



GHGT-12

## TG-FTIR Measurement of CO<sub>2</sub>-H<sub>2</sub>O co-adsorption for CO<sub>2</sub> air capture sorbent screening

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### Abstract

Capturing atmospheric CO<sub>2</sub> using solid sorbents is gaining interest. As ambient air normally contains much more (up to 100 times) water than CO<sub>2</sub>, a selective sorbent is desirable as co-adsorption will most likely occur. In this study, a convenient method based on an TG-FTIR analysis system is developed and used to characterize sorbents for their water and CO<sub>2</sub> adsorption capacity when exposed to ambient air. The method allows to determine quantitatively the co-adsorbed amounts of CO<sub>2</sub> and water from small sample sizes (10 mg range) and is expected to be a useful instrument in sorbent screening and evaluation.

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### 1. Introduction

CO<sub>2</sub> capture using solid sorbents is seen as a promising alternative to the current state of the art aqueous (amine-based) solvent processes for CO<sub>2</sub> capture and utilization or storage. The advantages of solid sorbents are found in a lower specific heat capacity, higher working capacities and avoidance of water evaporation during regeneration. A significant fraction of the sorbent material research in this field is concentrated on supported amine sorbents. This sorbent class is regarded promising for the high equilibrium capacities obtained, simple regeneration by temperature swing to modest temperatures and fast kinetics performance, which has been tested and confirmed under post-combustion CO<sub>2</sub> capture conditions [1,2].

Also for capturing CO<sub>2</sub> direct from air, solid sorbents are gaining interest. Direct Air Capture (DAC) can be seen as a pathway to compensate the anthropogenic CO<sub>2</sub> emitted from mobile and disperse sources. DAC provides flexibility in the location choice by breaking the link between the locations of the emission sources and capture sites, thereby releasing pressure related with the competition for land use [3]. In developing DAC technology based on solid sorbents, a first step is to identify suitable sorbents. Such sorbents preferably have a high CO<sub>2</sub> equilibrium capacity and show fast kinetics during adsorption at ambient conditions.

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Another issue is the sorbents' tolerance to humidity as ambient air normally contains much more water than CO<sub>2</sub>. At 20°C and 70% RH the molar ratio of water over CO<sub>2</sub> in ambient air is around 30-70 times higher than the ratio found in flue gas for post-combustion CO<sub>2</sub> capture conditions. Hence, a selective sorbent is required as water co-adsorption is likely to occur. Evaluating and screening sorbents for atmospheric CO<sub>2</sub> capture would benefit from a simple analysis method, applicable to small sample sizes, to determine the amounts of CO<sub>2</sub> and water captured.

## 2. Method development

In this study we aim to develop a method based on using a coupled TG-FTIR analysis system to screen sorbents for their water and CO<sub>2</sub> adsorption capacity. Normally, thermo gravimetric analysis (TGA) is used to determine sorbent capacity by measuring the mass increase by passing a gas stream of a non adsorbing gas like N<sub>2</sub>, containing a known amount of a single adsorbing component, here CO<sub>2</sub>. The mass increase is then uniquely related to the amount of CO<sub>2</sub> adsorbed and depends on the temperature and CO<sub>2</sub> concentration in the gas stream passing the sorbent sample. Upon increasing temperature, the sorbent will release CO<sub>2</sub> and the sample mass decreases again. The capacity can then be determined by both the adsorption step as well as the desorption step in the TGA.

For sorbent samples which are loaded in ambient air, the total mass loss during TGA analysis is due to desorption of carbon dioxide, water and possible other co-adsorbed species, as well as the possible loss of sorbent material. In most cases the major fraction of this weight loss will be caused by desorption of water and carbon dioxide. It was anticipated that with an FT-IR gas analysis coupled to the TGA outlet gas stream, this desorption process can be followed qualitatively and could be developed into a quantitative method to determine the separate contribution of CO<sub>2</sub> and water to the total weight loss.

### 2.1. Experimental

For this study a Netzsch STA 449 F3 Jupiter TGA analyzer coupled with a Bruker TGA-IR Tensor 27 is used. According to manufacturer specification, the FTIR cell is kept at 200°C. As sorbent during method development mainly 'Lewatit' (Lewatit, VPOC 1065; LennTech) was used; a macroporous, divinylbenzene crosslinked polymer with benzylamine groups on a polystyrene matrix, purchased as beads of 0.47-0.57 mm with 0.27 cm<sup>3</sup>/g pore volume and 25 nm pore diameter. Other sorbent materials tested include zeolite 13X (Sigma-Aldrich), PEI (Sigma-Aldrich) on styrene-divinylbenzene support (Sigma-Aldrich), K<sub>2</sub>CO<sub>3</sub>, Na<sub>2</sub>CO<sub>3</sub>, CaCO<sub>3</sub> and activated carbon (Sigma-Aldrich). For calibration purposes both N<sub>2</sub> (99.999% pure) and CO<sub>2</sub> (99.998% purity, Praxair) and a calibrated gas mixture of 20.9 vol% O<sub>2</sub>, 78.9 vol% N<sub>2</sub> and 2020 ppm CO<sub>2</sub> (Praxair) was used. Using the pure N<sub>2</sub> sample no mass increase was observed under adsorption conditions, indicating no (or negligible amount of) CO<sub>2</sub> in this gas.

