AFRL-RX-TY-TP-2009-4565

PREPRINT

ANALYSIS OF CHEMICAL OFF-GASSING FROM FILTERING FACEPIECE RESPIRATORS AFTER DECONTAMINATION

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AUGUST 2009

This document was submitted to Springer for publication in the Journal of Material Science.

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REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

PLEASE DO NOT RETURN YOUR FORM TO THE ABOVE ADDRESS.

1. REPORT DATE *(DD-MM-YYYY)*	2. REPORT TYPE	3. DATES COVERED *(From - To)*
31-AUG-2009	Journal Article PREPRINT	01-FEB-2008 -- 01-AUG-2009

4. TITLE AND SUBTITLE

Analysis of Chemical Off-Gassing from Filtering Facepiece Respirators after Decontamination (PREPRINT)

5a. CONTRACT NUMBER

FA4819-07-D-0001

5b. GRANT NUMBER

5c. PROGRAM ELEMENT NUMBER

99999F

6. AUTHOR(S)

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5d. PROJECT NUMBER

DODT

5e. TASK NUMBER

L0

5f. WORK UNIT NUMBER

Q220L7AD

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)

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8. PERFORMING ORGANIZATION REPORT NUMBER

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)

**Air Force Research Laboratory
Materials and Manufacturing Directorate
Airbase Technologies Division
139 Barnes Drive, Suite 2
Tyndall Air Force Base, FL 32403-5323

10. SPONSOR/MONITOR'S ACRONYM(S)

AFRL/RXQL

11. SPONSOR/MONITOR'S REPORT NUMBER(S)

AFRL-RX-TY-TP-2009-4565

12. DISTRIBUTION/AVAILABILITY STATEMENT

Distribution Statement A: Approved for public release; distribution unlimited.

13. SUPPLEMENTARY NOTES

Ref AFRL/RXQ Public Affairs Case # 09-139. Submitted to Springer for publication in the Journal of Material Science. Document contains color images.

14. ABSTRACT

A major concern among healthcare experts is a shortage of supplies during a pandemic. An item of particular interest is the N95 filtering facepiece respirator (FFR), which is responsible for protecting individuals from infectious aerosols. Most experts agree there will be a shortage of N95 FFRs if a severe pandemic occurs and one option for mitigating an FFR shortage is to decontaminate and reuse the devices. Many parameters must be studied to verify the effectiveness of this strategy: biocidal efficacy of the decontamination treatment, filtration performance, pressure drop, fit, and toxicity to the end user post treatment. The focus of this research effort was to measure chemical off-gassing of six types of FFRs following decontamination. Our data indicate that for disinfectants, such as hydrogen peroxide and bleach, the amount of residual decontaminants is below the Permissible Exposure Limit (PEL). Toxic by-products were also evaluated, and they were detected for ethylene oxide treatment of FFR rubber straps. These data are encouraging and may contribute to the evolution of effective strategies for decontamination and reuse of FFRs.

15. SUBJECT TERMS

pandemic, respirator, infectious, aerosols, H1N1, decontaminate

16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT	18. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON
a. REPORT	b. ABSTRACT	c. THIS PAGE			Joseph D. Wander
U	U	U	UU	24	19b. TELEPHONE NUMBER *(Include area code)*

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Standard Form 298 (Rev. 8/98)
Prescribed by ANSI Std. Z39.18

Analysis of Chemical Off-Gassing from Filtering Facepiece Respirators after Decontamination

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Abstract

A major concern among healthcare experts is a shortage of supplies during a pandemic. An item of particular interest is the N95 filtering facepiece respirator (FFR), which is responsible for protecting individuals from infectious aerosols. Most experts agree there will be a shortage of N95 FFRs if a severe pandemic occurs and one option for mitigating an FFR shortage is to decontaminate and reuse the devices. Many parameters must be studied to verify the effectiveness of this strategy: biocidal efficacy of the decontamination treatment, filtration performance, pressure drop, fit, and toxicity to the end user post treatment. The focus of this research effort was to measure chemical off-gassing of six types of FFRs following decontamination. Our data indicate that for disinfectants, such as hydrogen peroxide and bleach, the amount of residual decontaminants is below the Permissible Exposure Limit (PEL). Toxic by-products were also evaluated, and they were detected for ethylene oxide treatment of FFR rubber straps. These data are encouraging and may contribute to the evolution of effective strategies for decontamination and reuse of FFRs.

