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Ultrafast screening of commercial sorbent materials for VOC adsorption using real-time FTIR spectroscopy

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2 real-time FTIR spectroscopy

3 Natan Blommaerts,[1] Fons Dingenen,[1] Vesna Middelkoop,[2] Jan Savelkouls,[3] Marcel Goemans,[3]

4 Tom Tytgat,[1] Sammy W. Verbruggen,[1,*] Silvia Lenaerts[1,*]

5 [1] Sustainable Energy, Air & Water Technology (DuEL), Department of Bioscience Engineering, University of Antwerp,
6 Groenenborgerlaan 171, 2020 Antwerp, Belgium

7 [2] Flemish Institute for Technological Research (VITO), Boeretang 200, 2400 Mol, Belgium

8 [3] Europem, Duwijckstraat 17, 2500 Lier, Belgium

9 * Sammy.Verbruggen@uantwerp.be ; silvia.lenaerts@uantwerp.be; Groenenborgerlaan 171, 2020 Antwerp, Belgium

10 Abstract

11 Recovery of valuable volatile organic compounds (VOCs) from waste streams is of great industrial

12 importance. Adsorption on zeolites offers an economically and environmentally friendly alternative to

13 conventional activated carbon. When evaluating the suitability of a given zeolite for a particular

14 adsorption application, its adsorption capacity has to be determined. This is traditionally achieved

15 using gas chromatography as an analysis tool, yielding only a few discrete sampling points that

16 constitute the adsorption profile. Meanwhile, only low flow rates and low concentrations of volatile

17 organics can be used, rendering the procedure troublesome and time consuming. Herein, we propose

18 a tool for the fast screening of a large amount of zeolites using on-line and *quasi* real-time Fourier

19 Transform Infrared Spectroscopy (FTIR). The technique was used to determine the adsorption capacity

20 of three different commercial zeolites and two silica gels, for five industrially relevant VOCs: acetone;

21 methanol; isohexane; isopentane; and toluene. A series of rapid measurements of the individual

22 adsorption capacities were carried out to obtain a detailed overview of the versatility of the proposed

23 method for the characterization of multi-component and multi-sorption bed systems.

24 Keywords

25 Zeolite, Silica gel, Adsorption, VOC, FTIR, Air purification, fast screening

1. Introduction

Volatile organic compounds (VOCs) are an important class of air pollutants, especially in industrial environments.[1–3] The removal of these VOCs is crucial in view of air pollution control. Some important destructive methods that are commonly used are incineration[4,5], biofiltration[6–8], and advanced oxidation processes (AOPs) such as photocatalysis[9–13]. As an alternative, recuperative, non-destructive methods, such as condensation[4], scrubbing[14] or adsorption[15] are gaining in popularity due to a wealth of potential applications that these recovered compounds offer. For instance, recovered VOCs with low vapor pressure can be used as an alternative fuel source. The adsorption of VOCs on highly porous solids is an economically interesting technology since large amounts of organic molecules can be stored in small volumes of solid material, without high pressure requirements. Activated carbon (AC) is one of the most extensively used VOC adsorbents due to its relatively low-cost production.[16–18] 25 years ago, zeolites were identified as potential adsorbent materials for the removal of VOCs as abatement systems.[19] At present, extensive reports on zeolite adsorption technology are readily available.[20–23] Zeolites offer some important advantages over common AC. Their greatest advantage is the improved safety of zeolites as compared to AC. When heated above 120°C, there is a potential risk of fire ignition in the AC beds. In contrast, zeolites are capable of withstanding very high temperatures due to their inorganic nature. Furthermore, polymerization or oxidation of some compounds can be catalyzed due to the presence of inherent impurities in AC leading to the formation of potentially hazardous by-products. Regeneration is also much more difficult for AC. Once the surface of AC is saturated, the material is commonly discarded and replaced.[24] The final major advantage of zeolites is the fact that the adsorption capacity remains high even at high relative humidity, whereas in the case of AC, the adsorption capacity drops rapidly at a relative humidity of 50 – 60%.[19,25–28] In summary, an ideal adsorbent for VOC removal should have (1) high thermal stability, (2) high adsorption and regeneration capacity, (3) limited catalytic activity, and (4) low cost.

1 The selective adsorption of VOCs is of high industrial relevance for a variety of applications. For

2 instance, concentrated VOCs can be used as an alternative source of fuel. To select the best suited

3 zeolite for selective VOC adsorption, screening the adsorption capacity is required. The most common

4 technique for analysis of VOC adsorption profiles of materials is gas chromatography (GC).[7,15–

5 17,19,29–32] Some important advantages of GC for the detection of VOCs are its ability to measure

6 extremely low concentrations down to ppb levels and its effective high resolution separation of

7 different compounds. A major limitation of GC is that it is highly time consuming. The time between

8 two sampling points is at least 30 seconds and can be as long as a few minutes, depending on the

9 length of the column and the temperature program. As a result, the obtained adsorption profile relies

10 on a few discrete sampling points, taken over a prolonged period of time.

