_R H^0 = \text{-165 kJ/mol} \qquad (eq. \ 2)$$

As a consequence of the reaction being exothermic removal of the reaction heat is the main issue in designing the methanation reactor. Established are 2-phase fixed-bed and fluidized-bed reactors. If

fixed-beds are applied, typically 2 – 5 serially connected beds with inter-cooling are necessary. Fluidized-bed reactors offer a more effective heat removal but require abrasion resistant catalysts.

In terms of fluctuating feed streams 2-phase reactors suffer by quick cooling down if no gas is entering the reactor. This causes an additional heat demand in order to maintain reactor and catalyst temperatures above 200 °C.

Therefore, for the PtG concept a new methanation process with increased flexibility to variable feed streams has to be developed. Due to the additional heat capacity of its liquid phase, a 3-phase reactor (e. g. a Slurry Bubble Column Reactor (SBCR)) is less prone to quick cooling down in case of fluctuating feed streams. Further the 3-phase methanation facilitates temperature control and heat removal. The reactor types discussed are compared in Tab. 2.

Drawbacks of a 3-phase reactor are evaporation and especially decomposition of the heat transfer liquid as well as the possibility of mass transfer limitations in the liquid phase. Therefore, two important aims of the research activities are:

- Identification of appropriate heat transfer liquids
- Identification of operating parameters for minimized mass transfer limitations in liquid phase

Tab. 2: Comparison of reactor types for methanation (SBCR = Slurry Bubble Column Reactor)

	Fixed-bed	Fluidized-bed	SBCR
Advantages	• Low mechanical load on catalyst • Wide range of operation • Simple catalyst handling • Simple dimensioning and scale up	• Effective heat removal • Small temperature gradients • Good mass transfer • Only one reactor necessary	• Very effective heat removal • Isothermal conditions • Less sensitive to fluctuating feed streams • Only one reactor necessary • No heat exchangers inside the reactor
Drawbacks	• Removal of heat challenging • Temperature gradients • Multiple reactors in series • Mass transfer limitations due to particle size possible	• High mechanical load on catalyst (attrition) • Entrainment of catalyst	• Liquid-side mass transfer limitations possible • Backmixing possible • Evaporation and decomposition of heat transfer liquid

2.2.2 Fundamentals of Slurry Bubble Column Reactors (SBCR)

Especially in a 3-phase system the overall conversion rate can strongly be influenced by the mass transfer processes. For the feed (H_2 and CO or CO_2) four steps have to be considered (see fig. 6):

1. gas phase $\Rightarrow$ gas liquid interface
2. gas liquid interface $\Rightarrow$ bulk liquid
3. bulk liquid $\Rightarrow$ liquid solid interface
4. pore diffusion and parallel reaction

In a stationary system all steps have the same specific flow (rate) $^{n}\Phi_{i,j}/V_R$, and the slowest step determines the overall conversion rate. If a SBCR for methanation should reach the conversion rate of a 2-phase reactor, the steps 1 – 3 have to be so fast that the kinetics of the reaction is the limiting factor. The specific flow for each of the 3 steps can be calculated by eq. 3:

$$\frac{^{n}\phi_{i,j}}{V_R} = \beta_{i,j} \cdot a_{i,j} \cdot \Delta c \qquad \text{eq. 3}$$