The FTIR spectra of the pure compounds CO<sub>2</sub> and H<sub>2</sub>O show a promising peak separation (Figure 1). The highest sensitivity for CO<sub>2</sub> was found at a wavenumber of 2360 cm<sup>-1</sup> and for H<sub>2</sub>O at 1510 cm<sup>-1</sup>. To check whether using a single wavenumber for peak integration is sufficient as measure for the total amount of CO<sub>2</sub> released, both the integration of the signal at 2360 cm<sup>-1</sup> over time (here called: "2D" integration) as well as integration of the complete CO<sub>2</sub> peak signal over the wavenumber range of 2227-2397 cm<sup>-1</sup> ("3D" integration) and over time were used.

In the analysis it is assumed that the measured wavenumber  $\nu$  dependent absorbance ( $A_\nu$ ) measured is proportional to the CO<sub>2</sub> concentration ( $c$ ), the path length ( $d$ ) and the CO<sub>2</sub> specific molar extinction coefficient ( $\epsilon_\nu$ ), according to Lambert-Beer's law, for both single wavenumber (as in Eq. 1a) as for integration over a certain wavenumber range (see Eq. 1b).

$$\text{Single wavenumber:} \quad A_\nu = c \cdot d \cdot \epsilon_\nu = K c \quad (1a)$$

$$\text{Wavenumber range:} \quad \int_{\nu_1}^{\nu_2} A_\nu d\nu = c \int_{\nu_1}^{\nu_2} d \cdot \epsilon_\nu d\nu = K_{\nu 12} c \quad (1b)$$

To determine the total molar amount  $n$  released by the sample, an integration of the concentration dependent response over time  $I_t$ , and for the wavenumber range method also over the selected wavenumber range  $I_{t,\nu}$  is needed, taking into account the flow rate applied:

$$n = F \cdot \int_{t_1}^{t_2} c dt = F \cdot \int_{t_1}^{t_2} \frac{A_\nu}{K} dt = \frac{F}{K} \cdot \int_{t_1}^{t_2} A_\nu dt = \frac{F}{K} \cdot I_t \quad (2a)$$

$$n = F \cdot \int_{t_1}^{t_2} c \, dt = F \cdot \int_{t_1}^{t_2} \left( \frac{\int_{\nu_1}^{\nu_2} A_{\nu} \, d\nu}{K_{\nu 12}} \right) dt = \frac{F}{K_{\nu 12}} \cdot I_{t,\nu} \quad (2b)$$

As sample heating in a TG apparatus is not instantaneous, the CO<sub>2</sub> desorbed from a sorbent sample is not released instantaneously and hence, the FTIR signal must be integrated over a longer period of time covering the whole desorption process. Therefore, a high resolution is needed to be able to integrate the CO<sub>2</sub> related signal. Sample heating rate was varied between 1 and 20 K/min, increasing sample temperature to 130°C (for supported amine sorbents), at which it was kept for five more minutes. The sample heating rate and purge gas flow rate can be varied to optimize the peak (area or volume) integration. For detecting CO<sub>2</sub>, a heating rate of 5 K/min was found to give good results in view of creating a smooth response curve with good signal/noise ratio suitable for integration purposes.

For water the desorption process and signal integration procedure is in principle similar to that of CO<sub>2</sub>, but the response signal is less smooth in comparison with CO<sub>2</sub> (see Figure 1a), making integration over a range of wavenumbers less accurate. Alternatively, the amount of water adsorbed can be estimated by the difference between the total weight loss (as measured via the TGA) and the recorded CO<sub>2</sub> signal, when the loss of active compounds (“amine” in equation (1)) and other compounds or support material (“other”) is negligible.

$$\Delta m = \Delta m_{H_2O} + \Delta m_{CO_2} + \Delta m_{amine} + \Delta m_{other} \quad (3)$$

For the supported amine sorbents studied in this work it was found that at a temperature as high as 150°C, the mass loss due to the sorbent (support + amine) itself was less than 0.5%/h. Hence, considering the temperature profile applied, this effect is indeed marginal.