Introduction

Pandemic influenza outbreaks historically occur every 40 to 50 years. The last pandemic was the Hong Kong Flu in 1968 so the next cycle could be realized during the autumn flu season of 2009 if the spring outbreak of H1N1 "Swine Flu" reemerges in the more-virulent episode that health experts fear. A primary barrier used to protect healthcare workers and the general public from airborne infections is the National Institute for Occupational Safety and Health (NIOSH)-approved filtering facepiece respirator (N95 FFR; note – many types of FFRs are available. The focus of this report is the N95 FFRs. All further references to FFRs in this manuscript specify N95 FFRs). The FFR is rated to capture ≥ 95% of airborne particles and has been proven to remove infectious microorganisms from the air stream. [1 - 6] A looming concern among healthcare providers is the anticipated outbreak of an influenza pandemic. These fears were aggravated in the spring of 2009 with the onset of an H1N1 outbreak. [7, 8] On June 11, 2009, the World Health Organization (WHO) raised the pandemic alert level to six, which indicates the onset of a pandemic. WHO reported almost 30,000 confirmed cases of H1N1 and 145 deaths world wide as of June 12, 2009. [9] Over 13,000 cases and 27 deaths were reported in the United States. [9] While this outbreak did not have the severity of earlier pandemics, it is sufficiently similar to previous pandemics to merit concern. It is not certain that the current H1N1 strain can mutate into a more virulent strain, but healthcare workers are taking the possibility very seriously.

The modes for human-to-human transmission of influenza are actively debated [10-15], but there are data that support aerosol transmission. [10, 14] This information has led the Occupational Safety and Health Organization (OSHA) and the Centers for Disease Control (CDC) to recommend that workers wear a properly-fitted NIOSH-approved FFR during a pandemic influenza outbreak. [16, 17] To supply the general public with protection and help mitigate the 2009 H1N1 epidemic, the FDA issued an Emergency Use Authorization (EUA) that approved release of FFRs from the Strategic National Stockpile (SNS). [18] The Centers for Disease Control (CDC) estimates that during a pandemic lasting 42 days, over 90 million FFRs will be required for healthcare workers only. [19] These projections indicate that a shortage of FFRs

is likely to occur, which would leave healthcare workers exposed and might add to the severity of the pandemic.

A possible solution for alleviating an FFR shortage is to decontaminate and reuse the FFRs. [19] However, data describing the effect decontamination technologies have on the performance of FFRs are sparse. Filtration efficiency, pressure drop, fit, off-gassing of residual chemicals, and overall durability are key questions that must be addressed. NIOSH has performed limited studies that indicate that some technologies can be used to decontaminate FFRs without affecting performance. [20] However, other technologies, such as autoclaving, render the FFRs unusable. [20] These tests were performed on a limited number of FFR models, and more research is needed on a large number of FFRs to properly evaluate decontamination technologies. The Air Force Research Laboratory (AFRL) is currently leading an effort that examines the effects of several decontamination technologies on six commonly distributed models of FFRs from the SNS (Table I). The six models of FFRs represent both common particulate FFRs and those cleared by the FDA as medical devices. The focus of this report is chemical off-gassing following decontamination; the other parameters will be the focus of future reports.

Many technologies could be used for decontaminating FFRs; however, they are too numerous to permit an exhaustive evaluation. To narrow the scope, multiple characteristics of 10 diverse decontamination technologies selected for applicability in three scenarios—major hospital, small clinic, first-responder station—were evaluated: 1) Biocidal performance of the technology—historical data must demonstrate biocidal efficacy on surfaces; 2) Cost—single-use FFRs will be decontaminated only in the event of an FFR shortage caused by a pandemic influenza or similar disease, so it is impractical to allocate scarce resources to purchase specialized equipment; 3) Availability—commercially available technologies were the primary focus of this study; however, some emerging technologies were also considered. 4) FFR compatibility—many technologies were eliminated that were known to degrade the performance of FFRs. [20] Data describing how FFRs respond to decontaminants are sparse, but care was taken to select technologies that are not overly aggressive; and 5) End use—the decontamination technologies need to

provide useful solutions for end users ranging from very large hospitals to non-occupational users. Each end user will have different tolerances for throughput and regeneration time of the FFRs.

The decontamination technologies selected for this study comprise gaseous, energetic, and liquid agents (Table II). The large-scale/high-throughput technologies selected were vaporized hydrogen peroxide (VHP) and ethylene oxide (EO) sterilizers. The achievable throughput using these technologies is questionable, but since many hospitals already utilize these devices for low-heat sterilization they were a logical choice for evaluation in this study. Both VHP and EO sterilizers are relatively expensive technologies; however, organizations that own these devices have only a small burden of added operational costs. The medium-throughput devices selected for the study were energetic devices: conventional and microwave ovens routinely found in many organizations and homes, and ultraviolet (UV) light: UV devices for surface sterilization are commercially available (Ultra Violet Products, Upland, CA); however, distribution of these devices in hospitals and other clinical/first responder organizations is unknown. If ultraviolet irradiation was found to be useful for decontaminating FFRs, the technology might also have routine applications that could justify its acquisition by small or even large organizations.

