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Improved Exhaust System for Laboratory Work with Fluorine

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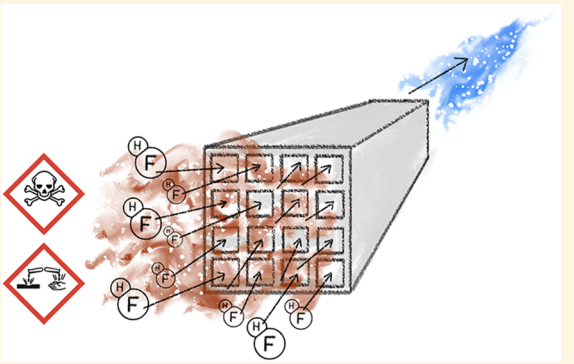
ABSTRACT: Hydrofluoric acid (HF) is a widely used chemical in laboratories as well as in the glass and ceramics industries. It is valued for its high reactivity; however, it poses significant disadvantages, including high toxicity to humans and corrosive effects on equipment. To prevent damage to laboratory and surrounding equipment as well as to ensure operator safety, an appropriate exhaust ventilation system must be used. Exhaust systems for hydrofluoric acid applications must meet stringent technical standards. Depending on the frequency of use, they may incorporate wet scrubbers, which necessitate considerable energy input and consumption of alkaline neutralizing agents and cause a substantial pressure drop. We here present an easy-to-handle alternative system based on calcium hydroxide honeycomb monoliths (HMs). We calculated and simulated the HF precipitation on the HMs based on thermodynamic data and with finite elements. We considered a liquid HF concentration of up to 50% in use within a standard laboratory fume hood. The HF concentration in the exhaust air after HMs throughflow was measured as well as the pressure drop. Theoretically and experimentally, HF was completely bound to calcium hydroxide HMs. Moreover, we find that the pressure drop in the fume hood was low compared to that of wet scrubbers. Calcium hydroxide HMs are a promising option for adsorbing HF from exhaust gas flows. From the economic viewpoint and from technical and organizational requirements, HMs offer significant benefits over wet scrubbers. Our findings can be directly implemented within experimental laboratories handling HF containing exhaust air.



KEYWORDS: *fluorine, laboratory, chemical safety, fume hood, honeycomb monolith, calcium hydroxide, finite element simulation*

INTRODUCTION

Soluble fluoride ions rank among the most hazardous chemicals encountered in laboratory practice due to their high toxicity and the ability to penetrate biological tissues. For example, their derivative, hydrofluoric acid (HF), is used for glass etching as well as in electronics manufacturing and various chemical applications. It is inexpensive and highly effective and is involved in numerous well-established standard procedures. Because of its toxic properties,[1−3] working with it in a scientific laboratory should only be undertaken after proper training and with the use of appropriate personal protection equipment, such as[4] a Teflon apron, special face shield, gloves, and rubber or PVC boots.[5] As a working material, preferably organic polymers can be used, such as PVC, PE, PP, and FPM (for example, Viton). Most rubbers are unsuitable, as are polysulfon or SAN (styrene-acrylonitrile). In the case of an HF contamination on the skin, calcium gluconate has to be applied immediately. Personnel trained in first aid should always be at hand, and medical treatment by medical professionals is needed. While skin and material can be readily protected from the liquid HF, fumes and vapors are not so easily handled. Thus, accidents associated with inhalation are more severe, and medical treatment is difficult. HF evaporates into the air from aqueous solutions. When working in a fume hood, its material and technique should therefore be carefully chosen. Ideally, HF fumes should be eliminated even before leaving the fume hood into the air or into the exhaust system of the building.