11 In this work an alternative technique is proposed based on Fourier Transform Infrared Spectroscopy

12 (FTIR). This, too, is shown to be a viable tool to perform adsorption measurements since VOCs can be

13 easily detected by FTIR, commonly at higher concentrations than for GC (ppm levels). In contrast to GC

14 detection, FTIR enables on-line and *quasi* real-time detection of gaseous components, making it

15 suitable for ultrafast screening studies. In this work, this is demonstrated by screening the adsorption

16 capacities of three commercial zeolites and two silica gels for five relevant VOCs including acetone,

17 methanol, isohexane, isopentane and toluene. It is, however, not the intention of this work to study

18 adsorption/desorption kinetic mechanisms, nor to select the most suitable adsorbent material or

19 process conditions for a given industrial application. Our aim is only to show that from a practical point

20 of view, FTIR as detection tool provides a much faster yet still very accurate screening of adsorbent

21 materials for VOCs in comparison with conventional GC detection.

22 In view of practical applications of recovered VOCs, desorption of these compounds and reusability of

23 the sorbent are equally important.[33,34] Therefore, as a final proof of concept of the proposed

24 methodology, a complete set of adsorption-desorption cycles has been performed on a multi-
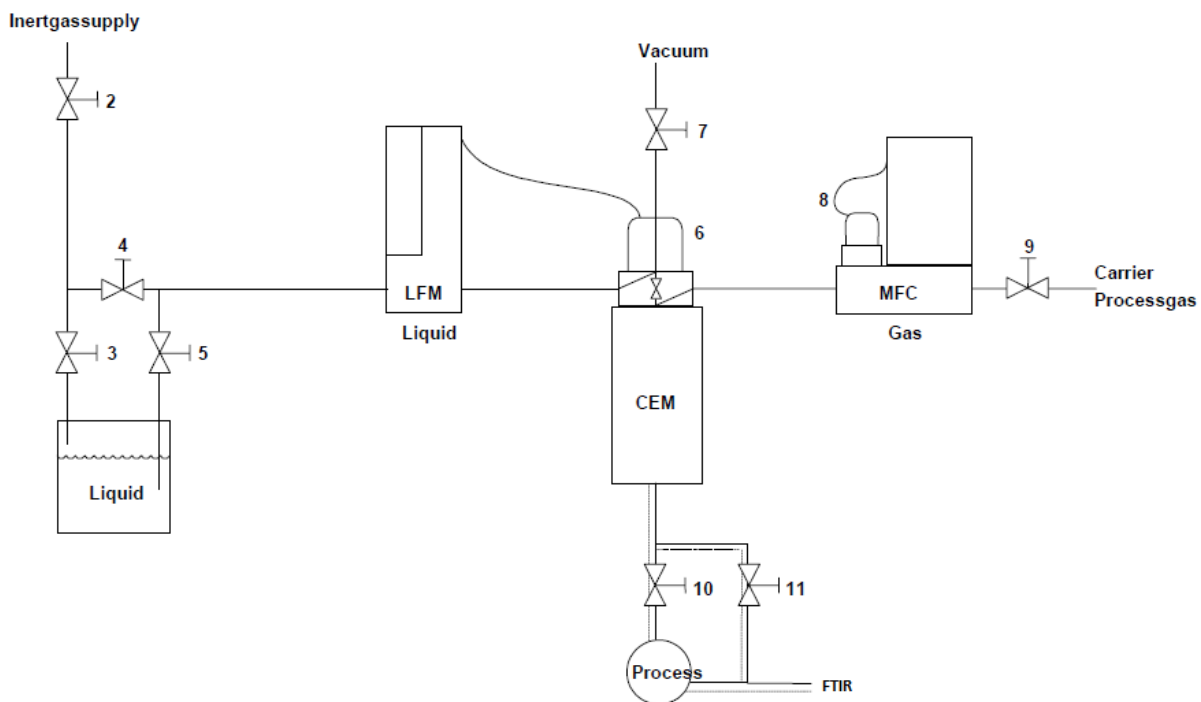
1 component gas flow and a multi-adsorbent bed system, showing the potential of this technique for full

2 characterizeation in terms of the adsorption and regeneration capacities of the adsorbent materials.

