Packed Bed Zeolite Experimental Setup To Study TCS Systems Up To 200ºC

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Abstract

Given the concern about the depletion of natural resources, some researchers have been motivated to study and develop new technologies of thermal energy storage (TES). It is well-known that thermochemical storage has a series of advantages compared to other kind of TES technologies (i.e. sensible or latent). This study starts with the design, construction, and start up of two experimental setups where the process of thermochemical storage by water adsorption/desorption of zeolitic systems can be measured. Afterwards, thermal behaviour of the process is investigated experimentally through operating factors that may influence it (such as degree of relative humidity, airflow inlet temperature and flow, particle diameter, amount of zeolite and setup). First results highlight the relevance of trade-off between different factors such as height of the packed bed system and the airflow.

Keywords: thermochemical energy storage, adsorption, zeolite, TES, experimental

1. Introduction

Methods of thermal energy storage (TES) have the objective to store heat or cold in form of thermal energy and release it when necessary. TES is mainly divided into three types: sensible, latent and thermochemical. Thermochemical storage (TCS) includes both chemical reaction and sorption storages. In sorption processes, thermal energy is stored thanks to an endothermic dissociation reaction that occurs when two reactant substances are separated. The energy is released when the reverse reaction takes place. Up to now, majority of TCS studies are related to solar applications (Mette et al. 2012; Yu et al. 2013). However, there are many other domestic or industrial applications and processes requiring thermal energy to operate but having waste heat flows that could be managed by a TCS system in a more efficient way. Many researchers have conducted experimental studies to investigate the equilibrium adsorption capacity, thermodynamic properties and energy storage capacity of a wide range of storage materials (Abedin and Rosen, 2011; Ding and Rifat, 2013). For the studies presented here, the adsorbent working pair chosen has been water and zeolite NaY. This working pair has shown a good cyclic behaviour without degradation over large number of cycles. Furthermore, zeolite NaY has demonstrated to have better results than other zeolites due to its higher energy density (Boggenbarg et al. 2002; Whiting et al. 2013). The main objective of this study is to design, construct and test an experimental setup where the process of thermochemical storage by water adsorption/desorption of zeolitic systems can be measured and thermal behaviour can be studied. Analysis of the experimental outcomes will be the basis to support the incorporation of such TCS systems in adequate applications (e.g. waste heat recovery in ovens, waste heat in engines, etc.).

2. Experimental Method

2.1. Thermochemical storage, basics

The term thermochemical storage is generally used to encompass two TES mechanisms: chemical reaction and sorption (adsorption and absorption). Both methods are based on a physical-chemical reversible reaction. According to Fig. 1, component C is the thermochemical material. Considering an input of heat, C is dissociated into A and B components, which can be stored separately. Both are reactants in the chemical reaction process or working pair in the sorption process. C can be formed again when A and B are in contact under certain conditions. The working principle of the process consists of the stages plotted in the figure 1.
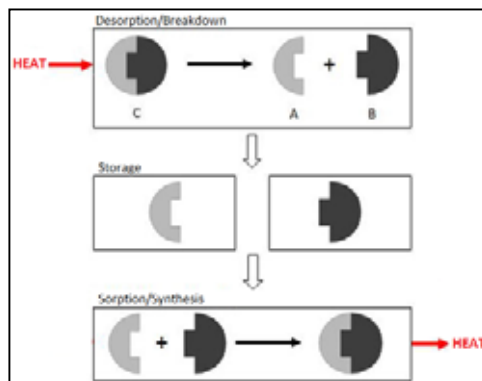

Fig. 1: Stages of thermochemical energy storage (Hauer, 2007)

2.2 Thermochemical storage by adsorption

Adsorption is a process that occurs when a gas o liquid stream gets in contact with a solid, and then one or more components of this stream adhere to the solid. Adsorption takes place by binding forces that are established between the solid and the stream component(s). A certain amount of heat is released as denoted in equation 1:

$$E_{adsorption} = \Delta H_{adsorption} \cdot m_{adsorbed} \qquad \text{(eq. 1)}$$

It depends on the enthalpy of adsorption and the adsorbed amount of adsorbate. Both, enthalpy and mass can be determined by specific measuring techniques (i.e. calorimetry, thermogravimetry). Besides, the amount of adsorbed water can be determined by differential weighing of the solid bed before and after the adsorption stage.

