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Evaluation of decontamination methods for commercial and alternative respirator and mask materials – view from filtration aspect

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ABSTRACT

This study aims to evaluate the filtration performance of three commercially available (3M 8210 respirator, Halyard 48207 surgical mask, and 3M 1820 procedure mask) and two alternative face mask and respirator materials (Halyard H600 sterilization wrap and Cummins EX101) after selected decontamination treatments, including isopropanol (IPA) treatments (soaking or spraying), ultraviolet germicidal irradiation (UVGI), and heat treatments (dry heat at 77 °C or steam heat). Both IPA soaking and spraying removed most electrostatic charges on all four electret materials (three commercial and one alternative), causing significant deterioration of filtration efficiency to unacceptable level. The other non-electret alternative material sustained its N95grade performance after both IPA soaking and spraying treatments, demonstrating the possible application of IPA disinfection for non-electret alternative respirator/mask materials. UVGI preserved the filtration of all three commercially available respirator/mask materials after up to 10 treatments, suggesting it can be a possible decontamination method for hospital and clinic use without compromising respirator/mask performance. The considerations of the practical implementation of this method was discussed. Between the two heat treatment methods tested, dry heat showed better compatibility with electret material by sustaining both filtration efficiency and fit (tested on commercial respirator only), although adding moisture was reported in favor of virus inactivation. Heat treatment is easily accessible method for general publics to implement at home, while it is recommended to maintain the moisture level below saturation. Comparing to size-integrated method, the size-resolved fractional efficiency measurement technique, although more time consuming, proved to be a better method for evaluating respirator/mask filtration performance after decontaminations by providing more sensitive detection of performance degradation and the capability of distinguishing charge loss to other mechanisms causing efficiency deterioration. Detailed descriptions are provided in methodology part to emphasize the cares needed for an appropriate efficiency evaluation. The limited results in this study on worn masks made of alternative sterilization wrap indicated possible performance degradation of electret material caused by normal human wearing activities, suggesting the need of assessing respirator/mask decontamination strategy by testing practically worn-and-decontaminated/ reused samples instead of unworn only-decontaminated counterparts.

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1. Introduction

The COVID-19 pandemic has led to a severe shortage of personal protection equipment (PPE) worldwide (World Health Organization, 2020c). According to World Health Organization (2020b), the novel coronavirus (SARS-CoV-2) is transmitted primarily through human respiratory droplets generated by coughing or sneezing (Leung et al., 2020; National Academies of Sciences E. & Medicine, 2020), either through direct contact when virus-containing droplets reach the nose, mouth, eyes of a person, or indirectly when a person touches virus-contaminated objects or surfaces and then touches his/her eyes, nose, or mouth. A respiratory protection equipment is therefore essential for frontline healthcare workers who may have direct contact with patients carrying COVID-19 virus. Nevertheless, the scarce inventory of N95 respirators and surgical masks in hospitals has been reported (Klompas, Morris, Sinclair, Pearson, & Shenoy, 2020). In light of the urgent needs and the exhausted national stockpile of PPEs, although not designed for, the possible decontamination and reuse of disposable filtering facepiece respirators (FFRs) becomes necessary (Centers for Disease Control and Prevention, 2020).

While tight-fitting respirators provide better protection against airborne submicrometer particles (Ollier et al., 2019; Zhou & Cheng, 2017), they are generally recommended to be reserved for healthcare workers during current pandemic (World Health Organization, 2020a). Loose-fitting masks, such as surgical masks and procedure masks, are designed mainly to be used as one-way protection, capturing large particles or droplets exiting from the wearer and preventing them from being spread to the environment. Surgical masks often have a fluid-resistant outer layer, which also protect the wearer against large droplets, splashes, or spray of bodily or other hazardous fluids. While being reported effective in preventing spread of influenza virus (Johnson, Druce, Birch, & Grayson, 2009), these loose-fitting masks are not typically considered as proper respiratory protection, since they do not provide the wearer with a reliable level of protection from inhaling small airborne particles, which can either penetrate through mask materials (insufficient capturing efficiency) (Chen & Willeke, 1992) or leak through the gap between mask perimeter and wearer's face (loose-fitting) (Oberg & Brosseau, 2008). Nonetheless, due to the severe shortage of FFPs and the recommendations of general publics to wear face-coverings when other social distancing measures are difficult to maintain (Centers for Disease Control and Prevention, 2020; Feng et al., 2020), loose-fitting face masks now become a major measure of respiratory protection for general publics, first responder workers, and even some frontline healthcare workers (mainly surgical masks in some low risk settings). While performance of respirators are well evaluated and many studies on respirator decontamination methods are reported (Centers for Disease Control and Prevention, 2020; N95DECON, 2020), the information of filtration performance and possible decontamination and reuse strategy for face masks is scarce.