The small-scale decontaminants were all aqueous solutions—bleach (diluted to 0.6% hypochlorite) and 3% hydrogen peroxide are common disinfectants that can be found in most homes in America. Mixed-oxidants and dimethyldioxirane (DMDO) were both developed as part of Department of Defense (DoD) projects, and represent emerging technologies that are not widely distributed.[21,22] They were included in this study in case both bleach and peroxide performed unsatisfactorily. The technologies of primary concern for off-gassing are the liquid and gaseous decontamination agents. Ultraviolet (UV) irradiation (at both 254 nm and 302 nm) was also analyzed for possible by-products from UV-initiated radical reactions. Since microwave and conventional ovens do not use chemicals, off-gassing analysis is not relevant.

The analytical methods for off-gassing analysis (Table III) were selected based on the chemical properties of each analyte. In most cases, head-space analysis using gas chromatography-mass spectrometry (GC-MS) would be the preferred method, as this would detect chemical compounds that were off-gassed from the FFR and likely to be respirable. EO was analyzed with this methodology using the guidance provided in ISO 10993-7 international standard for the biological evaluation of medical devices.[23] However, many of the disinfectants in this study are reactive and do not afford themselves to separation by GC. The hydrogen peroxide agents (VHP and 3% liquid), hypochlorite, and DMDO fall into this category. Also, whereas off-gassing is the primary concern, many of the decontaminants used for this study do not readily off-gas. The active species for bleach is a hypochlorite salt that will not elute on a GC column but can react with chloride to liberate chlorine as Cl_2 (g). In addition, aqueous solutions containing hypochlorite are very destructive to the stationary phase of the GC column. The mixed oxidants contain 6% sodium bicarbonate, 5% sodium chloride, and 10% potassium peroxymonosulfate (Oxone®). The initial oxidative capacity is provided by the Oxone®; however, it is also possible that Oxone® can react with NaCl to form Na^+ (aq) and ClO^-(aq). For the examples above, GC-MS would not be effective to quantify the chemicals. To quantify the amount of trace chemicals left on the FFR by these technologies, iodometric back titrations (IBTs) were carried out via the oxidation of sodium thiosulfate added to the samples. IBT is commonly used for quantifying oxidative capacity[24 - 26], and since all the chemical decontaminants used in this study are oxidative in nature, the use of IBT was appropriate. Pentane extractions of decontaminated FFRs were also conducted on specimens treated with any chemical disinfectant or with UV light to ensure that additional hazards were not created during the decontamination. Pentane is an organic solvent commonly used to extract volatile organic substances.

Materials and Methods

Liquid Decontaminants: Three FFRs of each model were submerged in liquid decontamination agents (Table II) in a chemical fume hood for 30 minutes at room temperature. A volume of 200 mL of

decontaminant per FFR was used. After the 30-minute soak, the FFRs were removed from the solutions, placed on trays, and allowed to off-gas for 18 hours in a chemical fume hood. Following the off-gassing period, ten 14-mm diameter samples were punched from areas equally spaced on each respirator and separately weighed in 20-mL glass scintillation vials. In addition, the straps, nose cushions, and metal nosepieces were cut into small pieces and separately weighed in scintillation vials. Iodometric back titrations were conducted as previously described. [24, 25] Three additional 14-mm samples were removed from each FFR and extracted with 10 mL of *n*-pentane for 3 hours. Extracts were analyzed using GC-MS as follows: 2-mL aliquots were added to standard GC vials, and separated on a GC with a Programmed Temperature Vaporization (PTV) injector in splitless mode. MS scans were taken from *m/z* 30.0–300.0 at 5 scans per second and a scan rate of 1807 (*m/z*)/*s*. Data were collected on a Thermo–Finnigan Trace GC (Thermo Scientific, Waltham, MA) fitted with a 30-m x 0.32-mm x 0.25-µm DB-5 column, using a Trace DSQ MS with a Leap Technologies CTC Combi PAL® autosampler. The ion source temperature was held at 225 °C and the detector gain was set to 1.0×10^5.

Gaseous Decontaminants: An Amsco Eagle 3017 Ethylene Oxide Sterilizer® was used to expose triplicate FFRs of each model to EO. The FFRs were packaged individually in sterilization pouches that contained sterilization indicator strips. The sterilization cycle was 3 hours at 54 °C, followed by a 12-hour aeration cycle at 54 °C. Following the off-gassing period, 14-mm diameter samples were punched from areas equally spaced on each respirator and weighed in a Supelco 20-mL headspace vial. In addition, the straps, nose cushions, and metal nosepieces were cut into smaller pieces and separately weighed in the headspace vials. GC-MS analysis for EO used guidance from the ISO standard AAMI/ANSI/ISO 10993-7. [23] Briefly, GC-MS analyses were carried out by headspace solid-phase micro extraction (HSSPME) with a PTV injector used as a desorber. The HSSPME fibers were Supelco 65-µm bonded phase polymethylsiloxane–divinylbenzene. The MS operated in scan mode from *m/z* 20.0–120.0 with 5 scans per second and a scan rate of 1807 (*m/z*)/s. For the HSSPME methods, an extraction time of 240 s was

used with a desorption time of 900 s. The GC temperature program began with a 40 °C isotherm for 4 min, followed by a ramp of 20 °C/min to 270 °C. The PTV injector was set to a base temperature of 250 °C, and the helium flow rate was 1.5 mL/min. Pentane extractions were also conducted as described in the liquid decontaminants section.

