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Supercritical CO_2 Sterilization of N95 Masks

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ABSTRACT

A preliminary investigation of the use of supercritical carbon dioxide for treating of 3M 1860 N95 masks was undertaken to evaluate a potential route to low-cost, scalable, sterilization of personal protective equipment for multiple reuse in hospital settings. Upon entering the supercritical regime, the normally distinct liquid and gaseous phases of CO_2 merge into a single homogeneous phase that has density, short-range order, and solvation capacity of a liquid, but the volume-filling and permeation properties that of a gas. This enables supercritical CO_2 to function as a vehicle for delivery of biocidal agents such peracetic acid into microporous structures. The potentially adverse effect of a liquid-to-gas phase transition on mask filter media is avoided by conducting cleaning operations above 31 C, the critical temperature for carbon dioxide. A sample of fifteen 3M 1860 N95 masks was subjected to ten consecutive cycles of supercritical CO_2 cleaning to determine its effect on mask performance. These 15 masks, along with 5 control samples then underwent a battery of standardized tests at the CDC NIOSH NPPTL research facility in Pittsburgh, PA. The data from these tests strongly suggest (but do not prove) that super-critical carbon dioxide do not damage 3M 1860 N95 masks. Additional tests conducted during this project confirmed the compatibility of supercritical CO_2 with ventilator tubing that, like N95 masks, has been in short supply during portions of the COVID-19 pandemic and cannot be sterilized by conventional means. Finally, a control experiment was also conducted to examine the effect of supercritical CO_2 on a BSL-2 surrogate virus, vesicular stomatitis virus (VSV), Indiana serotype strain. In the absence of biocidal additives, supercritical CO2 exhibited no measurable lethality against VSV. This surrogate virus experiment suggests that a biocidal additive such as peracetic acid will be necessary to achieve required sterilization metrics.

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EXECUTIVE SUMMARY

In response to the COVID-19 outbreak earlier this year, Sandia National Labs stood up several short-duration R&D projects to evaluate various technological countermeasures for this crisis. The present work is directed towards multiple reuse of normally disposable N95 masks used in hospital settings to ensure the safety of medical staff. Exhausting the supply of personal protective equipment (PPE) is one of the most likely scenarios for collapse of our nation's health care infrastructure during a wide-spread pandemic.

N95 masks, such as the 3M model 1860 widely used in the healthcare industry, were never meant to be reused. Accordingly, such masks were not designed to withstand the harsh conditions of autoclaving and other traditional sterilization protocols. In fact, the internal structure of such N95 masks is quite delicate because of the mechanism used to achieve high-efficiency particle filtration while minimizing restriction of air flow. High particle filtration efficiency is not achieved be using an extremely fine fabric weave, but rather via electrically poled polymeric fibers that capture particulates by electrostatic attraction (i.e., the induced dipole effect). The challenge is to develop sterilization process that does damage the delicate internal structure of the mask or cause other adverse effects such as degradation of the elastomeric band used to achieve a reliable seal between the mask and face.

We hypothesized that supercritical CO_2 processing in conjunction with a suitable biocidal additive will allow for multi-reuse processing of N95 masks using commercially available equipment that has been developed in the U.S. and abroad for the dry-cleaning industry. In the proposed application, a key attribute of a super critical fluid is that it possesses the characteristics of a gas from standpoint of its ability to permeate into microporous materials and completely fill the volume of internal recesses and cavities independent of aspect ratio (i.e. leaving no place for contaminants to hide), while also having the properties of a liquid from the standpoint of its performance as a solvent. Supercritical CO_2's properties as a non-polar solvent allow it to serve as a vehicle for delivery of a variety of candidate biocidal additives. The reason that super-critical CO_2 is suitable for use on fabrics having a delicate microstructure is that the strong surface tension forces that can alter fabric microstructure during liquid-to-gas drying at the end of a solvent or water-based washing cycle can be circumvented by keeping carbon dioxide above its critical temperature of 31 C, such that the liquid-to-gas phase change is prevented from ever occurring. Finally, the ability of super-critical CO_2 to also remove soil is highly desirable in PPE reuse applications.

Data obtained in this brief preliminary study strongly suggest, but do not prove, that processing of 3M model 1860 N95 masks in super-critical carbon dioxide will not impair the function of such masks. Fifteen such masks were subjected to ten consecutive 1-hour cycles of super-critical carbon dioxide cleaning at a temperature of 37 C and a pressure of 1200 psig. These masks, along with five control samples from the same manufacturing lot were sent to the CDC NIOSH NPPTL research facility in Pittsburgh, PA for standardized battery of performance tests. Interpretation of the test results was complicated by the fact the masks were mechanically deformed during insertion into our small pressure chamber. This degraded the ability of the masks to seal properly around the face, allowing some particulate material to bypass. But in quantitative terms, 100% of the reductions in masks particle collection efficiency was attributable to such leakage. We strong suspect that repeating the same experiment with a larger size pressure vessel will definitively demonstrate that super-critical carbon dioxide cleaning is benign to 3M model 1860 N95 masks.

