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Breakthrough Screening of Porous Materials: A Simple and Effective Tool for Database Generation

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The release of toxic and noxious gases in the atmosphere has become a big concern, due to its adverse effect on the health and life quality in general. The development of efficient filter materials can help to tackle this issue, considering that the production of such gases in various processes cannot be prevented. Metal-organic frameworks (MOFs) have emerged as a promising class of material to adsorb several toxic industrial chemicals (TICs). Nevertheless, identifying the suitable MOF material to adsorb a specific gas is a challenging task due to the lack of fast characterization methods. Using a mass spectrometer-detector, a simple setup has been developed and applied to register breakthrough curves and evaluate the retention capacity of selected MOFs against NH_3 and H_2S. The experimental results obtained using this *in-house* assembled device are in agreement with several literature reports, resulting in a fast tool for the development of a gas adsorption database for porous materials. Additionally, for the specific case of Ni-MOF-74, the breakthrough measurements correlate well with the surface areas of the material, providing an additional and fast quality control method to evaluate the material synthesis.

1. Introduction

The quality of the air has a critical impact on human health. In this context, various filters are deployed in different areas of the environment, aim to reduce human contact with toxic and noxious chemicals, such as common air filters are used for automotive cabins, fume hoods, air purifiers, and other purposes. The majority of these filters are based on activated carbons, their performances, however, are modest, being saturated within a relatively short timeframe. Additionally, activated carbon shows satisfactory adsorption capacity only for volatile organic compounds (VOCs), devoid of inorganic gases (NH_3, H_2S, CO, NO_x, SO_x, etc.).[12]

The quest for innovative sorbents has been carried out continuously in the last decades. Zeolites appeared to be promising candidates to replace or integrate activated carbon but their efficiency is drastically affected by humidity.[3] Furthermore and due to their superacidity, they catalyse other side reactions in the presence of water vapor, which leads to the release of other harmful compounds, such as crotonaldehyde.[4]

With this perspective, the implementation of metal-organic frameworks (MOFs) has been observed by the industry with a big hope for a decisive innovation in this field.[5] MOFs are crystalline porous materials consisting of metal ions or clusters linked together by organic bridging units.[6] The countless possible combinations of metals and organic groups make this material class extremely versatile and interesting for disparate applications such as gas storage[7] and separation,[8] catalysis,[9] sensors,[10] energy,[11] water harvesting[12] and remediation,[13] biomedical uses[14] and many others. The synthesis of MOFs and their scale-up is well advanced, allowing the supply of these porous compounds with a strongly reduced price compared to the first reported materials.[15]

Many reports deal with the use of MOF for toxic and harmful gas removal,[16] making this one of the most dynamic and advanced fields in the entire area of porous material research.[17] MOFs have exceptional porosity and incredibly high surface area, which promotes the adsorption of large amounts of gas molecules. Nonetheless, in the context of hazardous and reactive chemicals, this property is not always sufficient concerning strongly bound substances that are harmful even at very low concentrations. Therefore, the use of MOFs for TICs removal is mostly limited to those materials owning coordinative unsaturated metal sites (cus) and/or specific functional groups on the pore surface. The process of binding a guest molecule within the MOF pores is governed by coordination bonds, acid-base/electrostatic interactions, p-complex/H-bonding formation, etc.[18]

By measuring a standard gas isotherm, it is possible to calculate the adsorption capacity of a MOF toward a specific gas. However, this static information is not sufficient enough to verify whether or not the tested sorbent[19] would be useful in a real scenario where the concentrations of the harmful gases are very low. The standard method to evaluate the capacity of a

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sorbent material under these conditions consists of recording dynamic breakthrough curves,[20] which are usually measured using specific instruments, that are not easily accessible.

Adsorption processes on porous materials dealing with ammonia and hydrogen sulfide are probably the most investigated, due to the prominent importance of these two gases. Indeed, their use and concentration is strictly regulated by national international authorities.[21] Ammonia is commonly used as fertilizer, additive for cleaners or refrigerants and it is widely applied in chemical synthesis; indeed, millions of tons of Ammonia are produced per year. NH_3 is very harmful causing irreversible effects at a concentration higher than 500 ppm and can be lethal at a concentration higher than 3400 ppm (depending on the exposure time).[20] A vast amount of MOFs has been tested as selective ammonia adsorbers, specific materials have been tailored for ammonia capture[22] and well-known MOFs such as UiO-66[23,24] have been modified to increase the adsorption performances. The stability of the porous materials upon ammonia adsorption also has been deeply studied,[25] advancing the MOF technology for NH_3 removal close to commercial applications.