2.3 Working pair

One interesting working pair for heat storage system by adsorption is constituted by zeolite as adsorbent and water vapour as the adsorbate (Dawoud et al. 2007; Wang et al. 2009; Dieng and Wang, 2001; Baker, 2008). Water is a very suitable compound to use as thermochemical adsorbate in storage systems as it has a high enthalpy of evaporation, which pushed for high heat of adsorption. It is also a non-toxic compound and non-hazard to persons or to the environment when handled (Henninger et al. 2010; Heyden et al. 2009). Zeolites are a family of hydrous aluminosilicate minerals. They have a porous structure with uniform pore dimensions (Auerbach et al. 2003). When the zeolite is dehydrated it has a large internal surface which provides a great capacity for selective adsorption of any molecule that can penetrate into their cavities. Once water vapour is selected as adsorbed compound, it is necessary to choose a zeolite with a high hydrophilic character. The two classes of zeolites most widely used for this purpose are type A and faujasite NaX and NaY (Heyden et al. 2009; Levitskij et al. 1996; Henninger et al. 2012). In the study here, NaY faujasite is used.

A characterization of the zeolite NaY is performed to obtain values of certain parameters relevant for the study and to preliminary check its behaviour. A thermogravimetry analysis was performed at the Department of Chemical Engineering and Environmental Technology (University of Zaragoza) using a Mettler Toledo TGA/DSC 1 STARe System (Mettler product catalog, Thermal Analysis). From the thermogravimetry (TG) curve and its derivative (DTG) shown in Fig.2, the zeolite adsorption capacity (equation 2) and total drying temperature (250ºC) is obtained.

$$\text{Adsorption capacity } [\%] = \frac{W_{H_2O(v),ads}[mg]}{W_{dry\ zeolite}[mg]} \cdot 100 = 30.3\ \% \quad \text{(eq. 2)}$$

Later on, for pre-design purposes, the corresponding mass of zeolite required to store a specific amount of energy can be evaluated.
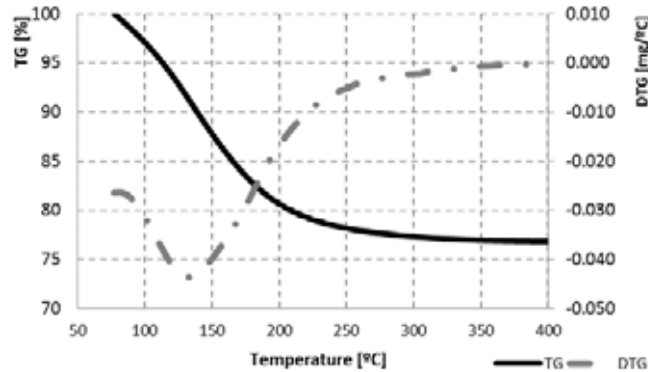

Fig. 2: Graph of thermogravimetry of NaY zeolite. Heating rate 10°C/min

2.4 The process

The thermochemical energy storage by adsorption process consists of two main stages, namely:

Desorption: Initially, zeolite saturated with water vapour is inside the reactor. As desorption is an endothermic process, it is necessary an external heat input. In the experimental setup, this heat is provided by a hot and dry air stream entering from the top of the reactor. When this stream contacts the saturated zeolite, two transfer phenomena occur:

    A. <u>Heat transfer</u>: air stream transfers the heat needed to evaporate the adsorbed water.
    B. <u>Mass transfer</u>: water is desorbed from the zeolite and becomes part of the air stream.

The air stream leaves the reactor at a lower temperature than at the inlet and with a higher relative humidity. While the zeolite is dried, the relative humidity at the outlet will increase. When the zeolite bed is completely dried, the humidity conditions at the inlet and the outlet are the same.