2. Experimental

2.1.   Gas test set-up

5 Figure 1 shows a schematic illustration of the gas set-up used in the adsorption experiments. When

6 opening valves 2 and 3 while keeping valves 4 and 5 closed, the N_2 carrier gas supply is sent through a

7 slightly over-pressurized reservoir filled with a liquid organic compound of interest. In this way, there

8 is a pressure build-up leading to the filling of the ducts with liquid up to valve 5. Gradually opening

9 valve 5 leads to the filling up of the liquid flow meter (LFM, mini CORI-FLOW^{TM}, Bronkhorst). This flow

10 meter regulates the amount of liquid VOC flowing to the controlled evaporator and mixer unit (CEM,

11 W-002-121-K, Bronkhorst). In this way, a very precise, known amount of VOC can be fed to the gas

12 stream. In the CEM unit the liquid compound is evaporated and mixed with N_2 carrier gas, of which the

13 flow rate is controlled by a mass flow controller (MFC, F-201CV-10K-RAD-22-V Bronkhorst, unit 8 in

14 Figure 1) and set at 2 L min^{-1}. Using valves 10 and 11, the gas stream can either be sent through the

15 reactor or immediately to the FTIR apparatus in bypass mode.

16

Figure 1. Schematic illustration of the experimental set-up for gas phase VOC adsorption measurements. Numbers 2-5, 7 and 9-11 indicate valves; number 6 indicates the CEM unit and number 8 indicates a gas MFC (reprinted with permission of Gefran/Bronkhorst).

2.2. Sample preparation
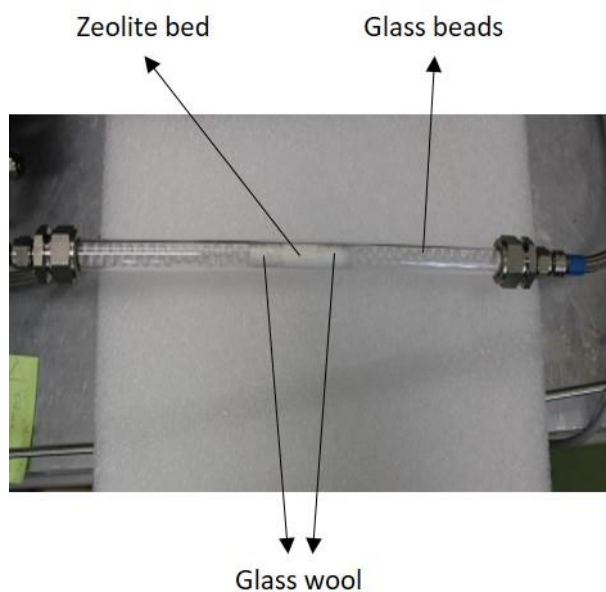
Commercially available ZEOflair zeolite powders (ZEOflair® 100, ZEOflair® 110 and ZEOflair® 300) were purchased from ZEOchem and compacted into disks by applying a pressure higher than 200 MPa, after which the disks were fragmented and sieved to obtain grains of sizes ranging from $300 - 630$ μm. Two commercial silica gels (SYLOBEAD® SG 127 and SYLOBEAD® SG H 127) were purchased from Grace, crushed and sieved to obtain grains in the same size range as the zeolite counterparts. Bead-shaped activated carbon (AC) was obtained from Kureha (A-BAC LP, large particles (0.6 ± 0.05 mm) bead-shaped activated carbon) as reference material. Acetone (VWR chemicals, ≥ 99.8 %), isopentane (VWR chemicals, ≥ 99 %), isohexane (Merck, ≥ 98 %), Methanol (Chemlab, ≥ 99.8 %) and Toluene (Aldrich, ≥ 99.8 %) were purchased in liquid form and used as target VOCs without further purification.

1 Prior to each adsorption experiment, the zeolite pellets or silica beads were activated at 350°C

2 overnight under an inert N_2 flow to ensure the complete removal of adsorbed water and organic

3 components. After cooling, an appropriate amount of sample (Table 1) was weighed and loaded in a

4 glass reactor tube (10 mm in diameter) to obtain a zeolite bed of 20 mm in length. The zeolite bed was

5 restrained in the middle of the tube with glass wool plugs and the remaining void space was filled with

6 4 mm glass beads to reduce the dead volume. Figure 2 shows a picture of the reactor used in the

7 adsorption experiments.

8 **Table 1**. Mass of adsorbent bed (g) for each adsorption experiment.

VOC Adsorbent	Mass of adsorbent bed (g)
Acetone	
ZEOflair® 100	1.191
ZEOflair® 110	1.085
ZEOflair® 300	0.891
SYLOBEAD® SG 127	1.216
SYLOBEAD® SGH 127	1.281
Activated carbon	1.329
Methanol	
ZEOflair® 100	1.203
ZEOflair® 110	1.275
ZEOflair® 300	0.881
SYLOBEAD® SG 127	1.326
SYLOBEAD® SGH 127	1.212
Activated carbon	1.202
Isopentane	
ZEOflair® 100	1.166
ZEOflair® 110	1.003
ZEOflair® 300	1.081
SYLOBEAD® SG 127	1.377
SYLOBEAD® SGH 127	0.743
Activated carbon	1.236
Toluene	
ZEOflair® 100	1.121
ZEOflair® 110	1.001
ZEOflair® 300	1.094
SYLOBEAD® SG 127	1.547
SYLOBEAD® SGH 127	1.747
Activated carbon	1.064
Isohexane	
ZEOflair® 100	0.984
ZEOflair® 110	1.193
ZEOflair® 300	1.196
SYLOBEAD® SG 127	1.546
SYLOBEAD® SGH 127	1.327
Activated carbon	1.055

9

Figure 2. Reactor tube filled with zeolite bed, glass wool and glass beads.

