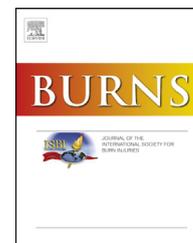


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Assessment of dermal hazard from acid burns with fire retardant garments in a full-size simulation of an engulfment flash fire



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ABSTRACT

There have been concerns that fire-derived acid gases could aggravate thermal burns for individuals wearing synthetic flame retardant garments. A comparative risk assessment was performed on three commercial flame retardant materials with regard to relative hazards associated with acidic combustion gases to skin during a full engulfment flash fire event. The tests were performed in accordance with ASTM F1930 and ISO 13506: Standard Test Method for Evaluation of Flame Resistant Clothing for Protection against Fire Simulations Using an Instrumented Manikin. Three fire retardant textiles were tested: an FR treated cotton/nylon blend, a low Protex[®] modacrylic blend, and a medium Protex[®] modacrylic blend. The materials, in the form of whole body coveralls, were subjected to propane-fired flash conditions of 84 kW/m² in a full sized simulator for a duration of either 3 or 4 s. Ion traps consisting of wetted sodium carbonate-impregnated cellulose in Teflon holders were placed on the chest and back both above and under the standard undergarments. The ion traps remained in position from the time of ignition until 5 min post ignition. Results indicated that acid deposition did increase with modacrylic content from 0.9 μmol/cm² for the cotton/nylon, to 12 μmol/cm² for the medium modacrylic blend. The source of the acidity was dominated by hydrogen chloride. Discoloration was inversely proportional to the amount of acid collected on the traps. A risk assessment was performed on the potential adverse impact of acid gases on both the skin and open wounds. The results indicated that the deposition and dissolution of the acid gases in surficial fluid media (perspiration and blood plasma) resulted in an increase in acidity, but not sufficient to induce irritation/skin corrosion or to cause necrosis in open third degree burns.

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1. Introduction

Concern has been voiced in the fire protection industry that protective textiles composed of synthetic fibers may represent a health hazard as the result of the gaseous acidic combustion products. Several bench top studies have looked at combustion gas generation from small areas of textiles and have reported the generation of hydrogen chloride, hydrogen cyanide, nitric oxides, hydrogen sulfur oxides, and other combustion constituents coming from flame retardant products containing Protex[®] modacrylic and nylon fibers [1,2]. A recent real-time analysis of gases generated during the full size engulfment simulations reported that synthetic flame retardant materials produced high concentrations of hydrogen chloride and cyanide with nominal amounts of nitrogen oxides and sulfur oxides [3]. However, this was not an issue as a toxicological inhalation hazard, because of the very high temperature and short duration of production [4]. Concerns remained that while the acidic gases may not represent an inhalation hazard, the ability of the gases to get trapped under the garments for prolonged periods of time might represent a hazard of dermal acid burns. Ackermann et al. [5] recently undertook a study where they concluded that acid deposition would certainly increase the severity of any burn received. Unfortunately, the authors did not directly examine the physiological implications of acid vapor exposure to substantiate their conclusion.

The purpose of this study was to address the issue as to whether acidic gaseous combustion products represent a potential dermal hazard to individuals exposed to a flash fire event. Using a full-sized simulator, three types of commercial fire retardant garments as full coveralls were exposed to propane-fired flash conditions of 84 kW/m² for either 3 or 4 s. Support manikins were fitted with sodium carbonate-impregnated cellulose ion traps to mimic skin deposition of dissociated acids. The results were used to determine both exposure and probability of adverse dermal injury to an individual who endures such an event.

