

# Improving the gas cleaning unit of a hydrogen production plant by using a temperature swing adsorption

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**Abstract**— Hydrogen production from dual fluidized bed (DFB) gasification of biomass has the potential to help to fulfill the aims of the UN and EU to reduce fossil fuel demand. To investigate the production of hydrogen from biomass a research plant producing 3 Nm<sup>3</sup> of hydrogen per hour was set up. First results showed the possibility to produce hydrogen from biomass with a purity of more than 99.99 %. However, techno economic analysis showed the need to reduce consumables. Therefore temperature swing adsorption (TSA) was investigated as an alternative to the biodiesel scrubber. First results show the good potential for using a TSA in the process chain. Nevertheless, additional work will have to confirm the long term stability and cost reduction potential for long-running commercial units.

**Key words:** gas cleaning; gasification; hydrogen production; temperature swing adsorption

## I. INTRODUCTION

Hydrogen usage is a key to a renewable future. Chemical industry and refineries have a high demand for hydrogen. The bulk amount of hydrogen in Europe is derived from fossil fuels, especially by natural gas steam reforming. By replacing the fossil hydrogen with renewable H<sub>2</sub> from biomass, a huge step towards a greener energy policy can be achieved. Together with other renewable hydrogen production routes, hydrogen produced from steam gasification of biomass is a promising way to reach these ambitious goals. [1-4]

BIOENERGY2020+ and TU Wien are working in this field since 2009. Since the beginning, experimental research was supported and extended with flow sheet simulations for hydrogen production out of lignocellulosic feedstock based on operating experience and industrially produced synthesis gas

from the 8 MW commercial dual fluidized bed gasifier (DFB) in Güssing.

For the present work, tar rich product gas was extracted from the combined heat and power (CHP) plant, after the dust removal step. A water gas shift stage applying a Fe/Cr based catalyst is used as first gas conditioning stage. After the water gas shift process step, tar and water are removed with a biodiesel scrubber. The scrubbed gas is compressed, and CO<sub>2</sub> is removed. To separate the hydrogen, a pressure swing adsorption (PSA) step is applied. The tail gas of the PSA can be reformed and recycled back into the water gas shift stage, to increase the hydrogen yield. [3-5]

To be competitive with major hydrogen production technologies (e.g.: natural gas steam reforming) further optimizations have to be done. By using biomass with lower quality – which is favourable for the economic viability of the commercial plant - the amount of impurities increases. These impurities can be hydrogenated over the water gas shift stage (WGS) or removed by gas cleaning with scrubbers as already investigated in [6]. Thus, gas cleaning by scrubber is too expensive, a more cost-saving way for gas cleaning must be investigated.

Tar can cause problems in different ways. Colder parts of the plant may be blocked by condensing tar and catalytic reactions may be less efficient because of blocking active catalyst zones. Therefore tar removal is a crucial part to ensure efficient operation for every gasification plant. [7,8]

Tars are formed during the pyrolysis step. The formation is highly dependent on temperature, fuel, residence time and used bed material [9]. Thus characterization of tar has been part of several research issues, a lot of classification systems exist.

Brage et al. define tars by their formation in primary, secondary and tertiary tar. However, this characterization does not include chemical properties or specific compound classes such as organic acids. Furthermore, tar can be defined as organic components with a higher boiling point than benzene. Additionally, classification distinguishing by gravimetric and GC/MS tar exists. [10-14,17]

Another method for tar classification is the characterization by molecular weight. According to ECN, tar can be categorized into five classes.

- Gravimetric tar (Class 1)
- Heterocyclic tar (Class 2)
- Aromatics (Class 3)
- Light poly-aromatic hydrocarbons (PAH) (Class 4)
- Heavy PAH (Class 5)

Class 1 tars are described as gravimetric tars. These tars have a boiling point too high for analyzing them by gas chromatography. Due to their high boiling point even small amounts of these class 1 tars (ECN definition) can cause plugging of pipes. Class 2 tars are heterocyclic tars with a high solubility in water. A typical representative compound for a class 2 tar is thiophene. Class 3 tars are aromatics like toluene. Class 4 tars are light PAH like naphthalene which can cause plugging problems if they are present in high concentrations. Class 5 tars also tend to cause condensation problems if they are present in low concentration. [15-17]