Generally, for acidic fumes, wet scrubbers are used in the exhaust system of a fume hood. Depending on the particular use, wet scrubbers have a high acid or base throughput and high energy consumption, and introduce various technical challenges, since a pressure drop of 400−700 Pa must be overcome.[6] Wet scrubbers often introduce a large pressure drop,[7] as they are operated with high gas velocities to induce turbulence, as this leads to elevated scrubbing efficiencies. However, this approach often causes problems with the adjustment of the air pressure in the laboratory. These problems, however, can be avoided by using honeycomb monoliths (HMs) as reactors to precipitate hazardous substances like acids. To our knowledge, HMs were used up

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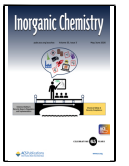

Table 1. Thermodynamic Data of the Reactants at a Standard Temperature of 298 K[13,14]

material	aggregation state	$\Delta^f \overline{H^0}$	$\overline{S^0}$	$\Delta^f \overline{G^0}$	$\overline{C_p^0}$
		standard enthalpy of formation	entropy	free enthalpy of formation	thermal capacity
		kJ/mol	J/mol/K	kJ/mol	J/mol/K
CaF_2	solid	−1225.91	68.57	−1173.53	67
HF	gas	−272.55	173.67	−274.64	
H_2O	gas	−241.83	188.72	−228.6	33.577
$Ca(OH)_2$	solid	−986	83	−987	

to now, for example, in industrial treatment systems to remove nitric oxides, volatile organic compounds, or to catalyze chemical processes.[8−10] Honeycomb monoliths have several advantages over packed beds: A good interphase mass transfer and a high specific surface[11] ensure efficient reaction on a large contact area.[12] The small pressure drop also at high flow rates,[11,12] and good thermal and mechanical properties make them easy to construct and apply in a laboratory situation, even with changing pressure and thermal conditions.

The aim of this work is to establish a method for the simple removal of HF from the exhaust gas stream of a laboratory fume hood. The method should be effective, inexpensive, and low-maintenance. This work evaluates the use of HMs made of calcium hydroxide ($Ca(OH)_2$) for this purpose theoretically and practically.

MATERIALS AND METHODS

Theory of the Thermodynamics to Estimate the Concentration of HF in Equilibrium after the Passage of the $Ca(OH)_2$ HMs

The partial pressure of water in the immediate vicinity of the reaction is assumed to be saturated at t = 25 °C compared to liquid water, as water is released during the reaction (eq 1)

$$2HF + Ca(OH)_2 \rightleftarrows CaF_2 + 2H_2O \quad (1)$$

The partial pressure of water (E_w) depends upon the temperature (eq 2),[13] and was calculated as being 3154 Pa.

$$E_w = 6.0328\ \text{hPa} \times \exp\left(\frac{17.1485 \times t}{234.69^\circ\text{C} + t}\right) \quad (2)$$

The energy released from the precipitation (eq 1) was calculated with the tabulated values of the free enthalpy of formation under standard conditions (Table 1) by using eq 3. Although water is liquid under standard conditions, we assume that only water vapor is involved in the reaction in the HMs. The airflow does not allow water to condense. For ease of calculation, the evaporation process of water has already been taken into account in the thermodynamic data.

$$\Delta^a \overline{G_r} = \sum (\text{energy output}) - \sum (\text{energy input}) \quad (3)$$

Under standard conditions (partial pressure 1 bar), the free enthalpy $\Delta\overline{G_r}$ is −94.45 kJ/mol.[14] The reaction does not take place under standard conditions. Actually, the partial pressure of H_2O is 3154 Pa (see eq 2). The free enthalpy has to be corrected using the relation of actual/standard pressure in eq 4. Thus, $\Delta\overline{G_i}$ is −112 kJ/mol.

$$\Delta\overline{G_i} = \Delta\overline{G_r} + 2 \times \left(R \times T \times \ln\left(\frac{p_i}{p_0}\right)\right) \quad (4)$$

When calculating the concentration of HF in the gaseous phase after the reaction, $\Delta\overline{G_r}$ equals 0. The reaction is now at equilibrium. Using eq 5, the partial pressure of HF in the gaseous phase is 1.55 × 10^{-5} Pa.

$$\Delta\overline{G_r} = \Delta\overline{G_i} + 2 \times \left(R \times T \times \ln\left(\frac{p_{HF}}{p_0}\right)\right) = 0 \quad (5)$$

The amount of HF in air can be calculated via the ideal gas law (eq 6).

$$p \times V = n \times R \times T \quad (6)$$

With p_{HF} = 1.55 × 10^{-5} Pa, V = 1 m^3, R = 8.314 J/(mol·K), and T = 298 K, n is 6.3 × 10^{-9} mol/m^3. Converted to the unit in which occupational safety standards are given, it amounts to 1.19 × 10^{-4} mg/m^3.