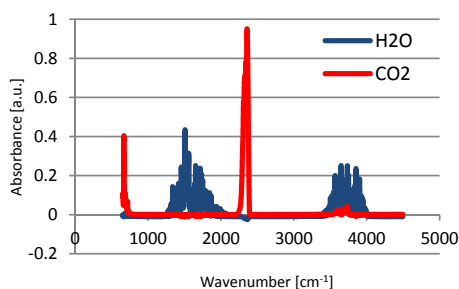


Figure 1a. FTIR spectra of CO<sub>2</sub> and H<sub>2</sub>O

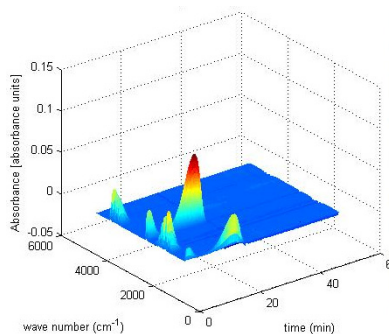


Figure 1b. FTIR response signal for CO<sub>2</sub> during sample desorption

## 2.2. Calibration

For calibration of the FTIR response different approaches were followed. In the first one, a pulse based calibration, block pulses of CO<sub>2</sub>-rich gas (with N<sub>2</sub> as balance gas) with defined flow rate, duration and CO<sub>2</sub> concentration were used (see Figure 2a), which were fed via the TGA to the FTIR. The CO<sub>2</sub> pulses were created from mixing pure CO<sub>2</sub> (quality 4.8; Praxair) and pure N<sub>2</sub> (quality 5.0) gas streams in the TGA. It can be seen in Figure 2a that the peaks show a bit of tailing, most likely caused by mixing issues in the TGA chamber or injection system and this is characteristic for this type of measurements [4].

The 2D and 3D integrated responses  $I_t$  and  $I_{t,\nu}$  were correlated to the total CO<sub>2</sub> amount fed to the system and especially for the 2D (single wavenumber) integration a very good linear dependency was found, see Figure 2b. For a typical analysis of a 10-20 mg sample, the peak height and amount of CO<sub>2</sub> released (around 0.02 mmol) are, however, significant smaller than the range covered in Figure 2b. To check reproducibility and sensitivity towards TGA temperature, 4×3 pulses of 1 ml/min for 1 min duration of pure CO<sub>2</sub> were analysed. Series A4 was done at a TGA temperature of 100°C, series A1,A2 and A3 at 30°C. The results presented in Figure 2c show a good reproducibility with average peak area  $I_t$  of 3.18 and a standard deviation of 0.03 (0.9%).

A further check using calcium carbonate samples was performed. During decomposition of CaCO<sub>3</sub>, CO<sub>2</sub> is the only product found, hence the total mass loss can directly be compared with the integrated peak area. The results presented in Figure 2d show indeed a good comparison between CO<sub>2</sub> measured as mass loss in the TGA sample and the amount of CO<sub>2</sub> as determined by peak

integration. The difference with the ‘theoretical’ value (assuming that the sample weight is pure CaCO<sub>3</sub>) is less relevant and may be due to sample impurity or incomplete decomposition.

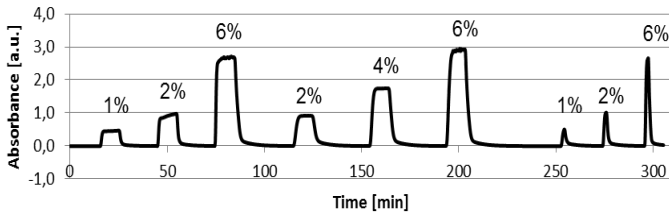


Figure 2a. Measured pulse signals at  $\nu=2360\text{ cm}^{-1}$ . Peaks 1-3 at 100 ml/min for 10 min; peaks 4-6: 50 ml/min, 10 min; peaks 7-9: 100 ml/min, 2 min; The labels represent the CO<sub>2</sub> concentrations in the gas pulses.