To remove tar a number of physical and chemical methods are used. Wet technologies like scrubbing or dry technologies like adsorption on activated carbon are commonly used in the market. Also, catalytic cracking is used in several plants to remove tar. [13]

Halser et al. describe the possibility of using activated carbon at higher temperatures for partial tar removal. [18]

In this work, the possibility of replacing the biodiesel scrubber with a temperature swing adsorption (TSA), to reduce operation costs, is investigated. Therefore activated carbon is tested with several representative tar components and adsorption, as well as desorption characteristics are examined.

## II. MATERIALS AND METHODS

To investigate the behavior of tar components in the product gas of a commercial gasifier after the dust filter, the product gas was analyzed (Table I) and model components were identified. With these model tars, a lab investigation was done by testing the properties of activated carbon (AC). Extensive chemical analyzes were carried out to measure the tar components.

### A. Dual fluidized bed gasifier

The CHP plant Güssing produces a high quality product gas in a DFB steam gasifier. The major components of this product gas are hydrogen, carbon monoxide, carbon dioxide and methane. The basic principle of DFB steam gasification technology is shown in Fig. 1.

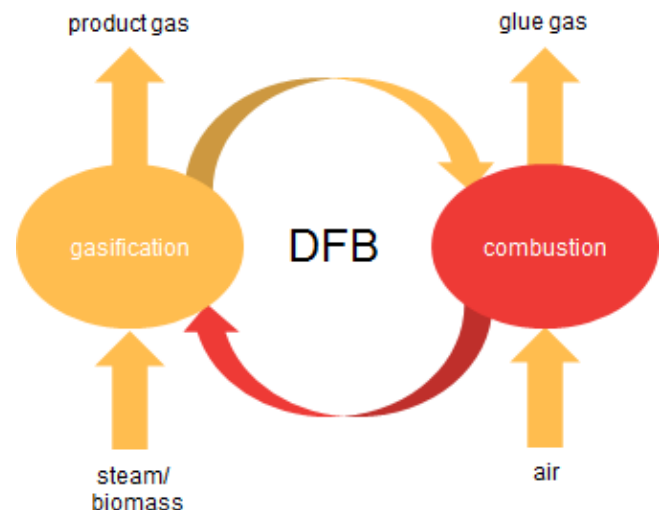


Fig. 1: Basic principle of the DFB process [6,19]

The DFB process consists of two reaction chambers, the gasification zone and the combustion zone. In the gasification zone, steam is used as fluidization and gasification agent. Hot bed material delivers the heat to gasify the lignocellulosic feedstock.

The combustion zone is operated with air in order to combust the non-gasified carbon (char), which is transported from the gasification zone into the combustion zone.

Generated by the two coupled reaction chambers, an almost nitrogen-free product gas is produced. This gas is suitable for synthesis applications or hydrogen production. The dust remaining in the gas is removed by a filter, resulting in a dust-free, hydrogen-rich product gas [see Table I].

TABLE I. IMPURITY CONTENT OF PRODUCT GAS AFTER DUST FILTER [5,6,20]

Impurity	Quantity	Unit
H <sub>2</sub> S	150-200	ppm
COS	5-9	ppm
Thiophene	20-25	ppm
Mercaptane	1-10	ppm
Gravimetric tar	1-2	g/Nm <sup>3</sup>
GC/MS tar	8-12	g/Nm <sup>3</sup>
BTX	15-20	g/Nm <sup>3</sup>

### B. Hydrogen production plant

Fig. 2 shows a simplified flow sheet of the hydrogen production plant.

The plant consists of a DFB biomass steam gasifier to generate a nitrogen free product gas, a product gas cooler, a bag house filter to remove dust particles, a WGS stage to enhance the hydrogen yield and an organic solvent scrubber to remove water and tar from the product gas. Further process steps include compression of the product gas, a CO<sub>2</sub> removal stage to separate sour gas components, a PSA to purify hydrogen and a steam reformer to enhance the efficiency of the overall process. With this plant setup, chemical efficiencies of

over 60% (based on net calorific values) and total efficiencies of up to 80% can be reached by the utilization of process heat [21].