A STERRAD© 100S system was used to expose triplicate FFRs of each model to VHP. The FFRs were packaged individually in sterilization pouches that contained sterilization indicator strips. The sterilization cycle was 55 minutes at 45–55 °C. Following the sterilization cycle, 14-mm diameter samples were punched from areas equally spaced on each respirator and weighed in a 20-mL scintillation vial. In addition, the straps, nose cushions, and metal nosepieces were cut into smaller pieces and separately weighed in vials. Samples were analyzed by IBT and pentane extractions as described in the liquid decontaminants section.

Energetic Decontaminants: Triplicate 38-mm diameter circles were cut from each FFR model. A multi-wavelength, 8-watt lamp (Ultra Violet Products, Upland, CA), was used to expose triplicate samples of each FFR model to UV light. Samples were placed 1 inch from the lamp source and were irradiated with 4.0 mW/cm^2 of UV-B (302 nm) and 3.4 mW/cm^2 UV-C (254 nm) for 1 hour each. A UV meter (Ultra Violet Products, Upland, CA) was used to measure irradiance. After exposure, samples were weighed in 20-mL glass scintillation vials and extracted with pentane as described in the liquids decontaminants section.

Data Analysis

Iodometric back titration (IBT): The data retrieved from this assay are initially reported in mmol of oxidant per gram, which is converted to mg of oxidant by multiplying by the gram-molecular weight of the decontaminant applied to the FFR. The mmol of oxidant recovered on untreated FFRs cannot be converted into mg because the chemical identity of the native oxidant(s) is unknown. To correct the data for the native amount of oxidant on the FFRs, the average amount of oxidant (mmol/gram) quantified

from the untreated FFRs was subtracted from the treated samples. The final formula for determining mg of oxidant per FFR is described in Equation 1. The IBT assay can produce negative numbers, which have no physical significance. In those cases, the negative numbers were viewed as below detection limit and were excluded from the analysis. The value for the amount of oxidant present on each triplicate FFR was imported in to Prism-5® software (GraphPad, La Jolla, California) and 95% confidence intervals were calculated.

[Equation 1]: mg of oxidant per FFR

Equation (1) $\sum [(T - U) * W * G]_{pi}$

T = Treated mmol of oxidant per gram

U =Untreated mmol of oxidant per gram

W = Weight of FFR component in grams

G = Gram-molecular weight of the decontaminant

$pi = p1, p2, p3, p4$ = Different FFR parts (FFR respirator material, straps, nose cushion, metal nosepiece

Ethylene oxide HSSPME data analysis: The ISO standard for the biological evaluation of medical devices provided much of the guidance for this analysis. [23] The ISO method uses direct injections of headspace gas quantified by external calibration using the ideal gas law. For this experiment, headspace analysis by SPME fiber was acceptable since all recoveries of EO were presumed to be well below the OSHA permissible exposure limit (PEL) of 1 ppm.[27] FFRs treated with EO were analyzed by HSSPME GC-MS as described above. Chromatographic analysis was carried out by manual recognition of Gaussian zones at an approximate signal-to-noise ratio of 3:1 or greater. Signals recorded below this ratio were not considered. A detection limit study for EO was used to determine a reasonable threshold value for the technique. Aqueous standards of EO purchased from Accustandard (New Haven, CT) were serially diluted to obtain concentrations of 10 ppm, 5 ppm, 1 ppm (PEL), 500 ppb, 50 ppb, 5 ppb and 0.5 ppb. EO was found to elute at t_R=5.60 min with qualifying ions of *m/z* 44, 43, and 42. FFR samples were analyzed

over a window from 4.0–6.5 minutes to account for any variances in chromatography due to potential by-products from EO alkylation. The detection limit for EO by HSSPME GC-MS was 500 ppb (half of the PEL).

GC-MS *n*-pentane extraction data analysis: GC-MS analysis of the *n*-pentane extracts provided chromatograms for each treated sample plus an untreated sample. Peaks present in the untreated sample or the normal instrument background for pentane were subtracted from the treated samples. Peaks still present were selected for investigation based on a visual comparison against the background signals of the instrument and procedural materials. Peaks that exceeded a signal-to-noise ratio of 3:1 were analyzed using Xcalibur® software (Thermo Scientific, Waltham, MA). The software provided peak identification by comparing the acquired mass spectra to several spectral libraries contained within the software. The first match provided by the software is not always the best match for the spectrum in question; however, peaks were labeled as the first match when it was consistent with species present in the procedure.