Simulation

Using a finite element simulation (Comsol), the precipitation of HF in one channel was also modeled. Four scenarios are assumed, which must be considered (Table 2): 1. A **worst-case scenario** is assumed to be an event in which all internal surfaces of the fume hood are contaminated with 50% HF. This could happen if a bottle containing HF explodes. The evaporation of 50% HF is fast. We experimentally found that the vertical surfaces are dry again after 20−30 min, and the roof surfaces after 10−20 min. The last traces of H_2O on the table surface have disappeared after 3 h. 2. In the case of an **average disaster**, the table surface of the fume cupboard is covered with 50% HF. This can happen if the content of a bottle of HF is spilled in the hood. Without further action, the accident continues until the HF has completely evaporated. Some of the HF flows into the sink. After 3 h, the entire HF should have evaporated. 3. An event with **maximum emission** in normal operation is defined by a few larger beakers filled with 50% HF standing openly in the fume hood. This can last for a longer period. 4. **Normal** emission when working with HF is defined by a few covered large beakers and a few open beakers with adhering HF residues. Only the worst-case scenario was considered in the simulation.

Table 2. Scenarios of Contamination in the Hood; Loss of HF, Mass Flow, and Evaporation Rate were Determined Experimentally, as Described in the Text Below

event	contaminated area [m^2]	loss of HF [g/h]	mass flow per area [g/m^2/s]	and [mol/m^2/s]	HF in air at enhanced exhaustion of 900 m^3/h [mg/m^3]
worst-case scenario	5.70	203	376 × 10^{-3}	18.8 × 10^{-3}	226
average disaster	1.05	37.5	69.4 × 10^{-3}	3.47 × 10^{-3}	41.6
maximum	0.20	7.14	13.2 × 10^{-3}	6.61 × 10^{-6}	7.93
normal	0.02	0.71	1.32 × 10^{-3}	6.61 × 10^{-7}	0.79

For the simulation, we assumed the following: air ventilation of 900 m^3/h in one channel (0.15 m^2 area, geometry 5 mm × 5 mm × 300 mm in the direction of flow). The HF contamination of the fume hood was calculated to be 18.8 × 10^{-3} mol/m^2/s in a worst-case scenario (Table 2). The honeycomb monolith was assumed to be fresh. The flow pressure drop was approximated to be 5 Pa in order to

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state an appropriate volume flow. Assuming a constant flow is difficult because volume changes with different pressures.

Real-Life Tests

As a test system, a fume cupboard of width × depth × height of 1.5 m × 0.7 m × 1.2 m (Waldner Scala, Germany) was used. When a wet scrubber is used, it is placed on top of a fume hood. The chemicals for neutralization usually occupy the working space of the hood. Due to the high airflow resistance and therefore resulting pressure drop, an additional fan is required in the exhaust system. The contaminated gas stream is brought into contact with the washing liquid. The contaminated droplets have to be collected from the gas stream afterward and disposed of.

In our case, the exhaust system was made of PVC, PE, and PP with 15 (3 × 5) calcium hydroxide ($Ca(OH)_2$) HMs (Steuler, Germany) placed above in the line of the contaminated gas stream (Figure 1).