Adsorption: When the two substances, zeolite and water vapour re-contact, the working material regeneration occurs. Water vapour enters the system when a stream of humid air at ambient conditions is fed in the reactor. Again two types of transfers occur:

    A. <u>Mass Transfer</u>: water vapour is attracted to the NaY zeolite surface where it adheres.
    B. <u>Heat Transfer</u>: the heat released due to condensation of water vapour is taken up by the air stream leaving the reactor.

While adsorbing, outlet air stream is hotter and dryer that at the inlet. While the zeolite is wetted, the relative humidity of the output current will be zero. Once the zeolite bed is fully wet, inlet and outlet humidity conditions will be the same.

3. Description of Experimental Setups

In order to test the thermal performance of sorption-based TCS reactors, two experimental setups have been developed. The first one (preliminary) allows a rough and coarse analysis, due to its simpler instrumentation (fish tank air compressor, handmade oven, laboratory impinger, rotameter). The second one is fine-tuned and all the operating conditions can be set with higher precision thanks to a Controlled Evaporation Mixing (CEM) and the corresponding mass flow controllers; it is based on Hongois et al. (2011) and on the initial results obtained with the preliminary setup. Both experimental setups are designed to work as open systems. An air stream transports different water contents (ranging from 0 to 100% relative humidity) inside a cylindrical packed bed reactor, glass made, and surrounded by a vacuum chamber. A series of temperature and humidity sensors (±0.8%HR, ±0.1K) to characterize the processes has been installed, not only for the air streams but also for the packed bed (multi-temperature sensor). Pressure drop has been measured for different reactor configurations.

3.1. Preliminary setup

The purpose of this preliminary setup is to qualitative study the adsorption process taking place between NaY zeolite and water vapour. The setup is designed with adequate, economical materials, easy and fast purchase, to perform a preliminary study of the process by varying the initial conditions, and trying to delimit the working range of modifiable system parameters. The obtained data are the starting point for the design and subsequent study of the second installation. Fig. 3 shows a block diagram of this preliminary setup and a photo marking the main components.
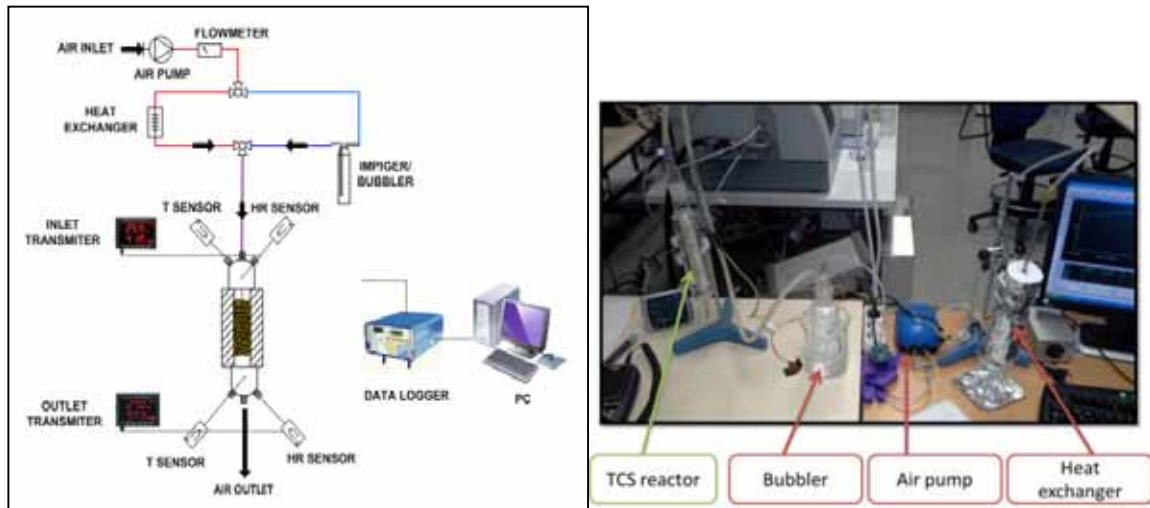

