
Developing an adsorption-based gas cleaning system for a dual fluidized bed gasification process

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Abstract

Biomass can make a major contribution to a renewable future economy. If biomass is gasified a wide variety of products (e.g.: bulk chemicals, hydrogen, methane, alcohols, diesel) can be produced. In each of these processes gas cleaning is a crucial factor. Impurities in the gas can cause catalyst poisoning, pipe plugging, unstable or poisoned end products or harm the environment. Especially aromatic compounds (e.g.: benzene, naphthalene, pyrene) have a huge impact regarding a stable operation of a syngas process. A removal of these compounds can be accomplished either with wet, dry or hot gas cleaning methods. Wet gas cleaning methods tend to produce huge amounts of waste water which needs to be treated separately. Hot gas cleaning methods provide a clean gas, but are often cost intensive due to their high operating temperatures and catalysts used in the system. Another approach is dry or semi dry gas cleaning methods including absorption and adsorption on solid matter. In this work special focus will be laid on adsorption based gas cleaning for syngas applications. Adsorption and desorption test runs were carried out under laboratory conditions using a model gas with aromatic impurities. Adsorption isotherms as well as dynamics were measured with a multi compound model gas. Based on these results a temperature swing adsorption process was designed and tested under laboratory conditions, showing the possibility of replacing the conventional wet gas cleaning by a semi-dry gas cleaning approach.

1. Introduction:

The usage of biomass in sustainable technologies is a key to a renewable future. As it is the only renewable carbon source, a strong focus should be set in the development of renewable processes for the production of carbon based chemicals.

Dual fluidized bed gasification (DFB) is used to produce a high caloric, nitrogen-lean product gas. With this product gas several syntheses like Fischer-Tropsch,

mixed alcohols, methanation and hydrogen production can be realized. Nevertheless, an efficient gas cleaning system is crucial for these downstream processes. Nitrogen based (e.g.: ammonia, hydrocyanic acid), halogen based (e.g.: hydrochloric acid), sulfur based (e.g.: H₂S, COS, mercaptane) impurities as well as higher hydrocarbons (tar) can cause problems in catalysis reaction. Therefore, they have to be removed from the gas before it is applicable in synthesis gas

(syngas) processes. To lower the total amount of tar, DFB gasifiers are operated at higher temperatures, leading to the reduction of the total tar amount, but enhancing the formation of higher aromatics and poly-aromatic hydrocarbons (PAH) due to reduction of oxygen in the structures [1-7]. Hydrogen is well known for tar reduction and reduces the formation of heavier tars. With increasing temperature higher molecular tars are formed. As tar formation is not topic of this work it shall be referred to other authors. [8-12]

To remove these problematic compounds several approaches exist. They can be divided into primary and secondary methods. As primary methods are not scope of this work it shall be referred to the authors cited above. Secondary methods can be divided into wet scrubbing (e.g.: baffle separators, cooling towers or venturi scrubbers), dry or semi dry methods (e.g.: Adsorption on solids, particle cyclones, cold filters) and hot gas conditioning methods (e.g.: hot filters, thermal and catalytic crackers, steam reformers). [12]

As wet tar removal methods tend to produce high amounts of waste water and hot gas methods are cost intensive this work focuses on dry methods, especially adsorption.

Thiophene removal from an oily phase as well as tar separation during the thiophene syntheses is well studied. Jeevanandam et al. studied the thiophene removal from hydrocarbons by using metal impregnated adsorbents. Yu et al. worked on the desulfurization (especially thiophene and dibenzothiophene) of oil using activated carbon. [13,14]

Edinger et al. investigated the thiophene removal from the gaseous phase using activated carbon as low temperature alternative to the hydro-desulfurization (HDS) at temperatures between 100-200°C. [15]

Several studies concerning the removal of tar from biomass gasifier gas have been done, focusing on the a gas cleaning for heat and electric power production. [16-18]

A comparable gas cleaning technology is employed in the Gothenburg Biogas plant (GoBiGas) were a four-adsorber system is used, to remove heavy tars, BTX and sulphur components to a achieve a syngas quality suitable for methanation after a biodiesel scrubber. [19,20]

This paper deals with the development of an adsorption based gas cleaning for the upgrade of a gasifier gas to syngas quality using adsorption technology.

2. Concept and methodology:

In laboratory scale a gas cleaning unit was investigated which focuses on reduction of operating costs by replacing the costly gas scrubbing using biodiesel (RME) by adsorption on activated carbon.

Therefore tar analysis, obtained from industrial DFB gasifiers were used to select model tar components. A classification of tars was introduced to represent each class in the mixture.

With this model tar, experimental research in terms of adsorption and desorption behavior was carried out.