SARS-CoV-2 is a large size virus with a total size of ~0.06–0.14 µm (Lim, Ng, Tam, & Liu, 2016; Zhu et al., 2020). Human exhalation droplets can be several micrometers or larger (Anfinrud, Stadnytskyi, Bax, & Bax, 2020; Bourouiba, 2020; Scharfman, Techet, Bush, & Bourouiba, 2016), but shrink while traveling in air due to water evaporation. Dried droplet nuclei are usually less than 5 µm in diameter (Hsiao, Chuang, Griffith, Chen, & Young, 2020), and therefore can remain suspended in air for a longer period of time, and travel a further distance away (Yu et al., 2004). Efficiency of filter media is a strong function of particle size and filtration velocity, as various filtration mechanisms (e.g. diffusion, interception, impaction, electrostatic capturing, etc.) work effectively at different size and velocity (Brown, 1993; Hinds, 1999; Ou, Maricq, & Pui, 2017), which yields a least efficient size, namely most-penetrating particle size (MPPS). Under the range of normal operation conditions, most mechanical filters have a MPPS between 100 and 300 nm, while electret filters, which most commercial respirators and face masks are made of, have smaller MPPS typically below 80 nm. In United States, respirators are approved by National Institute for Occupational Safety and Health (NIOSH), by testing well-sealed respirator at a flowrate of 85 L/min using charge neutralized polydisperse sodium chloride (NaCl) particles with a count median diameter (CMD) of 0.075 ± 0.02 µm and a geometric standard deviation (GSD) of less than 1.86 (NIOSH, 2019). A mass integrated efficiency is then determined by a light scattering based photometer. Surgical masks, differently, are cleared by the Food and Drug Administration (FDA), by reviewing data supplied by the manufacturers that is collected from third-part independent testing laboratories. A wide range of testing flowrate or face velocity is implemented for testing face masks, and different sometimes confusing efficiencies (e.g., PFE, BFE, VFE, etc.) are reported (Rengasamy, Shaffer, Williams, & Smit, 2017). In view of the lack of a universal standard for testing respirators and various face masks side-by-side, in our work, fractional efficiency measurement was conducted which provides efficiency at a series of particle sizes from 30 to 400 nm, covering the MPPSs of all samples tested.

Various disinfection methods have been tested for decontaminating filtering facepiece respirators, and a comprehensive review of these studies is available at N95DECON website, a scientific consortium for data-driven study of N95 filtering facepiece respirator decontamination (N95DECON, 2020). Among all these methods, US Center for Disease Control and Prevention (CDC) (2020a) has identified three most promising ones: vaporous hydrogen peroxide (VHP) (Battelle, 2016; Bergman et al., 2010), ultraviolet germicidal irradiation (UVGI) (Lindsley et al., 2015; Viscusi, Bergman, Eimer, & Shaffer, 2009), and moist heat (Bergman et al., 2010). Steam treatment (Edward M. Fisher, Williams, & Shaffer, 2011; B. K. Heimbuch et al., 2011) and liquid hydrogen peroxide (Bergman et al., 2010) are listed as promising methods with some limitations. All these methods are evaluated for respirators only, and are mainly targeted for reuse by healthcare workers. In our study, we attempted to extend the scope a little further to include the decontamination of loose-fitting face masks and some alternative materials that have been used for making respirators/masks under this crisis, and the reuse purpose for both healthcare workers and general publics. Because of that, among all applicable decontamination methods being reported to be efficient in deactivating COVID-19 and other pathogens, we excluded methods that require sophisticated equipment and professions (e.g. VHP), and chose UVGI in view of its wide availability in hospitals and small clinics and the heat treatments (both dry oven heat and water steaming) for simple household applications. Filtration performance were evaluated to assess if these decontamination methods can be implemented without compromising the function of respirators/masks. Although it has been well reported

that isopropanol (IPA) cannot be used to disinfect respirators as it removes charges so that lead to filtration deterioration, its possible application for disinfecting non-electret alternative respirator material was evaluated in this study.

2. Methodologies

2.1. Decontamination methods

For the ultraviolet germicidal irradiation (UVGI) method, the respirator/mask samples were hung between a Xenex LightStrike Germ-Zapping Robots and a UV reflective wall, both < 1m away from the samples. The UV-C light from the source machine and the reflected illumination irradiated the samples for 5 min. For the oven heating method, 77 °C (170 °F) was chosen because it is the lowest temperature setting for most household ovens, and Covid-19 virus is reported deactivated at 70 °C (Chin et al., 2020). At the target temperature, the respirators/masks were placed on a stack of coffee filters inside the oven without touching any metal surfaces to prevent thermal damage and heated for 30 min. For the steam heat treatment method, the respirators/masks were placed on the rack of a steamer with boiling water for 30 min. For isopropanol (IPA) soaking, test samples were soaked in 75% IPA-water solution for 10 min and let dry overnight.

2.2. Filtration performance testing

A round piece of filtration materials with 40 mm diameter was cut from each sample, and secured in a stainless steel filter holder with gaskets sealing around the edge on both sides to prevent air leakage. The breathing resistance of the materials was determined by measuring the differential pressure drop across test samples under testing filtration velocity of 10.5 cm/s, which is equivalent to 85 L/min across a filtration area of 135 cm². 85 L/min is the standard flowrate at which NIOSH certifies N95s, and 135 cm² is the lower end of filtration surface area of typical commercial N95 respirators. The choice of lower end value is to ensure our measurement represents the worst scenario, as filtration performance deteriorates with increasing filtration velocity. As a reference, 3M 8210 N95 respirator has an effective surface area of 150–170 cm², depending on how it is sealed for standard NIOSH N95 certification test.