Fig. 3: Diagram of the preliminary setup (left) and photo with main components marked (right)

3.2. Fine setup

The purpose of this installation is to study more quantitatively the adsorption and desorption processes taking place. This setup will get more fine measures and with greater accuracy than in the preliminary setup. Fig. 4 shows a diagram of this final setup and Fig. 5 a photo marking the main components.
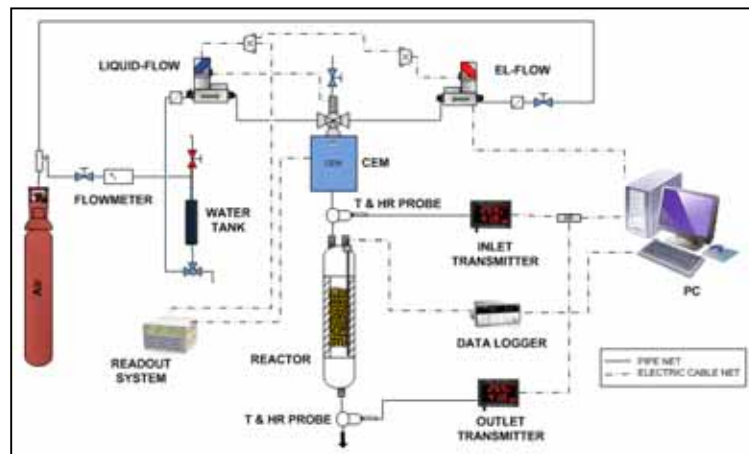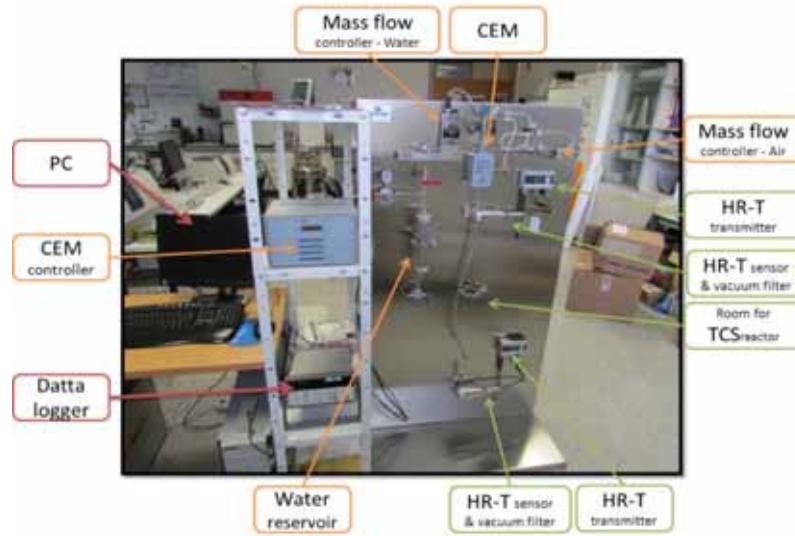

Fig. 5: Design of fine setup

Fig. 5: Photo of the fine setup and main components

One of the core differences between both setups is the CEM. This system is made up by three elements which show a series of advantages: accurately controlled gas ($\pm$0.8%) and liquid ($\pm$0.2%) mixture, fast response, high reproducibility (error <0.1%), very stable vapour flow, flexible selection gas/liquid ratio.

4. First results and Conclusions

The effect of the variation of a series of parameters and variables (air flow, particle size, height of packed bed) has been studied to analyse the thermal behaviour of the process. All the experiments reported here were done with the preliminary setup. As a reminder, when the inlet stream is in contact with the zeolite, the process does not take place in the whole bed at the same time. A zone is delimited. In this zone heat and mass transfers occur, and for this fact the zone is called transfer zone.

