

Emergency responses in smoke from Li-ion batteries

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Niels van Veen* & Arjen Koppen

National Institute for Public Health and the Environment &
University Medical Center Utrecht, National Poisons Information Center
Utrecht, Netherlands

ABSTRACT

This study aimed to determine health risks associated with firefighters' exposure to smoke from burning Li-ion batteries, focusing on 1) Hydrogen Fluoride (HF) concentration development in smoke and 2) the protective value of a fire hood. Smoke from Li-ion battery fires was collected in an airtight bag. Hydrogen fluoride (HF) concentrations in the bag were detected over time, showing a decrease to levels ranging between 8% to 50% of the initial concentration within 20 minutes. At the end of the experiment, a fluoride-rich deposit was observed at the bag's surface, suggesting deposition of HF from the smoke onto the bag.

HF absorbing filters covered by samples of fire hoods with particle barriers were exposed to the smoke. The tested fire hood did not prevent HF penetration, but the particle barrier of the fire hood retained larger fluorine-containing particles (aerosol) well. Based on the amount of HF penetrating fire hoods with particle barriers, skin damage cannot be excluded. On the other hand, systemic health effects due to skin absorption of HF are unlikely.

Keywords: HF concentration decrease, Fire hoods, health effects HF

Introduction

Fires with Li-ion batteries will increasingly become part of standard fire-fighting scenarios. Complicated indoor fire scenarios, such as fires in parking garages, can lead to prolonged firefighter exposure to smoke with relatively high concentrations of HF. Much research has been done on gas emissions from burning Li-ion batteries, but little is known about time-dependent changes of flue gas concentrations. Many compounds of smoke settle on, condense on, or are adsorbed by solids with which they make contact. Since HF is a reactive gas, it is expected that HF flue gas concentrations decrease considerably over time as a result of these processes. Since smoke can circulate in enclosed spaces before it is released into the open air, a possible decrease in concentration is important for a risk assessment for deployed firefighters.

The added value of personal protective equipment (PPE) is also an essential part of the risk assessment. Fire-fighting clothing has been tested for resistance to penetration of HF gas. Several layers of clothing tissues containing a layer of Gore-Tex appear to offer sufficient resistance to penetration of HF gas [1]. However, uncertainties remain about the protective value of the thinner fire hood. The fire hood with Nomex Nano Flex® particle barrier is considered to be a commonly used fire hood at fire brigades. This study aimed to gain insight into the development of HF concentration in smoke and the protective value of the fire hood against HF in smoke. Since considerable amounts of HF were detected behind the protecting fire hood, toxicologic interpretation is added to this paper.

Methods

Four experiments (numbered A-D) were performed using various Li-ion batteries, which were ignited in a vessel with a propane gas burner. The burner was removed when thermal runaway conditions appeared. The smoke was collected in a closed cylindrical bag once fluoride was detected in the smoke. The trapped smoke was measured for +/- 50 minutes with Fourier-transform infrared spectroscopy (FTIR) and sampled with gas washing bottles. The experiments were primarily focused on the properties of smoke and not on the Li-ion batteries fire behaviour.

FTIR measurements were performed continuously, and gas washing bottles sampled smoke in the period 0-10 minutes, 20-30 minutes, and 40-50 minutes from the start of the experiment. Gas detection tubes were tested in the first minutes for comparison with FTIR and gas washing bottle results. All sampling for analysis was located at the bottom of the bag. The burned prismatic batteries induced very dense smoke, which resulted in immediate clogging of the gas washing bottles of experiments B and D. As a consequence, no data of gas washing bottles is available for these experiments. The experiments were performed in outdoor conditions. Humidity levels ranged between 80-100%, the temperature was 10°C, and similar conditions were present in the smoke-filled bag.

Samples of fire hoods were placed in the bag to detect penetration rates of HF. Only the double-layered part of the hood, including particle barriers, were used for this experiment. Fire hood samples covered HF absorbing filters (HF filters) or polyvinylchloride (PVC) pipes. Fire hoods were exposed in both dry and slightly moistened conditions. An overview of exposure methods and sample types is available in Table 3.

Table 1 Overview of the experiments, bag number, number, and type of battery used. Please note that experiments C and D were performed in the same bag, resulting in the use of a total of three bags. The battery of experiments B and D is identical.