2. Materials and methods

2.1. Experimental design and measurement systems

Three fire retardant garments were tested for gas emissions in a full sized flash simulation. Materials were in the form of standardized work coveralls. The garments tested were all commercially available and identified as follows:

- FR Cotton: Blended 88/12 Proban CCTM cotton blended with nylon
- Med. Modacrylic: A medium content blend containing Protex[®] modacrylic
- Low Modacrylic: A low content blend containing Protex[®] modacrylic

The ion traps consisted of 90 mm Watman #41 filter papers soaked in a solution of 1 M sodium carbonate (pH 10.3) for 12 h and then dried at room temperature under ultrapure nitrogen

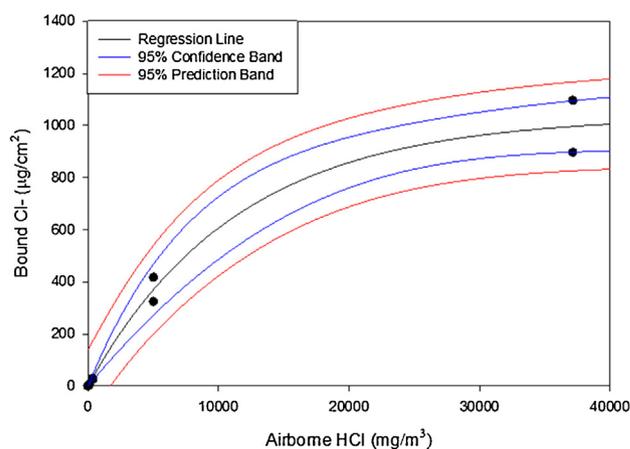


Fig. 1 – Ion trap calibration. Responsiveness of precision of the ion traps was determined by placing the ion traps in a controlled atmosphere of hydrogen chloride for 5 min.

for 6 h. The individual filters were placed in clean individual 100 mm petri dishes for storage before use.

The capacity of the ion traps was tested using a chamber of known atmospheric concentrations of hydrogen chloride. The ion traps were exposed to known concentrations for 5 min and then analyzed for chloride content. The relation showed an exponential rise to maximum ($r^2 = 0.983$) that was linear with the range of results attained in this study (Fig. 1).

The garments were pre-conditioned as required and then exposed to a simulated engulfment flash fire consistent with the requirements of ASTM F1930 [6]. As required, the manikin was dressed in 100% cotton t-shirt and briefs under the test work garment. Prior to testing, the ion traps were removed from the petri dishes, placed in clean Teflon holders, and wetted with deionized water. The holders were then affixed to the test manikin on the front and back of the trunk with one on the inside of the under garments, one on the outside (four total per test; Fig. 2). The traps outside of the underwear were intended to represent skin areas directly adjacent to the test garments. Two field blanks were included in the testing regime.

The full size fire exposure was performed in a 4.34 m by 3.78 m by 2.45 m high enclosure. The heat flux was provided by propane-fired jets with 12 burners on six stands. The system was calibrated to provide an average thermal input of 84 kW/m² (Table 1) and met the required standards of ASTM F1930-13.

Table 1 – Radiant energy calibration for the manikin used in the ASTM F1930 fire exposures.

Location group	No. of sensors	Mean	Minimum	Maximum	Std dev.
Head	8	2.079	1.384	2.799	0.428
Left arm	10	1.935	1.416	2.232	0.262
Right arm	10	2.042	1.605	2.477	0.296
Left leg	22	1.979	1.687	2.228	0.136
Right leg	22	1.956	1.579	2.278	0.159
Chest and abdomen	28	2.063	1.593	2.509	0.251
Back	22	1.988	1.639	2.465	0.201