4.1. Airflow
A series of tests were carried out varying the airflow in the range of 4 to 9 litre/min in all cases saturated with water. Fig. 6 shows the outlet temperature of these flow-tests, keeping other conditions fairly constant. The most significant results of the study are compiled in Table 1.
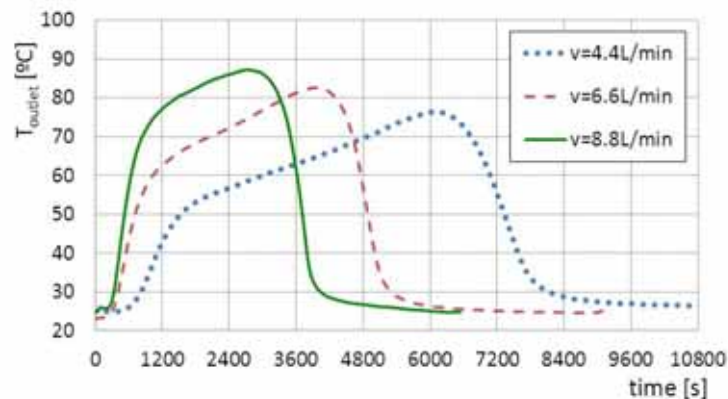

Fig. 6: Evolution of the outlet temperature for different flow rates

Tab.1: Results in experiments with different flow

Flow rate [L min^{-1}]	4.4	6.6	8.8
Maximum T_{outlet} [ºC]	76.42	82.65	87.26
Maximum ΔT [ºC]	51.24	57.95	61.92

There is a clear trend with the airflow: with this specific configuration, while air flow increases, adsorption is faster and temperature reached in the initial phase is higher. Also a particular shape of the out temperature curve is observed with 3 stages: ideal situation will be an step function (very steeper at the beginning, followed by a plateau at high temperature, and a quick drop in temperature with another steeper stage back to ambient temperature); in these cases what we can observe is that as air flow increases (meaning also increase in transported water), and the zeolite has the capability to adsorb, the shape of the curve becomes closer to the step function. There is a trade-off between a series of parameters that constrain mass and heat transfer (part of the energy will be used to heat up the air current, another to heat the zeolite, and some will be lost through the walls of the reactor), at macro level: water content in the air current, air velocity (flow), amount of zeolite, length of the packed bed, section of the packed bed.

By increasing the inlet flow, the water vapour flow rate is increased, which means that the transfer zone covers a larger bed length. The consequences are:
- Higher temperatures and ΔT obtained at the outlet as heat release is increased.
- Duration of the stage is shorter since the zeolite is saturated faster.

4.2. Bed Height

Height of packed bed was modified in the range of 3.5 to 15 cm, in four different positions as shown in Fig. 7. The following experiments were performed by fixing the inlet air flow, which means that the length of the transfer zone will be the same in all of them. When air leaves the transfer zone loses heat depending on the length of the remaining bed, due to the transfer of sensible heat between the air and the zeolite in the remaining bed. Therefore, it is expected that there will be an optimum of operation between bed height and airflow/relative humidity.
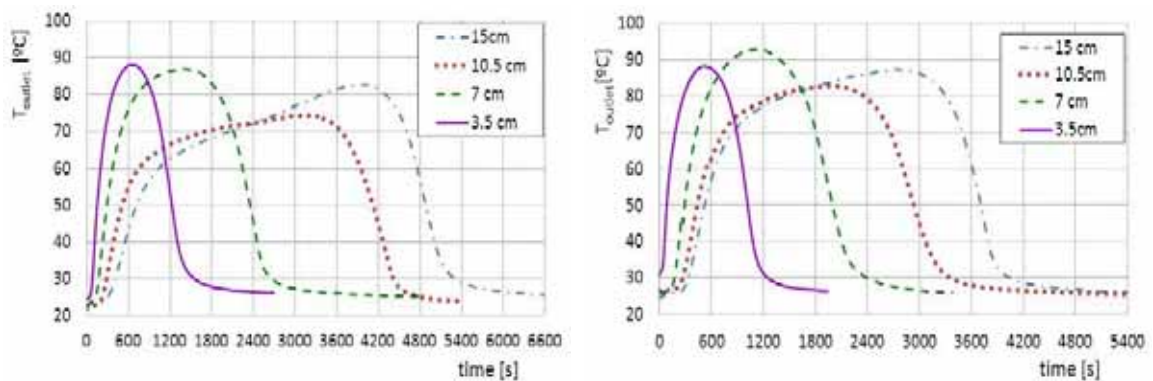