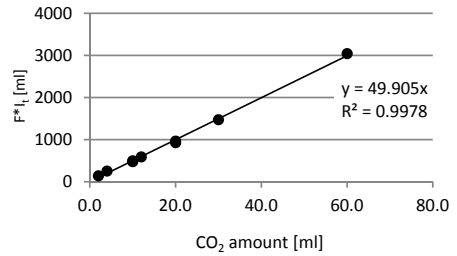


Figure 2b. Calibration using gas pulses from Fig. 2a

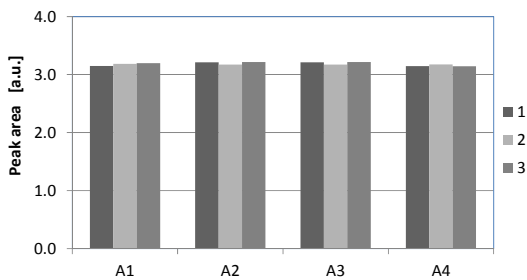


Figure 2c. Reproducibility of integrated gas pulse peak areas

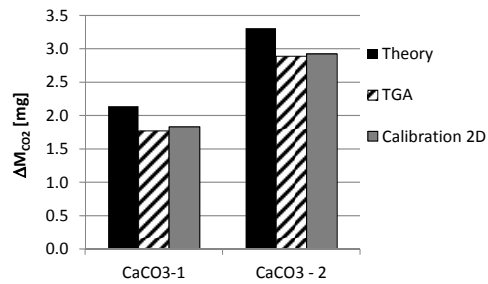


Figure 2d. Testing calibration with CaCO<sub>3</sub> samples

To calibrate and further validate the method at low amounts of CO<sub>2</sub> and at low concentrations, a calibration curve is made using a gas cylinder with diluted CO<sub>2</sub> instead of pure CO<sub>2</sub>. In this case a cylinder with a calibration mixture (20.9 vol% O<sub>2</sub>, 78.9 vol% N<sub>2</sub> and 2020 ppm CO<sub>2</sub>) was used to repeat the calibration curve measurement in the case of low amount of CO<sub>2</sub>. The pulses are taken at a total gas flow of 100 ml/min and calibration-gas pulses in ascending order of respectively 5, 10, 30 and 40 ml/min are given. The duration for each pulse is 30 min and all pulses are injected during one TG-FTIR run at a constant temperature of 30°C as shown in Figure 3. From this Figure, it is clear that the irregular shapes of the block pulses are more pronounced at these concentration levels and therefore less suited for calibration purposes as the concentration decreases.

With a separate CO<sub>2</sub> analyser (LI-COR 840A) temporarily connected to the outlet of the FTIR, the irregular response obtained was confirmed to be real. From the results, comparing Figures 3a and 3b, it was concluded that the preparation of the block-pulses was not ideal, but that the recorded signal from the FTIR combination is trustworthy. The response curves for the TG-FTIR combination and the dedicated LI-840A analyser show identical patterns.

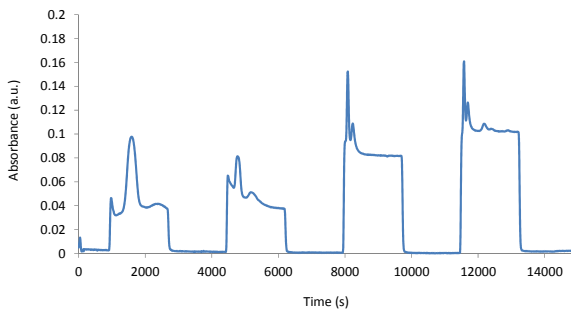


Figure 3a. Pulse signal recorded at  $\nu = 2360\text{ cm}^{-1}$ , the targeted CO<sub>2</sub> concentrations are resp. 100, 200, 600 and 800 ppm

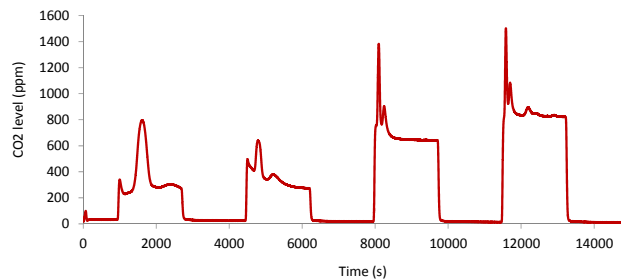
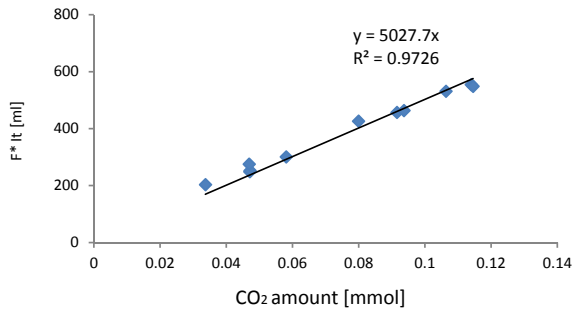
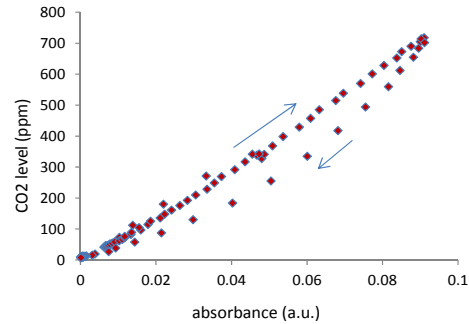