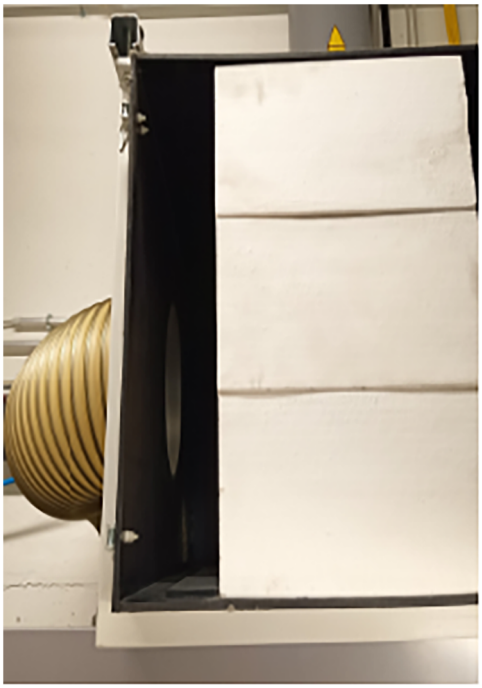

Figure 1. Honeycomb monoliths (white, in the black box) and the connecting exhaustion hose (yellowish, left side) on top of the fume hood. Photographed from the side with airflow from left to right.

The HMs are stacked in the box without further need for additional mounting. The polymer channel ends in a galvanized sheet metal collecting duct. Here, concentrations of HF in the air of >1 mg/m^3 would cause corrosion (experience from different laboratories).

The geometry of a single honeycomb monolith is 0.15 × 0.15 × 0.28 m with 400 channels (0.005 m × 0.005 m) each (Figure 2). Each monolith had a weight of 4.4 kg. The sum of all the front surfaces of the channels is (6000 × 0.00025 m^2 = 0.15 m^2), and the total front surface is 0.34 m^2. Thus, 44% of the total available area is open.

The pressure drop in the exhausted airflow before and after the HMs was measured using a multifunctional analyzer instrument testo 435−1 (Testo, Germany). To estimate the evaporation rate of HF, a photo developing tray of size 40 cm × 50 cm was completely wetted with 50% HF (Sigma-Aldrich, Germany). It was placed on a balance in the fume cupboard and left there for 1 h at 900 m^3/h (corresponding to the maximum exhaustion capacity). Our experiments were performed with the fume hood sash both opened and closed and with or without an increased ventilation rate. The mass loss was set equal to the emission of 50% HF. With the wetted area of the photo developing tray being 0.2 m^2, the emission of HF can be calculated in g/h/m^2.

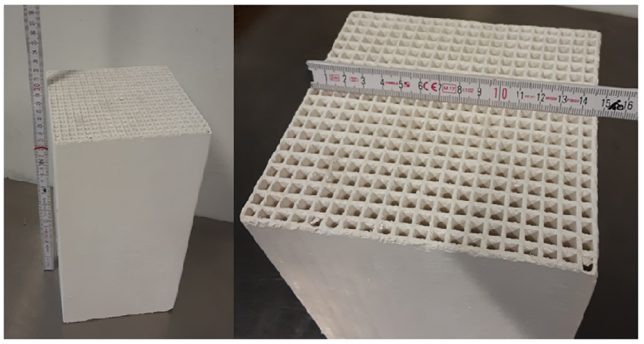

Figure 2. Calcium hydroxide honeycomb monoliths.

The HF content was measured in the exhaust airflow before and after the honeycomb. The detection methods and their sensitivity were checked by using different concentrations of liquid HF in Petri dishes. Detection devices and materials were exposed to the gas phase above the liquid. Fluoride test paper from Macherey-Nagel[15] and Crowcon Gasman S011433 0−10 ppm sensors with HF specification[16] were used for this purpose. Although the producer's specification for the lower limit of detection was 20 ppm for the test paper, we could trace it down to ± 1 ppm (Figure 3). The alarm of the Gasman sensor is by default set to 1 ppm.