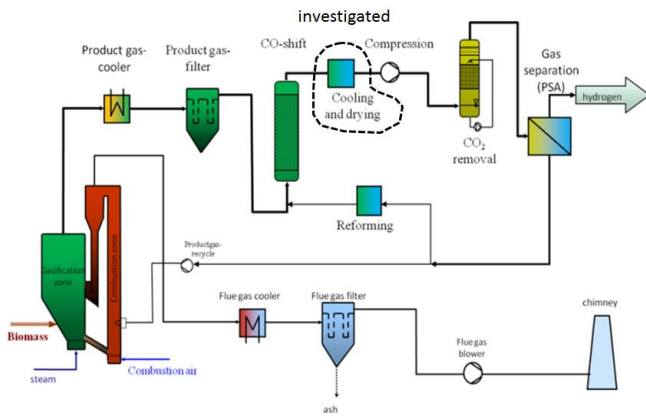


Fig. 2: Flow sheet of the hydrogen production plant [5,6]

To improve the quality of product gas, a WGS stage and a biodiesel scrubber were used. In these units the hydrogen yield can be increased and substances that are detrimental to the downstream processing can be removed. [8,22,23]

In this work, the possibility of using a TSA instead of the organic solvent scrubber is investigated.

### C. Tars and their classification

Several tar analyzes were done to estimate a valid tar concentration, used for the experiments. A total tar load of 25 g/Nm<sup>3</sup> (including BTX) was chosen as mean value for these investigations. A characterization by molecular weight [17] was done, to classify the tars and their condensation behavior.

To summarize the tar present in real product gas, the following model was used to classify tars. Thiophene, toluene and naphthalene were selected as model substances for the experiments. Table II shows the quantities which result in a dew point of 53 °C. Also the gas was saturated to 50 m-% with water to obtain near reality operation conditions.

TABLE II. CHOSEN MODEL TAR MIX FOR THE EXPERIMENTS

Tar	Class	Composition [g/g]
Thiophene	2	0.5 %
Toluene	3	74.6 %
Naphthalene	4	24.9 %

High boiling tars (e.g.: Anthracene, Pyrene) were not considered in this work because former investigations showed that these tars can be hydrogenated in a Fe/Cr based CO shift stage, which is situated upstream of the tar removal. [6,8]

### D. Adsorption test runs

The main aim of this test campaign was to determine an optimal temperature, where on one hand adsorption is still feasible and on the other hand temperature levels can be kept above the dew point of the product gas.

To test the AC a laboratory scale facility (Fig. 3) was used. This facility consists of

- a flow controller to adjust the nitrogen flow,
- an impinger bottle in a boiler to adjust the moisture of the gas
- a pump for dosing the tar mixture
- an evaporator to prevent condensation in the test tube

The designed test gas is transported to an AC test tube which is held at constant temperature. After this, a series of impinger bottles filled with isopropanol prevent venting and allow analysis of tars which were not adsorbed in the AC.

Each experiment started with filling of fresh AC into a test tube. After determining the weight of the AC inside the tube ( $m_{AC,unloaded}$ ), this test tube was put into an oven, to enable a stable temperature, and connected to the test gas line and clean gas line. Depending on the experimental setup the water saturator bottle was heated to a certain temperature or bypassed if no water content was necessary for the experiment. Several adsorption experiments according to Table III were done. In each experiment, a tar concentration of 25 g/Nm<sup>3</sup> ( $\pm 1.5$  g/Nm<sup>3</sup>) was used. Beside the variation of tar composition, also the temperature was varied for each tar composition.

The solution from the impinger bottles was analyzed by gas chromatography, to determine the tar concentration in the off gas of the AC.