Figure 3b. CO<sub>2</sub> concentration reading in outlet gas of TG-FTIR using a downstream LI-840A CO<sub>2</sub> analyzer

Figure 4a. Example of pulse calibration at low amounts of CO<sub>2</sub>;Figure 4b. Testing proportionality between ppm CO<sub>2</sub> measured and FTIR absorbance during sample analysis

For three calibration series of pulses, similar to the one shown in Figure 3a, the calibration factor  $K$  was determined to be  $5.0 \cdot 10^3$  l/mol with a standard deviation of 4%. At even lower concentrations, the responses of the FTIR signal from the TG-FTIR combination and that from the calibrated CO<sub>2</sub> analyser could be compared directly during an actual sample analysis run. The measured ppm CO<sub>2</sub> signal and the FTIR signal show a near linear dependency in the range of interest, see Figure 4b where data of both analysers during analysis of a sorbent sample are plotted versus each other. The deviating data points, lower curved branch, are due to small time differences, as the CO<sub>2</sub> analyser is downstream the FTIR apparatus and the concentration is rapidly changing in that regime.

When using the same calibration method for water adsorption, the  $K$  factor of water can be calculated by collecting the signal at the wavenumber of  $1510 \text{ cm}^{-1}$ . Unlike CO<sub>2</sub> calibration measurements, now the samples put into the crucibles consist of fresh demi water. Hence, from the mass loss, the molar amount of water was known. Figure 5a displays the spectrum at wavenumber  $1510 \text{ cm}^{-1}$  for a series of five tests. Each curve shows two peaks in the whole time range. The first peak may originate from the humidity in the surrounding air, introduced when opening the furnace for placing the sample. The second peak was caused by the water evaporation with increasing temperature of the sample. With this, the calibration factor of water was correlated to be 164 l/mol with a standard deviation of 4.5%, as shown in Figure 5b.

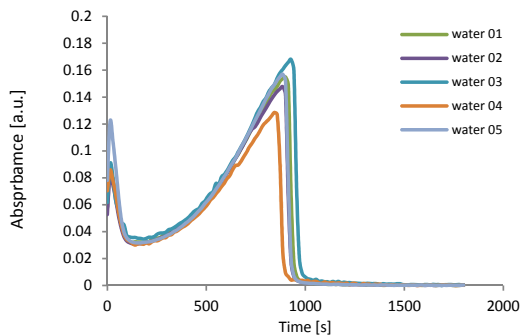
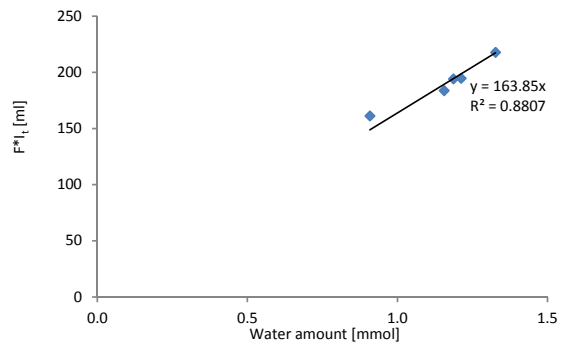
Figure 5a Spectrum at  $\nu = 1510 \text{ cm}^{-1}$  for the water factor calibration tests

Figure 5b Calibration of water factor from results showed in Figure 5a

### 2.3. Working with solid sorbent samples

When testing actual sorbent samples for CO<sub>2</sub> air capture, handling of sorbent samples is a point of attention. In the experiments in this work, sorbents are put into several, identical containers and exposed to the atmosphere in the lab. Prior to this, the sorbents have been desorbed by heating in an oven ( $105^\circ\text{C}$  for 45min) and cooled down in a flow of CO<sub>2</sub>-free nitrogen. The samples are exposed to air overnight for approximately 15 hours in total at ambient conditions. From earlier TGA experiments at low CO<sub>2</sub> concentrations, it seems that this duration is sufficient to approach equilibrium [5]. In the morning, all containers are covered with a lid to stop further sorbent loading. Subsequently, each sample is measured using the TG-FTIR apparatus and method described above.

Upon analyzing a typical sample the sorbent must be removed from the container and brought into the TGA apparatus. For this, the TGA must be opened to place the sample and closed again to start the measurement. During this handling, which can take a few minutes, ambient air can enter the TG-FTIR combination and may disturb the signal. Figure 6a illustrates a typical

recorded signal for two runs with different sorbents (Lewatit and PEI600). In Figure 6b once more the consistency between the TGA/FTIR result and an extra CO<sub>2</sub> analyser is shown. In the first few minutes of the run, the first peak detected is due to enclosed air during opening and closing the TGA machine, as evidenced by comparison with the “blank – 0 min” run, where a run was started without a sample and without having to open and close the TGA. In the “blank-5min” run, again without actual sample but now opening the TGA for 5 min and then closing it, the machine gave a similar first peak, illustrating the effect of this opening and closing of the machine. It was decided to standardize this period to exact 5 minutes for every series and to do a “blank 5 min” run prior to every measurement series for correction.