2.3. Characterization techniques

Brunauer-Emmett-Teller (BET) specific surface area was determined using a Micrometrics® Tristar Surface Area and Porosity Analyzer. Prior to each analysis, the samples were degassed overnight at 200°C.

To monitor the adsorption profile of the different VOCs, breakthrough curves were recorded using a Thermo Fisher Scientific Nicolet[TM] 380 FTIR spectrometer, by recording spectra in a wavenumber range of 4000-400 cm^{-1} at a resolution of 1 cm^{-1}. The MacrosBasic software (Thermo Fisher Scientific) was used for on-line monitoring of the peak heights of different characteristic bands of the corresponding VOCs (Table 2).

Table 2. Location of the FTIR bands of the different VOCs used for the online monitoring of the VOC adsorption capacity of

2 commercial zeolite structures.

VOC	wavenumber (cm^{-1})	Vibration mode
Acetone	1223	v_{as} (CCC)
Methanol	1060	ρ (CH$_3$)
Isopentane	1382	δ_s (CH$_3$)
Toluene	2939	v_{as} (CH)
Isohexane	1381	δ_s (CH$_3$)

3 ## 2.4. Adsorption experiment

4 A typical adsorption experiment is carried out in four stages: (1) The entire system is purged with N$_2$-

5 gas to ensure all possible compounds are eliminated from the system and an FTIR background

6 spectrum is recorded under these conditions; (2) Using the LFM-CEM unit described above, an

7 appropriate amount of VOC is fed into the system that results in a gas phase concentration of 0.5 vol%

8 in N$_2$ carrier gas for 30 min to ensure a stable by-pass concentration readout; (3) The gas flow is sent

9 through the reactor to start the adsorption stage, corresponding with a drop in the FTIR absorption

10 time profile, until breakthrough of the VOC is observed; (4) Finally the adsorption capacity can be

11 obtained by integrating the adsorption time profile and subtracting it from the bypass level. Since the

12 flow rate of VOC (g/h) is known exactly, it is straightforward to link this to the integrated value of the

13 breakthrough curves and to calculate the total adsorbed amount. The adsorption capacity is then the

14 adsorbed amount (in moles) divided by the mass of the used adsorbent bed (Table 1).

15 # 3. Results and discussion

16 ## 3.1. BET analysis

17 Typically, zeolite structures display high surface areas in the order of several hundred m^2 g^{-1}. Table 3

18 summarizes the BET surface area for the different zeolites and silica gels used in the adsorption

19 experiments. As expected all the tested zeolites and silica gels have lower BET surface area values

1 compared to AC. The two silica gels have a very similar value that is significantly higher than that of

2 the ZEOflair® zeolites. The latter show significant differences in BET surface area.

3 **Table 3**. BET surface area of different commercial zeolites and silica gels in comparison with activated carbon.

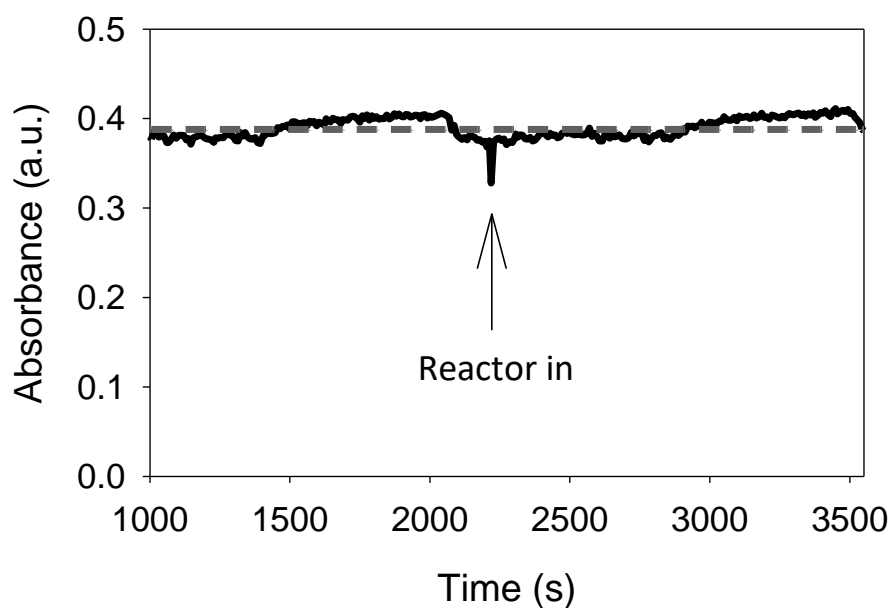| Adsorbent	Type	BET surface area ($m^2\ g^{-1}$)
Zeolite	ZEOflair® 100	253 ± 2 [A]
	ZEOflair® 110	338 ± 4 [A]
	ZEOflair® 300	442 ± 7 [A]
Silica gel	SYLOBEAD® SG 127	692 ± 7 [A]
	SYLOBEAD® SGH 127	649 ± 3 [A]
AC	A-BAC LP	949 [A]
		1100 – 1300 [B]