Hydrogen sulfide is used in several chemical processes as it is present in flue and natural gas streams. Due to its elevated toxicity, its removal is necessary and MOFs have also been widely applied as selective adsorbers.[26] Many experimental works that use MOF-74,[27] UiO-66[28] and derivatives,[29] MIL-101,[30] MIL-125,[30] fluorinated MIL-101(Cr),[31] HKUST-1[32], PCM-75,[33] MFM-300(Sc),[34] Mg[35] and Al[36] MOFs, and several other MOFs with soc-topology[37], have shown good to excellent performances in H_2S adsorption. In this respect, an *in-house* set-up, based on an MS-detector, has been successfully developed and used to provide a fast and reproducible screening and comparison of different materials based on their performance towards H_2S and NH_3 dynamic adsorption. The performed dynamic breakthrough tests provide with benchmark data of the analysed materials, defining essential references for the evaluation of newly developed materials.

2. Results and Discussion

The research in the field of MOFs has revealed methods for the fast production of hundreds of porous materials with specific characteristics in terms of porosity, specific surface area, and chemical properties. It is hence difficult to make a thorough selection of the most promising materials to be used for toxic gas adsorption. Aiming to test the *in-house* developed experimental setup we considered screening some commercially available and/or easily synthetized MOFs. In our system the gas stream is flowed through a packed bed, which contains the selected adsorber and it is coupled with a mass spectrometer used as detector to quantify the retention capacity of the materials (further details are reported in the experimental section).

Dynamic breakthrough measurements show the retention capacity of the sorbents against the target gases. All the samples were tested as powder in order to produce comparable results which are not influenced by the material form. Figure 1 reports the time duration (in minutes) needed to achieve 10% or 50% of the initial target gas concentration. These values provide useful indications on the gas adsorption in function of time, the complete breakthrough curves and the total adsorption capacity are reported in the SI. The time in the figures is normalized by the amount of sorbent used for each experiment. Respectively 100 ppm of NH_3 in N_2 and 500 ppm of H_2S flowed through the column, which contains the MOF material to register the breakthrough curves. The gas concentrations were selected using calculations made by the German Federal Office of Civil Protection and Disaster Assistance (BBK) for typical accidents with gas release in warehouses. Nitrogen was deliberately selected as carrier to avoid concurrent reactions, which could have hampered the reproducibility of the analytical method. Zirconium MOFs from the UiO class are an example of relatively robust and stable materials, based on cationic $Zr_6O_4(OH)_4$ building units and dicarboxylate linkers. Their chemical composition and porosity can be easily tuned using different derivatives of benzodicarboxylic (BDC) or biphenyldicarboxylic (BPDC) acid. Figure 1 shows similar results for both UiO-66 (based on BDC) and UiO-67 (based on BPDC), it can be assumed that the MOF cage sizes have no particular effect on the retention capacity. From the other hand BDC acids functionalized with either amino or hydroxyl groups show a much greater uptake of NH_3, considering the time before the NH_3 concentration reach 50% of the original gas stream an at least 10-fold increase has been registered. It is plausible that the hydrogen bonding interactions between the pendant groups of the BDC and the guest molecules are responsible for a stronger retention.[23]

Subsequent measurements were performed on the copper-trimesic acid MOF HKUST-1. In this case the retention time needed to reach a 10% NH_3 concentration in the output gas flow is relatively short, on the other hand the 50% breakthrough time is reached after a time comparable to the UiO-66-

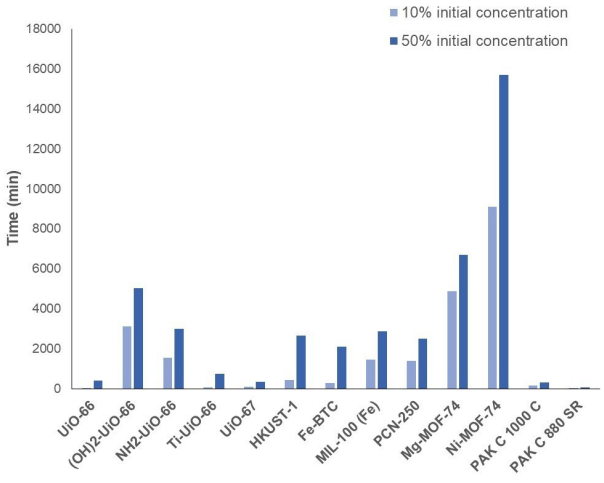

Figure 1. Breakthrough measurements of selected sorbents against NH_3 measured through *in-house* developed adsorption system. Time elapsed before 10% (pale blue) or 50% (navy blue) of the initial gas concentration is registered.