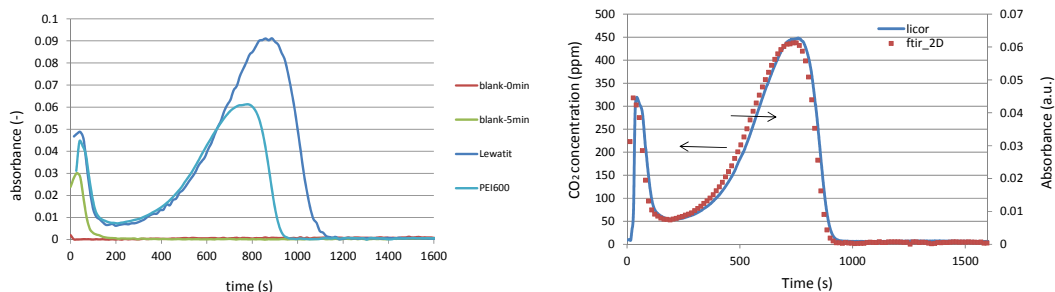


Figure 6a. Example of sorbent analysis and effect of TGA open/close Figure 6b. TG-FTIR and CO<sub>2</sub> analyser signal for PEI600 sorbent

### 3. Sorbent screening

Sorbent screening was done at ambient conditions in the lab, allowing for adsorption for 15 hours before analysis into the TG-FTIR. For analysis in the TGA/FTIR, it was chosen to quickly heat up to 130 °C and cool down as fast as possible to minimize time between analysis of the sorbent samples. The entire desorption procedure was designed to last 30 min and a desorption heating rate of 5 K/min was selected. The flow rate of the purge gas was selected to be 40 ml/min. In the series used for Figure 10, the time for lifting and closing the TGA furnace was not the same for all samples and for the comparative data analysis it was decided to integrate the FTIR signal neglecting the first three minutes. In all other series, a blank run was done with standardized open/close time of the machine and the integrated response (for the first five minutes) was subtracted from the results obtained for subsequent analysed samples.

#### 3.1. Reproducibility

To be able to compare different sorbents next to each other, it must be clear what is the reliability of evaluating a single or duplo sorbent sample. Therefore, for two of the most promising sorbents a reproducibility test is performed. The experiment is repeated 12 times for Lewatit and 11 times for PEI600 and the results are reported in Figure 7. In this Figure, the CO<sub>2</sub> capacities are determined by integrating the FTIR signal as described above. The water capacity is determined from the difference between total sample mass loss and mass loss due to CO<sub>2</sub> desorption. For Lewatit, the average CO<sub>2</sub> capacity found is 1.46 (±0.12) mole/kg and for water this is 5.75 (±0.71) mole/kg, whereas for PEI600, the capacity of CO<sub>2</sub> and water are respectively 1.43 (±0.15) mole/kg and 9.33 (±0.76) mol/kg. It should be noted that not all samples were measured on the same day. In Figure 7a, samples nr. 2 – 6 were adsorbing in parallel and in Figure 7b, samples nr. 3 – 7 were adsorbing in parallel. The standard deviation in the CO<sub>2</sub> capacities between those samples (for Lewatit this is 4% and for PEI600 6%) is around 5%. In short, these results show that the capacities thus determined are reasonably reproducible, with a standard deviation of less than 10%.

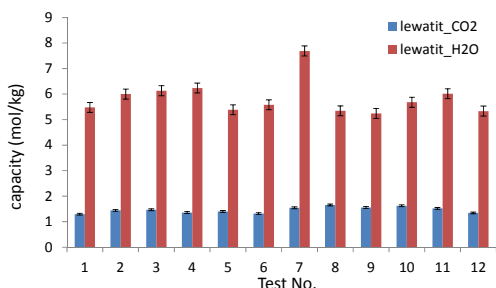


Figure 7a. Reproducibility test using Lewatit sorbent samples

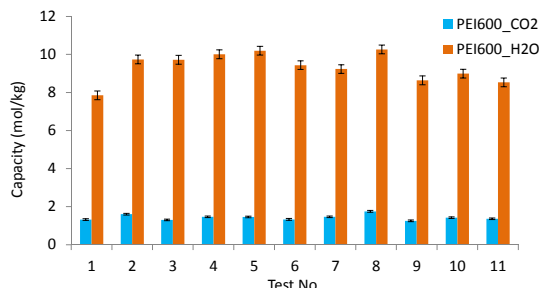


Figure 7b. Reproducibility test using PEI600 sorbent samples

Furthermore, it can be noticed that the capacity of Lewatit and PEI600 for CO<sub>2</sub> is almost the same, but the water capacity (in Figures 7a and 7b determined by difference between total mass loss and the mass loss assigned to CO<sub>2</sub> desorption, from integrating the CO<sub>2</sub> signal) of Lewatit is some 40% smaller than for the PEI600's. Since a higher amount of co-adsorbed water may negatively affect the energy consumption in the regeneration process, Lewatit is to be preferred over PEI600 in this regard.

