Development and Testing of a Thermo-chemical Energy Store
- Results of a Five year Research Project -

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Abstract

During the five-year research project EnErChem, a new solar heating system has been developed, aiming to supply the demand for space heating of energy efficient buildings completely based on solar thermal energy. Within the research project engineers and material scientists from universities and industry cooperated interdisciplinary. The aim was the further development of a concept of efficient long term heat storage based on improved storage materials. Composite storage materials based on the combination of hygroscopic salts and zeolite granulates have been developed. To fully use the properties of the developed storage materials a robust reactor design has been developed. Based on this design a prototype store has been built and experimentally investigated in a "hardware in the loop" (HIL) test environment. The developed design of the thermal energy store and the experimental results of the thermal energy store operation are presented in this paper.

Keywords: thermo-chemical heat storage, thermal energy store, adsorption, solar heating, high solar fraction, testing, hardware in the loop

1. Introduction

Solar assisted space heating systems are already well established on the market. These systems are usually able to cover between 30 % and 50 % of the building's total heat demand using solar radiation. To increase the solar fraction of space heating systems towards covering the complete heat demand using solar radiation as the only energy source, efficient heat storage is a crucial aspect. The main part of already well established systems is a sensible heat store based on hot water. Due to continuous heat losses during the storage period high efficient long term heat storage based on sensible heat is not possible.

For the purpose of highly effective long-term heat storage the technology of thermo-chemical heat storage has the advantage of high energy storage densities and minimal heat losses during energy storage over long time periods. Hence, adsorption heat storage, a sub category of thermo-chemical heat storage, has been under intensive research in recent years (Engel et al. 2017, Mette et al. 2014a, Kerskes et al. 2012, Cuypers et al. 2012, Zondag et al. 2012, Jähnig et al. 2006). Within the research project EnErChem, funded by the German Federal Ministry for Economic Affairs and Energy (BMWi), the development of this technology was conducted. In this project material scientists from ITC (Institute of Chemical Technology, University of Leipzig) partners from industry (Vaillant GmbH, Chemiewerk Bad Köstritz) and process engineers from TZS/ITW (Research and Testing Centre for Thermal Solar Systems, Institute of Thermodynamics and Thermal Engineering, University of Stuttgart) were working collaboratively on the development of enhanced sorption storage materials and on the development of the thermo-chemical heat store itself. Both scientific fields are crucial for the implementation of a thermo-chemical heat store with a high energy density.

Previous work on development of thermo-chemical heat stores stated the need for improvement regarding the robustness and efficiency of the charging and discharging process. Additionally enhanced sorption storage materials show a significant change of their thermal characteristics, influencing the charging and discharging process of the thermo-chemical heat store. This paper shows the improvement of the charge and discharge operation of the developed thermo-chemical heat store due to the newly developed reactor design and discusses the impact of the changed thermal characteristics of the storage material on the thermo-chemical heat store operation.

2. Enhanced sorption storage materials

With a commercial zeolite material as a basis, the sorption properties have been improved systematically by ion exchange and the impregnation with salts (Nonnen et. al. 2016). Figure 1 shows the energy storage density related to the material volume of the commercially available zeolite 13X (Na-X, KÖSTROLITH 13XBFK, Chemiewerk Bad Köstritz), the corresponding Ca^{2+}-ion-exchanged zeolite Ca-X, as well as the salt-impregnated zeolite composite 15 $CaCl_2$/Ca-X on the basis of the ion-exchanged zeolite Ca-X.

Due to the ion-exchange, the storage density Q_V of the zeolite can be increased by approximately 15 % to about 145-180 kWh m^{-3} (desorption at temperature of T = 110 °C and partial pressure of water vapor of p_w: <0.2 mbar; adsorption at T = 30 °C/p_w: 5-25 mbar). Furthermore a profound understanding on the interaction between zeolite and salt could be obtained from investigations on the composite material (Nonnen 2016). Experiments at partial pressure of water vapor below the deliquescence of $CaCl_2$ show, that adsorption centers of the zeolite were covered by salt. Consequently, the storage density is decreased compared to the pure zeolite material. For water vapor pressures exceeding the deliquescence humidity of $CaCl_2$, the storage density of the composite material increases. Due to the formation of a salt solution, the energy storage takes place by simultaneous water adsorption by the zeolite and water absorption by the salt solution.
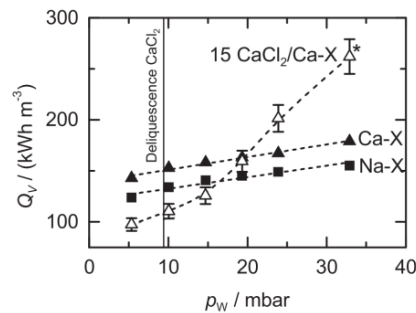

Fig. 1: Energy storage density Q_v of selected storage materials as a function of water vapor pressure p_w. Experimental conditions: desorption at 110 °C, < 0.2 mbar water vapor pressure, adsorption at 30 °C, 5-35 mbar water vapor pressure (Nonnen et al. 2016)