ACRONYMS AND DEFINITIONS

Abbreviation	Definition
SC-CO_2	supercritical carbon dioxide
NIOSH	National Institute of Occupational Safety and Health
CDC	Center for Disease Control (Atlanta, GA)
VSV	vesicular stomatitis virus
PPE	personal protective equipment
PRV	pressure relief valve
COTS	commercial off the shelf
COVID-19	Corona Virus Disease 2019

1. BACKGROUND AND MOTIVATION

1.1. Objective

The objective of this project is to investigate supercritical carbon dioxide sterilization as a non-destructive, high-throughput process for sterilization of N95 masks that can be stood up quickly, widely deployed, uses electricity as the only significant consumable, and that minimizes exposure of personnel to handling of contaminated masks. Multiple reuse of N95 masks has become an important objective for the protection of health care workers fighting the current COVID-19 pandemic, an anticipated 2nd wave of COVID-19 in the fall of 2020, and future communicable-disease public health crises.[1] There are also PPE sterilization applications beyond N95 masks that are driven by the same need to reuse equipment that is normally considered disposable because of current supply chain limitations. Most notably this includes tubing and other components for COVID-19 patient ventilators.

1.2. Technical background

There are many types of masks on the market with a rating of "N95", which denotes that such a mask blocks transmission of 95% of particles 0.3 microns or larger in size (95) and that such a mask is not (N) oil resistant. The main challenge in the design and fabrication of any such mask is providing high particle filtration efficiency without an objectionable level of air flow restriction. Such masks must also be very inexpensive to manufacture, because under normal circumstances disposability is a critically important element of the safety provided by such PPE. For example, a physician may change his or her mask before meeting with a new patient as a simple but effective precaution against transmission of disease.

Figure 1: Health care worker wearing personnel protective equipment (PPE) that includes a 3M 1860 N95 mask. These masks are normally considered disposable PPE. Accordingly, such masks were not designed to withstand sterilization processes such as autoclaving.

Applications for N95 masks vary widely as does their design. For example, an N95 mask used on a construction site may feature a one-way valve to reduce flow resistance for worker exhalation because the purpose of such PPE is minimizing inhalation of dust. On the other hand, a mask design that prevents transmission of air-borne contaminants in both directions (inhalation and exhalation) is required in a hospital setting. The present study emphasizes the 3M model 1860 N95 mask because it is the most commonly used mask by health care workers confronting the current COVID-19 crisis. Moreover, other N95 masks that have been employed as alternatives in hospital settings are similar in design.

Such masks are constructed with a multi-layer fiber mesh that is coarse enough to allow adequate air flow, employing electrically poled polymeric

filaments to capture particles that would otherwise pass through such a coarse mesh. This electrostatic capture feature is critically important to mask breathability. A frozen-in-place charge separation present in the woven filaments of the inner mask material creates numerous locations among filaments with electric field gradients strong enough to trap particles via the induced dipole effect. Electrical poling of these fibers is carried out by applying a strong electric field during a specific stage of the fiber fabrication process. To avoid destroying the mask, it is vitally important that a candidate sterilization technique not electrically de-pole the fiber material by allowing charge migration, nor leave behind a residue having any significant electrical conductivity.

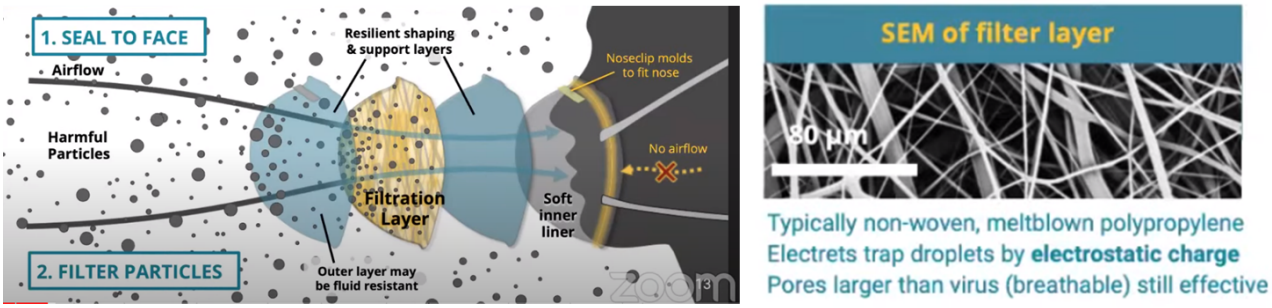

Figure 2: Details of N95 mask design. Any proposed sterilization process must not compromise the integrity/efficacy of the filtration layer, nor compromise mask features that ensure proper fitting of the mask to the face. Source: N95 Decontamination & Reuse Webinar | N95DECON & MGB, https://www.youtube.com/channel/UCG05UXgAI7mtRT_pN9TdSzg