Due to economic considerations, this paper focuses on the fine gas cleaning (substitution of the second biodiesel scrubber, see Fig. 1), as it has a 10 times higher biodiesel consumption as the first biodiesel scrubbing stage [21].

DFB steam gasification

DFB steam gasification is an allothermal gasification technology, using steam as gasification agent. Two reaction zones coupled by a slide and a cyclone are applied for this process. A steam fluidized reactor is used to gasify biomass in a bubbling bed. The fuel reacts with the steam in presence of catalytic active bed material under consumption of heat to the

main gas components hydrogen, carbon monoxide, carbon dioxide and methane [22].

The non-gasified biomass and the bed material are transported over a slide to the combustion zone. There a fast fluidized bed is applied by fluidization with air. The non-gasified biomass is burned there and the bed material is heated up again. Through a cyclone bed material is separated from the flue gas stream and transported back into the gasification reactor. The resulting gasification product is an almost nitrogen-free gas that is well suitable for syngas applications, like hydrogen production, Fischer-Tropsch synthesis or methanation.

For the use in syngas applications impurities have to be removed.

Impurities can be hydrogenated over the water gas shift stage (WGS) or removed by a two staged biodiesel scrubbing unit [23,24], which is the state-of-the-art in biomass to chemical processes.

Figure 1 shows the gas cleaning setup of a Fischer-Tropsch plant based on DFB gasification of wood. The two staged scrubber (biodiesel scrubber “warm” and biodiesel scrubber “cold”) allows an almost complete removal of tar components. The temperature swing adsorption is used to remove high volatile hydrocarbons as well as sulfur components.

Thus, gas cleaning by scrubbing is expensive; a more cost-saving way has yet to be developed.

Table 1 shows the impurity amount in the syngas before and after the first biodiesel scrubber stage (biodiesel scrubber “warm”, Fig. 1). A high removal efficiency of gravimetric tars and a poor removal efficiency of BTX components can be observed. Applying a two staged biodiesel scrubber would lead to high biodiesel consumption, even if the solvent is regenerated [21].

Therefore an adsorption based gas cleaning unit will be developed. First the biodiesel scrubber “cold” and the temperature swing adsorption will be replaced to remove tar and sulfur components more efficient.

To design a temperature swing adsorption (TSA) a detailed investigation regarding the behavior of tars during adsorption and desorption process was carried out.

	Inlet scrubber	Outlet scrubber
H ₂ O [vol-%]	35-45	8-12
Gravimetric tar [g/m ³ _{STP}] (db)	2-5	0-0.2
BTX [g/m ³ _{STP}] (db)	15-20	5-15
Sulfur components [ppm]	175-250	150-250
NH ₃ [ppm]	200-1500	100-400

Tab.1: Impurities before and after the first biodiesel scrubber stage [25-27]

STP... Standard temperature and pressure (273.15 K, 10⁵ Pa)

Classification of tars

Thus characterization of tar has been part of several research projects, a lot of classification systems exist. Baker et al. [28] 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 boiling point higher

than benzene. Additionally, classification distinguishing by gravimetric and GC/MS tar exists. [12,29-32]