Experiment	Bag nr	Number of batteries	Battery type	State of charge
A	1	1	Pouch (LFP)	0-25%
B	2	1	Prismatic (LFP)	75%
C	3	5	Cylindrical 18650 3500mA (NMC)	100%
D	3	1	Prismatic (LFP)	75%

The bags were cut open after the experiments. The inside of the bag was sampled with swabs (surface of each swab: 100 cm²) on both ceiling and bottom of the bag. Fire hoods were removed from the PVC pipes, and the surface of the PVC pipes (100 cm²) was wiped with swabs. The swabs and HF filters were then analysed by ion chromatography for the fluoride content. Inside the bag surface, samples were also taken on metal elements which were analysed by IC-PMS. Experiment C indicated low concentrations of HF. In order to ensure sufficient exposure of the fire hood samples, another battery was ignited after experiment C, for which the same bag (no 3) was used. As a consequence, the deposition fraction of bag 3 increased in comparison to bag 1 and 2.



Figure 1 The cylindric smoke bag filled with smoke from experiment B. Tripods were placed in the bag to attach fire hood samples. In between the tripods, the PVC pipes which were covered with fire hoods. After the experiment, the bags were cut open, and surface samples were taken at the bottom and top of the bag. The positions for the metal swabs are indicated in red, the ion swabs in green.

All fluoride concentrations are reported as HF in this paper. However, we expect that in some occasions, high concentrations of other fluoride-containing compounds than HF are released. From a risk assessment point of view, HF will generally cause a larger threat for skin exposure of firefighters than most other fluoride-containing compounds. For this reason, reporting fluoride as HF may be regarded as a worst-case approach.

Results

HF concentration development

The HF concentration in the collected smoke decreased strongly, especially in the initial phase, while other flue gases such as CO₂ hardly decrease. The HF concentration decrease differs per experiment and varies between a 50% to 92% decrease compared with the first measurement value at the start of the experiment. Stronger decreases are observed in gas washing bottles in comparison with FTIR measurements. After approximately 20 minutes, the HF air concentration stabilized.