While the adsorption kinetic remained unchanged after the ion-exchange in the zeolite, the salt-impregnated zeolite changed its adsorption kinetic vastly. Figure 2 demonstrates this change, showing the results of an adsorption experiment in a packed bed reactor at a water vapor partial pressure of 24 mbar, an air mass flow rate of 0.3 kg h^{-1} resulting in a mass flow rate per cross section area storage material of 330 kg m^{-2} h^{-1}.
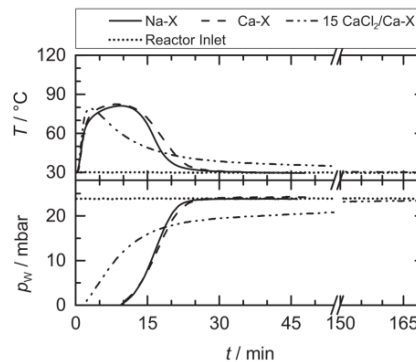

Fig.2: Comparison of a temperature T and water vapor pressure p_w at the outlet of a fixed bed reactor during adsorption for the storage materials Na-X, Ca-X and 15 $CaCl_2$/Ca-X (Nonnen et. al. 2016)

All three materials are showing a maximum temperature lift of about 50 K. While the water vapor breakthrough for the Na-X and Ca-X zeolites is begins after 9 min, it starts already after 2 min for the composite 15 CaCl2/Ca-X and extends over a time period more than 5 times as long as for the salt free zeolites. This characteristic is a challenge for the application of the material in a thermo-chemical heat store.

In addition to the steps of material development and laboratory characterization, the process for large-scale manufacturing of the composite has been developed by the industrial partner Chemiewerk Bad Köstritz. Material batches of 150 liters up to 200 liters have been produced. The produced material was inserted in the newly developed store and experimentally investigated in a "hardware in the loop" (HIL) test facility under representative operation conditions described in section 5.

3. Development of improved storage design

The implementation of continuous charging and discharging process of the thermo-chemical energy store has been realized as an open adsorption process with material transport. The thermo-chemical energy store consists of three main parts: The storage vessel, a material conveyor system and an external reactor unit (cf. Figure 3). Charged and discharged material is stored separately in a thermally uninsulated, water vapor proof vessel. For charging and discharging the material is transported via the material conveyor system (e.g. vacuum exhauster transport system) to the external reactor unit. In the reactor unit the storage material flows gravity driven through the reactor and leaves the reactor unit at the bottom where it is collected and transported back to the appropriate part of the storage vessel.
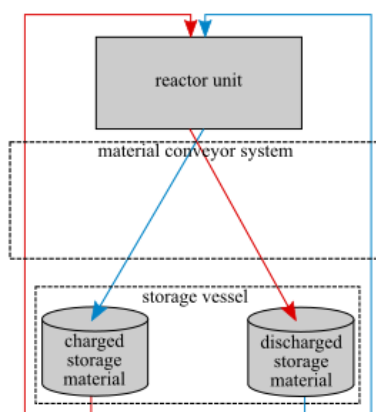

Fig. 3: Schematic view of the thermo-chemical store components

This concept has been applied by Kerskes et al. (2012) and Mette et al. (2014a). It shows a great potential for the realization of a high thermal performance thermo-chemical heat storage. The earlier work also addresses challenges regarding the robust operation and efficiency during charging and discharging of the storage material in the external reactor. A semi batch operation mode has been suggested by Kerskes et al. (2012) to implement an interruption-free operation. The experimental work on the semi batch operation has been limited to operation modes with a constant storage material mass flow rate. An introduction of a process controlled storage material mass flow rate based on the temperature measured in the reactor has been identified as the most promising improvement for a stable reactor operation. The implications resulting from a temperature based storage material mass flow control in a single stage reactor as used in previous work and further challenges regarding the efficiency of the charging and discharging process led to the development of an improved reactor design based on a two stage reactor.

Improvement of the reaction behaviour through reactor splitting

During charging and discharging the reactor is operated in a semi batch operation mode (see figure 3 left). Small portions of storage material are extracted discontinuously at the bottom of the reactor. Fresh storage material falls from a buffer vessel at the top of the reactor into the reactor bed. The air streams through the reactor bed in cross flow.

To ensure that the storage material always leaves the reactor completely charged/discharged it has to stay in the reactor bed until the adsorption process is completed everywhere in the material batch. In practice this state is hardly achievable. Due to grain size deviations, zones with unevenly distributed air flow are present. The increased friction between the storage material and the reactor walls creates zones with lower material flow. These influences lead to different timings of water vapor breakthrough in different parts of the reactor. Therefore a buffer zone at the bottom of the reactor is necessary where the charging/discharging of all parts of the material is completed. Furthermore the buffer zone provides the possibility to transfer the sensible heat of the storage material to the air stream during discharging.