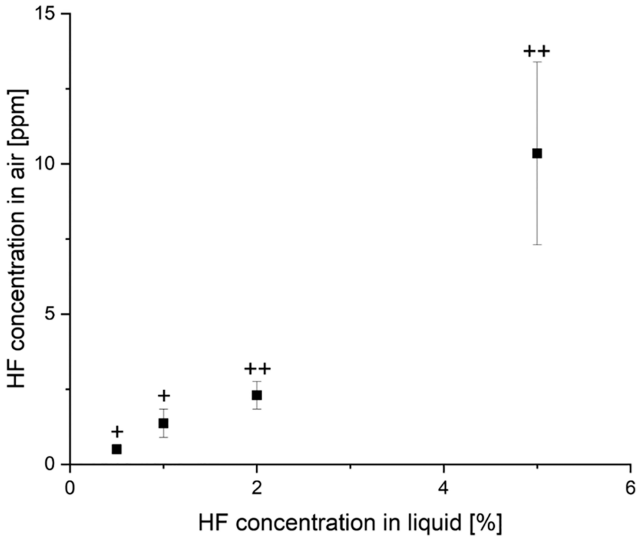

Figure 3. HF concentration in vapor above a liquid prepared containing different concentrations of HF. Concentrations in vapor measured using a Gasman sensor. Detection of HF with test paper is indicated with + or + +.

In ordinary wet scrubbers, the depletion of the HF-eliminating system is irrelevant because water and base are continuously added in surplus. For the honeycomb monoliths, those responsible must monitor the exhaustion of the reactant in order not to have an HF breakthrough. We calculated the maximum of HF neutralized as follows:

Stoichiometrically, 1 mol of $Ca(OH)_2$ binds 2 mol of HF (eq 1). This means that ideally, 1.85g of $Ca(OH)_2$/g of HF is required for neutralization. Experimentally, the more than triple-fold amount of calcium hydroxide (6.48 g $Ca(OH)_2$/g HF) was needed.[17] This value seems to be plausible because not all $Ca(OH)_2$ molecules can react. Diffusion of HF into the HM material can be hampered physically.

In our laboratory, we installed 15 HMs of 4.4 kg each, resulting in 66 kg of $Ca(OH)_2$. Thus, the maximum amount of HF that can be precipitated on the HMs is about 10 kg (= 66/6.48).

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RESULTS

Amount of HF in the Exhausted Air

The mass loss in the photo developing tray was 14.3 g/h of the 50% HF solution. Initially, more than 50% HF will transition into the gas phase, that is approximately 90%.[18] As the evaporation process continues, this value will continuously decrease until the azeotrope is reached in the liquid phase at approximately 39% HF in H_2O. This then also applies to the gas phase.

On average, however, 50% HF and 50% water evaporate, as all liquid transitions into the gas phase in the event of an accident. This equals an average of 7.14 g pure HF/h, normalized to 35.7 g/h/m^2 HF. With this value, the amount of HF reaching the HMs was between 0.8 and 226 mg/m^3, depending on the event. These values are documented in Table 2, as we calculated further with the results.

Simulation

A finite element method simulation was calculated by assuming an average laminar flow velocity of 0.8 m/s in the channel (Figure 4). In contrast, the flow is turbulent in the adjacent channels. This fact is important when comparing the measured and the simulated pressure differences. The pressure drop was 5 Pa over the length of the HM channel.

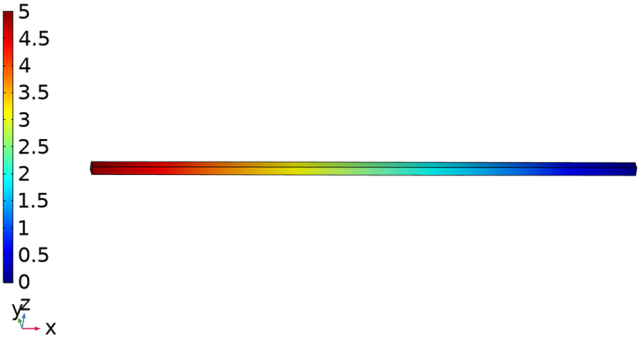

Figure 4. Pressure drop in Pa in the channel of the honeycomb monolith along the entire length of the channel. The ambient pressure of 101,000 Pa is subtracted for better illustration.