The capacity for water and CO<sub>2</sub> can also be estimated by applying the factor obtained from the water calibration tests presented in Figure 5. As can be seen in the Figure 8, applying this factor to two Lewatit measurements conducted as duplo, the results by using the K factor for water (K<sub>H2O</sub>) always overestimates the water capacity as determined from total mass loss and using K<sub>CO2</sub>. This deviation can be as high as 40% (sample nr.2) and results in a negative value for the resulting CO<sub>2</sub> capacity, when calculated by difference from total mass loss and mass loss associated with the water desorption as determined by integrating the water signal. Integration of the water signal is therefore considered to be less reliable.

In another test, the initial sample heating rate was reduced in the range from 30°C to 50°C to 1K/min and was kept at 5K/min from 50°C onwards (see Figure 9). There is clearly mass reduction in the time range from 4 min to 30 min, to be attributed mainly to water desorption, whereas there was hardly any peak recognized in water absorption range during this time period. This is related to the fact that the water signal in the absorption spectrum is more complicated than that for CO<sub>2</sub>. As the water molecule may vibrate in a larger number of ways [6] including combinations of symmetric stretch, asymmetric stretch and bending of the covalent bonds. Using a single wavenumber is apparently not good enough for reliable determination of the amount of water.

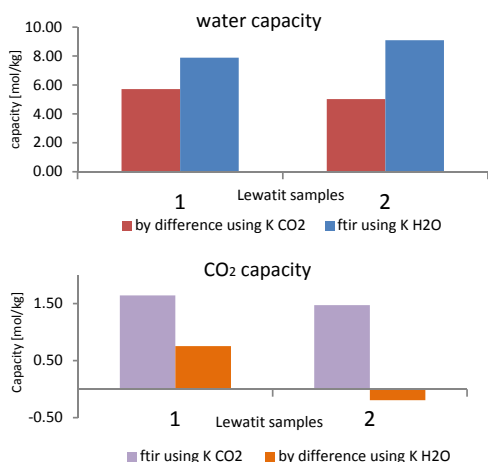


Figure 8 Calculated water (above) and CO<sub>2</sub> (below) capacity by using K factor of CO<sub>2</sub> and water

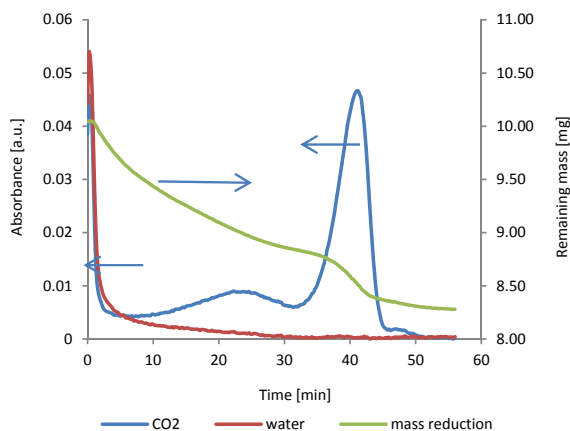
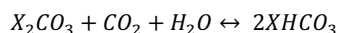


Figure 9 Spectra signal at  $\nu=2360\text{ cm}^{-1}$  and  $\nu=1510\text{ cm}^{-1}$  and mass reduction over time

### 3.2. Sorbent screening

The capacity and selectivity (CO<sub>2</sub> vs. H<sub>2</sub>O) of different types of sorbents is measured at ambient conditions. Basically, the sorbents tested can be categorized, according to their properties, into physical sorbents, dry carbonates and supported amine sorbents. The physical sorbents evaluated are zeolite 13X and activated carbon (AC). 13X is a well-known zeolite, which enjoys the highest capacity among zeolite sorbents. Activated carbon (AC) is a common material for various adsorption processes and possesses small, low-volume pores and a high internal surface area available for adsorption. In these physical sorbents the CO<sub>2</sub> is not chemically bonded, leading to relatively low energies and –temperatures for regeneration. However, the adsorption capacities obtained for DAC under ambient conditions are not very promising, see Figure 10. To what extent the co-adsorbed water was negatively affecting the CO<sub>2</sub> capacity was beyond the scope of this study.