For particle filtering efficiency, a method called fractional efficiency measurement was used. As depicted in Fig. 1, NaCl nanoparticles generated from a Collison-type atomizer (TSI 3076, Shoreview, MN) were employed as challenging aerosol for fractional efficiency measurement. The test particles were first passed through a diffusion dryer to remove excess moisture from atomizer solution and bring the humidity level of the aerosol below deliquescence relative humidity of NaCl. They were then sent into a Po-210 radioactive neutralizer (0.5 mCi) and a Differential Mobility Analyzer (DMA), which act as a size selector and only allow NaCl particles with a narrow mobility range to exit. To save time, the NaCl solution concentration was not changed during the measurement, but the number mode was kept at slightly below 65 nm to minimize the multiple charge effect in mobility classification for large particles. The size-selected particles were then passed through another Po-210 neutralizer (0.5 mCi) to bring the charge level of these charged particles to a Boltzmann equilibrium charge distribution (Liu & Pui, 1974; Wiedensohler, 1988), a steady state distribution best representing the charge level of ambient aerosols. This second neutralizer is particularly important for testing respirators and masks, as most commercial units employ electret media which show much higher efficiency on small (<100 nm) charged particles. The importance of testing with neutralized (Boltzmann equilibrium) aerosol was overlooked by a recent publication with similar topics (Konda et al., 2020), in which un-conditioned aerosols from a mechanical nebulizer was employed, whose inherent high charge level would cause overestimation of filtration efficiency. The neutralized test particles were then mixed with dilution air and sent into filter holder, whose number concentrations upstream and downstream of test sample were measured by a Condensation Particle Counter (TSI 3776, Shoreview, MN) with the assistance of a pneumatic three-way switching valve. The fractional efficiency (FE) of the sample was determined as $FE = 1 - \frac{C_{dn}}{C_{up}}/\frac{C_{blank,up}}{C_{blank,up}}$, where C_{up} and C_{dn} are the number concentrations of NaCl particles at upstream and downstream of the sample, and $C_{blank, up}$ and $C_{blank, dn}$ are the number concentrations of NaCl particles at upstream and downstream of the filter holder when no sample presents. The calculated FE accounts for particle transportation loss in the test system. FE was determined for 8 sizes spanning from 30 to 400 nm, which covers the MPPS of both electret and mechanical filtration materials.

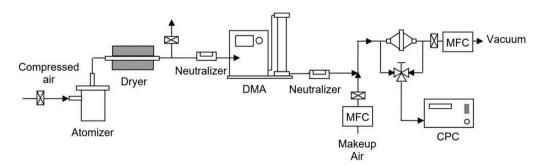


Fig. 1. Schematic of the fractional efficiency measurement setup.

3. Results and discussions

Three commercially available respirator and mask samples were selected as the major data set of this study, namely 3M 8210 N95 respirator, Halyard 48207 surgical mask, and 3M 1820 procedure mask. Additionally, 2 other materials that are reported being used for making respirators/masks under Covid-19 pandemic were selected. One is Halyard H600 one-step fabric sterilization wrap, which was first proposed as alternative mask material by a team at University of Florida who has a pending patent on this (University of Florida, 2020). The other material, named as EX101, was donated by Cummins Inc. to University of Minnesota as part of a project to supply masks to Minnesota's M Health Fairview network (Businesswire, 2020; University of Minnesota, 2020).

All the 3 commercial respirator and masks and Halyard H600 fabrics have electrostatic charges on their fiber surface, which help to boost filtration efficiency without causing extra breathing resistance. However, these charges may be completely or partially removed by some sterilization methods, which could negatively impact the selection of decontamination and reuse strategy for these materials. EX101 is a pure mechanical filtration material with no electric charge on it. It was made of polymer fibers: PBT polyester, Nylon 6-6, and PET polyester in different layers.

3.1. Base performance before decontamination

Filtration efficiency and breathing resistance of all the five materials are measured first without any decontamination treatment was conducted, which is summarized in Fig. 2(a). Among all five materials, N95 respirator material and EX101 media have the highest level of efficiency, greater than 95% over the entire size range between 0.03 and 1.0 µm. All three commercial respirator and masks show similar trends of increasing efficiency with particle size, suggesting they all possess high level of electrical charge on their fiber surface. Unlike N95 respirator having efficiency greater than 95%, surgical mask and procedure mask tested have efficiency around 80-90% and 70-80% respectively in the range of $0.03-0.4~\mu m$. Their efficiency increased sharply after $0.4~\mu m$, to greater than 95% at 1.0 µm, attributed to the improved capture by interception and inertia impaction. Since the efficiency keeps increasing and stays high after 0.4 µm for all 5 materials, only the results between 0.03 and 0.4 µm are reported in the rest part of the paper. EX101 has a MPPS at ~0.1 µm, which is typical for pure mechanical filter with small fiber diameter at this face velocity (10.5 cm/s). The shortcoming of achieving N95 level of efficiency without an assistance from electrostatic charge is the highest breathing resistance (323 Pa @ 10.5 cm/ s) among all, which though is still below NIOSH's limit of 343 Pa for an N95 respirator. This breathing resistance can be further reduced by making masks with larger effective filtration area. H600 sterilization wrap has the lowest efficiency and the second highest breathing resistance among all. It is a surprise that its efficiency is only marginally lower than 3M procedure mask, given that this material is not originally designed as a filtration material, which is mainly because it has certain level of electrical charge on its fibers. This is known by comparing its filtration with that of a 75% isopropanol-soaked sample as discussed later. It is worth mentioning that the sterilization wrap is more plastic than most non-woven face masks, potentially providing better facial fit than loose-fitting masks, although this advantage is partially offset by its higher breathing resistance (causing more pressure driven facial leak). All the three commercial respirator and masks have flow resistance around 50-75 Pa, which agrees with previous measurement (J.-H. Kim et al., 2015). A recent study (Konda et al., 2020) measured significantly lower flow resistance at as low as 10–15 Pa may suggest leaks in their test apparatus.