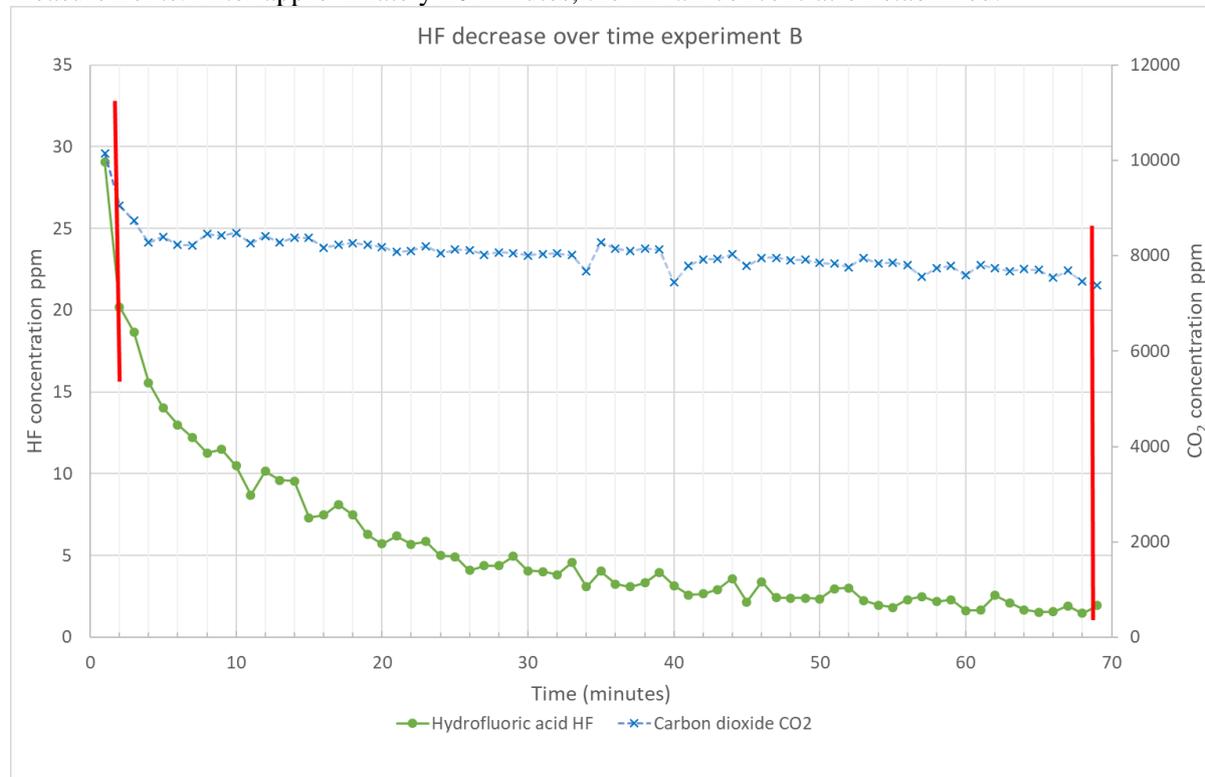


Figure 2 FTIR measurements of experiment B. The HF decrease is considered representative between the red lines, the closure of the bag, and the end of the experiment.

In experiment B, the HF concentration decreased exponentially with a half-life time of roughly 10 minutes. After 20 minutes, the half-life time increases. The CO₂ concentration decreased slightly in a linear fashion. Since experiment A had very low fluoride concentrations, phosphoric acid (based on PO₄³⁻ ions) air concentrations are shown to illustrate decreased levels of other smoke compounds.

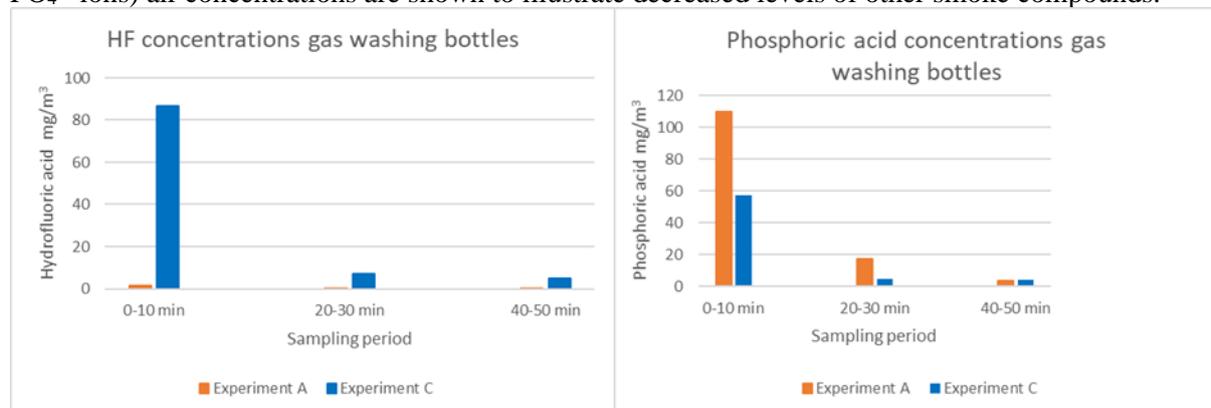


Figure 3 Sampling results of gas washing bottles. Left-hand panel: the F concentrations reported as hydrofluoric acid, right panel: PO₄³⁻ concentrations reported as phosphoric acid. The x-axis shows the sampling time of gas washing.

The results from the gas washing bottles confirm the substantial concentration decrease of HF observed with FTIR measurements in other experiments.

Table 2 Sampling results of a surface of 10x10cm inside the bag. Metals are shown when elevated concentrations are detected.