$\beta_{i,j}$ = Mass transfer coefficient, i,j: (G = gas, L = liquid, S = solid)
$a_{i,j}$ = specific contact area
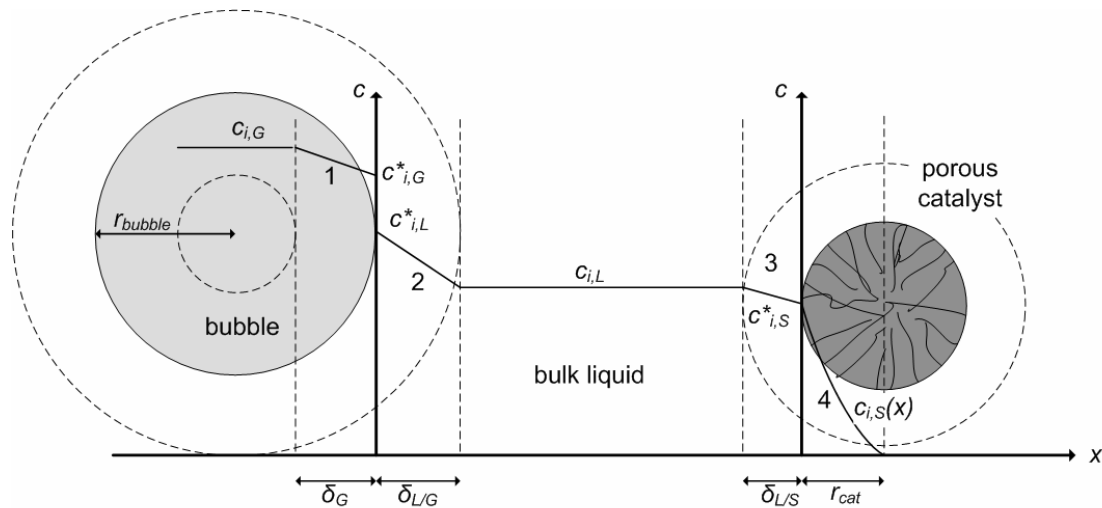Δc = concentration difference

Fig. 6: Concentration profiles for a 3-phase system with a porous catalyst according to the film model

According to literature [6 - 9] and to our own calculations step 1 is almost never rate controlling. Therefore, minimizing mass transfer limitations in liquid phase (especially in step 2) is the development aim. For this aim the mass transfer coefficient $\beta_{i,j}$ and the specific contact area $a_{i,j}$ have to be increased.

Both parameters strongly depend on the flow regime of the system. The flow regime map in fig. 7 exhibits 3 flow regimes (at methanation reaction temperature the liquids have a low viscosity). Small reactor diameters d_R and high gas velocities u_G result in large bubbles whose diameters can be as large as the reactor diameter. This regime is called **slug flow** and it has to be avoided because of its poor mass transfer characteristics. By increasing the reactor diameter and/or reducing the gas velocity the **heterogeneous regime** can be reached. Here, large and small bubbles exist in parallel. Many technical reactors operate in the heterogeneous regime because of a comparably high mass transfer coefficient $\beta_{i,j}$. For small gas velocities the **homogeneous regime** is observed where the bubbles have a small and almost the same diameter and the distribution of the bubbles is homogeneous.
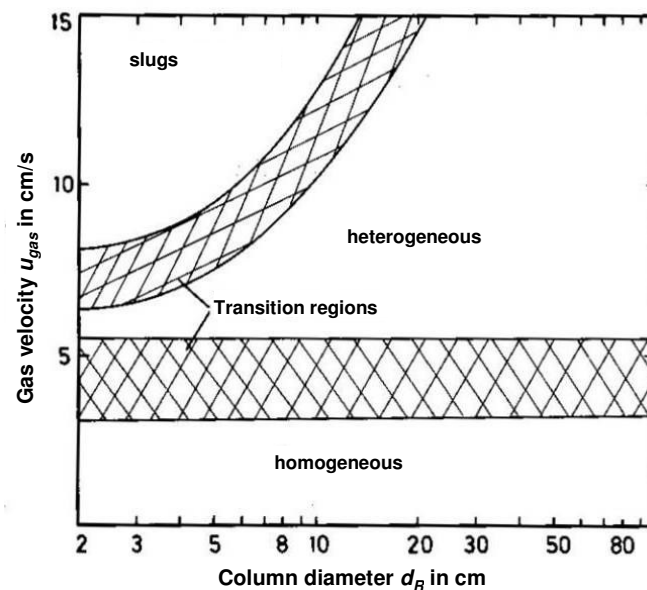

Fig. 7: Flow regime map for low-viscosity media and sinter plate gas feed [10]

The advantage of operating in the homogeneous regime is a large specific gas liquid contact area $a_{G,L}$. It can be easily calculated from the bubbles' geometry (eq. 4):

$$a_{G,L} = \frac{6 \cdot \varepsilon_G}{d_{bubble}}$$

eq. 4

The gas holdup ε_G is conventionally defined (eq. 5):

$$\varepsilon_G = \frac{V_G}{V_G + V_L + V_S}$$

eq. 5

V_i = Volume of phase i (G = gas, L = liquid, S = solid)

To reduce or even avoid liquid-side mass transfer limitations the gas holdup has to be increased while decreasing the bubble size.