The equilibrium concentration of 1.19×10^{-4} mg/m^3 HF was taken from the Materials and Methods section (see above). The simulation shows that the removal of HF from the gas flow occurs in the first 10 mm of the channel (Figures 5 and 6).

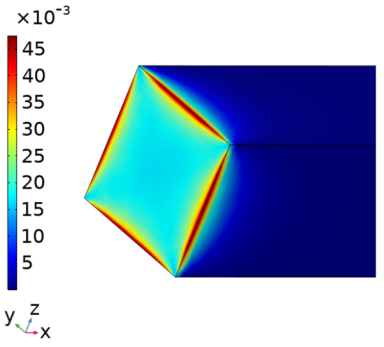

Figure 5. Mass flow of HF at the interfaces between exhaust gas and $Ca(OH)_2$, model shown for one channel inlet only (5 mm × 5 mm opening). Total flux magnitude is given in mol/(m^2·s).

Pressure Drop

A pressure drop of about −28,000 Pa was measured in the piping system between the hood and the exhaust line, including the HM filter as designed in our system. The pressure drop in the HM filter was 80 Pa at a flow of 1000 m^3/h and 15 Pa at a flow of 220 m^3/h, respectively. The contribution to the pressure drop of the HMs is thus only about 0.05%. According to technical data provided by the manufacturer[19] or documented in literature,[6] for wet treatment hoods, the pressure drop is at least 10-fold.

Amount of HF in the Exhausted Air

While the exhausted air was considerably contaminated with HF before flowing through the HMs, afterward, HF could not be detected in any case (Table 3). A visual inspection of the metallic collection duct behind the HMs also revealed no signs of corrosion damage.

DISCUSSION

In laboratory environments, a sophisticated pressure regulation regime is needed to prevent contaminants from either entering or leaving the laboratory. The pressure drop resulting from the passing of the contaminated air through the HMs is negligible and therefore is of a huge practical benefit, while ensuring good working conditions and requiring minimal technical expertise.

The calculated concentration in the airflow after HM treatment is >5600 times lower than the initial concentration ($0.83/1.46 \times 10^{-4}$). Experimentally, such low concentrations could not be confirmed because of the limited sensitivity of the available equipment. Nevertheless, neither the quantitative analyzer nor the semiquantitative test paper was able to detect any HF in the gas flow after treatment. Neither did we face problems with corrosion in the metallic exhaust system. The occupational exposure limit for HF of 1 ppm (0.83 mg/m^3) or 1.5 ppm (1.25 mg/m^3) can obviously be complied with.[20,21]

The calculated maximum capacity of the HMs in our laboratory of 10 kg should be sufficient for the assumed common workload for many years. Experience has shown that most of the HF is removed from the fume hood with the wastewater or collected in waste containers. Even if 1 L of HF (50%) were to evaporate over a certain period of time, this would be 0.5 kg of HF, i.e., only 5% of the potentially absorbable quantity for our HM system. In the case of evaporation of 0.5 kg of pure HF per year, the filter would be saturated after 20 years. To err on the side of caution, we recommend exchanging the honeycomb monoliths after 10 years.

If we consider the cost-effectiveness of wet scrubbers and HMs, the HMs clearly have the advantage: the purchase cost of a wet scrubber is around €20,000. For the system with HMs presented here, the total cost is around €600. Of this, €300 is for the box, and another €300 is for the 15 HMs. The running costs for the wet scrubber are around €1000/year and include maintenance, chemicals, electricity, and water. The HMs, on the other hand, are maintenance-free and should be replaced after 10 years. This equates to €30 in consumption costs per year. In terms of both acquisition and consumption costs, the ratio is 33:1 in favor of the HMs.