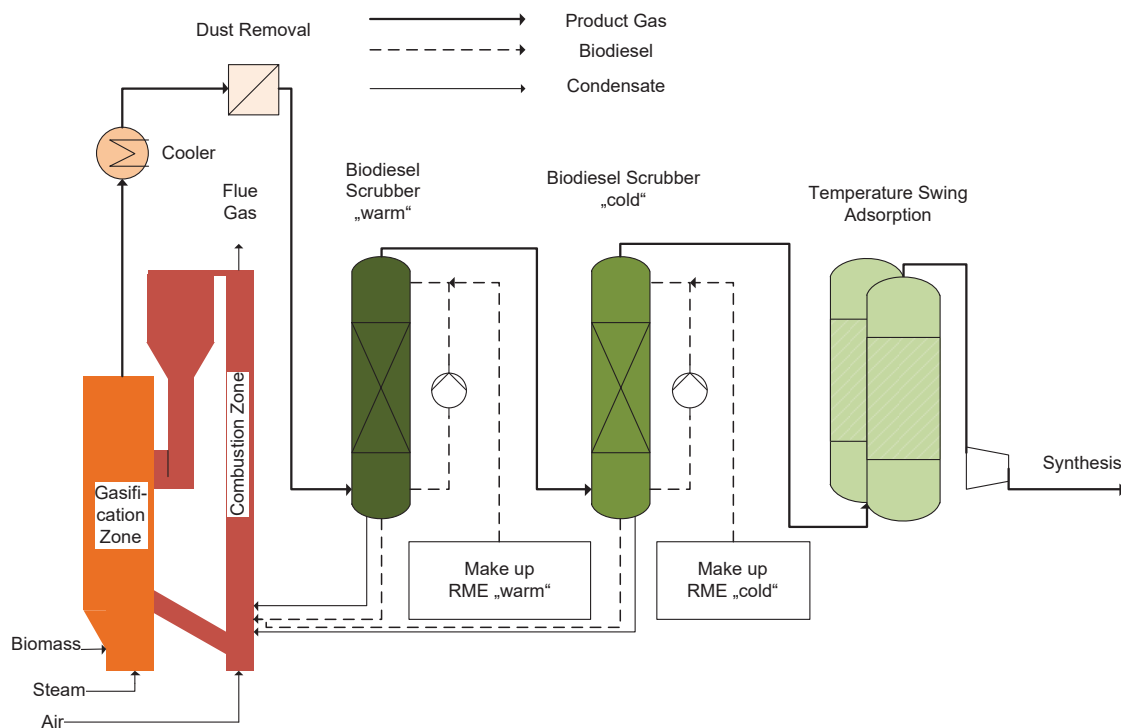


Fig.1: Flow scheme of the gas cleaning of a DFB based syngas plant

Another method for tar classification is the characterization by molecular weight and physical properties, defining five classes. Class 1 tars are described as gravimetric tars. Class 2 tars are heterocyclic tars with a high solubility in water. A typical representative compound for a class 2 tar is phenol. Class 3 tars are light aromatics like toluene. They are described as single ring components. Class 4 tars are light PAH (2-3 ring structures), like naphthalene. Class 5 tars have a poly aromatic structure of 4-7 rings. (e.g.: pyrene). [32]

One of the definitions states that long-chain hydrocarbon compounds having a larger molar mass than benzene (78.1 g / mol) can be referred to as tar. [33]

The tar protocol, defines tar as the total of all organic compounds in the synthesis gas with the exception of permanent gaseous hydrocarbons and benzene. [32-33]

Another definition describes tar as the condensable fraction of organic gasification products and as aromatic hydrocarbons including benzene. [34]

As the aim of this work is to clean up a gas to syngas quality, a removal of benzene due to its condensability during compression is highly recommended. Therefore the classification system using five tar classes [32] including benzene as class 3 tar, will be applied.

Based on the classification approach with five tar classes and the real gas composition after the biodiesel scrubber, a model gas composition was selected.

The model tar after the biodiesel scrubber consists of thiophene, representing aromatics with a hetero atom, toluene representing the BTX fraction (benzene, toluene, xylene), styrene representing aromatics and naphthalene, representing light polycyclic aromatics. Class 5 tars were not considered, as they

are removed in the first biodiesel scrubber stage. Table 2 depicts the model tar composition.

Selection of model tars composition

	Tar composition [%]
Thiophene	0.3
Toluene	85.4
Styrene	7.1
Naphthalene	7.2

Tab.2: Model tar composition after the first biodiesel scrubber stage

This mixture has a tar dew point of 38.9°C, at concentrations of 25.91 g/m³_{STP} which represents the tar dew point of DFB syngas after a one staged biodiesel scrubber. [21]

Activated Carbon

A commercial available activated charcoal was used for the experiments. This coal has a bulk density of 500 kg/m³ and a pellet diameter between 0.5 – 1.6 mm. As measured by BET surface method, an inner surface between 890 - 940 m²/g could be determined for the examined activated carbon. The BJH method gives a pore size between 3.1 to 3.5 nm (adsorption/desorption) for the examined activated carbon. An average pore volume of 0.13 to 0.19 cm³/g (desorption/adsorption) could be determined by BJH the method. The measurements of pore size, pore volume and BET surface were done with a TriStar II 3020 analyzer.

Adsorption setup and design of experiments

To develop an efficient gas cleaning for syngas applications several adsorption

experiments were executed. Figure 2 shows the flow scheme of the adsorption test rig used for the experiments. A mass flow controller (MFC) was applied to adjust a proper carrier gas flow. A syringe pump was used to adjust the amount of water and tars fed into the system. These two streams were mixed in an evaporation column situated in an oven. The mixture was sent to an adsorber, also situated in the oven to guarantee a stable temperature. After the gas left the adsorber several measurements were carried out. Tar components with a higher boiling point than naphthalene were measured by a liquid tar sampling method (according to tar protocol). Tar components with a boiling point of naphthalene and below were measured directly with gas chromatography (FID detector). Sulfur components (thiophene) were also measured by gas chromatography (SCD detector). Adsorption behavior in terms of adsorption isotherms and isobars was determined experimentally. As adsorption reactor a stainless steel cylinder with an inner diameter of 9 mm and a height of 50 mm was used. A superficial velocity of 0.1 m/s was adjusted. Each measuring point was measured multiple times. Adsorption isotherms were established at a standard temperature of 40°C by varying the inlet tar concentration between 1.5 and 50 g/m³_{STP}. Adsorption isobars were measured by adjusting a constant tar concentration of 25 g/m³_{STP} and varying the temperature between 40-220°C.