4 [A] measured value
5 [B] Manufacturer data

3.2. Adsorption experiments

3.2.1. Single component adsorption

8 Prior to the actual adsorption experiments, a blank measurement was performed in the absence of

9 any adsorbent material, only using glass wool plugs and glass beads in order to determine the

10 displacement of dead volume in the reactor tube. Measurements showed that this value was always

11 far below 1% of the adsorbed amount in all experimental cases. Figure 3 shows an example of this

12 blank experiment for acetone. After 2200 s, the gas stream is switched from bypass to the reactor,

13 showing only a very brief, line-shaped and insignificant drop in absorbance. Therefore, the blank

14 measurement can be neglected in all the adsorption capacity calculations.

1

2

Figure 3. Blank measurement with acetone as compound (black solid line). The gray dashed line indicates the bypass level.
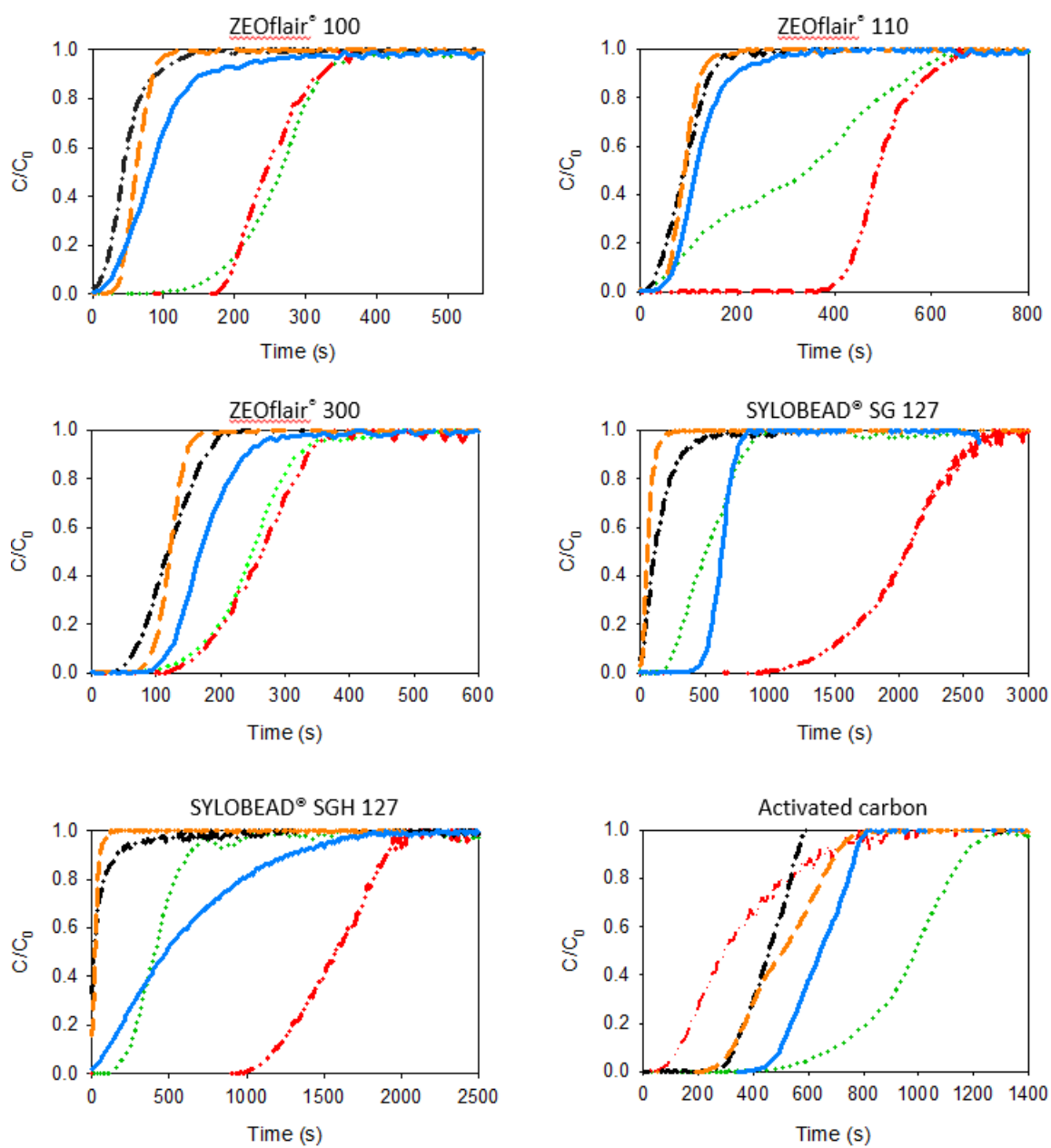
After 2200 s, the gas stream was switched from bypass mode to the reactor.