For a better comparison among all the five materials, their quality factor (Q) is determined by

$$Q = -\frac{\ln(1 - Eff)}{\Delta P},$$

where Eff is the fractional efficiency at certain size, while ΔP is the differential pressure drop (breathing resistance). Quality factor

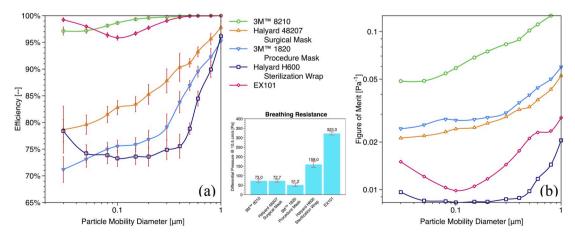


Fig. 2. Fractional efficiency, breathing resistance, and quality factor of 3M 8210 respirator, Halyard 48,207 surgical mask, 3M 1820 procedure mask, Halyard H600 sterilization wrap, and Cummins EX101 material, before any decontamination treatments.

represents a tradeoff between efficiency and resistance of a filtration material under a specified operating condition (e.g., face velocity, temperature, etc.), with a higher value preferable. It is clearly seen from Fig. 1(b), that 3M 8210 respirator has much higher quality factor than the other materials as it is one of the highest efficient and the least resistant material among all five tested. With the assistance of electrostatic capturing, both surgical mask and procedure mask have decent quality factor that is higher than typical filtration materials at this face velocity. As a pure mechanical material, the quality factor of EX101 media is among the top tier, and is even slightly higher than H600 sterilization wrap which has electrical charge on it. The non-electret nature of EX101 makes it more tolerant to various decontamination methods, which will be demonstrated later.

3.2. Decontamination by isopropanol alcohol

It is well reported that isopropanol alcohol and soap cause filtration degradation of N95 respirators (Viscusi et al., 2009), and the purpose of this set of test is mainly to confirm if similar degradations can be found on other face mask materials and alternative respirator/mask materials. As shown in Fig. 3, after only one treatment, significant decay in efficiency was found in all the materials except for EX101 which is a non-electret media. For all four electret media, the degree of efficiency degradation increases with particle size, resulting in MPPS shift towards larger size from below 30 nm (except for sterilization wrap at 100 nm) before the treatment to 150–300 nm afterwards. The shift of MPPS towards larger size is a strong indication that the drop in efficiency level is due to charge loss rather than structural change or integrity degradation. Another evidence of this conclusion is the unchanged breathing resistance level, which is also shown in Fig. 3. Exposure to organic solvents such as isopropanol, acetone, xylene, toluene, and benzene has been reported to increase charge mobility in polypropylene fibers, subsequently reducing the electrostatic charge on the fibers and their ability of capturing particles (Jasper et al., 2006; J.; Kim, Hinestroza, Jasper, & Barker, 2009).

Although not as significant as others, EX101 also shows a slight drop in efficiency with an unchanged MPPS at 100 nm. At this size, it is 90.5% efficient after treatment comparing to 95.7% of untreated media. The change is likely from the loss of small fibers when IPA soaks into the material's micro-structures or evaporates from its fiber surface. It is worth noting that although the IPA soaked EX101 sample has an efficiency below 95% at MPPS (100 nm), it may still pass N95 certification for two reasons: (1) photometer based NIOSH N95 certification test puts more weight on the efficiency at $\sim 300 \text{ nm}$; and (2) the sample would have been tested under a low face velocity if a respirator was made with greater than 135 cm^2 effective filtration area, which is the case for most commercial respirators. For further justification, an integrated mass efficiency was calculated by assuming a lognormal test aerosol distribution with a count median diameter at 75 nm and a geometric standard deviation of 1.86. The calculated mass weighted efficiency was 95.18%.

Instead of soaking, a gentler treatment was then implemented by spraying 75% isopropanol-water solution onto media surface using a small garden sprayer. Almost identical level of efficiency degradations were found on all four electret materials, suggesting that the small amount of IPA droplets generated is sufficient to almost fully discharge all electrical charges on electret fiber surface. This result suggests that attentions should be paid to avoid direct contact of alcohol liquid or vapor with respirators and face masks, especially in hospitals and clinics where alcohol or other disinfection agents are used routinely. One example of that is adjusting/touching respirators/masks immediately after using hand sanitizer. Unlike IPA soaked case, no efficiency degradation was observed on IPA sprayed EX101 sample, indicating the nanofibers are strong enough to retain their structure when contacting with small amount of IPA droplets.