For both, adsorption isotherms and isobars, the adsorption capacity was calculated after their full loading by equation 1.

$$X_{Ads,Max} = \frac{m_{AC,out} - m_{AC,in}}{m_{AC,in}} \quad (1)$$

To get a more accurate result for the adsorption (respectively desorption) enthalpy the Clausius-Clapeyron equation

(2) was applied. By using the adsorption isothermes the adsorption enthalpy could be calculated. [36]

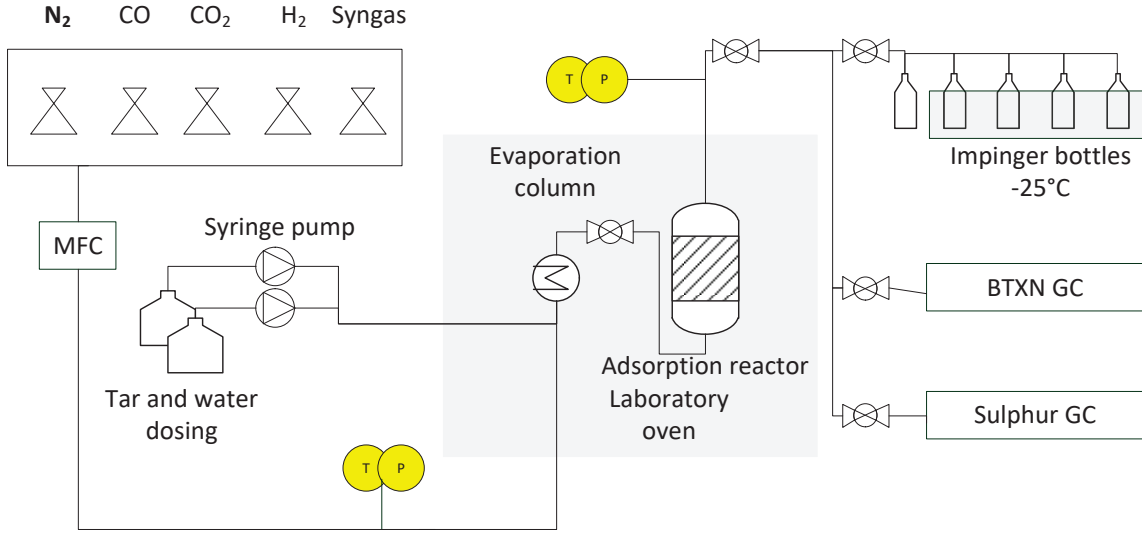


Fig.2: Flow scheme of the lab test setup

$$\ln \frac{p_2}{p_1} = \frac{\Delta H_{Ads}}{R} \left(\frac{1}{T_1} - \frac{1}{T_2} \right) \quad (2)$$

Adsorption dynamics were measured at tar concentrations of $25 \text{ g/m}^3_{\text{STP}}$ by monitoring the total tar amount fed into the system and monitoring the total tar amount leaving the system. The difference between inlet and outlet tar amount was defined as adsorbed tar amount (equation 3).

$$Y(t)_{ads} = Y(t)_{in} - Y(t)_{out} \quad (3)$$

Also the thiophene adsorption dynamics were measured by analyzing the outlet gas in a SCD gas chromatograph.

Based on the adsorption dynamics the specific tar adsorption amount (X_{BT}) was calculated by using the breakthrough time (t_{BT}), the mass of adsorbent (m_{AC}) and the tar inlet concentration (Y_{in}) (equation 4).

$$X_{BT} = \frac{Y_{in} t_{BT}}{m_{AC}} \quad (4)$$

Based on these data, the adsorption part of a temperature swing adsorption was designed.

Desorption setup and design of experiments

To investigate the desorption behavior loaded activated carbon (AC) was used and investigated in a thermo gravimetric analyzer (TGA). In the TGA a temperature ramp was adjusted to measure the mass reduction of the AC over time, respectively the desorption of tars. The temperature, where the highest mass loss occurred was defined as characteristic temperature.

3. Results and discussion

Adsorption

Adsorption isotherms were measured to describe the adsorption behavior of AC used to remove tar components from syngas.

Figure 3 shows the adsorption isotherm measured at 40°C . This isotherm correlates with the pure toluene adsorption at similar temperatures.