The impact of introducing a buffer zone in a single stage reactor shall be explained with an adsorption example. The example sequence is based on a dynamic simulation of the adsorption process. The used storage material is the zeolite Na-X. The storage material enters the reactor with a temperature $T_{s-in} = 20$ °C and a water loading of $X_s = 84$ g/kg. The reactor is crossed with a humid air stream with a mass flow rate of $\dot{m}_f = 300$ kg/h. The humid air enters the reactor with a temperature of $T_{f-in} = 30$ °C and the specific humidity of $x_{f-in} = 6$ g/kg corresponding to water vapor pressure of approximately 9.4 mbar. At the beginning of the adsorption sequence the reactor is completely filled with fresh storage material. The reactor control initiates an exchange of one storage material portion (1.0 liter) if the trigger

temperature T_{ME} is lower than $T_{f\text{-in}}$ + 1.8 K. Figure 3 shows a scheme of the single stage reactor as well as the time series for specific humidity, temperatures and the binary signal for storage material exchange (ME) in the reactor.
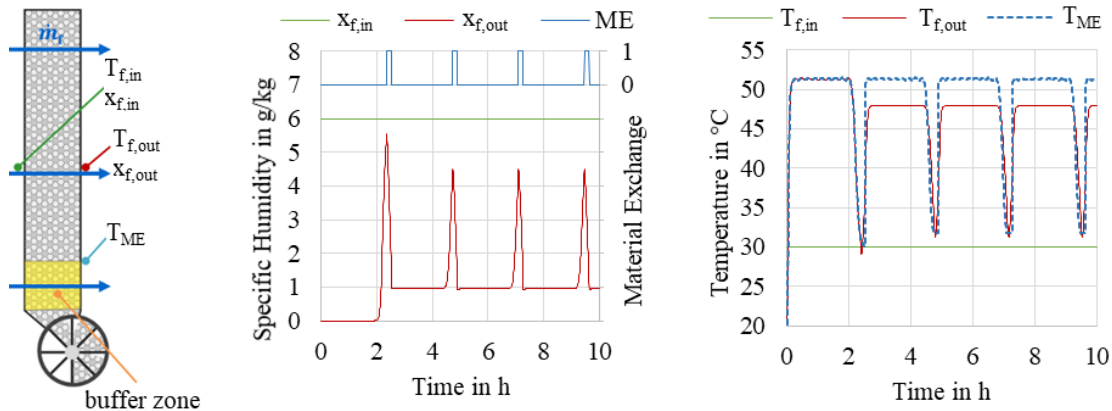

Fig. 3: Simulation results of an adsorption sequence in a single stage reactor

The process illustrated in figure 3 shows that in a single stage reactor the introduced buffer zone has multiple disadvantages. Since the reactor depletes as a whole, all storage material portions above the buffer zone are exchanged after each other. This leads to a short drop of the outlet temperature $T_{f,out}$. Since the buffer zone is not filled with fresh material the specific humidity of the air stream is not adsorbed in the buffer zone. Therefore after the initial period the specific humidity in the mixed air stream behind the reactor has a value of at least 1.0 g/kg. Since there is no temperature lift in the buffer zone the temperature in the mixed air stream behind the reactor ($T_{f,out} \approx 48$ °C) is also lower compared to the temperature during the initial period of 51 °C.

To overcome the disadvantages of the single stage reactor the reactor has been split up into two stages: a main reactor and a pre-reactor (see figure 4 left). In this two stage reactor the air first flows through the pre-reactor, is redirected at the outlet of the pre-reactor and then enters the main reactor. The storage material enters the main reactor first and is moved to the pre-reactor after leaving the main reactor. The introduced buffer zone is now placed in the bottom part of the pre-reactor. Analog to the single stage reactor the storage material portions will be exchanged based on the trigger temperature at the top of the buffer zone.

For the comparison of the single stage reactor with the double stage reactor the same adsorption sequence has been applied. The sum of the reactor bed thickness of the double stage reactor equals the reactor bed thickness of the single stage reactor. The cross area of each, the main reactor and pre-reactor is equal to the cross area of the single stage reactor. Therefore the pressure drop over the whole reactor remains similar. Figure 4 shows the calculation results. In the double stage reactor the specific humidity and the temperature at the outlet of the pre-reactor are specified in addition to the quantities at the inlet and outlet of the whole reactor.
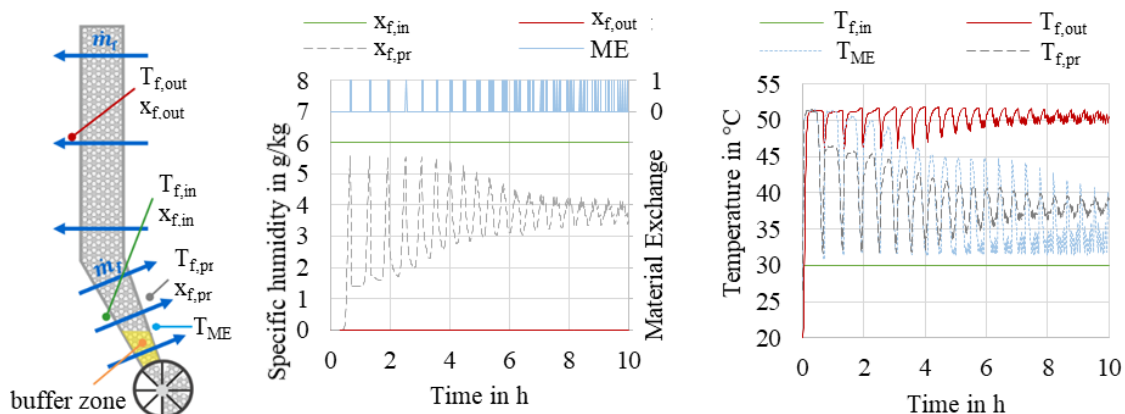