TABLE III. EXPERIMENTS AND THEIR SETUP

Experiment	Procedure
1	100 m-% toluene
2	98 m-% toluene, 2 m-% thiophene
3	98 m-% toluene, 2 m-% thiophene, water vapor (50 m-% saturated)
4	74.6 m-% toluene, 24.9 m-% naphthalene, 0.5 m-% thiophene, water vapor (50 m-% saturated)

After each experiment, the mass of the AC inside the test tube ( $m_{AC,loaded}$ ) was determined and the maximum adsorption capacity ( $X_{AC}$ ) was calculated according to equation 1.

$$X_{AC} = \frac{m_{AC,loaded} - m_{AC,unloaded}}{m_{AC,unloaded}} 100 \quad (1)$$

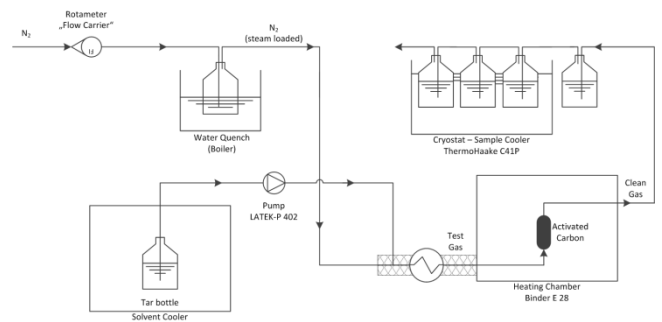


Fig. 3: Flow scheme of activated carbon test facility

The mass of the AC test tube was measured before and after each test run, to calculate adsorbed amount of tar. To identify which tars were adsorbed the solvent in the impinger bottles were analyzed by a GC with FID detector.

As adsorbent, activated carbon Desorex K47 was used. This adsorbent has an inner surface of  $> 900 \text{ m}^2/\text{g}$  [24]. In addition, a screening of other adsorbents will be done in a future separate work.

#### E. Desorption test runs

To investigate the desorption behavior of AC, the same test facility (see Fig. 3) was used. A defined amount of AC was fully saturated with tar (Table II) and stored under air exclusion in sealed vessels. This AC was then used for desorption investigations. Each loaded AC ( $m_{AC,loaded}$ ) was used once for desorption experiments. After each test run, the mass difference was measured ( $\Delta m_{AC}$ ).

Several test runs investigating desorption temperature and superficial velocity were executed. Desorption efficiency was calculated according to equation 2.

$$X_{DE} = \frac{\Delta m_{AC}}{m_{AC,unloaded} (X_{AC} - 1)} \quad (2)$$

#### F. Analysis of the tar

To analyze the tars, which were not captured in the AC, impinger bottles filled with isopropanol were used. The first impinger bottle was operated at ambient temperature ( $25 \text{ }^\circ\text{C}$ ). Bottle 2-4 were operated at temperatures of  $-25 \text{ }^\circ\text{C}$ . Each of these four impinger bottles was filled with 50 ml isopropanol. An additional empty impinger bottle was used as condensation trap.

The collected solvent was analyzed by gas chromatography, using a Clarus500 GC from Perkin Elmer. A liquid injection with a  $1 \mu\text{l}$  autosampler was done. As column a Restek MXT-1 (dimethyl polysiloxane) with a length of 60 m and an inner diameter of 0.53 mm and  $0.25 \mu\text{m}$  df was used. Helium 5.0 with a flow of 6 ml per minute was used as carrier gas. A split of 49:1 was adjusted. As detector, a FID with an operation temperature of  $420 \text{ }^\circ\text{C}$  was used.

#### G. Economics

A first economic analyze was done to compare the state of the art process, biodiesel scrubbing with Rapeseed methyl ester (RME), with a TSA operation. For first estimations, several simplifications were used. The electric energy was considered as lump sum. Cooling energy was set to be the same because both processes need to cool gas and remove water, which is the most energy consuming step. As RME, heating energy, electric energy activated carbon and strip gas consumption are the main consumables, these were considered for the economic evaluation.

Process heat was assumed to be used for district heat ( $57 \text{ €/MWh}$ ) when not used for biodiesel regeneration and as regeneration energy for the TSA operation.

The price for electric energy was assumed to be  $42 \text{ €/MWh}$ . A cooling efficiency of 4 was assumed.

The economics were calculated on basis of a DFB plant with a fuel feed power of 10 MW. As previous investigations showed a total of 25 l/h of biodiesel is needed for removal of heavier tars (class 4). Another  $1 \text{ l}_{RME}/\text{Nm}^3_{PG}$  is needed for the removal of BTX.