It is important to appreciate that such masks were never intended to be cleaned and reused. They are of delicate construction and can easily be ruined by exposure to traditional sterilization methodologies such as autoclaving or direct application of alcohol. It is also important to remember that mask degradation may comprise (1) a significant reduction in particle filtration efficiency, (2) structural changes that impair proper fitting of the mask when donned, (3) degradation of specific components such as the elastic band used to hold the mask firmly in place, (4) the presence of biocidal agent residue that is toxic or irritating to the respiratory tract, or various combinations thereof.

During the past few months of the COVID-19 crisis, there are three sterilization methodologies that have been pressed into service for reuse of N95 masks on an emergency basis. These include hydrogen peroxide vapor treatment, UVC ultraviolet light treatment, and thermal treatment. A comprehensive review of research conducted on these three methodologies as of May 1, 2020 can be found in the form of a 2-hour video at the Mass General Brigham Center for COVID innovation.[2]

None of these three methodologies provide a fully satisfactory solution for multiple reuse of N95 masks. In the case of thermal methods, researchers have found that the temperature and time duration of heat exposure required to achieve sterilization (defined as a 10^6 reduction in viral load) is in excess of what can be tolerated by the mask. The lesser goal of

"decontamination", defined as a 10^3 reduction in viral load appears to be feasible, but this does not meet application requirements.

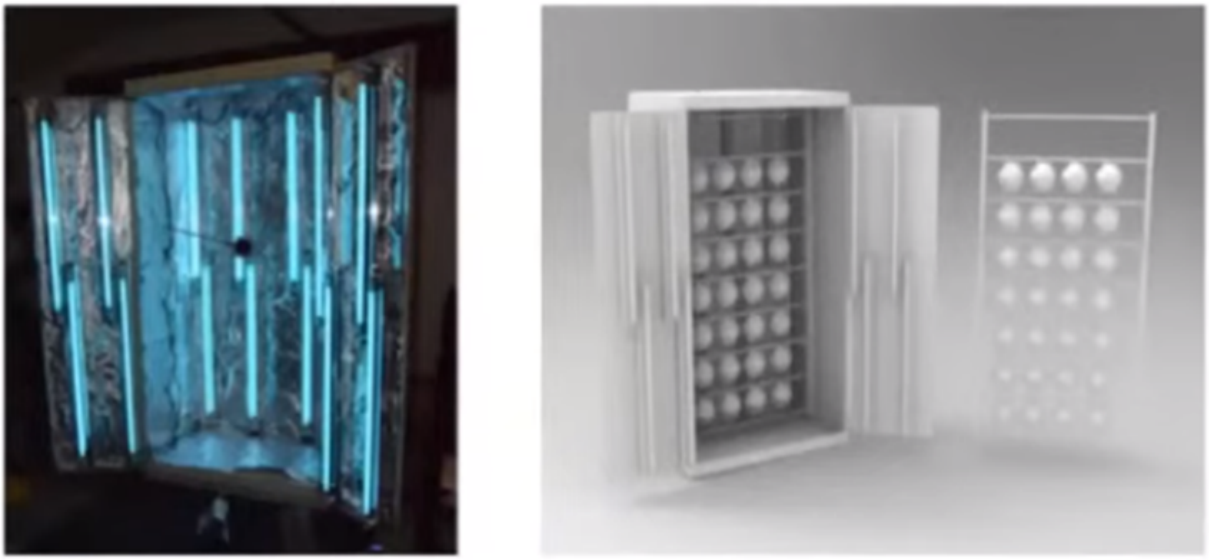

Figure 3: Examples of UVC chambers constructed for N95 mask sterilization.