A modified Langmuir model was applied to describe the adsorption isotherm. Adsorption isobars were measured at 40°C, 65°C, 85°C and 180°C. Based on these measurements and the fitting Langmuir model, adsorption isotherms for 65°C, 85°C and 180°C were calculated and are shown in Figure 3. These isotherms were validated at other temperatures and show a good correlation at higher tar concentrations (20-30 g/m³_{STP}), and higher deviation at low tar concentrations. A maximum error of 20% could be obtained at low tar concentrations. Based on the adsorption isotherms, the Clausius-Clapeyron equation was used to calculate the adsorption enthalpy. An adsorption enthalpy of 93 kJ/mol could be calculated for the desired process parameters. This value is highly dependent on the process temperature and inlet tar concentration. The measured value correlates with literature data. [35-37]

The adsorption dynamics were measured by a gravimetric approach. The amount (mass) of tar injected into the system was adjusted with a syringe pump. The mass increase of the adsorbent was measured. Figure 4 shows the breakthrough curve measured gravimetrically. It can be seen, that the theoretical mass increase (inlet) and the mass increase of the adsorbent are similar, until a maximum loading of the adsorbent is reached. A maximum adsorption capacity of 0.34 g_{tar}/g_{AC} respectively 34% could be measured. Based on these data a specific tar adsorption amount of 0.30 - 0.33 g_{tar}/g_{AC} could be calculated running several experiments. Nevertheless, this approach gives no information about the outlet concentration of the components itself. So thiophene content was analyzed.

The gravimetric breakthrough curve and the thiophene breakthrough curve

show deviations in terms of breakthrough time. As thiophene is a minor component of the gas, it does not contribute to the gravimetric breakthrough curve in a high amount. A concentration of 25 ppm thiophene was adjusted at the inlet of the system. A complete breakthrough of thiophene could be detected after 50 min, which is similar to the complete saturation time measured by the gravimetric breakthrough approach. However, a further increase beyond the feed concentration could be measured, which indicated displacement effects.

If the thiophene breakthrough curve is compared with the gravimetric breakthrough curve (Figure 4 and Figure 5) it can be seen that thiophene fully breaks through when the activated carbon reaches the maximum loading. Further loading of the AC leads to displacement effects and thiophene is desorbed and replaced by hydrocarbons with a higher evaporation temperature.

Desorption

Desorption behavior was analyzed by TGA experiments. Several temperature ramps, from 20°C to 700°C were measured. Test runs with a heating ramp of 5 K/min, 10 K/min and 20 K/min were carried out. A maximum mass decrease could be observed at a temperature of 152°C. Heating ramps of 10 and 20 K/min showed other results, due to the fast temperature increase. Figure 6 shows the TGA curve including the variation in a temperature range of 30 to 300°C with a heating ramp of 5 K/min.

Based on the characterizing temperature and the adsorption properties a temperature swing adsorption was designed and operated.