For the economic evaluation no CAPEX were considered. The estimation is based on the operational costs.

Economics were calculated for several operation scenarios:

Case I: Operation with a two staged RME scrubber, were the first stage is responsible for a coarse cleaning of the gas at  $40 \text{ }^\circ\text{C}$  and the second stage is used for fine cleaning of the gas at  $0 \text{ }^\circ\text{C}$ .

Case II: Case I with regeneration of the solvent used in the second stage

Case III: TSA operation with water removal by cooling

Case IV: Operation of a one staged RME scrubber  $40 \text{ }^\circ\text{C}$  and a TSA for fine cleaning. Water removal is done by cooling

### III. RESULTS AND DISCUSSION

Experiments regarding the adsorption and desorption behavior of AC were executed. Desorex K47 was used as adsorbent. Research focused on the adsorption behavior to gain a better understanding of the adsorption process at different temperatures.

#### A. Adsorption behavior

First experiments were executed with toluene as standard tar component. In order to simulate real product gas, thiophene, naphthalene and water were added to the mixture. All these mixtures were tested using different temperatures. Table IV shows the adsorption capacity of Desorex K47 at different temperatures. The measured adsorption capacities of Experiment 1 to Experiment 4 are summarized in this table. These results seem to be valid, as they match with the adsorption isotherm from the AC manufacture ( $20 \text{ }^\circ\text{C}$ ).

TABLE IV. ADSORPTION CAPACITY OF DESOREX K47 AT DIFFERENT TEMPERATURES AND TAR MIXTURES

Exp.	Temp. [ $^\circ\text{C}$ ]	Ads. capacity [kg/kg]	Std. deviation [kg/kg]
1	25.0	24 %	0.05 %
1	43.5	20 %	*
1	63.5	20 %	0.02 %
1	81.0	16 %	1.32 %
2	25.5	24 %	1.59 %
2	41.0	24 %	0.87 %
2	65.0	21 %	3.68 %
2	84.5	16 %	*
3	25.5	27 %	*
3	41.0	26 %	1.91 %
3	62.0	22 %	*
3	83.0	19 %	2.25 %
4	67.0	18 %	0.89 %
4	88.5	19 %	1.86 %

\* ... single sample, no standard deviation can be given

A clear decrease of the adsorption capacity was seen with increasing temperature. This trend was also observed when different tar mixtures were used. Fig. 4 shows different

adsorption capacities, which are achievable under different temperatures and tar qualities. It can be seen that the possible adsorption time reduces with rising temperature.

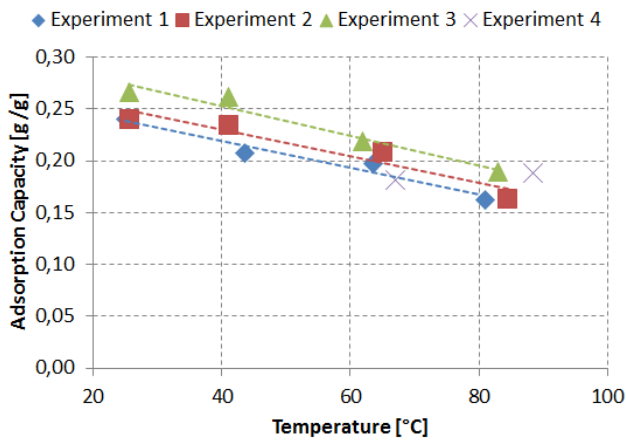


Fig. 4: Adsorption capacity in dependency to temperature and tar quality

Using the tar mixture of experiment 4 (see Table III) an adsorption capacity of 18 % was measured at the desired operation temperature.

As depicted in Fig. 4 the adsorption capacity increases slightly when water vapor (compare experiment 2 and 3) is added to the mixture. This effect is already reported for sulphur adsorption in several studies [25-27].