RESULTS

Iodometric back titrations: The concentration of oxidant remaining on the FFRs varied depending on the FFR model and decontamination technology (Table IV). All FFRs treated with 3% hydrogen peroxide retained similar amounts of oxidant with the exception of the S3 FFR, which had no detectable oxidant. The S1, S2, P1 and P2 models treated with VHP retained ~3X more oxidant than the other two models. All FFR models treated with 10% bleach retained similar amounts of oxidant with the exception of the S3, which held no detectable amount of oxidant. The P2 FFR retained more oxidant than the others, but the data had large confidence intervals, which indicate the result is somewhat questionable. The same is true for the mixed-oxidant-treated P2 and P3 FFRs, which retained large quantities of oxidant compared to the other FFRs. The DMDO-treated FFRs stand out from the others because all FFR models retained ~5X more oxidant than their counterparts treated with the other disinfectants.

GC-MS analysis of *n*-pentane extracts: Many unique peaks were identified in the *n*-pentane extracts (data not shown); however, most of these were found in fewer than three FFRs, which suggests that they are random events unrelated to the disinfection technologies. Table V provides data for the unique peaks that were found in more than three of the 18 FFRs tested. For the chemical disinfection agents, a total of 11 unique peaks were identified, one a ubiquitous plasticizer and the remainder attributable to solvent contamination or column background. UV irradiation produced the greatest number of unique peaks; however, many of these appear to be constituents of the pentane solvent.

Ethylene oxide HSSPME results: EO was not directly detected in any of the respirators or respirator components tested (Table VI). The total ion chromatograms were reviewed over a window from 4.0–6.5 minutes because time to elution of EO itself gradually decreased from ~5.6 to 5.2 minutes as removal of contaminated sections at the front of the column decreased the working length of the GC column. Furthermore, this large time window accommodated variations in chromatography such as retention time shifts or peak fronting/tailing. Diacetone alcohol was found in 11 samples, 2-hydroxyethyl acetate appeared in 15 samples, and cyclohexanone was identified in 2 samples. The 15 occurrences of 2-hydroxyethyl acetate were all at or below the signal-to-noise ratio; however, the fact that they occurred so frequently and were strap-specific warrants mention.

Discussion

The presence of oxidant on the FFRs following decontamination was not surprising. The critical question, however, is whether enough decontaminant remained on the FFRs to cause health concerns to the user. The data collected were compared to NIOSH's recommended exposure (REL = time-weighted average [TWA] concentration for up to a 10-hour workday during a 40-hour workweek) and/or the Short Term Exposure Limit (STEL = 15-minute TWA exposure that should not be exceeded at any time during a workday). [27] Using the mean value for this comparison would be appropriate but a more-conservative

approach is to use the upper 95% Confidence Interval (CI). The REL for the nonvolatile hypochlorite salt (bleach) is "not established" according to the Clorox® MSDS. NIOSH reports the REL for chlorine, the off-gassing product from hypochlorite, as 1.47 mg/m^3 and the PEL as 3 mg/m^3. Under the assumption of complete and instantaneous dissociation into Cl_2 (g), the upper 95% CI for two FFR models would exceed the REL (Table IV). However, the equilibrium constant disfavors formation of chlorine so the preponderance of the oxidant is expected to remain on the FFR as a hypochlorite salt and act only as a potential skin irritant. Also, the REL is a TWA of exposure during a 10-hour period. If the slightly less-improbable assumption is made that all of the oxidant is transformed into chlorine and inhaled at constant concentration during one 10-hour period, the accumulated exposure would not exceed the REL. Because the active agent in the mixed oxidants technology is hypochlorite, the previous discussion of bleach also applies for this decontaminant.

The peroxide-based decontaminants (VHP and 3% hydrogen peroxide) left the lowest observable amount of oxidant on the respirators. Only two models of respirators treated with VHP exceeded the REL of 1.4 mg/m^3. As peroxide will be slow to off-gas, the previous discussion on hypochlorite is also relevant for the peroxide-based decontaminants.

An analysis of the amount of oxidant retained by the various FFR parts reveals delineation between the particulate and surgical FFRs (Figure 1). For the particulate FFRs a majority of the oxidant was recovered from the filtering media for all five decontaminants that were evaluated. However, two of the surgical FFRs (S2 and S3), retained very little oxidant on the filtering media. This is likely due to the hydrophobic coating that is applied to the surgical FFRs to provide resistance to blood splatter and other body fluids. As mentioned previously, the S3 FFR retained very little oxidant overall and this is partially due to its simple design: it does not contain a nose cushion. The nose cushion in the S2, which is very large compared to the nose cushions on the other FFRs, was responsible for retaining a majority of the oxidant using the traditional decontamination methods. The data for the mixed oxidant decontaminant is somewhat skewed due the overall low retention of oxidant. The DMDO is also a special case as it was