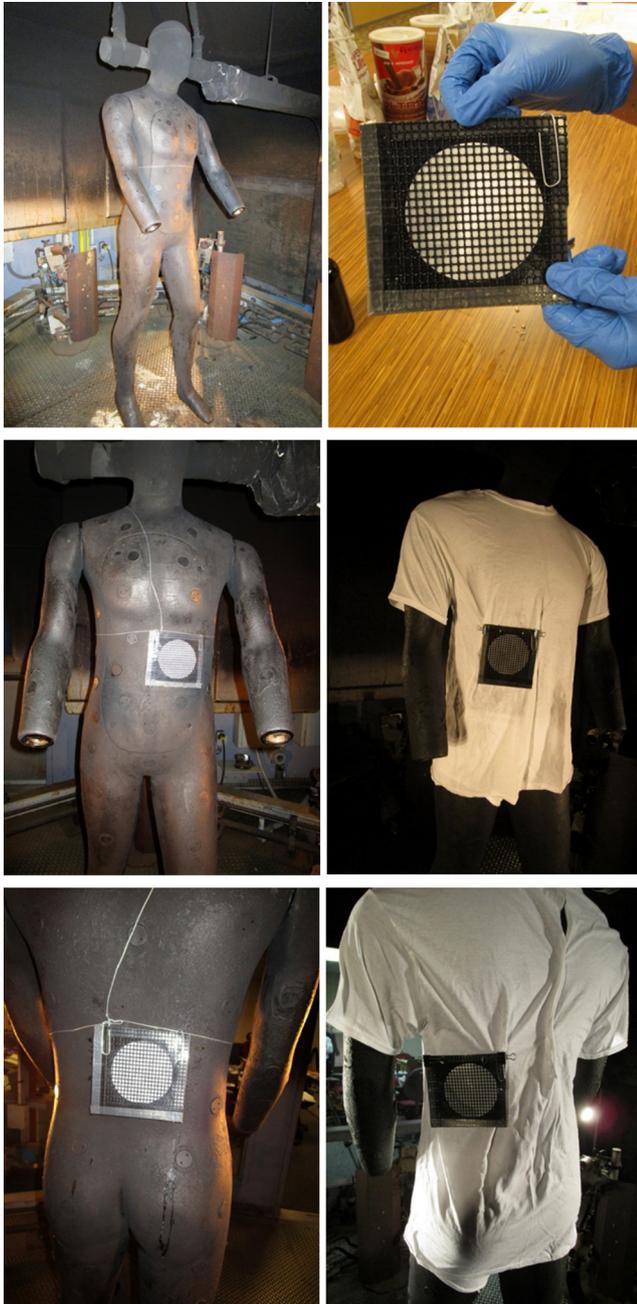


Fig. 2 – Test procedure used in this dermal deposition study. Top-left: test facility used for the ASTM F1930 Standard Test Method for Evaluation of Flame Resistant Clothing for Protection against Fire Simulations Using an Instrumented Manikin Standard Test Method for Evaluation of Flame Resistant Clothing for Protection against Fire Simulations Using an Instrumented Manikin. Top-right: ion trap within its holder. Middle: Placement of the ion traps on the front of the trunk both inside (left) and outside (right) of the undergarments. Bottom: placement of the ion traps on the back of the trunk both inside (left) and outside (right) of the undergarments.

All materials were tested at fire exposure durations of 4 ± 0.2 and 3 ± 0.2 s. The flame retardant garments were tested in a random order.

After fire treatment, the garments and ion traps were left in place for a period of 5 min post ignition. During this time, no ventilation was applied. After the 5-min period, the room was ventilated and the ion traps were removed from the Teflon holders and placed in their individual petri dishes for delivery to the laboratory.

The ion traps were analyzed by Exova Laboratories in Santa Fe Springs, CA. Ion traps were removed from the sealed protective petri dishes, placed in plastic bottles, and extracted in 100 mL of water with sonication for 1 h. The extracts were then diluted to a minimum of 1:10 with water and analyzed for chloride, nitrate/nitrite and sulfate/sulfite by ion liquid chromatography in accordance with method EPA 300.0 [7]. Cyanide content of the eluents was determined by EPA Method 9013A [8].

The detection limits are adjusted for the minimum dilution for analysis. Further dilution with water was necessary to bring some of the results for hydrochloric acid into the range of the calibration. The result for nitrite on one sample required further dilution to avoid interference from the large chloride peak.

2.2. Dermal risk assessment

There have been no studies that have developed a dose response curve based on the exposure of either damaged or intact skin to acid gas vapors. However, a comprehensive study was undertaken by Bjornberg [9] where human subjects were exposed to aqueous hydrochloric acid in skin patches for 24 h. The degree of irritation/corrosion was evaluated using the primary dermal injury index (PDII) developed by Draize [10]. The PDII is a systemic method that is the sum of the erythema and edema scores (1–4) divided by the number of observation intervals. The result is a scale between 0 and 8 that