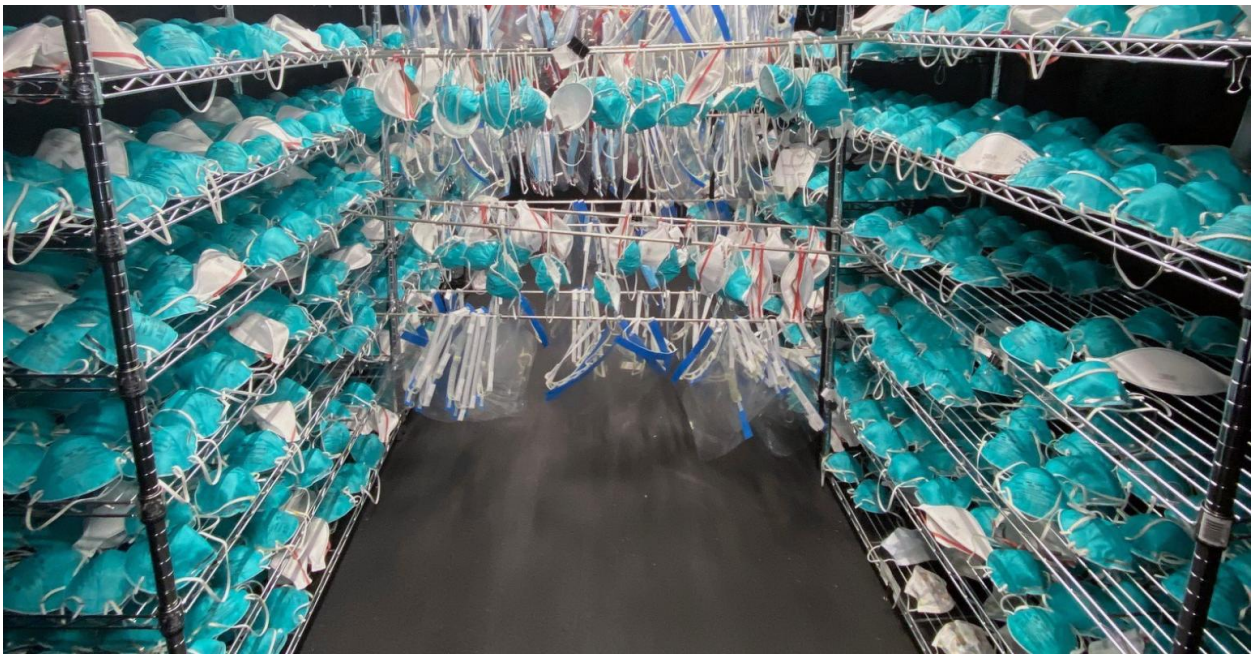

Figure 4: 3M 1860 masks being racked for Batelle's hydrogen peroxide vapor sterilization process.

UVC irradiation at 254 nm by exposure to low-pressure mercury discharge lamps can achieve a 10^6 reduction in viral load, but small numbers of masks must be individually racked to carefully avoid shadowing effects, and a secondary sterilization process must be applied to the elastic band. A further complication is that contaminants deep inside the microporous structure of the

masks are somewhat shielded from UVC light because of absorption and scattering by multiple layers of mask materials. The extent to which this shielding effect reduces the reliability of the UVC method is currently under study.

The hydrogen peroxide vapor (HPV) method provides a 10^6 reduction in viral load but also shares the work-flow impediment that contaminated masks need to be individually racked via manual labor. A lengthy aeration cycle is also required to ensure complete removal of hydrogen peroxide following sterilization because H_2O_2 is very irritating to the respiratory tract. HPV systems are also expensive to build. Batelle was recently awarded a contract to build sixty HPV sterilization modules at a cost of 6.9 million dollars each.[3]

Unlike thermal treatment, in HPV and UVC systems, the precautions required to ensure that all portions of the mask receive adequate biocidal dosage accounts for much of the complexity and tedium of applying these methods. Moreover, personnel tasked with individually racking thousands of contaminated masks by hand each day must exercise extreme caution because of potential COVID-19 transmission. And a major deficiency of all three methods is their inability to remove soil; thermal, UVC, and HPV seek to kill COVID-19 virus in place, but none of these methods clean the mask in a manner that removes foreign material. From the standpoint of aesthetics alone, this discourages the reuse of masks when visible soil is present. But the accumulation of foreign matter such as dried saliva is also a concern from standpoint of reduced air flow, especially in scenarios where individual masks may be reused 10 or more times. Finally, in the case of the UVC and HPV methods, both visible and invisible soil can function as barrier to biocidal infiltration, thereby introducing another unwelcome source of uncertainty.

1.3. Proposed Approach

We hypothesize that supercritical CO_2 processing in conjunction with a suitable biocidal additive will allow for multi-reuse processing of N95 masks using commercially available equipment that has been developed in the U.S. and abroad for the dry-cleaning industry. Supercritical carbon dioxide (CO_2) is a unique solvent that is becoming increasingly popular for industrial cleaning and chemical extraction processes. Upon entering the supercritical regime, the normally distinct liquid and gaseous phases of carbon dioxide merge into a single homogeneous phase that has the density, short-range order, and solvation capacity of a liquid, but the volume-filling and permeation properties that of a gas.