Fig. 7: Evolution of outlet T during adsorption for $\dot{v}$ = 6.6 litre/min (left) and $\dot{v}$ = 8.8 litre/min (right) at various bed heights

The most significant values of the study are shown in Table 2:

Tab.2: Results in experiments with different flow rates and bed heights

	Bed height [cm]	15	10.5	7	3.5
$\dot{v}$=6.6 L min^{-1}	T_{max} [ºC]	82.65	74.48	86.88	88.15
	ΔT_{max} [ºC]	57.95	51.47	61.14	62.29
$\dot{v}$=8.8 L min^{-1}	T_{max} [ºC]	87.26	82.81	92.86	88.24
	ΔT_{max} [ºC]	61.92	56.24	66.37	61.88

For a fixed air flow, as bed height decreases:
- The remaining bed length after the transfer zone is smaller. Therefore, heat transferred from the air stream to the rest of the zeolite is lowered.
- The duration of the stage is shorter. As the amount of zeolite is reduced, it is saturated earlier.

For the same bed height, as the flow rate increases, the outlet temperature profiles resemble and the stage duration decreases. Presumably, if the rate continues to increase, at some point the temperature profiles will have almost the same behaviour. When a flow rate of 8.8 litre/min is set, the maximum ΔT and outlet

temperature are achieved with a bed height of 7 cm and not with the lower bed height. It could mean that we have found an optimal operation given the design of the installation and the conditions established. Again, a trade-off between airflow and bed height is noticeable. When setting up a bed height of 10.5 cm we always get the worst results. In general terms there is not a clear trend so a deeper study will be carried out with the final setup in order to verify or not this behaviour.

It is observed that there is a maximum ΔT which could be translated into an optimum point of operation according to the requirements of a final application of the TCS system. There is a trade-off between design parameters (reactor), amount of zeolite and content of water available in the air flow. Here, only first order relations have been studied (factor: bed height; response: ΔT), but second order studies or non dimensional analysis will be of interest.

4.3 Particle size

A series of experiments were done working with two zeolite bead sizes: 1.6-2.5 mm range, and 2.5-5 mm range. All other conditions remain fixed. As shown in Fig. 8, the trend of the outlet temperature and the duration of the stage are almost equal for both experiments.
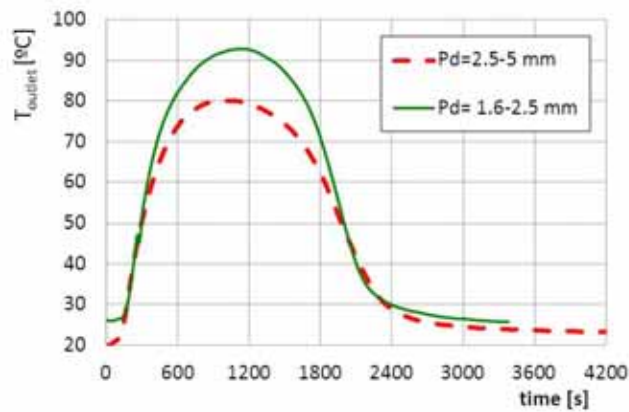

Fig. 8: Evolution of outlet temperature for different particle size

Tab.3: Results in experiments with different particle diameter

Particle diameter [mm]	1.6 -2.5	2.5- 5
Maximum T_{outlet} [ºC]	92.86	80.08
Maximum ΔT [ºC]	66.37	55.33
Amount of adsorbed water [g]	4.506	4.327

The temperature difference (ΔT), as well as the maximum temperature reached, is higher when working with smaller particles (Table 3). As the particle diameter decreases, the potential for adsorption of the bed increases because the external mass transfer resistance (found at the external surface of the zeolite bead) is reduced as the total external surface is increased.