Bag no	Experiment	Surface position	HF μg	Li μg	Al μg	Mn μg	Fe μg	Ni μg	Co μg	Cu μg
1	A	bottom	9.3	0.37	1.21	2.07	0.26	0.46	0.06	0.45
		ceiling	3.5	0.28	1.28	2.82	0.26	0.55	0.07	0.87
2	B	bottom	15.1	0.11	0.88	1.56	0.25	0.25	< LOQ	0.11
		ceiling	4.7	0.06	0.77	1.36	0.2	0.28	< LOQ	0.07
3	C + D	bottom	195	160	48.4	13.9	90.4	205	30.9	23.7
		ceiling	77.1	85.9	12.1	2.65	6.19	63.8	10.9	7.95

Surface analysis of the bags after the experiments showed approximately three times higher contamination values at the bottom compared with the ceiling (Table 2). In addition, important differences between all detected elements are visible between bag no 3 and the other bags. Experiments A, B and D were LFP batteries, of which B and D were identical batteries with the same state of charge. Experiment C was performed with cylindrical NMC batteries, producing black smoke. The NMC batteries are likely to be mainly responsible for the high surface contamination of bag 3. The total amount of disappeared HF as found in the experiments with FTIR and the gas washing bottles corresponded roughly with the detected surface contamination.

Fire hood test results

The fire hoods were exposed to changing concentrations of HF. In order to translate the exposure to a dose relevant for deployed firefighters in HF containing smoke, the area under the curve of the FTIR measurements was used to calculate the total HF exposure reduced to a time of 20 minutes. The time frame of 20 minutes was chosen since most fire-fighting air supply systems do not provide longer air during average heavy work conditions for firefighters.

Table 3 Test results of the HF filters, PVC surface, the sum of the surface contamination of all experiments derived from Table 2. The FTIR concentrations for 20 minutes of exposure are derived from the area under the curve of the FTIR measurements. Results below the detection limit are shown in red.

Experiment	Sample type	Protection methodology	Detected amount of HF μg	FTIR measurements extrapolated to 20 min equal concentration in mg/m^3
A	HF filter	Unprotected	<5.0	0.7
		Dry fire hood	<5.0	
		Wet fire hood	<5.0	
B	HF filter	Unprotected	16	15.3
		Dry fire hood	9.0	
		Wet fire hood	<5.0	
C + D	HF filter	Unprotected	16	11.5
		Dry fire hood	8.6	
		Wet fire hood	<5.0	
Exposed to all experiments	HF filter	Unprotected	28	27.5
		Dry fire hood	25	
		Wet fire hood	12	
	Sum of bottom surface contamination 100 cm^2	Unprotected	219.3	
	Sum of ceiling surface contamination 100 cm^2	Unprotected	85.3	
	PVC swab	Dry fire hood	<1.0	
	Wet fire hood	<1.0		

The HF filters show clear penetration values of the fire hood while the HF values of the PVC surface remain below the detection limit (Table 3). The difference between the measurement results of HF filters and PVC surface can be explained by the fact that:

1. Gaseous HF penetrates through the fire hood while fluoride-containing particles (aerosol and soot) are retained.
2. The receiving medium influences the quantity of absorbed HF. A resistant medium such as PVC does not or hardly bind HF, whereas an absorbent medium binds HF to a large extent.

The difference between the two measurement results shows that fluoride-containing particles (aerosol) in the smoke do not or hardly pass through the particle barrier of fire hoods. If this were the case, fluoride-containing components should be demonstrably present on the PVC surface. The test results of the fire hood in experiments C + D match with the relative low HF concentrations in experiment C. If HF concentrations in experiment C exceeded $80\text{mg}/\text{m}^3$, higher values in the HF filter would be expected.

A dry fire hood allows more HF to pass through in comparison with a wet fire hood with the same exposure. However, a higher accumulation of harmful substances is detected in the wet fire hood. It is therefore not certain whether a wet fire hood offers better protection than a dry fire hood. In addition, the functionality of the particle barrier and the transfer of harmful substances to human skin from a wet fire hood may be different compared with a dry fire hood. This difference was not investigated.

For toxicologic purposes, the worst-case HF contamination scenario of $220\mu\text{g}$ for 100cm^2 skin surface can be derived from Table 3 during 20 minutes of exposure time at $27.5\text{ mg HF}/\text{m}^3$. In this worst-case scenario, the fire hood is assumed to offer no protection and all detected F^- ions originate from HF.

After the experiments, a piece of 10x10 cm was cut out of the two fire hoods which protected the PVC pipe. The pieces were weighed and submerged in an 8mmol Na₂CO₃ and 1 mmol NaHCO₃ solution. The liquid was analysed with ion chromatography and ICP-MS. The added value of the particle barrier disappears by immersing the fire hood sample in the solution. As particles are retained by the particle barrier, exposure to substances in tables 4 and 5 are not necessarily an important issue when these occur as aerosol in smoke.