Fig. 4: Simulation results of an adsorption sequence in a double stage reactor

Due to the lower reactor bed thickness of the pre-reactor the specific humidity at the outlet of the pre-reactor rises and the temperature falls already after a period of about 0.5 h. The adsorption in the pre-reactor is completed and the first storage material portion is exchanged. Since the main reactor is still filled with fresh material this has no impact

on the specific humidity at the outlet of the reactor. The exchange of the storage material portion causes a short reduction of the outlet temperature. The entering storage material in the upper part of the reactor has a temperature of 20 °C and has to be heated up to the process temperature. During the whole sequence the water vapor is completely adsorbed in the two stage reactor and the mixed air stream temperature at the reactor outlet remains nearly at the maximum of 51 °C. In this way the introduced buffer zone does not have a negative impact on the reactor performance.

4. Experimental investigation of the improved storage design

For demonstration and performance testing of thermo-chemical energy storage device with the newly developed reactor unit with a two stage reactor, a "hardware-in-the-loop" (HIL) test facility has been built. The HIL test facility consists of an air loop, a heat load emulation loop and an electrical power source. With the air loop a humid air stream with a defined specific humidity in the range from 3 to 16 g/kg, a temperature in the range of 15 to 30 °C and a mass flow rate up to 200 kg/h can be provided to the reactor of thermo-chemical energy store. In the heat load emulation loop thermal oil is circulated with a mass flow rate between 100 and 250 kg/h. For tempering of the oil an electrically heated thermostat is used for heating with a power up to 4 kW and for cooling with a power up to 2.5 kW in a temperature range from 20 to 200 °C.

In the HIL test facility the thermo-chemical storage has been operated under real application conditions. Adsorption and desorption experiments have been carried out in application scale. All ingoing and outgoing energy fluxes were measured based on the measurement of temperatures, mass flow rates and humidities, which allowed an energetic balancing under dynamic operating conditions.

Developed reactor unit with a two stage reactor

The newly developed reactor unit consists of four key components: the two stage reactor (consisting of main and pre reactor), air heat exchanger, charge heat exchanger (or electrical heating rod) and discharge heat exchanger. All key components are placed in a common thermal insulation (cf. Figure 5). During operation ambient air enters the reactor unit through the air heat exchanger and is preheated by the exiting air stream. Thus the air heat exchanger decouples the reactor operation temperature level from the ambient temperature. Afterwards the air flows through the charge heat exchanger, which is only active during charging, and enters the reactor through the pre-reactor. The air steam is deflected in a duct behind the pre-reactor and led through the main reactor. Finally it flows through the discharge heat exchanger, only active during the discharge operation, which is placed directly after the main reactor.
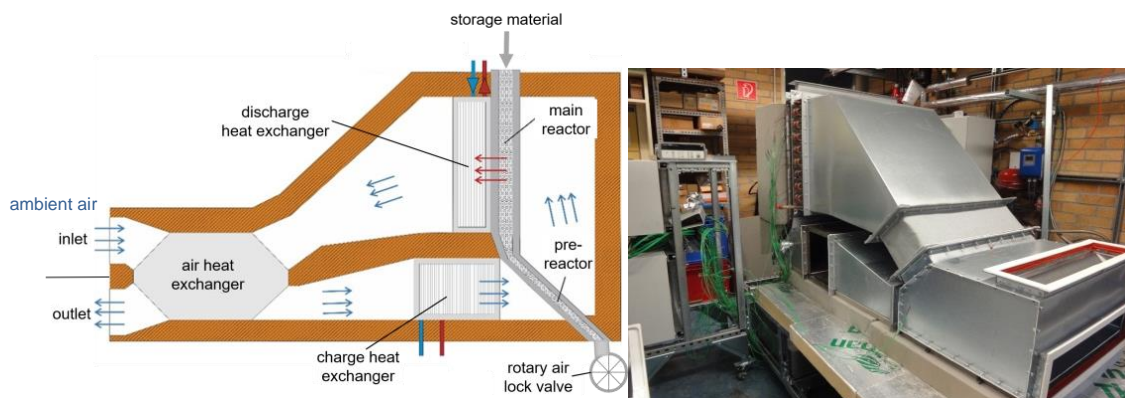

Fig. 5: Sketch of the reactor unit with a two stage reactor (left) and the reactor unit during construction integrated in the "hard ware in the loop" (HIL) test environment (right)

The reactor accommodates approximately 24 liters of storage material. The pre-reactor has a cross area of 0.245 m^2 and a bed depth of 0.02 m. The main reactor has a cross area of 0.49 m^2 and a bed depth of 0.05 m. A buffer vessel at the top of the reactor unit contains additionally 90 liters of storage material. The storage material enters the reactor unit from the buffer vessel and is conveyed through the reactor driven by gravity. The dosing of the storage material is handled by a customized rotary air lock valve. The rotary air lock valve is operated discontinuously triggered depending on the temperature behind the pre-reactor. The volume of each rotary air lock valve chamber is 0.7 liters, which corresponds to the storage material portions which are extracted at once.