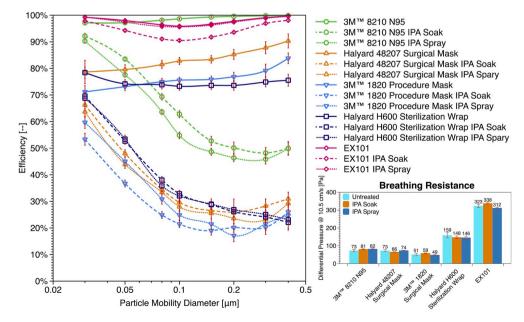


Fig. 3. Fractional efficiency and breathing resistance of the 5 filter materials before and after IPA treatments (soaking or spraying).

3.3. Decontamination by UVGI

Within the spectrum of ultraviolet light (10–400 nm wavelength), ultraviolet-C (100–280 nm) has the highest disinfection efficacy as UV-C is absorbed by DNAs and RNAs of microbes and viruses which in turn induces changes in DNA/RNA structures (Lindblad, Tano, Lindahl, & Huss, 2019). It is reported that it is more difficult to disinfect virus on respirators than on a flat object surface, and a UV-C dose of 1000 mJ/cm² is necessary to disinfect (≥3-log inactivation) viruses similar to SARS-CoV-2 on N95 FFRs (Mills, Harnish, Lawrence, Sandoval-Powers, & Heimbuch, 2018). The exact UV-C dose in one treatment cycle in this study is not available, but it is estimated to be sufficiently higher than 1000 mJ/cm². As shown in Fig. 4, no systematic efficiency drop was found on either of three commercial respirator and face masks after up to 10 cycles. Since the filtration performance testing in this study is a destructive method, to save precious PPEs, only one sample was prepared for each decontamination method and treatment cycle combination. The small fluctuations (within 2% for N95s, 11% for surgical mask, and 8% for procedure mask) observed can be mainly attributed to variation among different pieces of samples. N95s were reported to keep integrity and fit at higher dose of UV-C light (B. Heimbuch & Harnish, 2019; Lindsley et al., 2015). Despite of sample variation, which is shown as efficiency fluctuation evenly over size range of 30–400 nm, no efficiency decay associated with charge loss can be identified, which would otherwise show higher efficiency reduction in larger sizes. This results are in agreement with previous experiments (Bergman et al., 2010; Viscusi et al., 2009, 2011), suggesting UVGI can be an option to decontaminate respirators and face masks under the shortage caused by Covid-19 without causing degradation in filtration.

As one of the standard disinfection methods used in hospitals, UVGI generally has larger availability than other recommended respirator decontamination options (e.g., VHP) for hospitals. Question however remains open about UV-C's ability of penetrating deeply into melt-blown materials, which is made of polypropylene, a UV absorber. The amount of penetration was found to vary largely among N95 models (E.M. Fisher & Shaffer, 2011). Higher UV-C dose may be necessary to disinfect other pathogens that are less susceptible. Both concerns require higher UV-C dose, leading to longer irradiation period. Some studies (B. Heimbuch & Harnish, 2019; Mills et al., 2018) demonstrated residual virus on respirator straps after UVGI treatment (likely due to the factor that the straps twist so as be shielded from UV-C irradiation), suggesting a subsequent disinfection of straps are necessary. The consideration of UV shading also requires that respirators and masks cannot be stacked during the treatment, which further limits the decontamination

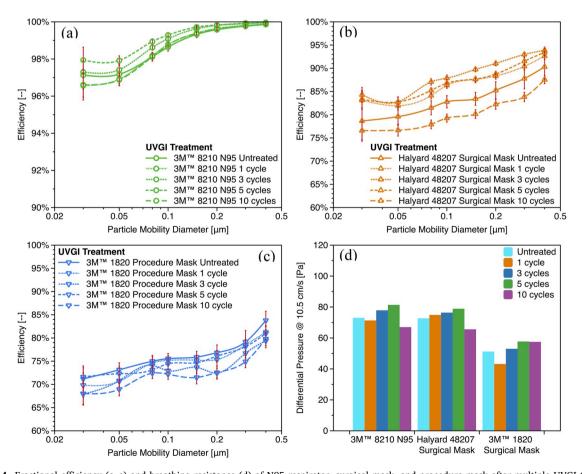


Fig. 4. Fractional efficiency (a–c) and breathing resistance (d) of N95 respirator, surgical mask, and procedure mask after multiple UVGI treatment cycles.

throughput of this method.

3.4. Decontamination by thermal treatments

Among all potential decontamination methods, thermal treatment is the most applicable one for the general public to implement in household setting, because of its simple procedure and the high availability of various ways of heat generation at home. For the same reason, methods requiring sophisticated humidity control are not evaluated due to its relatively low feasibility at home, although there is evidence showing that higher humidity will better inactivate viruses similar to SARS-CoV-2 (McDevitt, Rudnick, First, & Spengler,