retained by the filtration media for all six FFRs. The outlier in the group is the S1 which is a surgical FFR that retained oxidant on the filtration media. It did perform better than the particulate FFRs, but it is unclear why the S2 and S3 FFRs retained no oxidant on the filtration media and the S1 retention varied from 30% -50% for the traditional decontamination methods. GC-MS analysis of the *n*-pentane extracts revealed many minor peaks (data not shown). Only 20 unique peaks were discovered that occurred on at least three of the 18 FFRs evaluated for each decontamination method (Table V). Eleven of those peaks were attributed to the chemical disinfectants and nine were discovered following UV disinfection. Many of the peaks appear to be species related to the solvent (*n*-pentane) and are not due to the disinfectant. Some products, such as tetramethylsilane and dodecamethylpentasiloxane, are known artifacts of column bleed. Common laboratory contaminants such as butyl phthalate and bis (2-ethylhexyl) adipate used in the synthesis of polymeric materials were also detected. While these were included in the results, it is important to note that they probably are not derived from the decontamination technologies. Although background subtraction was performed via the negative controls, some treated samples indicated the presence of additional chemicals similar to those found within the controls. It was expected that these peaks would have been discovered in the negative controls (untreated FFRs and pentane control), but many of these peaks were at the instrument detection limit and may have been overlooked. The peaks that are due to column bleed are inherently random, although they will always increase in concentration as the oven temperature increases. Solvent-derived compounds that did not match the controls were also discovered in some of the treated samples. As the study progressed, GC maintenance necessitated trimming several inches off the front end of the capillary column. While we have tried to account for shifts in retention time caused by this procedure, some of the peaks might not have been precisely tracked through the entire course of the study.

Although the respirators were found to be entirely free of EO, several of the models and components tested contained diacetone alcohol and 2-hydroxyethyl acetate. Diacetone alcohol is a Class II combustible liquid with a REL of 50 ppm.[27] While it is uncertain that an adequate amount of this

compound was present to affect human health, further studies should be conducted to ascertain the exposure threat before this technology is considered for disinfecting respirators. Because it is classified as a possible carcinogen, no REL or PEL is listed for 2-hydroxyethyl acetate (acetic acid, 2-hydroxyethyl ester). [28] This compound might have formed via EO alkylation of vinyl acetate, a common component of rubber. Rubber straps were components of every FFR tested with the exception of S1 (straps composed of an elastic material containing rubber) and P2 (straps composed of a thermoplastic elastomer which contains rubber). As with any unfamiliar compound, caution is warranted until the exact nature of the substance is determined. Although the concentration recovered was minute, avoiding human exposure to this compound would be prudent when there are clearly safer alternatives.

Summary and Limitations of the Study

The data from this study demonstrate readily available decontamination technologies that do not leave significant quantities of toxic residues on the FFRs. UV irradiation and the peroxide-based technologies (VHP and 3% hydrogen peroxide) provided favorable results for all FFR models tested. Diluted household bleach (0.6% hypochlorite) also produced acceptable results; nevertheless, it should be noted that all FFRs treated with bleach retained a bleach odor following the off-gassing period. At a minimum, the odor is unpleasant and may cause adverse health effects in users with certain asthmatic conditions. Also, bleach rusted the metal parts on the FFRs (staples, nosepieces, etc.) and discolored others. For these reasons, bleach is not recommended for decontaminating FFRs. The two emerging technologies, DMDO and mixed oxidants demonstrated similar problems. DMDO retained the greatest amount of oxidant, but no PEL is available for DMDO, thus human safety concerns cannot be evaluated.

Both gaseous sterilizers (EO and VHP) left very little of the active species on the FFRs following decontamination and off-gassing. However, both techniques have undesirable traits that limit their use for decontaminating FFRs: EO treatment of FFRs produced 2-hydroxyethyl acetate: a hazardous chemical

by-product, possibly formed by a reaction of EO with rubber parts of the respirator. Further studies are needed to clarify these observations. Additionally, EO requires a long off-gassing period that will limit throughput. Throughput is also a problem for the VHP technology—our experience during testing with the VHP sterilizer was a sterilization cycle abortion if more than six FFRs were loaded in the chamber during the one-hour sterilization cycle. It is know that cellulosic material will absorb peroxide [29], but the masks do not appear to contain cellulose. The main component of the FFRs appear to be polyester (40 - 70% by weight, mmm.com). We could not find any data to support that polyester absorbs peroxide. It is unclear why the FFRs would have resulted in abortion of the VHP cycle.

This study is an initial look at the potential toxicity of FFRs following decontamination. The authors do not endorse any method for decontaminating FFRs. More data are needed to measure the effect of candidate decontaminants on filtration efficiency, fit, and the ability of each method to decontaminate the influenza virus *in situ*. These studies are in progress and will be reported in the near future. Additional work is also needed on other models of FFRs. This study focused on six models of FFRs, but hundreds exist and each must be tested before conclusions can be made about compatibility with specific decontamination technologies.