In the experimental setup a vacuum exhaust transport system has been added at the outlet of the rotary air lock valve.

This system transports the charged/discharged storage material into a storage vessel, where it is stored water vapor tight until the next test sequence. As storage vessels for the charged and discharged storage material two metal bins have been used. The transport from the storage vessel to the buffer vessel is done manually before the beginning of a test sequence.

The newly developed reactor unit has been operated in a fully automated mode under realistic conditions in approximately 50 adsorption and desorption experiments, each with a duration between 4 and 20 hours. During discharging the set value for the reactor unit is the flow temperature of the heat load emulation loop. The reactor control adapts the air mass flow rate through the reactor to match the reactor adsorption output to the heat demand of the discharge heat exchanger. The mass flow rate and the return temperature of the heat load emulation loop were applied as boundary conditions. During charging the temperature at the inlet of the pre-reactor is the set value. To attain the required pre-reactor inlet temperature in this operation mode the air mass flow rate is adapted to the available charging power and the outlet temperature of the main reactor.

5. Results of HIL experiments

The following questions have been experimentally investigated in the HIL test facility:

- Is the reactor unit able to provide the expected thermal power?
- Is the reactor unit operating robust when dynamic heating demand is applied during discharging?
- Can the theoretically available energy storage density of the storage material be fully used in the reactor unit?
- Is efficient operation with the commercially available Na-X zeolite and the newly developed composite material $15CaCl_2/Ca-X$ in the reactor unit possible?

Reactor unit operation with different storage materials

A series of adsorption and desorption experiments have been successfully conducted with the commercially available zeolite Na-X and with the newly developed composite material 15 CaCl2/Ca-X.

In the results of the desorption experiments shown in figure 6 the storage material is desorbed at a temperature of 180 °C. The air mass flow rate through the reactor unit is controlled to provide a defined fluid temperature measured at the reactor inlet of $T_{f\text{-in}} = 192$ °C. A portion of the storage material is released, when the fluid temperature measured at the outlet of the pre-reactor bed $T_{f\text{-pr}}$ reaches 180 °C. Figure 6 shows the temperatures and the specific humidity during desorption of the zeolite Na-X and during desorption of the composite material $15\ CaCl_2/Ca-X$.
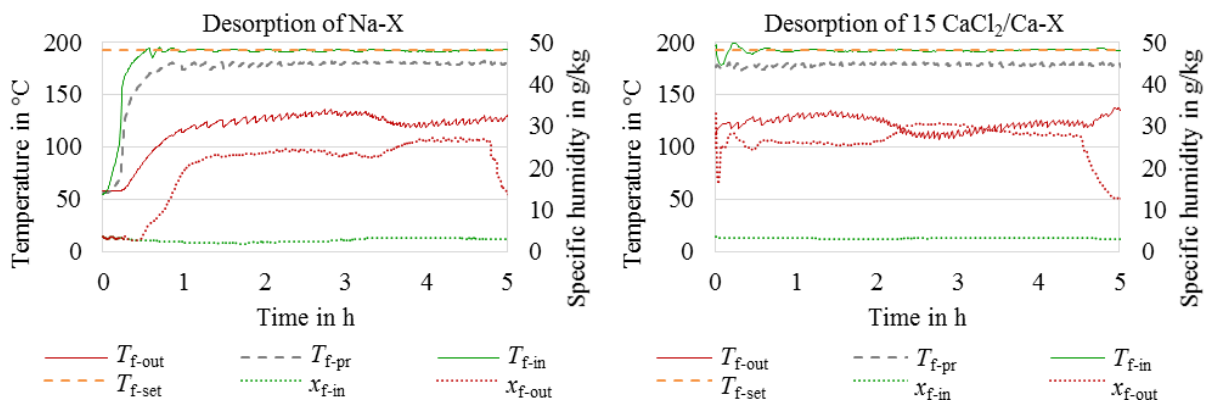

Fig. 6: Mean temperatures in the reactor unit as well as specific humidity and temperatures at the inlet and outlet of the reactor unit during a stationary desorption of Na-X zeolite (left) and the composite 15CaCl2/Ca-X (right)

As shown in figure 6 a stable desorption process continues over a period of about 5 hours for both storage materials. In both sequences similar fluid output conditions are reached over the major part of the shown period. The fluid temperatures at the reactor outlet ($T_{f\text{-out}}$) are in both cases in the range from 110 °C to 140 °C. The reactor outlet specific humidity is in both experiments in a similar range from 22 g/kg to 30 g/kg. After a starting period of approximately 1 hour (not included in the shown desorption of 15 CaCl$_2$/Ca-X) a stationary operation of the reactor is reached and continues until the buffer vessel at the top of the reactor runs out of storage material.