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NH_2 MOF. The ammonia adsorption in HKUST-1 is probably due to the affinity of the unsaturated copper moieties in the framework.[38] The unsaturated metal sites interact with the stream of the target gas and they are gradually coordinated, and therefore no longer available for adsorption. This process is translated into a continuous increase of the ammonia concentration in the output gas stream. Where several interaction mechanisms between the sorbent and the gas are simultaneously taking place, like in the case of the UiO-66-NH_2, which also contains unsaturated metal sites (due to defects), the process is more likely to be much effective at the beginning, with a longer retention time before reaching the 10% breakthrough. However, once the adsorption sites are occupied, the concentration of the target gas rises rapidly in the output stream. Three topological different Iron MOFs were also tested against ammonia. The performances are comparable to the UiO-66-NH_2 MOF. Obviously, the adsorption mechanism is different for each material and a detailed discussion is quite difficult. Larger adsorption capacities have been registered using two MOF-74 materials (also known as CPO-27). In particular, the Ni-MOF-74 displays the longest retention time against NH_3 of all the tested materials. Nickel and Magnesium have certainly different affinity to NH_3, however, most probably the longer retention time for Ni-MOF-74 in comparison to Mg–MOF-74 can be attributed to the larger specific surface area (1200 and 900 m^2g^{-1} respectively). Indeed, the presented method provides useful insights into the quality of the samples, which, considering the lack of reproducibility by many MOF synthetic pathways, is a fundamental issue. In Figure 1 the breakthrough measurements of two commercially available activated carbon (mesoporous PAK C 880 SR and meso-microporous PAK C 1000 C, CarboTech AC GmbH, Essen Germany) are also reported. The ammonia retention of these materials is quite modest compared to the MOFs.

In Table 1, the retention times for both 10% and 50% gas release of two different batches of Ni-MOF-74 are reported. The remarkable differences can be ascribed to the different surface areas of the samples. The breakthrough measurements are therefore a fast and powerful tool to evaluate the quality of a material, based on the application performances.

The results of the H_2S breakthrough measurements are shown in Figure 2. Very low to almost no retention has been observed using the UiO materials. An improved, but still modest retention has been observed with UiO-66-$(OH)_2$ and UiO-66-NH_2, most probably due to the instauration of hydrogen

Table 1. Comparison of B.E.T. Surface Area and breakthrough measurements for two different Ni-MOF-74 batches.

Ni-MOF-74	B.E.T. Area [a]	Breakthrough	NH3[b]	H2S[c]
Batch 1	1220	10%	9087	3128
		50%	15709	5757
Batch 2	950	10%	7165	1911
		50%	13364	4224

[a] m^2g^{-1} obtained through B.E.T. measurements. [b] Initial ammonia concentration 100 ppm, time reported in minutes. [c] Initial hydrogen sulphide concentration 500 ppm, time reported in minutes.

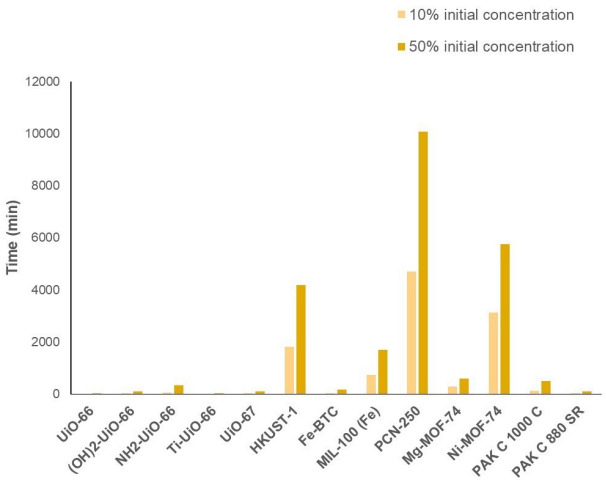

Figure 2. Breakthrough measurements of selected sorbents against H_2S measured through *in-house* developed adsorption system Time elapsed before 10% (light taupe) or 50% (yellow ochre) of the initial gas concentration is registered.