In the dry carbonate category [7,8], Na<sub>2</sub>CO<sub>3</sub> and K<sub>2</sub>CO<sub>3</sub> are investigated at ambient conditions, as operating at more common temperatures for CO<sub>2</sub> adsorption with (supported) carbonates (60-90°C) is considered to be unattractive in view of the enormous heat demand required. For carbonates, the presence of water in air is beneficial as it is needed in the bicarbonate formation.



However, from the results in Figure 10, it is clear that (unsupported) dry carbonates, as Na<sub>2</sub>CO<sub>3</sub> and K<sub>2</sub>CO<sub>3</sub> do not provide advantages in CO<sub>2</sub> absorption capacity at ambient conditions. Additionally, for potassium carbonate the high water adsorption



capacity is an issue, both energy-wise as with respect to solids handling, as the  $K_2CO_3$  sorbents used became very sticky after 15 hours of exposure to air. This high affinity of (unsupported)  $K_2CO_3$  to water makes it a less suitable candidate as sorbent for  $CO_2$  air capture.

Lewatit, PEI 600 and PEI 10k are tested in the group of hybrid sorbents. Lewatit is a commercial sorbent; a macro porous, divinyl benzene cross linked polymer with benzylamine groups on a polystyrene matrix. PEI 600 and PEI 10k are supported amine sorbents prepared by impregnation of polyethyleneimine polymers (PEI) into a porous material with a high thermal stability and high internal surface area (Diaion™, HP-20 a styrene-divinylbenzene based support). For the impregnation, the PEI was dissolved in a volatile solvent, here: methanol. The support material was submerged in this solution and subsequently the volatile solvent was evaporated, leaving behind the PEI impregnated on the internal surface of the porous support material. The number behind PEI such as 600 and 10k represents the average molar weight of the PEI. Compared to the physi-sorbents and dry carbonate sorbents studied, these supported amine sorbents show a high capacity at ambient conditions and during the same time of adsorption (15h). The class of supported amine sorbents seems most promising for  $CO_2$  air capture, among the options studied.

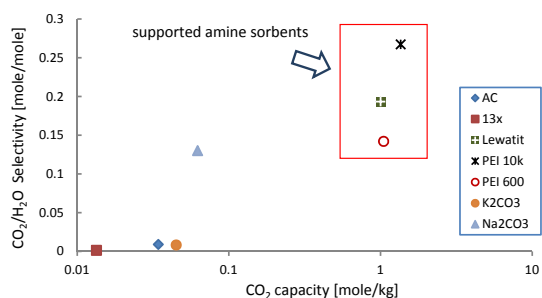


Figure 10. Screening sorbents for direct air capture at ambient (lab) conditions for 15h

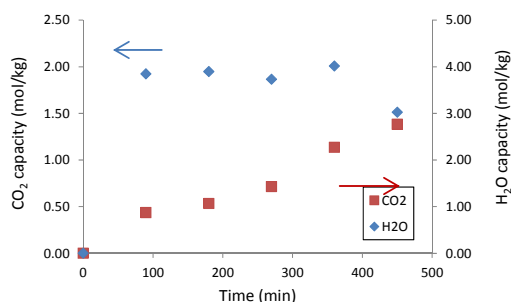


Figure 11. Lewatit sorbent loading vs. time

In Figure 11, the  $CO_2$  and  $H_2O$  loading versus adsorption time is plotted for the Lewatit sorbent. Figure 11 shows that the sorbent is quickly saturated with water vapor, while the  $CO_2$  adsorption requires a much longer time span. It also shows that possibly the maximum capacity for  $CO_2$  is not yet reached and hence, the results presented in Figure 10 are not necessarily the maximum (equilibrium based) capacities. Hence, the method developed in this work seems not only useful for comparative screening of sorbent performance under identical ambient conditions, but also enables to study these transient effects.

#### 4. Conclusions

A simple method was developed, utilizing a TG-FTIR apparatus, to determine quantitatively the amount of  $CO_2$  and  $H_2O$  co-adsorbed from ambient air on small sorbent samples (10-20 mg). Using a gas pulse-based calibration, integrating the absorption spectrum at single wavenumber ( $2360\text{ cm}^{-1}$ ) over time, the  $CO_2$  capacity can be determined with around 5% accuracy. Integrating over more wavenumbers did not improve accuracy. Whereas for  $CO_2$  this method is successful, for water the capacity is best determined by difference from the TGA mass loss and the determined  $CO_2$  capacity. The method seems especially useful for comparative screening of different sorbents, subjected to the same adsorption conditions.

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