Figure 4 provides an overview of the experimental breakthrough curves for the different VOCs obtained for each of the commercial zeolites and silica gels. As expected, most of the breakthrough curves are showing a characteristic sigmoidal shape. Based on these curves, the adsorption capacity for each VOC-sorbent combination can be calculated as explained above. The resulting adsorption capacities are summarized in Figure 5. The three ZEOflair® zeolites display similar adsorption characteristics, with ZEOflair® 300 demonstrating the best overall performance, followed by ZEOflair® 110 and ZEOflair® 100. These results can be explained by two main sorbent characteristics: BET surface area and selectivity toward hydrophilic/hydrophobic VOCs. The latter parameter (Table 4) shows that the ZEOflair® zeolites have a similar hydrophobicity. The main parameter determining the difference in adsorption capacity is the BET surface area. As shown in Table 3, the BET surface area can be ranked as follows: ZEOflair® 300 > ZEOflair® 110 > ZEOflair® 100. The same trend can be found in the adsorption capacity for the five different VOCs and confirms the reliability of the method. The two

silica gels clearly have a higher adsorption capacity for the smaller compounds (methanol, acetone, toluene) in comparison with the ZEOflair® zeolites. This can also be explained by the higher BET surface area of the silica gels. The difference in adsorption behavior between the zeolites and the silica gels towards alkanes can be attributed to the stronger hydrophobicity of the zeolites compared to the silica gels. AC shows the largest adsorption capacities except for methanol. Some literature reports already suggested that activated carbon is not a good sorbent for alcohols.[29]

To verify the reliability of the presented ultrafast screening method, the adsorption capacities measured in this study were compared with literature values obtained by conventional detection methods. Zaitan et al.[35] determined the adsorption capacity of methanol and toluene, amongst others, on different types of adsorbent materials. For toluene, adsorption capacities were obtained in a range of 0.8 – 2 mmoles g^{-1}, whereas for methanol a value of 8 mmoles g^{-1} was reported. These are both in the same order of magnitude as the adsorption capacities in this study for toluene and methanol. Zhao et al.[26] compared the adsorption capacity of a hydrophobic zeolite with AC for the adsorption of benzene. For AC, a capacity of 370 mg g^{-1} was determined, corresponding to 4.75 mmoles g^{-1} which is in very good agreement with the adsorption capacity of AC in this study for a similar compound, i.e. toluene (4.3 mmoles g^{-1}). Hu et al.[31] performed similar experiments on the adsorption of benzene for different types of organofunctionalized SBA-15 materials and achieved adsorption capacities in the range of 0.29 - 0.63 mmoles g^{-1}. Brosillon et al.[32] used different compounds to compare experimental and simulated breakthrough curves. For acetone, a maximum adsorption capacity of 1.28 mmoles g^{-1} was achieved which is again of the same order of magnitude as our results for the Zeoflair® series. It is clear that the adsorption capacities determined by FTIR are very similar to adsorption capacities detected by conventional GC detection. The main difference between the two techniques is the time that it takes to perform the entire adsorption experiment. With our method, we were able to finish the experiments in an average time of 12 min. Based on similar experiments in literature, the average experimentation time using GC as detection tool is roughly 10 times longer.[19,25–28,31,32,34–36]
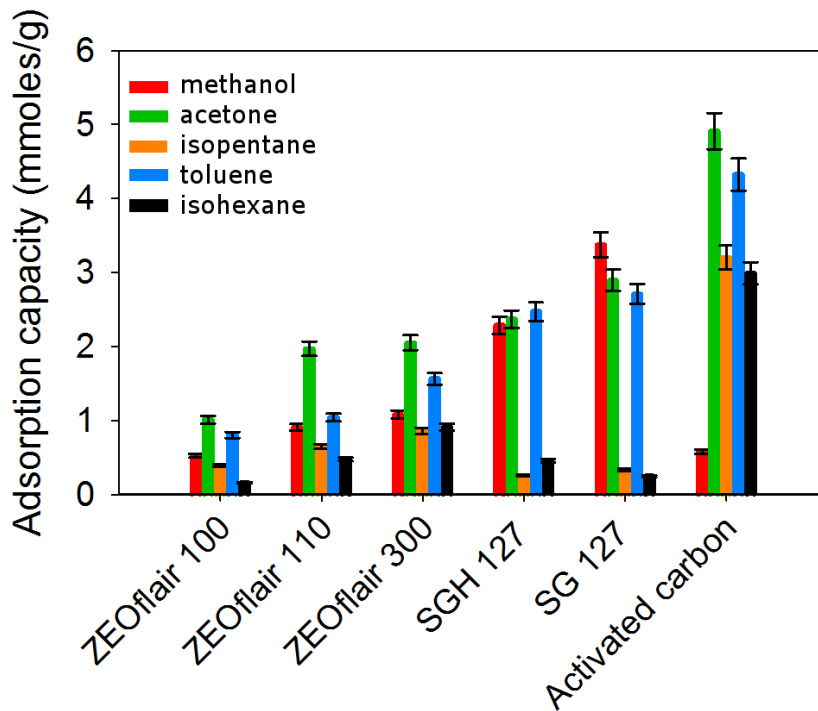
1