In the adsorption experiments shown in figure 7 the storage material has been discharged during an adsorption process with an air mass flow rate through the reactor unit of approximately 110 kg/h. The mean thermal output

power of the reactor unit during the adsorption process was 680 W. Due to control errors the specific humidity deviates slightly in the two shown experiments resulting.
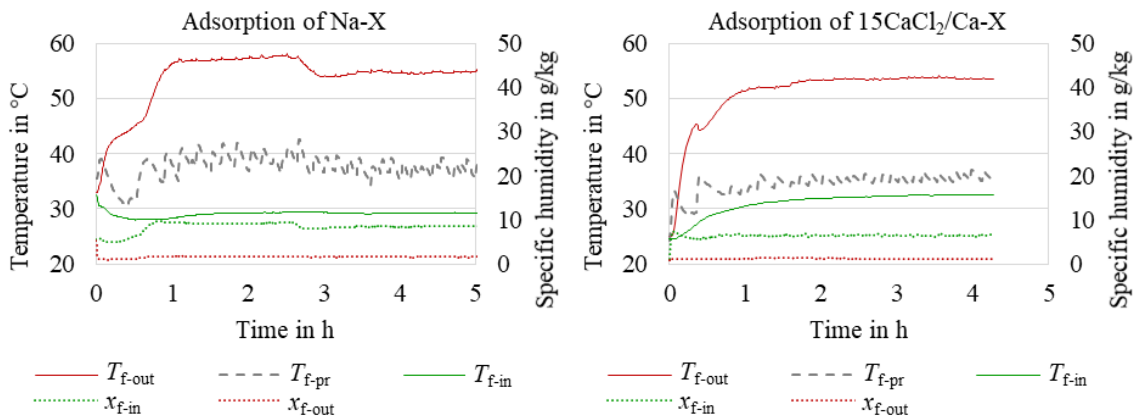

Fig. 7: Mean temperatures in the reactor unit as well as specific humidity and temperatures at the inlet and outlet of the reactor unit during a stationary adsorption of Na-X zeolite (left) and the composite 15CaCl2/Ca-X (right)

As shown in figure 7 in both experiments a stationary outlet temperature of approximately 54 °C has been reached after 3 hours of operation. After a period of approximately 20 to 30 minutes the material exchange starts in both cases. The process is stationary after approximately 1 hour. The temperature $T_{f\text{-}pr}$ at the outlet of the pre-reactor is slightly lower during the adsorption of the 15 $CaCl_2$/Ca-X compared to the adsorption of Na-X. This behavior is consistent with the results measured in a fixed bed reactor, which have been shown in figure 2. Although the zeolite-salt composite 15 CaCl2/Ca-X has a slower reaction kinetic, the double stage reactor design compensates this limitation and allows a stationary charging and discharging process.

Measured reactor unit output power

To determine if the reactor unit is able to provide the expected heat flow rate, multiple adsorption experiments have been conducted. In this series of experiments the specific humidity has been varied in the range from 5.5 g/kg to 13.5 g/kg. The heat load demand has been varied in the range from 500 W to 1800 W. As reference the theoretical heat flow rate is used which is calculated based on the measured water uptake from the air stream and the standard adsorption enthalpy of the Na-X zeolite of h_{ads} = 3456 kJ/kg determined by Mette et al. (2014b). The reactor unit output power is determined based on measured inlet and outlet temperatures and mass flow rate of the heat load emulation loop. Figure 8 shows the comparison between the theoretical heat flow rate and the measured reactor unit output power.
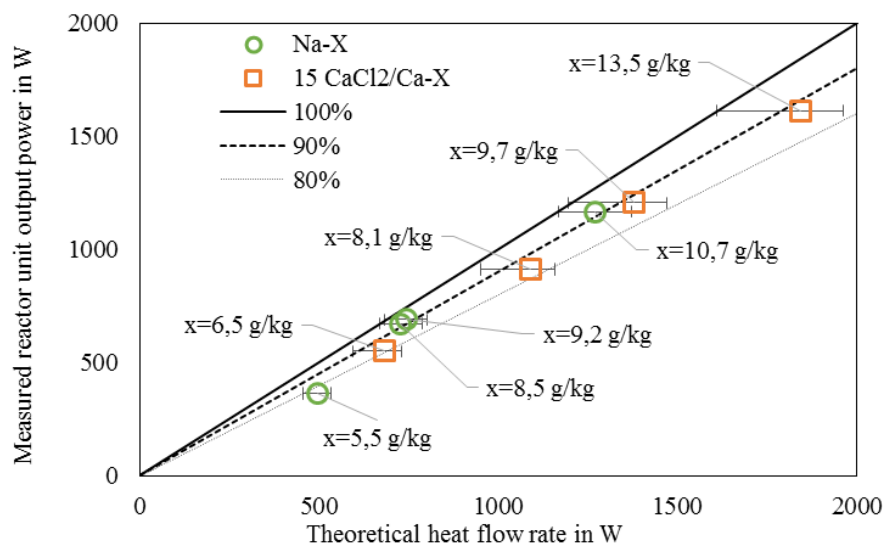

Fig. 8: Comparison of the measured reactor unit output power to the theoretical heat flow rate or power respectively obtainable for Na-X zeolite and the composite 15CaCl2/Ca-X at given boundary conditions

As shown in figure 8 in the most experiments the measured reactor unit output for the zeolite Na-X has reached

approximately 90 % of the theoretical heat flow rate. Operation conditions with lower humidity and operation with the zeolite salt composite 15 CaCl$_2$/Ca-X leads to a slight degradation of the efficiency. In this cases the measured reactor unit output is about 80 % of the theoretical heat flow rate.