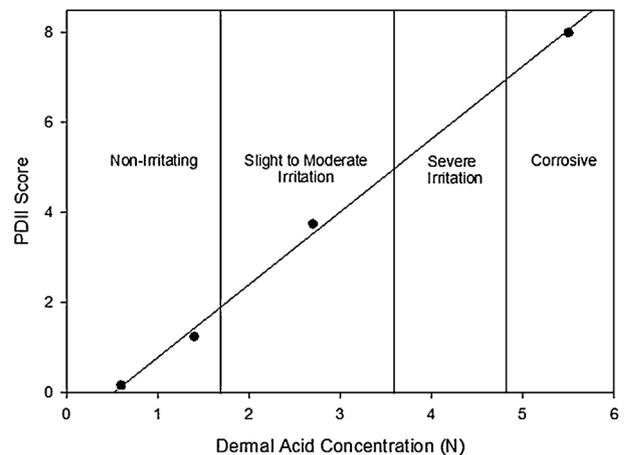


Fig. 3 – The relation between 24-h HCl exposure and adverse skin reaction on the primary dermal irritation index (PDII) based on the study of Bjornberg [9]. Classification of severity is based on the interpretation of the PDII by the USEPA's Office of Pollution Prevention and Toxics. [20].

gauges the irritation and damage to the dermal layers. Bjornberg's relation between the acid concentration and the PDII yielded a linear response between 0.1 and 5.5 N HCl, illustrated in Fig. 3 and described as follows:

$$(PDII) = 1.617 \cdot [H^+] - 0.833 \quad r^2 = 0.998 \quad 0.1 < [H^+] < 5.5 N$$

Because the assessment is based on exposure to acid gases and not aqueous acid solutions, an extra consideration of deposition must be taken into account. Acid gases produced in this study are highly hydrophilic and will dissolve and dissociate rapidly in water even at high concentrations. The acid concentration corresponding to the amount of acid gas absorbed is a function of the molar amount of acid deposited, the volume of fluid into which it is dissociated, and the buffer capacity of the fluid. For intact skin, the target of the acid toxicity is the viable cell layers in the basal lamella and reticular dermis. The minimum thickness of impact is best described as the thickness of the dermal layer which varies from 950 to 3900 μm [11]. Water content varies with the nature of the skin layers from 15–35% in the stratum corneum, rising to 65 to 70% in the basal reticulum and reticular dermis, respectively [12]. For considerations of surface hydration in the assessment of impact to intact skin, a model based on the projected pH of the perspiration was applied. Parameters on buffer capacity and volume of distribution are provided in Table 2. For the assessment, the gases were assumed to dissolve in 8.37 μl of perspiration per cm^2 of skin area. After accounting for the buffer capacity, the concentration was compared to the dose-response of Bjornberg [9].

For open wound exposure, the deposition was assumed to occur in plasma as opposed to perspiration. Research by Opie indicates that the level of acid exposure to intradermal tissue necessary to cause necrosis is between 0.1 and 1 μmol in 100 μl of water [13]. Hence, the no-effect level for cellular necrosis was assumed to be 0.01 μmol . The volume of 100 μl equates to an area of 1 cm^2 with a depth of 1 mm. This is within the range of dermal thickness (average of 770 μm) [11] and therefore is comparable to deposition and distribution of the gases within an injury. Effect of plasma buffer capacity was determined using the Figge-Fencel model [14].

3. Results and discussions

The amount of acid accumulation on the ion traps is detailed in Table 3. Regardless of the garment tested, the overwhelming

Table 2 – Values and sources for the determination of buffer capacities in biological receiving fluids.

Assessment scenario	Volume ($\mu\text{l}/\text{cm}^2$)	Buffer capacity	Reference
Perspiration	8.37	0.124 M/pH	Thurmon et al. [18]
Intercellular fluid (dermal)	76.6	$3.83[H^+] - 4.4.186$	Zheng and Maibach [19]
Injured tissue	100	$\text{pH} = 31.08[H^+] + 7.4$	Figge-Fencel [14]