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Figure 4. Breakthrough curves for the five different VOCs on the zeolites and AC: acetone (green; •); methanol (red; –••–);

isohexane (black; –•–); isopentane (orange; – –); and toluene (blue; –).

1

2

3 **Figure 5**. Comparison of adsorption capacities for the different zeolite – VOC combinations. From left to right: methanol (red);

4 acetone (green); isopentane (orange); toluene (blue) and isohexane (black).

5 **Table 4**. Adsorption selectivity toward hydrophilic/hydrophobic VOCs for the different ZEOflair® zeolites (data from

6 manufacturer ZEOCHEM®).

	Selectivity to	
	Hydrophilic VOC	Hydrophobic VOC
ZEOflair® 100		+++
ZEOflair® 110		++++
ZEOflair® 300	+	++

7
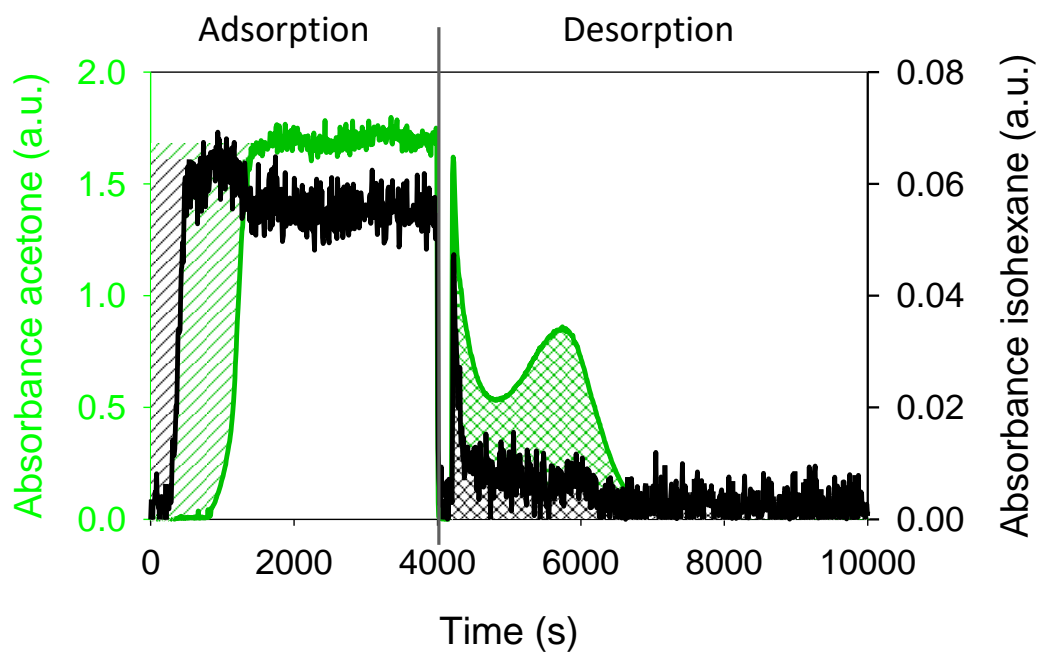
3.2.2. Multi-adsorbent packed bed, multi-component adsorption and desorption

10 For practical applications of VOC recovery, it is essential that adsorbed VOCs can be desorbed from the

11 zeolite surface at moderate temperatures. At the same time, the zeolites should be regenerated for

the next adsorption cycle. Using the presented on-line FTIR detection method, such a full adsorption/desorption/regeneration and reusability cycle can be monitored fast and easily. As a proof of concept, two of the most promising materials were packed in a multi-sorbent bed and exposed to a mixture of two VOCs. More specifically, ZEOflair® 300 and SYLOBEAD® SG 127 were applied in a two-bed system for the simultaneous adsorption/desorption of acetone and isohexane. The bed is organized in such a way that the gas mixture first passes SYLOBEAD® SG 127 and then ZEOflair® 300. For the desorption step a tube furnace was mounted around the dual sorption bed, with a programmed temperature profile (from room temperature to 150°C at a rate of 300°C/h, then from 150°C to 250°C at a rate of 90°C/h and a dwell time of 30 min). The direction of the flow during the desorption step was reversed in comparison with the adsorption step.