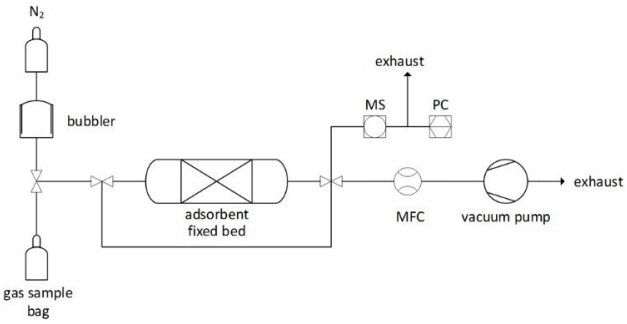

Figure 3. Flowchart of the developed setup for the material characterization through breakthrough measurements.

bonds between the guest molecule H_2S and the functional -OH and -H_2N groups. The latter has shown the largest retention time for the UiO-MOF family; indeed the presence of basic groups allows the adsorption of the sulfhydric acid. Good retention for H_2S has been observed in the case of the HKUST-1 material. This MOF contains copper coordinatively unsaturated metal sites, owing the high affinity of Cu for the H_2S guest molecule. The experimental results are coherent.

Concerning the iron MOFs, the results range over different scales, from the modest retention of Fe(BTC) to the exceptional high of PCN-250. Moderate adsorption has been registered in the case of MIL-100 (Fe). The very large retention time observed in the case of PCN-250 is probably ascribable to its peculiar structure, containing several sites for hydrogen bonding. Indeed, the H_2S can establish a bond with the Fe3(μ_3-O)$(CH_3COO)_6$ metal clusters, the Fe-OH groups and the 3,3′,5,5′-azobenzenetetracarboxylic acid (H_4ABTC) ligands.

Concerning the two MOF-74, the differences in adsorption capacity are even larger in the case of hydrogen sulfide compared to the case of ammonia. Here the different surface

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areas again have an important effect on the retention capacity, furthermore, the stronger affinity of nickel towards H_2S compared to Mg is probably responsible for the observed results.

The two activated carbons show a modest retention capacity towards H_2S. However, it is significant to highlight a longer retention time in this case, compared to the ones registered for NH_3 breakthrough measurements, especially since the concentration of the guest molecule is higher.

3. Conclusion

A simple method for the registration of dynamic breakthrough measurements of solid sorbents has been developed and tested. The system provided useful data on the adsorption capacity of selected materials. The experimental results consent a valid comparison of the investigated materials, allowing to make rational assumptions on the performance under real scenario conditions. Furthermore, in the case of Ni-MOF-74, the breakthrough measurements correlate well with the B.E.T. areas, providing an additional characterization method to evaluate the quality of the MOF material. The establishment of the benchmark data for the standard sorbents will allow the comparison with composites or modified materials, accounting for an additional tool for the evaluation of new materials and their synthetic processes.

This analytical system, which is based on a mass spectrometer detector, is simple and can be set up rapidly without the need for massive resources. This set of easy conditions will allow collecting data about a large number of adsorbent and gases within a short time-frame. Examining such a database using machine learning and artificial intelligence will provide a new potential strategy to generate important knowledge on the material properties.

Supporting Information Summary

The detailed experimental information concerning synthesis, characterization and breakthrough measurements (including breakthrough curves and gas loading capacities) of the adsorbents are reported in the supporting information.

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Conflict of Interest

The authors declare no conflict of interest.

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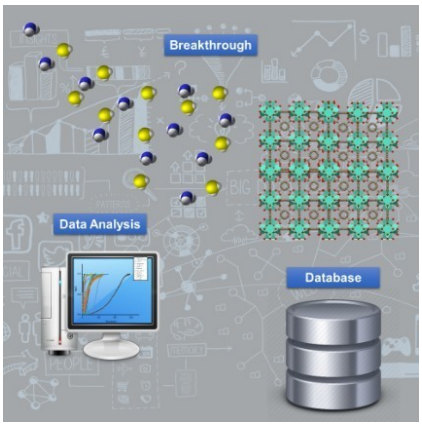
COMMUNICATIONS

Identifying suitable materials to adsorb a specific gas is a challenging task due to the lack of fast characterization techniques. A simple method for the registration of dynamic breakthrough measurements of solid sorbents has been developed and tested. This analytical system, based on an MS-detector, allows to collect data about a large number of adsorbent and gases and provides with useful information to evaluate the material quality.



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