### B. Desorption behavior

To evaluate the best desorption behavior with respect to an economic utilization of process heat, a parameter screening was done. A fixed volume of gas used for regeneration was set to keep the results comparable. Temperatures between 110 °C and 190 °C were used for desorption. As second parameter the superficial gas velocity, respectively desorption time was varied. Therefore gas flow was adjusted to obtain superficial velocities between 0.87 and 0.01 m/s. Different AC bed heights were tested, which resulted in similar desorption efficiencies. Changing the amount of AC did not affect the desorption efficiency significantly. These results show that the amount of AC, is not affecting the desorption efficiency, as long as the temperature in the AC bed is constant. Each AC used for desorption experiments was fully saturated with tars, before the desorption investigations.

Fig. 5 shows the desorption efficiency in dependency to the desorption temperature and superficial velocity. Test runs were performed by using the same amount of desorption gas each time. A higher superficial gas velocity resulted in shorter desorption experiments.

Results show that an increase of temperature increases the desorption efficiency linearly. Also, an increase of the desorption efficiency can be seen by decreasing the superficial gas velocity. At 0.03 m/s an optimum can be seen, with the desorption efficiency at its maximum. Further decrease of the superficial velocity leads to a decrease of the desorption efficiency, due to an increase of the partial pressure. Based on

these results a parameter study regarding the desorption duration was done.

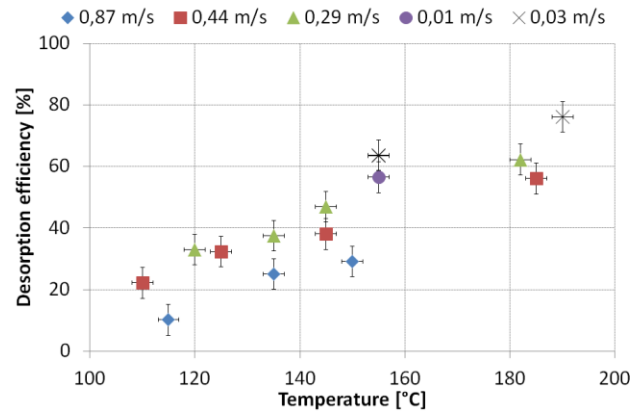


Fig. 5: Desorption efficiency in dependency on the temperature and superficial velocity

As desorption temperature, 155 °C was chosen. It can be seen (Fig. 6), that with increasing desorption time, desorption efficiency is increasing. After approximately 1 day of operation an adsorption efficiency of 70 % was observed, when a superficial velocity of 0.03 m/s is established. Test runs with a superficial velocity of 0.01 m/s proof the assumption that 0.03 m/s yield optimum results.

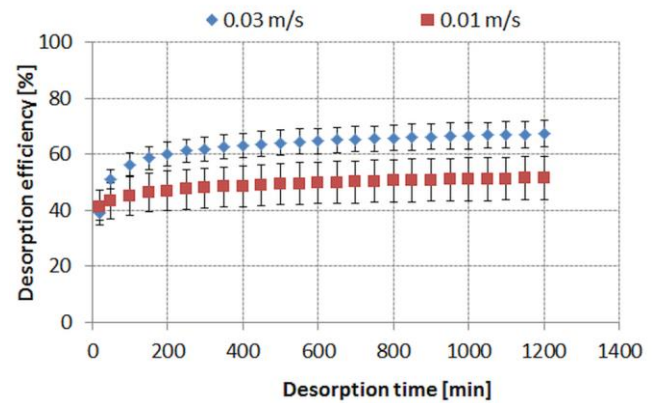


Fig. 6: Desorption efficiency in dependency on the desorption time at 155 °C

Nevertheless, a desorption efficiency of only 70 % would lead to an inefficient TSA. Therefore, higher desorption temperatures were investigated.

Fig. 7 shows the study on desorption time for temperatures of 190 °C. As superficial velocity 0.03 m/s was chosen. It can be seen that the desorption efficiency increases significantly with temperature.

As reported by Itaya et al. a temperature gap between adsorption and desorption of 150 °C is sufficient to remove sulphur components efficient. As experiments show an even lower temperature gap can be sufficient for tar removal.

After 20 h of operation, 90 % of the fully loaded AC was regenerated. However, these experiments were done in batch operation, which means it is still necessary to run long term tests, to get detailed information about the behavior of activated carbon during the TSA operation.