source of acidity was gaseous hydrogen chloride. Hydrogen cyanide represented a significant portion of the acid gases only for the FR cotton/nylon blend and that was most attributable to very low hydrogen chloride production rates. For the Protex[®] modacrylic garments, the amount of hydrogen chloride produced was proportional to the amount of modacrylic in the blend. The amount of hydrogen cyanide relative to hydrogen chloride was less than 1%. This is consistent with past studies that examined the composition of off-gases from these types of garments [4]. Very little nitrate/nitrite or sulfate/sulfite was detected in the ion traps. Past studies suggest that these are only minor acid constituents and are the result of gases given off during the burning of these types of fire retardant blends. The total average acid gas concentration under the garments varied based on the material tested. Generally, the higher the modacrylic content and the longer the burn period, the higher the acid gas generation (expressed as HCl equivalents) with outer levels being higher than inner levels (Fig. 4).

The process of thermal skin damage is a process of biochemical oxidation, protein denaturation, and cellular necrosis. The process follows the thermal gradient from the surface epidermis, causing cellular damage in the basal layer (first degree or superficial burns), through the damage and necrosis of the underlying dermal layer (second degree, medium, partial thickness, or deep partial thickness burns), then the ablation of the dermis and damage to the underlying muscle and connective tissue (third degree or full thickness burns) and finally damage and necrosis of organ and bone tissue (fourth degree or progressive full thickness burns). It is within this context of the thermal burn process that the impact of acid gas vapors is considered, because the severity of the thermal burn dictates the type of biological fluid that are available for contact by the acid vapors and the types of adverse effects that are likely to occur. In the superficial or partial thickness burns, the dermal layer initially remains intact although the layer is usually sloughed off subsequent to this injury (after acid gas exposure) with partial thickness burns. Therefore, the contact point between the environmental acid gases and the individual is the dermal layer. Blistering may impact the efficacy of acid depositions, but this is not expected to be significant within the timeframe considered in this assessment and would likely reduce the potential hazard by diluting any acid depositions that were to occur. Hence, for consideration of first and second degree burns, it is assumed conservatively that the acid deposition occurs on intact dermis.

The outer epidermal surface is normally an acid environment with a pH between 5.5 and 4.5. [15]. This is due to the selective secretion of lactic acid and free amino acids, which are typically found in concentrations 10 times greater in dermal exudates than in plasma or intercellular fluids [16]. In order to predict the deposition of acidity on the skin's surface, we assume that the stratum corneum is hydrated. To assume otherwise would result in an environment where the acid gases could not depositing on the skin relative to their airborne concentration. Therefore, this assumption is conservative. The assessment for the potential impact of acid deposition on intact skin indicates that the added acid content to epithelial perspiration ranged from negligible (i.e., fully buffered) to

Table 3 – Acid content measured on the ion traps. Ion traps located above (Out) and under (In) the cotton undergarments were analyzed for chloride (HCL), cyanide (HCN), nitrate/nitrite (NO_x), and sulfate/sulfite (H_xSO_y) content. Total concentrations on the filters were standardized on the single side surface area of the ion traps. Values are a composite of filters located on the front and back of the manikin.

Test material	Loc. ^a	Duration (s)	H ⁺ μmol/cm ^{2b}				Total
			HCL	HCN	NO _x	H _x SO _y	
Field Blanks			(0.0127)	(1.14 × 10 ⁻⁵)	0.0131	(0.00627)	0.0289
FR Cotton/Nylon	In	3	0.0253	0.00202	0.0131	0.00784	0.0482
FR Cotton/Nylon	Out	3	0.0190	0.00176	0.0131	0.00627	0.0401
FR Cotton/Nylon	In	4	0.0127	0.00543	0.0131	0.00941	0.0406
FR Cotton/Nylon	Out	4	0.0232	0.01075	0.0131	0.01411	0.0611
Med Modacrylic	In	3	0.295	(1.14 × 10 ⁻⁵)	0.0131	0.00627	0.315
Med Modacrylic	Out	3	1.22	0.01559	0.0131	0.00627	1.26
Med Modacrylic	In	4	4.93	0.02091	0.0131	0.00627	4.97
Med Modacrylic	Out	4	12.4	0.00361	0.0164	0.00627	12.4
Low Modacrylic	In	3	0.422	0.00222	0.0131	0.00627	0.443
Low Modacrylic	Out	3	1.81	0.00122	0.0131	0.00627	1.83
Low Modacrylic	In	4	3.92	0.01812	0.0131	0.00627	3.96
Low Modacrylic	Out	4	7.63	0.00913	0.0131	0.00627	7.66