Robustness of reactor unit operation

To ensure a robust operation of the reactor unit adsorption experiments with varying heat demand have been conducted. The heat demand variation has been applied through variation of the mass flow rate in the heat load emulation loop. Small deviations of the specific humidity in the air stream entering the reactor constitute an additional disturbance representing a dynamic environment. Figure 9 shows the results of an adsorption experiment.
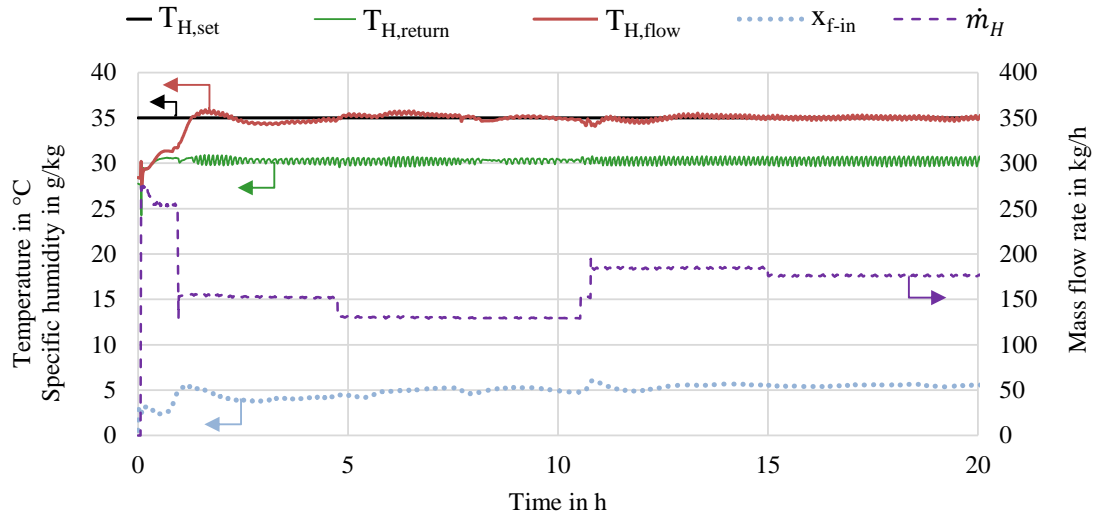

Fig. 9: Temperatures and the mass flow rate in the heating loop of the reactor unit and specific humidity at the inlet of the reactor unit during adsorption of Na-X zeolite with variations of the heat demand

The results show that the reactor unit managed to provide the required flow temperature of $T_{H\text{-set}} = 35$ °C with only small deviations. The reactor unit control adapted the mass flow rate of the air through the reactor adequately and with it the required thermal power of the reactor. Thus both deviations due to the variation of the humidity and the variation of the heat demand were compensated.

Degree of storage material capacity utilization

The introduction of a pre-reactor with a buffer zone at the bottom theoretically allows to utilize the full storage material capacity. Therefore experiments have been conducted to investigate the degree of storage capacity utilization.

During adsorption and desorption operation of the reactor unit in the HIL test facility material samples of approximately 10 g have been extracted for analyses from the storage vessel. The samples have been taken from the storage vessel after finishing the experiment from volume portions of 10 l each. The water loading of the samples has been determined using a halogen moisture analyzer from the manufacturer Mettler Toledo. All samples were desorbed in the moisture analyzer to reference conditions ($T_{ref} = 180$ °C, $x_{f\text{-ref}} = 7.2$ g/kg), where the change of the water loading between the state after the experiment and the reference conditions has been measured. From the change in the water loading the water loading at the end of the experiment has been calculated.