In particular cases such as university laboratories, the degree of exhaustion of such an HM system can often not be easily estimated: experimental interests and setups are subject to frequent change, as indeed are the personnel responsible. Additionally, the individuals responsible for the operation of

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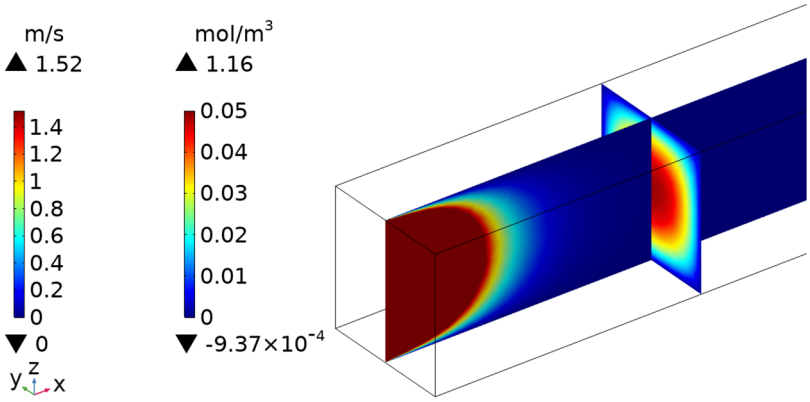

Figure 6. Velocity in m/s (perpendicular to the flow) and HF concentration in mol/m^3 (in the direction of flow), channel inlet only. Channel area: 5 × 5 mm^2. For better visibility, the concentration is scaled to lower values.

Table 3. Amount of HF in Exhaust Air before and after Treatment

	before treatment	after treatment	limit of detection (mg/m^3)
gasman	>32 mg/m^3	0 mg/m^3 (<ld[a])	0.1 ppm (83 μg/m^3)
test stripes	yellow	no coloring (<ld)	1 ppm (0.83 mg/m^3)

[a]ld: limit of detection

the HF fume hood should be able to test exhaustion without special training or elaborate equipment. Therefore, we recommend using an easy test with a volatile acid to inspect the retention capacity of the HMs. The reaction product with $Ca(OH)_2$ should ideally be easily soluble, in contrast to CaF_2, which has poor solubility. The acid should be allowed to evaporate in an open dish in a fume hood. The acid content in the exhaust gas stream can be easily determined using moistened pH paper before and after the HMs. The equilibrium concentration of the acid in the gas phase due to adsorption on the honeycomb is only determined by the neutralization capacity of the $Ca(OH)_2$ that has not yet reacted. For example, HCl or acetic acid (CH_3COOH) applies to both conditions: Both are volatile, and the reaction products, $CaCl_2$ and $Ca(CH_3COO)_2$, are easily soluble salts. Another advantage of these acids is that no toxic F^- or HF has to be handled. Thus, a test with these acids will already indicate exhaustion of the neutralizing capacity due to the acidic reaction of the gas phase, although HF is still retained by precipitation as CaF_2.

One distinct advantage of our approach using HMs is that they require no active maintenance, unlike wet scrubbers, and can easily be integrated into an existing fume hood system. There is no need for sophisticated measures in the case of removing the hood from service. In the case of recommissioning, the above-mentioned test can be executed easily before starting work with HF again.

CONCLUSIONS

We have demonstrated that honeycomb monoliths are an easy and effective method to eliminate hazardous acids, in this case HF, from the exhaust of a chemical lab. The system with HMs is low-cost in both outright purchase and maintenance. This is especially the case because sophisticated pressure regulation is not necessary, and thus only minimal technological expertise is needed.

From our calculations, we find that the HMs are especially suitable in laboratories with low or highly varying frequencies of HF use. Depending on the working conditions and experiments, the HM-extraction system is scalable. It could be used for small experiments in closed systems as well. In laboratories with high HF throughput, a wet scrubber might be the superior method. In these laboratories, the technical expertise should also be available.

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Author Contributions

CRediT: **Ralf Keding**: conceptualization, methodology, thermodynamics, FEM simulation, technical realization, investigation, validation, writing - review & editing. **Clara Moedl**: investigation, validation, writing - review & editing.

Notes

The authors declare no competing financial interest.

ABBREVIATIONS

HMhoneycomb monolith

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