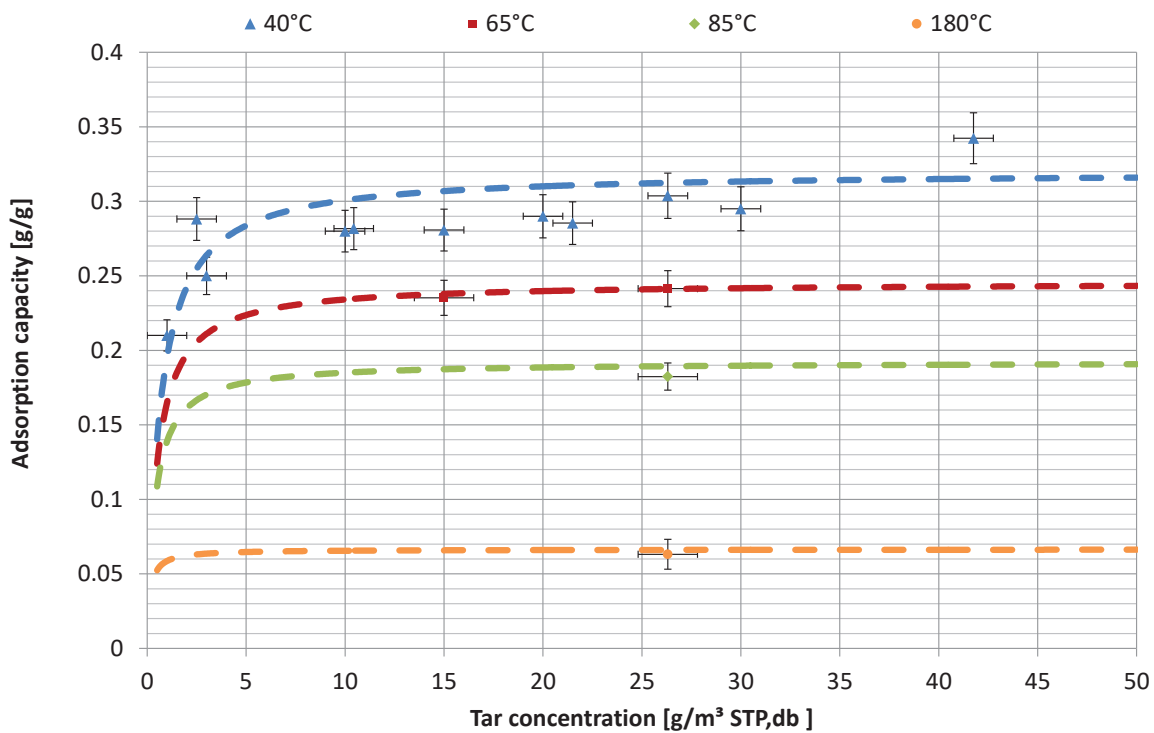


Fig.3: Adsorption isotherms at different temperatures

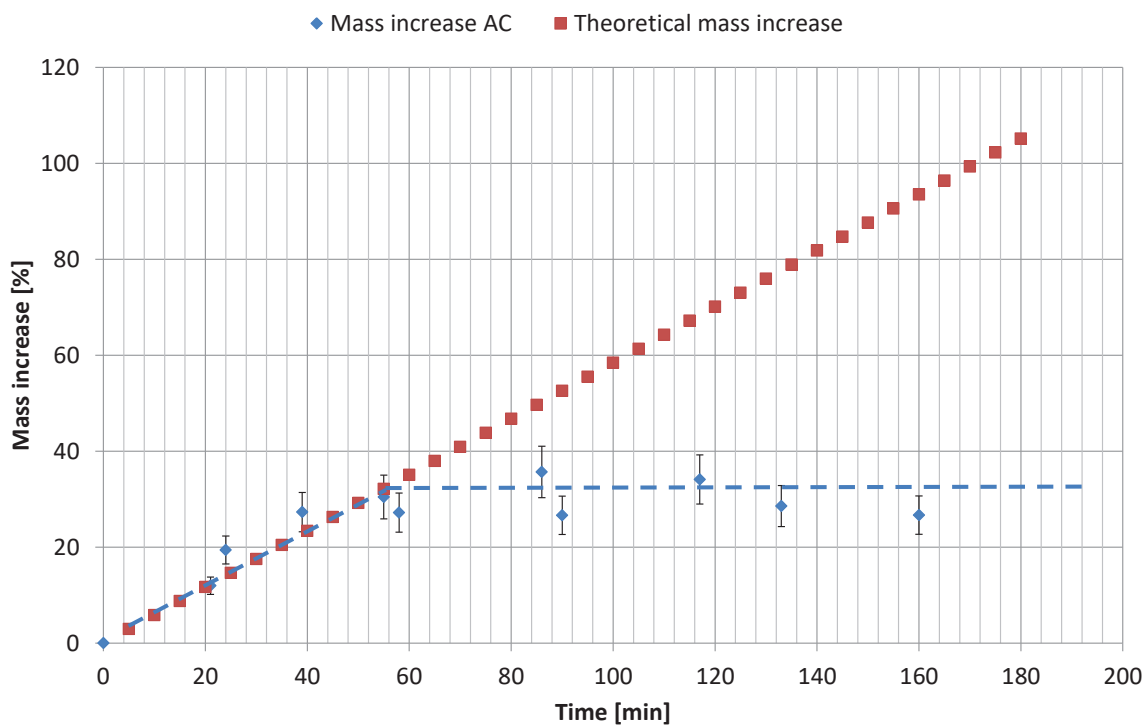


Fig.4: Gravimetric breakthrough curve

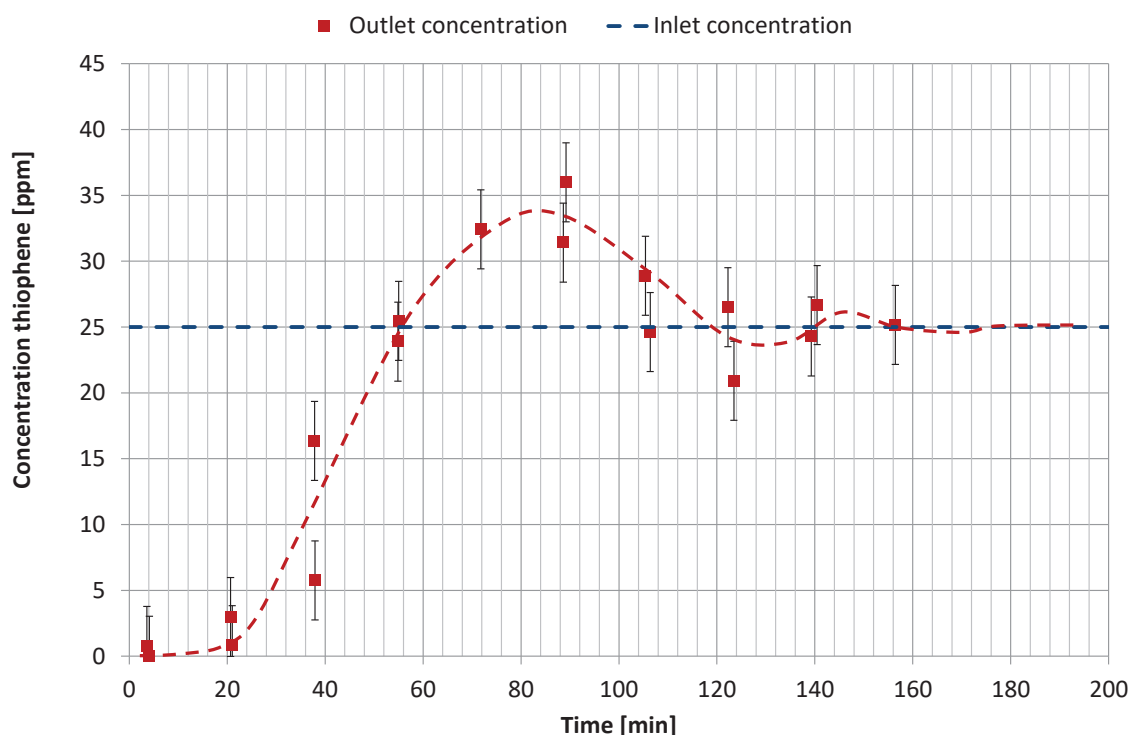


Fig.5: Thiophene breakthrough curve

Temperature swing adsorption

The designed TSA was operated at adsorption temperatures of 40°C and a tar concentration of 25 g/m³_{STP}. Desorption temperatures of 180°C were adjusted for the first test runs, to be comparable to the achieved adsorption isotherms which were also measured at 180°C.

The TSA was operated with the same setup described for the adsorption experiments. The calculated adsorption capacity of 0.3-0.33 g adsorb-able tar per 1 g activated carbon, could be proven throughout several experiments. Figure 7 depicts the tar outlet concentration over 24 hours of operation. It can be clearly seen, that during the desorption phase, operated at 180°C tar components are desorbed and the adsorbent is regenerated. During the adsorption phase (40°C) tar is adsorbed with an efficiency of over 99%. A total of 1.4 m³ of gas per 1 m³ of flush gas (N₂, off-gas) could be cleaned with this setup. However, aging effects of the adsorbent can be observed. Over 200 hours of

continuous TSA operation the adsorption efficiency of the TSA was reduced to 95%. This means further optimizations have to be done.

Displacement effects for the TSA were not considered in this work, as only the sum of tar could be detected.

4. Conclusion and outlook

An adsorption based gas cleaning for the syngas applications has been developed. Due to economic considerations the gas cleaning setup described in Figure 1 was redesigned to replace the “cold” biodiesel scrubber. Adsorption isotherms and isobars were measured to obtain the optimal operation conditions for the expected tar loading in the gas. Desorption experiments were carried out in a TGA to gain the optimal desorption temperature. However this temperature was set higher (from 152 to 180°C) for the first TSA experiments.

Based on these data a TSA was designed and tested in a single reactor

setup, switching between adsorption and desorption operation. First results indicate the possibility of using a two-reactor-setup with a clean gas to flush gas efficiency of $1.4 \text{ m}^3_{\text{clean gas}}/\text{m}^3_{\text{flush gas}}$.

In further work the replacement effects, observed in this study will be

analyzed in more detail. Also a further optimization of the TSA, regarding a lowering of the desorption temperature and an increase of the clean gas to flush gas ratio will be performed.