4.4. Determining the exchanged energy

In order to check how far we are from the theoretical limits we compared the energy exchanged during the adsorption process according to equations 3 (almost theoretical) and 4 (from experimental results). Table 4 shows the results. For such a comparison, an error analysis has been carried out to determine the experimental error associated to indirect measurement of the total exchanged energy. Uncertainties propagation has been accomplished in accordance to the GUM guide (2008). Table 5 compiles measuring equipment errors and equations 5 and 6 state the functions used to estimate air properties depending on average air temperature inside the reactor.

- Theoretical- experimental: $E_{Theo} = \Delta H_{adsorption} \cdot m_{adsorbed}$ (eq. 3)

- Indirect- experimental: $E_{Ind-Exp} = \sum \dot{v}_{air} \cdot \rho_{air} \cdot c_{p,air} \cdot \Delta T \cdot \Delta t$ (eq. 4)

Tab. 4: Comparative chart of total energy exchanged theoretical-experimental and indirect-experimental

	Bed height [cm]	15	10.5	7	3.5
$\dot{v}$= 4. 4 L min^{-1}	E$_{THEO}$ [kJ]	36.2	24.7	17.4	8.6
	E$_{IND-EXP}$ [kJ]	20.7±0.488	15.9±0.375	12.0±0.283	6.5±0.154
	Difference	43%	36%	31%	24%
$\dot{v}$= 6. 6 L min^{-1}	E$_{THEO}$ [kJ]	33.8	24.9	17.5	8.5
	E$_{IND-EXP}$ [kJ]	25.2±0.433	21.2±0.365	14.2±0.244	7.6±0.130
	Difference	25%	15%	19%	11%
$\dot{v}$=8.8 L min^{-1}	E$_{THEO}$ [kJ]	34.3	23.7	16.8	8.6
	E$_{IND-EXP}$ [kJ]	26.5±0.369	19.2±0.267	15.0±0.208	7.8±0.108
	Difference	22%	19%	11%	9%

Tab. 5: Errors derived from measuring equipment and functions

Variable / Parameter	$m_{adsorbed}$	$\dot{v}_{air}$	ρ_{air}	$c_{p,air}$	ΔT
Unit	g	L/min	kg/L	J/(kg·K)	K
Measurement / Function	M	M	f$_1$(ΔT)	f$_2$(ΔT)	M
Error	±0.001	±0.1	±0.002	±0.001	±0.48

$$f_1 = \rho_{air} = 1.29733 - 0.00425 \cdot T_{average} \qquad \text{(eq. 5)}$$

$$f_2 = c_{p,air} = \frac{28960 + 9390 \cdot \left(\dfrac{\dfrac{3012}{T_{average}+273.15}}{\sinh\left(\dfrac{3012}{T_{average}+273.15}\right)} \right)^2 + 7580 \cdot \left(\dfrac{\dfrac{1484}{T_{average}+273.15}}{\cosh\left(\dfrac{1484}{T_{average}+273.15}\right)} \right)^2}{28.951} \qquad \text{(eq. 6)}$$

A first approach to estimate heat losses through the reactor walls has been done. Although the glass reactor was designed with a vacuum chamber, there is a clear thermal bridge where the tubes are joint. Therefore, depending on the experiment conditions, heat losses through walls can be noticeable. They have been estimated taking into account natural convection and radiation correlations while measuring reactor external surface temperatures. However, none of them matches the real situation (not isothermal surface neither uniform heat flux) but serve as a first approach. Following equations (7-11) have been used:

$$Ra \equiv g\beta(T_s - T_\infty)L^3/(va) \qquad \text{(eq. 7)}$$
$$\overline{Nu} \equiv \bar{h} \cdot L/\lambda \qquad \text{(eq. 8)}$$
$$\overline{Nu} = 0.59 Ra_L^{1/4} \qquad \text{(eq. 9)}$$
$$q_{nat.conv} = A \cdot \bar{h}(T_s - T_\infty) \qquad \text{(eq. 10)}$$
$$q_{rad} = \varepsilon \cdot A \cdot \sigma \cdot (T_s^4 - T_\infty^4) \qquad \text{(eq. 11)}$$

Heat losses through the reactor walls have been estimated at every time as the addition equations 10 and 11 while measuring external wall temperatures: for the whole set of experiments in the range of 22% up to 38% of total energy exchanged. This means that although a vacuum chamber is provided, better reactor design and/or materials to take full advantage of the system thermal capacity is necessary.