^a Location of the ion trap being either between the manikin and the underwear (In) or between the underwear and the test garment (Out).
^b Deposition concentration on the ion traps. Values in parenthesis were below the limit of quantification and assumed to be at that limit. Values less than the field blanks were assumed to have a value equal to the field blanks in the determination of the total acid deposition.

1.35 N (Fig. 5). This equates to a PDII range of 0–1.75 based on the Bjornberg relation. The threshold between no irritation and slight to moderate irritation for acid deposition is 1.75 N, and the threshold for cellular necrosis and corrosion is 4.8 N. The highest observed acid skin concentrations (Medium Protex[®] modacrylic blend, 4 s, outside undergarments) was not sufficient to cause irritation and was more than 3.5 times lower than necessary concentrations to induce corrosion. Therefore, when considering unburned, or intact, skin having suffered first or second degree burns, the acid deposition is insufficient to either intensify pain perception, or contribute to the heat-induced dermal damage.

In full thickness burns, the epidermal and dermal layers are no longer intact and as such the atmospheric gases will be contacting damaged sub-dermal tissues. In these situations, acid gases will not dissolve and dissociate in dermal exudate such as perspiration, but rather in a mixture of blood, plasma, and intercellular fluids. Irritation is not an issue in such injuries because the nociceptors responsible for pain signaling

are destroyed at this level of burn severity [17]. Therefore, the principal concern in the aggravation of full thickness burns is the promotion of damage and the inhibition of repair through attributable cellular necrosis.

Early studies by Opie showed that the introduction of 100 μl of 0.01 M (1 μmol/cm²) hydrochloric acid to the sub-dermal layers of rats induced cellular necrosis within 24 h. However, introduction of 0.001 M HCl (0.1 μmol/cm²) had no effect. Using plasma as the dissolving medium for the acid gases produced in the burns suggests a range in the final plasma acid content between negligible (fully buffered) at 4.04 × 10⁻⁸ to 2.8 × 10⁻⁴ μmol/cm² (Fig. 6). This maximum amount of acid deposition for the projected was more than 350 times less than the no-effect threshold reported by Opie [13].

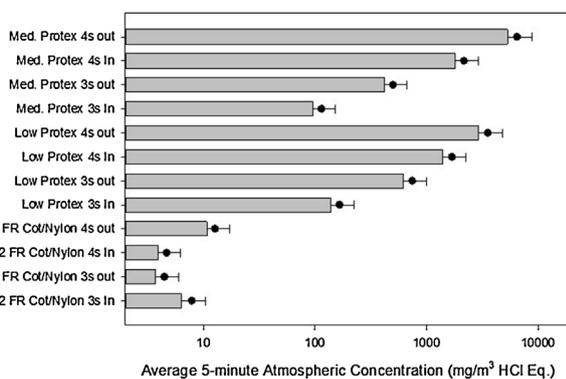


Fig. 4 – Projection of acid gas concentrations under the garments during the 5-minute burn testing period. Bars and whiskers represent the upper/lower 95% UCL for prediction with the circles (●) the mean of the estimate.