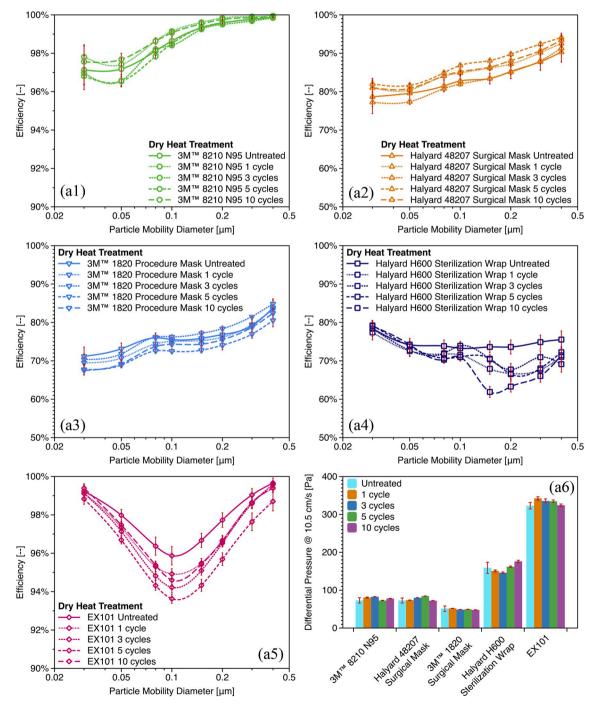


Fig. 5. Fractional efficiency and breathing resistance of all five filter materials after dry (a) and steam (b) heat treatments.

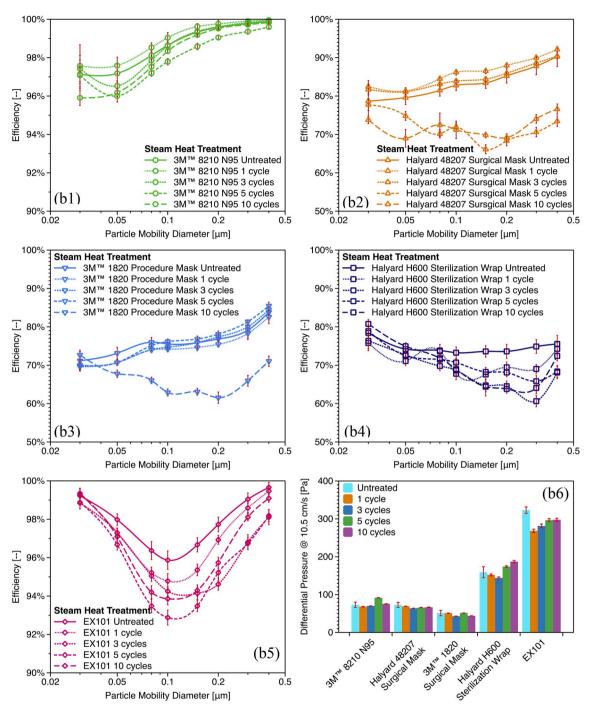


Fig. 5. (continued).

2010). A dry heat temperature of 77 °C(170 °F) was chosen as it is the lowest temperature setting of most household ovens, and a recent evidence showed that dry heat at 70 °C for 30 min can achieve 1.9-log activity reduction, which further increased to 3.3-log if the treatment duration was extended to 60 min (Fischer et al., 2020). Each treatment cycle was chosen to be 30 min in this study considering a slightly higher temperature was used and the relatively low exposure risk for general publics. As shown in Fig. 5(a1-a6), dry heat at 77 °C did not cause any observable efficiency degradation of the 3 commercial respirator/mask materials after 10 cycles. Similar to UVGI method, small fluctuations were observed which can be attributed to sample variation rather than any systematic efficiency deterioration for the reason explained above already. A slight change of MPPS from below 30 nm–50 nm was found on 3M 8210 N95 respirators, implying a subtle loss on charge level which is not sufficient to yield any practically meaningful performance

degradation. For sterilization wrap, after the first treatment, \sim 10–15% of reduction was observed at sizes greater than 100 nm, suggesting certain degree of charge loss occurred at high temperature. Further dry heat treatment for multiple cycles (up to 10 cycles) did not seem to cause further filtration degradation. Similar to sterilization wrap, EX101 experienced slight (<5%) efficiency drop after first dry heat treatment, but further reduction was not observed after multiple cycles (up to 10). No MPPS change was observed on EX101, indicating the efficiency drop is likely due to small amount of structure change (likely loss of nanofiber).

Although a precise control of humidity in thermal treatment is not an easy implementation in household, steam heat treatment can be a feasible alternative high humidity option which keeps the moisture level near or above saturation. Recent studies demonstrated SARS-CoV-2 (Kumar et al., 2020) and bacteriophage MS2 (Massey et al., 2020) viruses can be deactivated by autoclave or microwave generated steam in shorter treatment time (≤15 min). As illustrated in Fig. 5(b1-b6), although 3M 8210 N95 respirator retained its efficiency after 10 cycles, efficiency decay was observed on surgical mask (after 5 cycles), procedure mask (after 10 cycles), and sterilization wrap (after 1 cycle). The efficiency reduction is more pronounced at larger sizes, causing shift of MPPSs from below 30 nm to ~200 nm, which is a strong indication of charge loss during the treatments. The almost unchanged breathing resistance provided further evidence that change of material integrity or micro-structure is not the major source of efficiency degradation. Polypropylene is hydrophobic, but water can condense on polypropylene fiber surface under super-saturation, which could potentially deteriorate surface charge by an unknown mechanism that requires more study. Accelerated charge degradation on corona-charged polypropylene fibers has been reported when conditioned at higher humidity levels (Motyl & Łowkis, 2006). Similar to dry heat case, steam heat induced small (<5%) efficiency drop on EX101 samples, which is likely attributed to loss of nanofibers.