Table 4 Results of ion chromatography of the solution in which the fire hood was immersed. Anions are reported as acids.

Piece of fire hood 10 x 10 cm exposed to all experiments	Hydro fluoric acid µg	Phosphoric acid µg	Nitric acid µg	Hydro chloric acid µg
Wet fire hood	223	208	765	1070
Dry fire hood	212	112	118	518

Table 5 Results of ICP-MS analysis of the solution in which the fire hood was immersed.

Piece of fire hood 10 x 10 cm exposed to all experiments	Li µg	Al µg	Mn µg	Ni µg	Co µg	Cu µg
Wet fire hood	316	196	72	126	16	1077
Dry fire hood	156	101	65	52	7	855

Detection devices for HF in smoke from Li-ion fires

HF gas sampling tubes were tested in two different types: tubes based on the reaction with zirconium hydroxide (not cross sensitive) and tubes based on the reaction of the H⁺ of the acid (cross sensitive to other acids). During these tests, the air humidity levels of 80-100% were beyond required operation conditions <80%. As a result, colour change of the tube appeared in some cases after finishing the measurement, leading to oversaturation of the tube. The dense smoke of tests B and D led (like the gas washing bottles) to clogged tubes. Tube sampling could take several minutes and an increasing time between pumps appeared to be an indication of unreliable measurement results.

During another performed experiment with smoke with air humidity rates of 30% and a temperature of 30°C, gas sampling tubes provided reasonable results compared to FTIR analysis [4]. The reading of the zirconium tube could in this experiment be complicated as sharp lines on the tube did not appear during every test. The other tube (based on the reaction of the H⁺ proton), provided overestimated HF (weak acid) levels since smoke also contained HCl (strong acid) because the tube was calibrated for weak acids.

Fluoride test paper was demonstrated to provide a useful indication of fluoride contamination in smoke and deposit. False-negative fluoride readings were observed in the caustic cooling water of the burned batteries. Fluoride test paper should be acidified before use for more precise and faster reading. However, in HF containing smoke, a colour change was also clearly observed in various experiments when fluoride paper was not acidified in advance. Moistening the paper with water or a 0.1 M HCl solution led to initial colour change around the edges of the paper in concentrations <20ppm within 1 minute while the dry paper showed a more equal and less clear colour change. In elevated HF airborne concentrations, faster and equal colour change is observed, also in wet

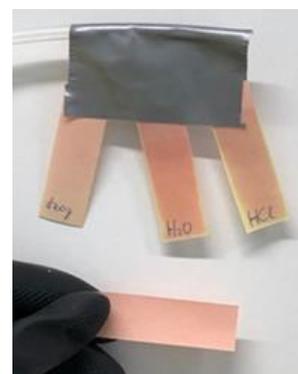


Figure 4 Fluoride detection paper exposed to HF vapours in a fume hood. The paper on the left was exposed in dry conditions, the paper in the centre was moistened with H₂O, and the paper on the right with HCl solution. At the bottom is a non-exposed paper. Fluoride papers exposed to HF containing smoke show an identical colour change but might be blackened by smoke particles.

fluoride testing paper. Paper moistened with HCl solution generally provides faster, sharper and clearer colour change.

Toxicologic consequences of HF gas exposure

Since HF is one of the specific combustion products of fires with Li-ion batteries, first responders should be aware of the risks involved in exposure to HF gas or hydrofluoric acid (the solution of HF in water) that can arise in moistened fire hoods or skin. HF is a weak acidic (pKa 3.19) and apolar molecule which dissolves quite easily in both water and organic solvents. This combination of physical-chemical properties underlies the potentially severe health effects associated with exposure to HF. Health effects may occur via different routes of exposure and are primarily caused by the corrosive effects of HF. Inhalation of HF gas may result in severe pulmonary problems. Mild symptoms include coughing and sore throat, while more severe symptoms consist of dyspnoea, bronchospasms, stridor, asphyxia and lung oedema. The severity of effects depends on both the HF concentration and exposure time (Table 6). Skin exposures to HF, usually in the form of hydrofluoric acid, may lead to local tissue damage. Due to its apolar nature, HF penetrates the skin more deeply compared with other acids (permeability coefficient 1.4×10^{-4} cm/s [3]). As a consequence, local skin damage after HF exposure usually starts mildly, with irritation. Subsequently, after a latency period, severe pain occurs, sometimes without visible skin defects. Oedema, characteristic white skin discolouration and deep tissue necrosis may follow. Again, the HF concentration and exposure time are important determinants in the severity of tissue damage (Table 6).