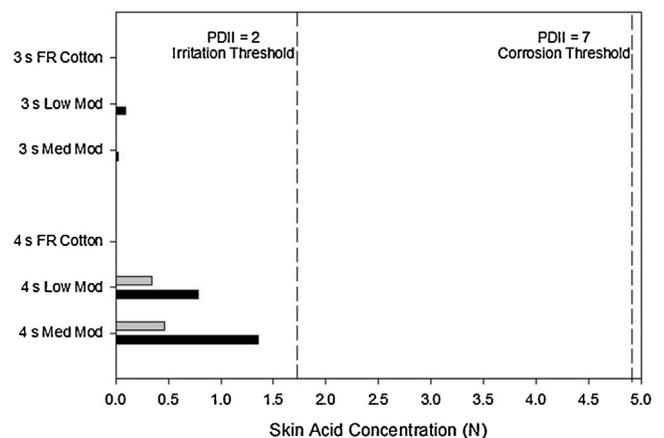


Fig. 5 – Acid concentrations within the perspiration-hydrated epidermis after exposure to acid gases. Bars represent the range between the traps inside and outside of the undergarments for the respective garment material and test duration. Vertical dashed lines represent the corrosion threshold (PDII = 7; [H⁺] = 4.88 N) and pain threshold (PDII = 2; [H⁺] = 2.75 N) based on the regression of the data of Bjornberg [9].

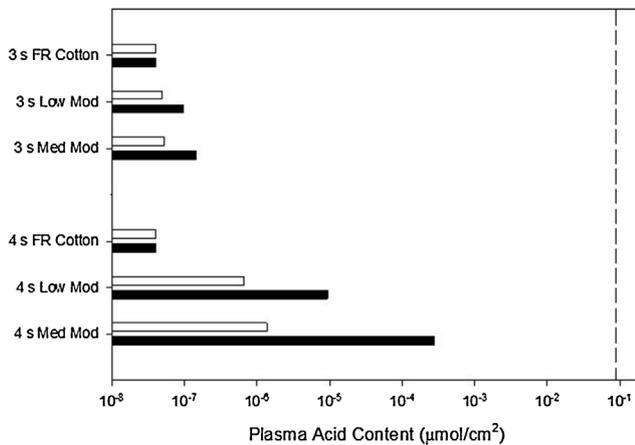


Fig. 6 – Acid concentration in an open subdermal wound hydrated with blood plasma. Bars represent the acid content range between the traps inside and outside the undergarments for the respective garment material and test duration. The vertical dashed line represents the threshold concentration of the no-effect level for tissue necrosis base on the results of Opie [13].

In order to ensure that the assessment does not under-predict the potential hazards, levels of compounding conservative assumptions were used to counter any identified uncertainty in the exposure or effects characterization components of this assessment. First, the conditions of the test were consistent with the standards of ASTM F1930 and as such represent a very extreme, worst case, borderline survivable event. Thus, it is more likely that an individual relying on these types of garments for fire protection will encounter significantly lower heat flux rates and thereby lower levels of off-gassing from the FR garments. Hence, the ability to show a lack of adverse effect under these extreme conditions indicates the large margin of safety between acid gas production rates and dermal susceptibility to injury. Additionally, the volume of perspiration and plasma were absolute minimal values. The perspiration volume was taken as the minimal potential hydration passed on average epidermal profiles. When compared to the method of Bjornberg who used wetted patches of acid in saline, reliance on the volume of water in the epithelium dramatically increased the prediction of acid concentrations, since acid concentration is inversely proportional to the volume of the receiving medium. In the case of the third degree burn assessment, equal volumes were assumed and the assessment was performed on a per area basis. Furthermore, in the cases of both the Bjornberg and Opie thresholds, the observed adverse effects were minimal and not likely to have resulted in significant attribution on injury. For Bjornberg, the endpoint was slight to moderate irritation. For Opie, it was necrotic lesions less than 5 mm in diameter. Finally, both studies considered injury after 24 h of continuous exposure whereby the exposure scenario assessed here involved prediction of acute impacts during and shortly after a 5-min exposure. For these reasons, it is with confidence that we conclude that a finding of no potential adverse impact is representative and conservative.

4. Conclusions

Gases produced during the flash fire combustion of the Protex[®] modacrylic garments will result in the production of acid gases, particularly hydrogen chloride. Prolonged exposure to high concentrations of acid gases could cause adverse impacts to exposed skin. However, where such garments were subjected to the most extreme conditions of 84 kW/m² for even 4 s, it was found that the amount of acid gas produced was insufficient to represent a risk of incurred dermal acid burns. Therefore, it is apparent from the results of this study and assessment, and in contradiction with the position of Ackerman et al. [5], that acid burns or complications resulting from dermal exposure to combustion gases does not represent a demonstrable health risk to individuals wearing such garments.

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