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420 **TABLE I.** Filtering Facepiece Respirators Selected for Decontamination Study

Number	**Class**	**Shape**
S1	NIOSH and FDA approved N95 Surgical FFR	Cup-shaped
S2		Flat-fold
S3		Duck-bill
P1	NIOSH approved N95 Particulate FFR	Cup-shaped
P2		Cup-shaped
P3		Cup-shaped

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423 **TABLE II.** Disinfection technologies

Large Scale (gaseous)	Ethylene oxide
	Vaporized hydrogen peroxide
Medium Scale (energetic)	Moist heat (65 °C, 85% RH)
	Desiccation (<10% RH)
	Microwave/steam
	Ultraviolet light (254 and 302 nm, ~$2.7X10^5$ J/M^2)
Small Scale (liquid)	Hydrogen peroxide (3%)
	Sodium hypochlorite (0.6%)
	Mixed oxidants (10% oxone, 6%, sodium chloride, 5% sodium bicarbonate)
	Dimethyl dioxirane (10% oxone, 10% acetone, 5% sodium bicarbonate)

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426 **TABLE III.** Analytical Methods for Quantifying Decontamination Agents on FFRs

Decontamination Agent	**Concentration**	**Analysis Method**
Untreated	N/A	Iodometric back-titration, GC-MS HSSPME Pentane extraction
Hydrogen peroxide	3%	Iodometric back-titration, Pentane extraction
Sodium hypochlorite	0.6%	Iodometric back-titration, Pentane extraction
Mixed oxidants	10% oxone, 6%, sodium chloride, 5% sodium bicarbonate	Iodometric back-titration, Pentane extraction
Dimethyl dioxirane	10% oxone, 10% acetone, 5% sodium bicarbonate	Iodometric back-titration, Pentane extraction
Ethylene oxide	Amsco Eagle 3017	GC-MS HSSPME, Pentane extractions
Vaporized hydrogen peroxide	Sterrad 100S System	Iodometric back-titration, Pentane extraction
Ultraviolet light (254 & 302 nm)	~$2.7X10^5$ J/M^2	Pentane extraction

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TABLE IV. Oxidant (mg) Remaining on FFRs Following Decontamination and 18-Hours Off-Gassing

3% Hydrogen Peroxide						
Respirators	S1	S2	S3	S4	S5	S6
Average	0.59	0.36	ND	0.43	0.53	0.70
Lower 95% CI	0.14	0.28	----	0.12	0.20	0.38
Upper 95% CI	1.04	0.45	----	0.74	0.87	1.02
Vaporized Hydrogen Peroxide						
Respirators	S1	S2	S3	S4	S5	S6
Average	1.23	0.43	0.36	1.09	0.81	0.35
Lower 95% CI	0.68	0.29	-0.11	0.64	0.29	0.04
Upper 95% CI	1.77	0.57	0.83	1.53	1.34	0.66
10% Bleach						
Respirators	S1	S2	S3	S4	S5	S6
Average	0.37	0.70	ND	0.32	1.66	0.45
Lower 95% CI	0.00	0.29	----	-0.31	-2.03	-0.64
Upper 95% CI	0.73	1.11	----	0.95	5.34	1.54
Mixed Oxidants						
Respirators	S1	S2	S3	S4	S5	S6
Average	0.14	0.08	ND	0.25	1.72	8.10
Lower 95% CI	-0.05	-0.08	----	-1.53	-1.38	3.06
Upper 95% CI	0.32	0.24	----	2.03	4.82	13.14
DMDO						
Respirators	S1	S2	S3	S4	S5	S6
Average	7.38	7.72	4.53	5.53	7.19	5.14
Lower 95% CI	6.87	-0.09	2.52	5.11	6.50	3.95
Upper 95% CI	7.89	15.53	6.53	5.94	7.87	6.33
ND - none detected						

TABLE V. GC-MS Unique* Peaks of Pentane Extracted FFR Found in Greater than 3/18 of the FFRs tested

Decontaminant	Unique Peaks Retention Time (min)	Peak Occurrence Among All FFR Models	Peak ID*
Hydrogen peroxide (3%)	3.98	14 / 18	Hexane
	13.13	4 / 18	Siloxane derivative
	14.63	4 / 18	Siloxane derivative
Vaporized hydrogen peroxide	3.98	14 / 18	Hexane
0.6 % Hypochlorite	4.46	5 / 18	Aliphatic hydrocarbon
Mixed oxidants	3.97	7 / 18	Hexane
	3.98	10 / 18	Hexane
Dimethyldioxirane	7.28	12 / 18	Siloxane derivative
	8.68	13 / 18	Siloxane derivative
	13.75	6 / 18	Siloxane derivative
Ethylene oxide	11.42	5 / 18	Aliphatic hydrocarbon
Ultraviolet (254 and 302 nm)	3.71	4 / 18	Aliphatic hydrocarbon
	3.72	8 / 18	Aliphatic hydrocarbon
	4.67	4 / 18	Aliphatic hydrocarbon
	5.7	15 / 18	Aliphatic hydrocarbon
	7.06	4 / 18	Aliphatic hydrocarbon
	7.07	10 / 18	Aliphatic hydrocarbon
	11.09	5 / 18	Aliphatic hydrocarbon
	13.22	5 / 18	Butyl phthalate
	13.23	11 / 18	Butyl phthalate