Table 6 Relation between concentration of a solution of hydrofluoric acid, exposure time of the skin and local effects.

Concentration HF in solution		Exposure time	Effect
Vol %	g/L		
0.01%	0.1	5 minutes	Visible skin damage (rabbit) [2]
0.03%	0.3	long lasting	Microscopically visible skin damage
<1%	10	long lasting	No pain, no clear corrosive effects
2%	20	1 minute	No skin damage (rabbit) [2]
1-5%	okt-50	short term	Local redness, pain hours later, no clear corrosive effects
>5%	>50	short term	Corrosive effects may occur
6-11%	60-110		Follow-up study of 237 patients exposed to 6-11% HF. Around 50% of the patients showed redness and oedema, 5% showed blisters, 23% only experienced pain
15-20%	150-200	short term	Severe pain (after a latency period), delayed occurring of corrosive effects
50-70%	500-700		Immediate pain, fast occurring of corrosive effects

Table 7 Relation between concentration HF, time of exposure and inhalation effects

mg/m ³	ppm	Exposure time	symptoms
0.83	1	30 minutes	AEGL-1 (Acute Exposure Guideline Level-1)
26	32	3 minutes	Exposure of 2 unprotected volunteers: "uncomfortable" [3]
28	34	30 minutes	AEGL-2 (Acute Exposure Guideline Level-2)
50	60	1 minute	Exposure of 2 unprotected volunteers: Worse than uncomfortable, pain of mucous membranes, nasal cavity and eyes. No pain of the skin [3]
51	62	30 minutes	AEGL-3 (Acute Exposure Guideline Level-3)
100	120	1 minute	Exposure of 2 unprotected volunteers: pain of the skin, conjunctiva, respiratory irritation, sour taste. Highest concentration tolerable during 1 minute [3]

Systemic effects, i.e., effects occurring in tissues distant from the site of contact, may occur after HF exposures with higher absorbed doses of HF (~200 mg for an adult of 75 kg). Systemic effects are caused by the direct interaction of fluoride with calcium in tissue and blood. When HF penetrates the skin, entering underlying tissue, and HF is deprotonated, F⁻ ions interact, among others, with Ca²⁺ ions to form insoluble fluoride-calcium salts. As a consequence, free soluble Ca²⁺ ions, which are needed for several cellular processes, are withdrawn from tissues and the circulation, leading to hypocalcaemia. In turn, hypocalcaemia is associated with several clinical symptoms, of which the severity depends on the degree of hypocalcaemia. Symptoms include thirst, weakness, paranaesthesia, muscle cramps, spasms, convulsions and arrhythmia.

The main question during emergency responses is whether exposures to smoke from burning Li-ion batteries may cause health problems for firefighters. With respect to local effects, the main concern will be unprotected skin. In case of firefighters in PPE for fire-fighting, the skin underneath the fire hood is considered to be the most vulnerable part of the skin. Assuming the helmet provides full protection to the skin underneath, the skin surface that is only protected by fire hood is estimated to be 500cm². As was shown, fire hoods with particle barriers block fluorine-containing aerosols, but HF gas is able to diffuse through the material. According to the fire experiments, exposures of 27.5 mg/m³ during 20 minutes are possible during a fire of a Li-ion battery in an enclosed space. As may be appreciated based on Table 7, irritation or pain of the skin cannot be excluded at this concentration. When multiple battery modules with elevated HF mass flow burn simultaneously in a small space, as could be the case with fires of several electric vehicles, the HF concentration may exceed 1000 mg/m³ in worst case scenario's in enclosed spaces [6], indicating that immediate local effects on unprotected skin are expected during long responses with regular PPE for fire-fighting.