Based on the results depicted in Figure 6, both acetone and isohexane are readily adsorbed by the multi-bed system. The clear benefit of the proposed FTIR detection method, is that both compounds can be measured simultaneously in *quasi* real-time, at industrially relevant concentrations and over a short experimental timeframe. This would never be possible with traditional GC analysis. In the desorption step, by reversing the direction of flow and starting the temperature program, a first desorption peak is almost immediately observed which can be attributed to weakly bound acetone and isohexane molecules. At slightly elevated temperatures, around 150°C, a second broad desorption peak is observed.
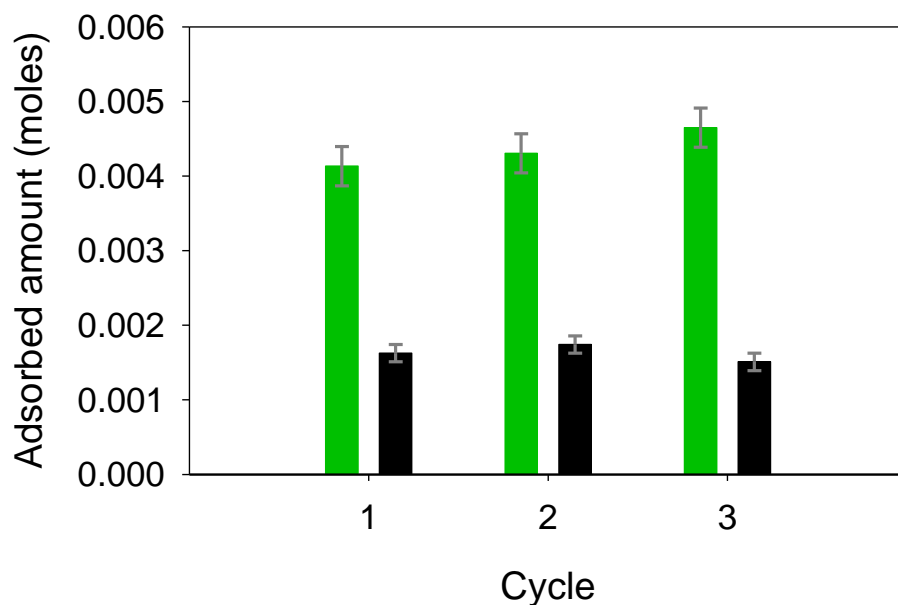
Figure 6. Adsorption-desorption cycle of acetone (green) and isohexane (black) on a combined bed of ZEOflair® 300 and SYLOBEAD® SG 127. The gray line indicates the transition from adsorption to desorption and thus the start of the temperature program. The dashed area indicates the amount of acetone/isohexane adsorption. The double dashed area under the desorption curve indicates the amount of acetone/isohexane desorption.

To evaluate the regeneration of the zeolites, three adsorption cycles were performed with the same multi-bed, each time after the abovementioned temperature program. From the three adsorption cycles (Figure 7), it is clear that the adsorption capacity of the multi-bed remains constant, and the sorbent surface is thus fully recovered.

Figure 7. Adsorbed amount of acetone (green) and isohexane (black) in the multi-bed system in three consecutive test runs.

After each adsorption cycle, the zeolite multi-bed was degassed at elevated temperatures up to 250°C.

4. Conclusion

In this work, it is shown that FTIR can be used as a valuable tool for ultrafast screening of zeolites based on their adsorption capacities towards VOCs. The adsorption capacities of three different commercial zeolites and two silica gels were determined for five industrially relevant VOCs (acetone, methanol, toluene, isohexane and isopentane). The obtained values are of the same order of magnitude as those reported in literature for similar sorbent-VOC combinations and determined by conventional detection methods. FTIR detection is shown to be much more time-efficient than GC analysis, and enables experiments at higher and more realistic concentrations in view of industrial applications. Finally, a multi-bed system was evaluated for a mixture of two compounds (acetone and isohexane). The on-line FTIR detection method enabled *quasi* real-time detection of both components simultaneously, both during the adsorption as well as desorption cycle. Three adsorption-desorption cycles were performed to confirm that the zeolites can be fully regenerated, showing that FTIR is a fast and reliable detection tool for the screening of adsorbent materials for VOC adsorption and recovery.

5. Acknowledgement

6. Competing interests

The authors declare no competing interests.

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