The theoretical storage capacity of the storage material is being utilized when the material reaches the equilibrium water loading at the given conditions. The equilibrium water loading has been calculated from the isotherms (Mette et al. 2014b) of the zeolite Na-X for each sample. The equilibrium was calculated according to the conditions ($T_{f\text{-pr}}$, $x_{f\text{-in}}$) in the lower part of the pre-reactor during the residence time of the material sample in the pre reactor. Figure 8 shows the results of the comparison of water loading X_s measured in samples and the calculated equilibrium water loading during an exemplary adsorption and desorption sequence. The samples are displayed over the normalized volume fraction. The normalized volume fraction is the total storage material volume used in the sequence divided by the reactor volume.
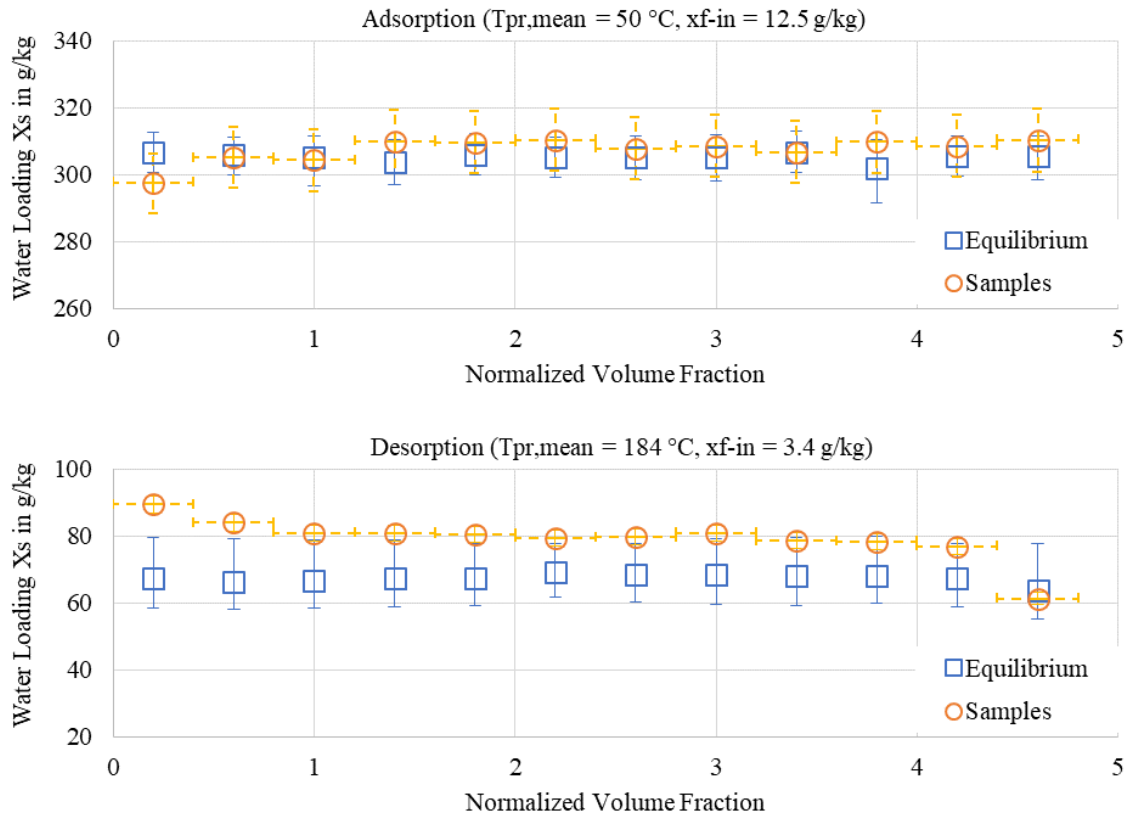
Fig. 1: Measured water loading of Na-X after an adsorption and desorption sequence in the reactor unit compared with the water loading of Na-X reached in equilibrium

The results for the adsorption experiment show that the measured and calculated equilibrium water loading matches well within the error margin of the experiment. In the shown desorption sequence the measured water loading is approximately 10 g/kg higher than the calculated equilibrium water loading. The measured water loading lift between the shown desorption and adsorption is 228 g/kg. Compared to the maximum water loading lift of 238 g/kg this shows a 96 % degree of storage capacity utilization. This experiment shows further that the charging and discharging of the storage material is robust over multiple filling volumes of the reactor. Only the first probe shows lowered water loading compared to the average values.

The shown experiment has been conducted for a combination of more than 10 adsorption and desorption sequences. The degree of storage capacity utilization in all experiments is in the range from 92 to 98 %.

6. Conclusions

Zeolites and zeolite salt composites are promising energy storage materials applicable for seasonal heat storage. Both storage materials have a high energy storage density but show different characteristics regarding the adsorption kinetic. Therefore a new reactor unit design has been developed. In this design the reactor is split into two stages: the main reactor and the pre-reactor. This two stage reactor allows an efficient and robust charging and discharging of the storage material under dynamic operation conditions.

For experimental investigation of the newly developed reactor unit a HIL test facility has been built up. The experiments carried out in the HIL test facility are an important step from laboratory to demonstration scale. The developed thermo-chemical store has been operated in the HIL test facility in a fully automated mode under realistic conditions. In stationary adsorption and desorption experiments using the pure zeolite Na-X and the zeolite salt composite 15 $CaCl_2$/Ca-X the expected operation behavior of the reactor unit has been confirmed. During discharging the rector unit shows an efficient operation: More than 80 % of the theoretical rector unit output power has been obtained for all investigated settings of humidity. Furthermore it has been shown, that approximately 95 % of the theoretical value of energy storage density provided by the storage material could be utilized with the newly developed two stage reactor design.

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