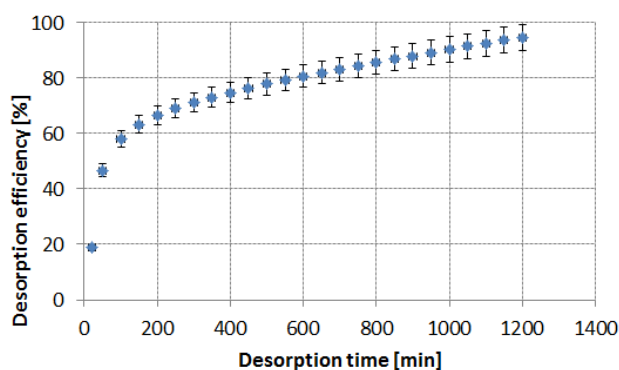


Fig. 7: Desorption efficiency in dependency on the desorption time at 190 °C

### C. Economic potential

OPEX were estimated using data gained from the test runs (AC) and using literature [29] for the RME scrubber. The process heat used in these cases is assumed to be used for district heat, if not needed in the regeneration process. For all four cases an electric energy consumption for cooling energy was estimated to 105 k€/a for a 10 MW<sub>Fuel Power</sub> hydrogen production plant.

#### 1) Case I

Through the high absorption capacity of the solvent at higher temperatures RME scrubbing used at 40 °C can remove the bulk amount of tars (especially higher tars) with relatively low operation costs. Yearly costs of 20 k€/MW<sub>Fuel Power</sub> are necessary to run the first scrubber stage. When a fine cleaning is needed a second scrubber stage operated at 0 °C has to be used. Through the low absorption capacity of RME at these temperatures a high amount of biodiesel is necessary to obtain a BTXN free gas. Yearly 1.6 M€/MW<sub>Fuel Power</sub> are needed to operate the second scrubber stage. This results in 16.3 M€ OPEX for a 10 MW DFB gasification plant.

#### 2) Case II

By adding a regenerator RME consumption can be reduced by up to 90 %. Nevertheless high amounts of process heat are needed to run the regeneration process. RME costs can be reduced to 180 k€/MW<sub>Fuel Power</sub> per year. Additional heat needed for the process is estimated to 100 k€/MW<sub>Fuel Power</sub>. This results in total yearly costs of 2.9 M€ for a 10 MW plant.

#### 3) Case III

Using only a TSA for the removal of BTXN and tars in one stage yearly operational costs of 104 k€/MW<sub>Fuel power</sub> could be calculated. The bulk amount is the strip gas, which was nitrogen for the lab test runs. A replacement of nitrogen by CO<sub>2</sub> or tail gas can reduce OPEX drastically. Total yearly operation costs for a 10 MW plant would be 1.1 M€.

#### 4) Case IV

Using a RME scrubber as first gas cleaning stage, followed by a TSA seems to be a feasible option. OPEX of 20 k€/MW<sub>Fuel Power</sub> for the scrubber and 56 k€/MW<sub>Fuel Power</sub> for the TSA could be calculated. The bulk amount of TSA costs is the usage of nitrogen as strip gas (35 k€/MW<sub>Fuel Power</sub>). If tail gas or CO<sub>2</sub> is used an even higher cost reduction is possible. Total yearly operational costs of 0.87 M€ seem to be feasible.

## IV. CONCLUSION

Comparing these results with a biodiesel scrubber, it seems feasible to replace a biodiesel scrubber with an activated carbon guard for the removal of volatile tar components up to naphthalene. Biodiesel scrubbers in this setup are able to remove about 70 % of tar [6]. An AC guard is able to remove up to 99.9 %. If this carbon guard is regenerated continuously (TSA), by utilization of process heat, an economic advantage could be realized. This gas cleaning setup seems also to be a promising plant configuration for the EC project ROMEO.

Based on the gained data, a setup using a warm (40 °C) RME scrubber for removal of tar components and a TSA for the fine gas cleaning seem to be technical and economical most feasible.

Future challenges will be, to bring this TSA process to the technical readiness level 5 (TRL 5) using real synthesis gas, including tar, in a small test facility, as well as long term tests which should be executed in a 5 Nm<sup>3</sup>/h scale. The behavior of sulfur over the TSA must be investigated in detail as well.

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