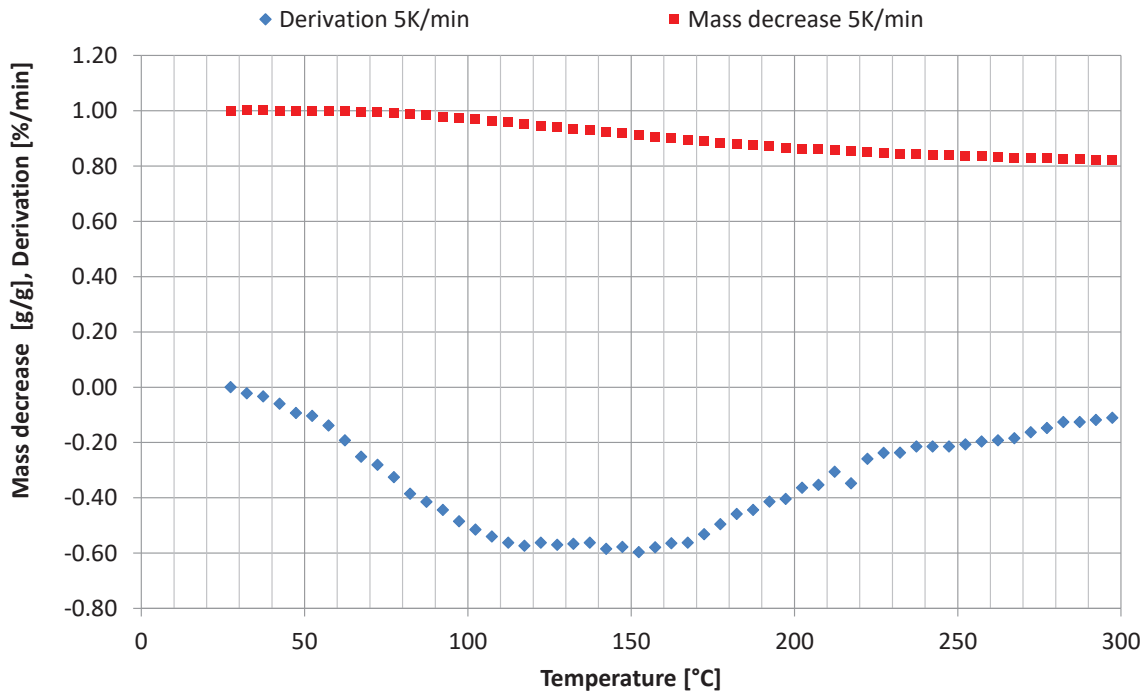


Fig.6: TGA analysis of the loaded AC

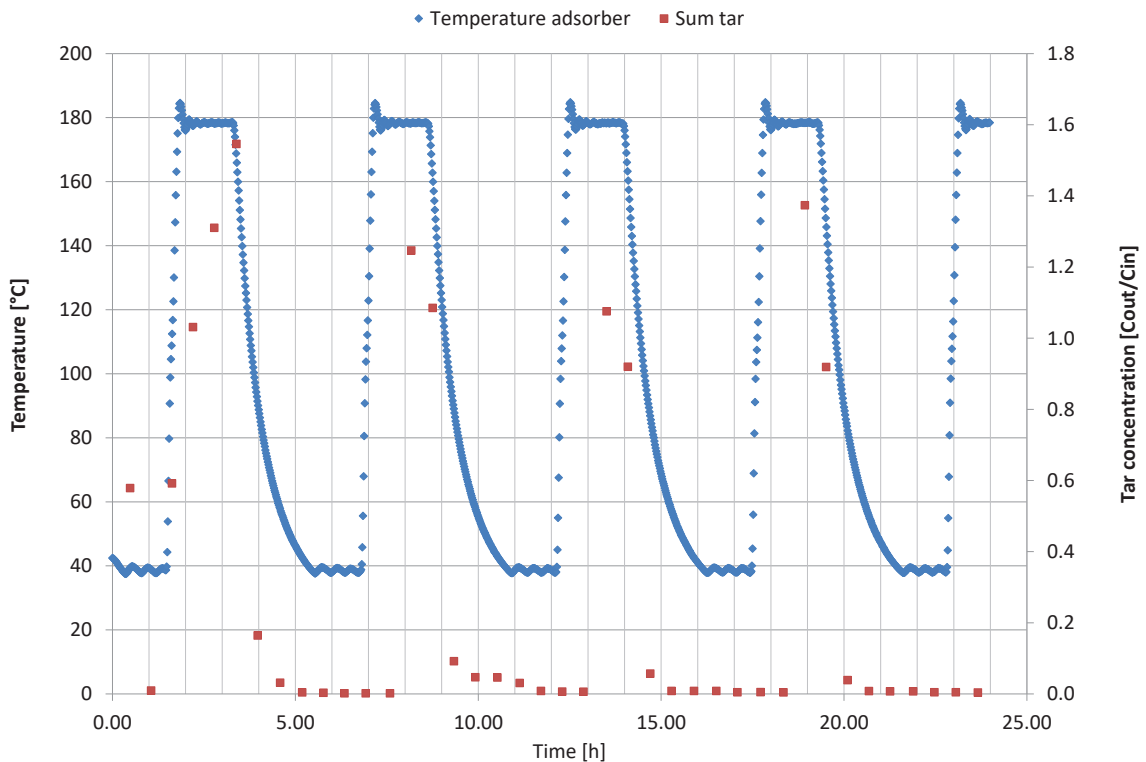


Fig.7: TSA performance over time after 24 hours of operation

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