2.2.3 Experimental setup
Heat transfer liquid
The heat transfer liquid should be used directly inside the methanation reactor and therefore needs a high thermal (at least 250 °C) and chemical stability under the given reaction conditions and with the reactants involved. Moreover, low toxicity and viscosity are desirable and a low vapour pressure for avoiding product contamination. From a literature study two groups of liquids - ionic liquids and high performance heat transfer oils - were identified as promising candidates. Ionic liquids are salts with a melting point below 100 °C. Due to their ionic character they have very low vapour pressures. Being most promising 9 ionic liquids and 19 heat transfer oils were tested more in detail (more ionic liquids are currently being investigated). The thermal stability was studied by thermogravimetric analysis with a Netzsch TG 209 F1 Iris thermo-balance.

Flow regime and specific gas liquid contact area
In a non-reactive environment experiments were performed for finding the homogeneous flow regime. In a gassed glass reactor with an inner diameter of 24 mm bubble sizes and gas holdups were measured. The test parameters were varied as given below.

Temperature: 20 – 300 °C
Pressure: 1 – 5 bar
Gas velocity   0 – 5 cm/s
Catalyst particle size: 50 – 400 µm
Particle concentration: 0 – 10 % by mass
Gases: N_2, H_2, CO, CO_2
Liquids: dibenzytoluene, polydimethylsiloxane, [BDiMIM][BTA]

Methanation experiments
Methanation experiments were carried out in a pressure-resistant steel reactor (steel 1.4571) with an inner diameter of 24 mm and a height of 650 mm for the two most promising liquids identified in the preceding experiments. A H_2/CO ratio of 4 was applied to avoid deactivation of the catalyst by coke formation. The following operating parameters were applied:

Temperature: 200 – 300 °C
Pressure: 1 – 20 bar
Gas velocity   0.5 – 1 cm/s
Catalyst: Ni-based
Liquids: dibenzytoluene, polydimethylsiloxane
Particle size: 50 – 100 µm, 100 – 200 µm
Particle concentration: 10 % by mass
Gas composition: 66.6 % H_2, 16.7 % CO, 16.7 % inerts (each by volume)

2.2.4 Results
Heat transfer liquid

In contrary to values for thermal stability given in older literature [e. g. 11, 12] no ionic liquid with a long-term stability at temperatures above 250 °C could be identified. Results for three of the most stable ionic liquids are shown in fig. 8. During TG-analysis at temperatures below 300 °C evaporation dominates the mass loss, but decomposition also occurs. The figure shows the change in colour of the ionic liquids after thermal treatment (all samples were originally colourless). At 400 °C even no liquid phase remained. From the ionic liquids investigated a combination of imidazolium-based cations and [BTA]-based anions leads to relatively stable ionic liquids. Furthermore it was found that the alkylation of the C_1-position of imidazolium-based ionic liquids increases the thermal stability. Therefore, [BDiMIM][BTA] is the most stable of the investigated ionic liquids and its thermal stability is on the limit for what is necessary for an application as heat transfer liquid for methanation.
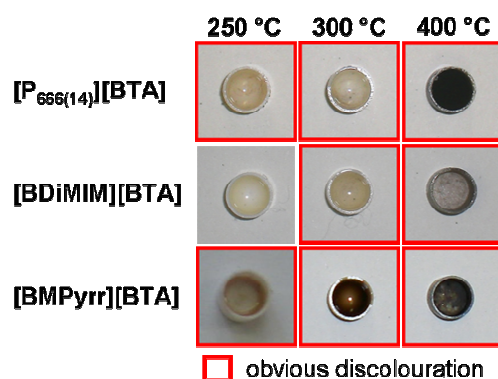