Besides the filtration efficiency of the material, another important property of a respirator or mask is the fit, which measures the seal between the respirator/mask and the wearer's face. An annual fit test is required when a healthcare worker needs to wear a tightfitting respirator, such as a 3M 8210 N95 respirator. The quantitative fit tests were performed using a TSI PortaCount® Pro+8038 by a specific researcher in this study. The fit factor, defined as the ratio of ambient particle concentration to the particle concentration inside the respirator, should be equal to or above 100 to pass the test. The fit tests were first performed with a new 3M 8210 N95 respirator and then performed after 1, 3, 5, and 10 cycles of 77 °C dry heat treatments with the same respirator. A second 3M 8210 N95 respirator was fit tested after 0, 1, 3, 5, and 10 cycles of steam heat treatment. As shown in Table 1, dry heat treatment deemed safe for the integrity and the fit of the respirator, while the steam heat treatment caused the respirator fail in fit test after 5 treatment cycles. It is worth noting that although all the fit tests were performed with the same person wearing the respirator in order to minimize variation of wearers, the results reported here should be considered as subject-dependent. Different fit factors should be expected if the tests were performed on a different wearer, even if with the same respirator. The impact of thermal treatment on respirator fit also varies by respirator model (Anderegg et al., 2020; Bergman et al., 2010; Fischer et al., 2020; Kumar et al., 2020). Loose-fitting face masks were not fit tested in current study. Although a fit test is not required, the overall efficacy of a face mask's respiratory protection is strongly affected by its ability to minimize facial leak around edges. With more loose-fitting masks are used by frontline healthcare workers as (sometimes the only) respiratory protection PPE, more study is urgently needed to quantify the fit factor of these face masks over a wide particle size range in order to provide a comprehensive assessment of their respiratory protection efficacy.

In summary, in terms of retaining both filtration efficiency and fit (tested for respirators only), dry heat seems to be a better option over steam heat treatment, while evidences (Fischer et al., 2020; Edward M.; Fisher et al., 2011; B.; Heimbuch & Harnish, 2019; Kumar et al., 2020; Lore, Heimbuch, Brown, Wander, & Hinrichs, 2011; McDevitt et al., 2010) suggested steam heat or moist heat (50–85% RH) disinfect SARS-CoV-2 or similar viruses at shorter exposure time. Respirators and masks can be heat treated in an enclosed rigid heat compatible container with small amount of water added (less than 1 ml for 1.25 quart container size) to help maintain a favorable 60–85% RH (Anderegg et al., 2020). It is suggested to keep the water amount low to avoid steam generation which may negatively impact efficiency and fit. The authors claim this method is scalable, so it can also be potential decontamination options at medium or large quantities after the viral inactivation of SARS-CoV-2 on FFRs under the given conditions can be confirmed.

3.5. Autoclave treatment on unworn and worn masks made of sterilization wrap

Since first introduced as a N95 alternatives, the blue sterilization wrap has been well adopted to make face masks over many hospitals and clinics in United States. As discussed above, although not really N95 grade, this material does provide good protection

Table 1Quantitative fit testing results of the new N95 respirator and after oven and steam heat treatment cycles.

| Oven Treatment | | | | | | Moist Heat Treatment | | | | |
|--------------------|--------------|--------------|--------------|--------------|--------------|----------------------|--------------|--------------|-----|-----------|
| Exercise | New | Cycles | | | | New | Cycles | | | |
| | | 1 | 3 | 5 | 10 | | 1 | 3 | 5 | 10 |
| Normal Breathing | 200+ | 200+ | 200+ | 200+ | 200+ | 200+ | 200+ | 191 | 109 | 141 |
| Deep Breathing | 200 + | 200 + | 200 + | 184 | 179 |
| Head Side to Side | 200 + | 200 + | 92 | 43 | 51 |
| Head Up and Down | 200 + | 165 | 101 | 72 | 99 |
| Talking | 135 | 134 | 124 | 170 | 125 | 135 | 86 | 80 | 56 | 58 |
| Bending Over | 200 + | 200 + | 151 | 197 | 200 + | 200 + | 144 | 136 | 47 | <i>37</i> |
| Normal Breathing | 200 + | 157 | 180 | 112 | 50 |
| Overall Fit Factor | 188 | 188 | 177 | 195 | 185 | 188 | 152 | 124 | 70 | 66 |

with efficiency similar to 3M procedure masks at about doubled breathing resistance. Unused and worn masks made of this sterilization wrap were provided by a local clinic, with all samples underwent an autoclave treatment at 132 °C for 30 min after the last use (if any) to minimize possible transmission of SARS-CoV-2 virus. Worn masks were used in real clinic activities and were treated with autoclave after each work shift and were then reused by the same person. 3 masks each were randomly selected after being wore and autoclaved for 1 or 2 times. Selected unworn masks were autoclaved up to 4 times as control groups. As can be seen in Fig. 6, autoclaved unworn masks had slight efficiency deterioration after only 1 treatment, mainly in large sizes, due to charge loss. Multiple treatments caused further degradation, but the impact seemed to fade out after 2 treatments, with very similar efficiencies observed for samples with 2, 3, and 4 autoclave treatments. The largest efficiency reduction after 4 treatments were within 20%.