To gain more insight into the possible systemic effects after HF exposure, the total HF dose to which the unprotective skin was exposed was further analysed. HF exposure at the measured HF air concentration of 27.5 mg/m³ was 220 µg/100 cm² (Table 3). At a total exposed skin surface of 500 cm², the total exposure would amount to 1.1 mg HF. In case of an air concentration of 100 mg/m³, the concentration at which skin pain is expected after only short exposure, an extrapolated total skin exposure of $(100/27.5) * 1.1 \text{ mg} = 4.0 \text{ mg}$ would be expected. It is unlikely that the absorption of 4.0 mg HF will lead to systemic effects since mild effects are only suspected at an absorbed dose of ~200 mg for an adult. It is therefore expected that local skin damage will be the first revealing thread for fire responses in standard PPE.

The severity of the initial local skin effect depends on the concentration of HF. In addition, a prolonged exposure time will also result in more severe effects like increased skin damage. Adequate and rapid skin decontamination after HF exposure is an important measure to reduce the impact of the exposure.

Conclusion and Discussion

The performed experiments demonstrated a considerable decrease of HF concentrations over time during a Li-ion battery fire in an enclosed space, leading to relevant changes in smoke consistency. The concentration decrease may depend on many factors, including relative humidity, smoke density and smoke consistency, but their relation is not clear yet. More research is necessary to define the circumstances that impact the speed of concentration decrease.

The HF concentration decrease over time leads in general to lower concentrations of HF in the smoke, but at the same time results in an increase in pollution of surfaces exposed to the smoke. The total amount of disappeared HF as found in the experiments with FTIR and the gas washing bottles corresponded roughly with the estimated total detected surface contamination.

In experiment C, considerably larger amounts of HF were measured with the gas washing bottles (86.5 mg/m^3) compared to the FTIR data (3ppm). High surface contamination levels, probably mainly derived from experiment C, were observed in bag 3, which is in line with the high fluoride concentrations detected with gas washing bottles. The HF filters used in experiments C+D have similar amounts of detected HF compared with experiment B, despite the fact that the filters were exposed in 2 experiments. This suggests that the HF concentrations in experiment C were low compared with the other experiments according to the HF filter measurements in the fire hood. This suggestion was further supported by the low FTIR results. This in turn, suggests that the gas washing bottle method is not completely specific for HF, but also measures other dissolvable fluoride containing compounds. The tested NMC batteries are likely to release high amounts of fluoride-containing compounds other than HF.

The experience with the handheld detection means demonstrated that fluoride detection paper is an easy-to-use method to quickly assess the presence of fluoride in the air. For clear interpretation of the colour change, a comparison with a non-exposed fluoride detection paper is recommended. Elevated exposure times of fluoride paper in HF containing smoke are best avoided since blackening of the paper might affect the visibility of the colour change. Moreover, the paper will also change colour when exposed for a long time to low concentrations of HF which are not necessarily harmful to firefighters in regular PPE for fire-fighting. In the tested smoke, acidifying the detection paper was not required to observe a colour change.

During the burning of NMC Li-ion batteries, large amounts of lithium (hydr)oxide (LiOH) were formed. This suggests that acids, originating from smoke, in moist fire hoods may be neutralised by LiOH, resulting in higher (neutral or even caustic) pH values. Indeed, measurements of fire hood moist after the experiments showed a neutral pH instead of acidic pH values. At higher pH values, the dissociation equilibrium of HF is shifted towards H^+ and F^- ions (dissociated HF). Since dissociated HF is unable to penetrate the skin, the pH increase caused by LiOH in smoke may prevent absorption of HF from the fire hood. Detecting the pH levels and fluoride presence in the fire hood after responses may be a useful assessment to estimate the likeliness of possible skin damage by HF. At low pH levels and a positive response of the fluoride paper, skin absorption of HF may be more likely.

The health effects after exposure to HF depend on both the concentration of HF and the duration of exposure. This research has shown that it is likely that short-term exposure to HF containing smoke after Li-ion battery fires may lead to local effects of the skin (irritation, pain). Rapid decontamination and rinsing of the skin (+/-30 minutes, lukewarm water) after exposure are essential to reduce health effects. Calcium gluconate gel may be applied to reduce further skin absorption of residual HF. Rapid availability of this gel is, therefore, important after responses with risks of severe HF exposure. When calcium gluconate gel is applied, a shorter rinsing time (+/- 5 minutes) may be applied.

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