Fig. 8: Ionic liquids after thermal treatment (isothermal conditions for 6 h); $[P_{666(14)}]$ = trihexyl(tetradecyl)phosphonium; [BTA] = bis[(trifluoromethyl)sulfonyl]imide; [BDiMIM] = 1-butyl-2,3-dimethylimidazolium; [BMPyrr] = 1-butyl-1-methylpyrrolidinium

Because of the poor thermal stability of the ionic liquids investigated so far hydrocarbon and silicon based oils were investigated more in detail. In the end two oils were chosen because they combine a low vapour pressure with a high thermal stability. Dibenzytoluene has the best thermal stability of the tested liquids (decomposition starts at about 350 °C) and a moderate vapour pressure. Polydimethylsiloxane has lower thermal stability but has one of the lowest vapour pressures of the 19 tested oils. Tab. 3 compares the chosen oils with a comparatively stable ionic liquid.

Tab. 3: Comparison of suitable heat transfer liquids

property	unit	[BDiMIM][BTA]	polydimethyl-siloxane	dibenzytoluene
molecular formula	-	$C_{11}H_{17}F_6N_3O_4S_2$	$(C_2H_6OSi)_n$	$C_{21}H_{20}$
decomposition temperature	°C	< 250	≈ 300	≈ 350
vapour pressure	mbar (at 250 °C)	0,008	6	37
molar mass	g/mol	433	≈ 100 000 building block: 74	272
density	kg/m³ (at 250 °C)	1 236	775	874
viscosity	mPa·s (at 250 °C)	2.4	2	0.55

Flow regime and specific gas liquid contact area
The homogenous flow regime was found for gas velocities below 2 cm/s as shown in fig. 9. For gas velocities above 4 cm/s the unwanted slug flow could be observed. The experiments also revealed that for temperatures above 200 °C the influence of the physical properties of the applied gases and liquids as well as the pressure is small compared to the influence of the gas velocity.
According to eq. 4 an increase in gas holdup leads to an increase in gas liquid contact area. Fig. 10 shows the gas holdup increasing with gas velocity. Therefore, the reactor should be operated at the

highest gas velocity whilst remaining in the homogeneous flow regime. A high gas velocity will result in higher mass transfer coefficients $\beta_{i,j}$ additionally.

However, fig. 10 reveals that measurements in a gas-liquid system do not represent a 3-phase system sufficiently. A higher catalyst loading leads to a lower gas holdup. Moreover, transition to the slug flow occurs earlier. For a system without catalyst and above 200 °C a homogeneous regime can be observed up to 2 cm/s. If the concentration of catalyst is above 10 % by mass no homogeneous regime can be observed anymore and for gas velocities of 1 cm/s or more slug flow prevails (for dibenzyltoluene).
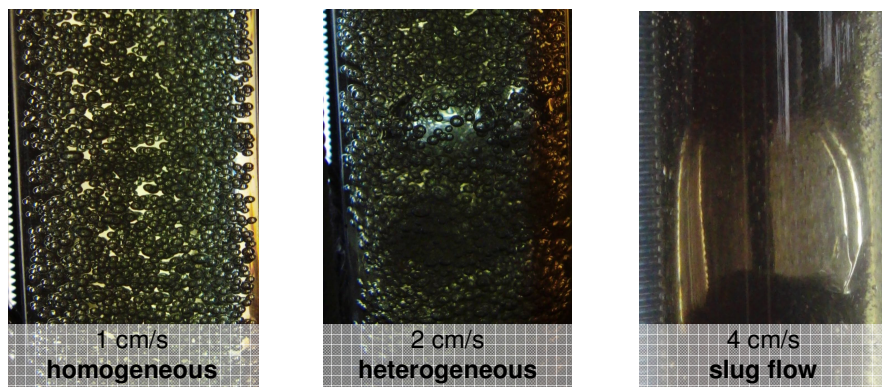