On the other hand, worn masks showed much severer efficiency degradation even after only one worn-autoclave cycle. The fact of no significant change in breathing resistance and the shift of MPPS towards larger size both suggested the filtration deterioration was from charge loss, The efficiency of the 3 masks worn by 1 shift reduced by 18, 19, and 35% respectively at 300 nm. After 2 shifts, the reductions further developed to 23, 36, and 43% respectively. The worst sample of 2-shift cases (#1) showed similar efficiency to the IPA-soaked sterilization wrap (the same material the mask is made of), indicating this sample lost almost all the electric charge on it. Respirators/masks used in real world may be contaminated by saliva and mucus from human body or chemicals and particles from ambient environment. These foreign objects may negatively impact the efficiency of electret media by neutralizing or shading their electrical charges (Ji, Bae, Kang, & Hwang, 2003; Mahdavi, Haghighat, Bahloul, Brochot, & Ostiguy, 2015; Raynor & Chae, 2004), which cannot be recovered by decontamination. Another possible mechanism causing charge loss is the moisture (from human exhalation) condensation on filter fibers. The subsequent water molecule evaporation from fiber surface could potentially lead to electron exchange, which neutralizes the electric dipoles gradually over repetitive exhalation (condensation) and inhalation (evaporation) cycles. This hypothesis may also help to explain the mild filtration degradation after multiple steaming treatment cycles. Although testing of more field-used respirators/masks is needed to confirm this observation and further investigation is necessary to validate our hypothesis or any other potential mechanisms, the result from limited field-used samples here rises a question that if decontamination tests conducted on unworn respirators and masks are sufficient to provide appropriate representation of their real-world counterparts being worn and subsequently decontaminated for multiple times. Various decontamination methods may be proven to not cause filtration degradation by themselves even after 10-20 cycles. The normal human wearing activities, however, may cause irreversible filtration deterioration that cannot be recovered by these decontamination methods. If this is proven true, it will negatively impact the current decontamination and reuse strategy of respirators and masks, and may largely limit the number of times a respirator or mask can be reused.

4. Conclusions

This research studied the effect of various decontamination methods on the filtration performance of five selected commercial and

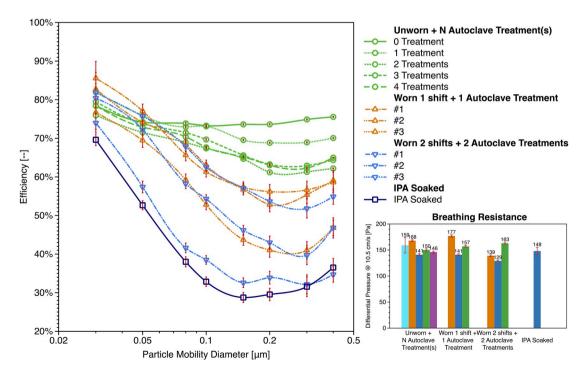


Fig. 6. Filtration performance of unworn and worn H600 sterilization wrap masks after one or multiple cycle(s) of autoclave treatment. IPA soaked same material is included for reference purpose.

alternative respirator/mask materials. Both UVGI for 5 min and dry heat at 77 °C for 30 min were found not to cause observable performance degradation after up to 10 cycles on commercial respirators/masks tested. Steam heat treatment for 30 min was found to cause charge loss on 3M 1820 procedure mask and Halyard 48207 surgical masks, which led to efficiency deterioration after 5 or 10 cycles. Although similar efficiency decay was not observed on 3M 8210 N95 respirators, the respirators' fit factor decreased with steam heat treatment cycles and dropped below 100 (fail) after 5 cycles. As an easily accessible method, heat treatment can be implemented by general publics in household setting, but our results suggested to keep the moisture level below saturation if masks made of electret materials are decontaminated. Both IPA soaking and spraying caused significant performance deterioration of all electrically charged materials, including the three commercial respirators/masks and Halyard H600 sterilization wrap, by largely removing charges from material surface. No change of material integrity or structure was observed on any combination of filter materials and decontamination methods tested, suggesting the stability of electrical charge is the major factor (together with facial fit) that limits the ability of a material being decontaminated and reused for multiple times. Non-electret filter materials (e.g. EX101), therefore, pose their advantages of wider compatibility with a variety of decontamination methods so more attention can be put on retaining the fit and the functionality of other auxiliary components (e.g., straps, nosepiece, etc.).

Fractional efficiency measurement approved to be a better method for respirator/mask performance evaluation than any size integrated methods (e.g. NIOSH N95 certification test), by providing size-resolved efficiency information (Rengasamy & Eimer, 2012). When used for evaluating decontaminated respirators/masks, this method is capable of distinguishing efficiency degradation due to charge loss from that caused by structure or integrity deterioration, which is not otherwise possible by a size integrated method.

The current study focused on the filtration performance of the filter materials, with fit testing conducted only on N95 respirators with selected decontamination methods. With more loose-fitting masks being used by frontline healthcare workers, assessing the facial fit performance of these face masks, which is equally important to material filtration testing, has become urgently needed and requires more immediate efforts. Moreover, the limited results on sterilization wraps in this study implied that normal human wearing activity could potentially cause irreversible charge loss and performance deterioration of electret materials, which has been overlooked by most respirator/mask decontamination and reuse studies. Decontamination tests on real-world used samples, together with the material durability evaluation especially after extended reuse and multiple donning/doffing cycles, are necessary to fully assess current decontamination and reuse strategy of respirators and face masks.

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