* Most likely match by Xcaliber™ software

TABLE VI. Unique Peaks from HSSPME Analysis of Ethylene Oxide Treated FFRs Present on Greater than 3/18 FFRs

Unique Peaks Retention Time (min)	Peak Occurrence Among All FFR Models	Peak ID*
5.3-5.33	11 / 18	Diacetone alcohol
5.49	15 / 18**	2-Hydroxyethyl acetate
* Most likely match by Xcaliber™ software		
** Only detected on straps		

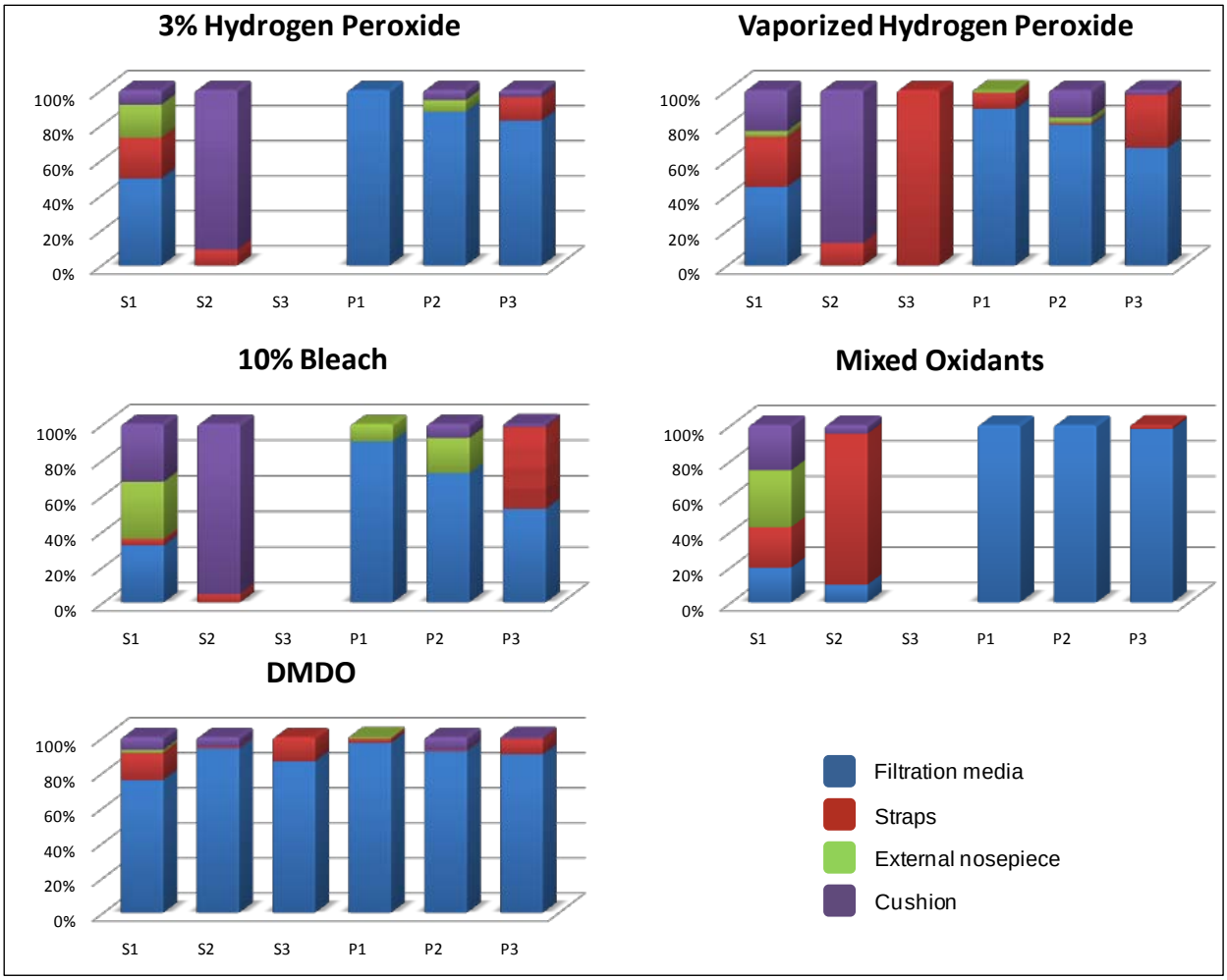

FIGURE 1. Percent Oxidant Recovered From FFR Components.