Fig. 9: Flow regimes for N_2 in polydimethylsiloxane at 250 °C and 1 bar

For polydimethylsiloxane the observed negative influence of the catalyst was lower than for dibenzyltoluene. For a catalyst loading of 10 % by mass transition from homogeneous to heterogeneous regime starts at 1 cm/s. Therefore, polydimethylsiloxane was used later on as heat transfer liquid.
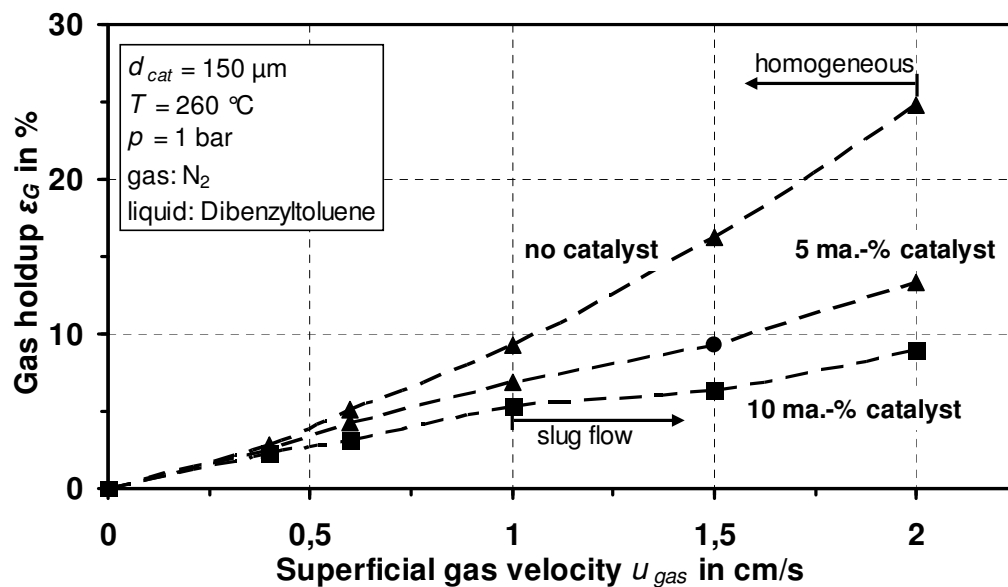

Fig. 10: Influence of catalyst loading on gas holdup (gas: N_2; liquid: dibenzyltoluene)

Methanation experiments

Aiming to operate in the homogeneous flow regime (see fig. 10) the superficial gas velocity was set to 1 cm/s for the methanation reaction. As modified residence time $T_{mod,CO} \approx 100$ kg·s/mol was chosen because this is a typical value for methanation. The modified residence time is defined as the mass of catalyst divided by the mole flow of CO in the feed. With these parameters a CO conversion of 84 % at a reaction temperature of 225 °C and at a pressure of 10 bar was achieved (tab. 4). By increasing the

temperature to 250 °C a conversion of 94 % can be reached. Hence, the 3-phase reactor offers an activity what is in the order of magnitude of a 2-phase system at similar conditions. For future experiments the operating pressure will be 20 bar which should increase the conversion further.

Tab. 4: Excerpt from the results of the methanation experiments

Temperature T	°C	225	250
Pressure p	bar	10	
Liquid	-	polydimethylsiloxane	
$d_{catalyst}$	µm	50 – 100	
$T_{mod,CO}$	kg·s/mol	109	
$^{V}\Phi_{gas}/m_{cat}$	m³/(h·kg)	4.5	
u_{gas}	cm/s	1	
c_{kat}	g/l liquid	95	
Conversion X	%	84	94

2.3   CONCLUSION AND OUTLOOK

Experiments were performed in a glass reactor to investigate the optimal operating parameters for a 3-phase methanation. Thereby one important aim was to increase the specific gas liquid contact area by decreasing the bubble size. By using these optimal operating parameters an activity of the 3-phase reactor in the order of magnitude of a 2-phase system was achieved.

In further experiments the operation at higher pressures will be performed. Moreover a new methanation reactor with a larger inner diameter (≈ 5 cm) is under construction. With the new reactor the influence of reactor diameter on flow regime and bubble size distribution can be investigated.

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