For a fixed flow rate:
The two values of exchanged energy decrease with bed height reduction. If mass of zeolite is smaller, the total amount of water that can be adsorbed is smaller as well. Thereby, the heat released and the stage duration decrease. If the process is faster, certain heat losses are lower. Therefore, the difference between the energy values determined by equations 3 and 4 decreases.

For a fixed bed length:

The theoretical stored energy remains almost constant (obviously).. However, the indirect experimental energy increases as flow does.. By increasing the flow rate the process is faster, favouring reduction of heat losses through the reactor walls. Therefore, the difference between the two energy values is smaller.These differences are caused mainly by the heat losses through the reactor walls. In addition, there are errors associated to the measurement devices and there is another contribution due to the adsorption enthalpy datum for NaY zeolite (taken from Whiting et al, 2013).

Therefore next actions will be: repetition of tests with the second experimental setup (fine measuring and control), get the value of enthalpy of adsorption of NaY zeolite, and reduce heat losses in the reactor (thermal insulation, selection of other reactor materials…).

Finally, as a brief remark on pressure drop: pressure difference has been measured coarsely between the inlet and the outlet of the TCS reactor. A baseline has been established for the empty reactor and then the pressure drop has been measured for different bed lengths. Fig. 90 shows the baseline and a pressure drop evolution for a specific test (1.6-2.0 mm particle size; 8.8 L/min; 7.5 cm height).
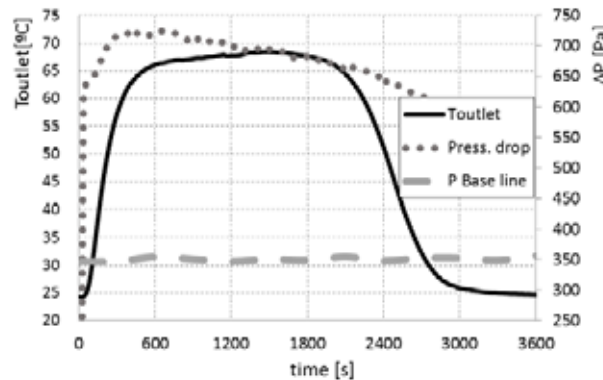

Figure 9. Pressure drop for the tested reactor (arbitrary conditions) and baseline

Initial tests remark the importance not only of pressure drop fine measurements (as this is closely link to the electrical consumption of the auxiliary systems to provide an airflow to the TCS system in the corresponding application later on), but also on variation of pressure drop while sorption process occurs.

In this paper two experimental setups have been designed, constructed and started up to characterize the thermal behaviour of air-water-zeolite systems for sorption-based TES. The combined installation allows operating with open systems at atmospheric pressure from room temperature up to 200ºC, from 0.08 L/min up to 10, and for any relative humidity. Both stages, adsorption and desorption can be tested. A packed bed cylindrical reactor has been tested using zeolite NaY faujasite. Parametrical studies have been accomplish for air flow, bed height and particle size: given the rank of experimental validity and the specific reactor design, the best results in terms of released heat have been obtained for the highest flow (8.8 litre/min), filling half of the reactor (7 cm bed height) with the smaller NaY zeolite (1.6-2.5 mm). With this conditions, we have reached $\Delta T = 66.37$ °C. Comparing theoretical limits for stored thermal energy and experimental values there can be differences from 9 up to 43% depending on the experiment. The main reason of these differences seems to be on the heat losses through the reactor walls. An improvement to reduce these losses will boost the thermal capacity of the system.

5. Acknowledgement

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