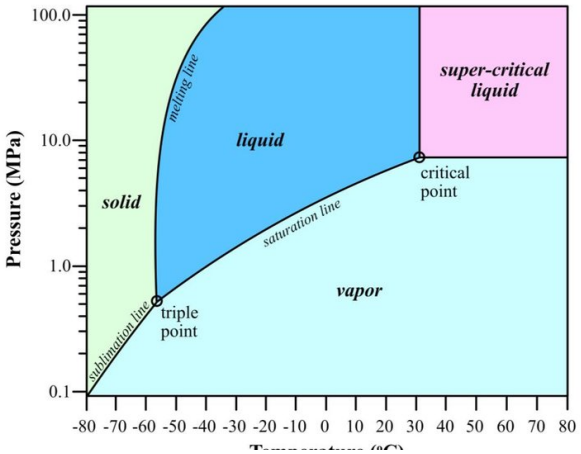

Figure 5: Carbon dioxide phase diagram

Supercritical CO_2 cleaning is becoming increasingly popular in the dry-cleaning industry[4] because it eliminates the need

for hazardous solvents such as perchloroethylene, it is suitable for use on even the most delicate fabrics, it can perform the function of a solvent for delivery and removal of chemical additives such as surfactants, and it leaves behind no dampness or chemical residue. It should also be noted that super critical CO_2 processing has long been used for various chemical extraction applications such as decaffeination of coffee beans on an industrial scale. The fact that supercritical CO_2 cleaning equipment is already deployed in the dry-cleaning industry is important to the question of wide-scale, cost-effective, rapid deployment.

Figure 6: COTS SC-CO_2 washing machine

In the proposed application, a key attribute of a super critical fluid is that it possesses the characteristics of a gas from standpoint of its ability to permeate into microporous materials and completely fill the volume of internal recesses and cavities independent of aspect ratio (i.e. leaving no place for contaminants to hide) while also having the properties of a liquid from the standpoint of its performance as a solvent. Supercritical CO_2's properties as a non-polar solvent allow it to serve as a vehicle for delivery of a variety of candidate biocidal additives. The reason that super-critical CO_2 is suitable for use on fabrics having a delicate microstructure is that the strong surface tension forces that can alter fabric microstructure during liquid-to-gas drying at the end of a solvent or water-based washing cycle can be circumvented by keeping carbon dioxide above its critical temperature of 31 C, such that the liquid-to-gas phase change cannot occur. Finally, as alluded to earlier, the ability of super-critical CO_2 to also remove soil is highly desirable in PPE reuse applications.

1.4. N95 mask sterilization workflow

We envision a very simple and safe workflow for super-critical CO_2 sterilization of N95 masks. Hospital workers would discard used masks into a container that is lined with a Tyvek bag, perhaps comparable in size to large garbage bag. When the Tyvek bag reaches capacity, it is heat sealed closed, after which its exterior surface is decontaminated (alternatively the Tyvek material could be pretreated with a biocidal agent). A collection of such sealed bags would then be transported to a local CO_2 dry cleaning facility, a CO_2 washing machine installed on site, or a mobile truck-mounted machine. The unopened bags would then be perforated (or perhaps unzipped) to allow rapid ingress/egress of CO_2, processed in super-critical carbon dioxide for tens of minutes, and then resealed for transport or storage. At the end of the cycle, the CO_2 is pumped back into a storage tank, and converted from supercritical fluid to back to gas as the pressure inside the cleaning vessel ramps down. The resealed sterilized bag and its sterile contents would then be returned to the hospital unopened. If desired, health care workers

could write their name on masks when they are newly issued. Alternatively, a passive RFID tag could be applied to each mask to facilitate sorting or barcoded masks could be used.

It will be noted that the proposed sterilization process requires very little labor, avoids exposing personnel to contaminated masks, and preserves masks in an uncontaminated state once sterilized. It is in fact very similar to the workflow used for sterilizing surgical instruments. Used surgical instruments are placed in a heat-sealed Tyvek bag, and then subjected to a penetrating sterilization process such as autoclaving, after which the unopened bags are returned for later use. As with the proposed supercritical CO_2 sterilization scheme, there are no shadowing effects or other uncertainties regarding portions of the mask that may have evaded adequate sterilization. The absence of surface tension in the supercritical state ensures that that even the most remote microporous regions and crevices receive treatment, regardless of the hydrophobicity of hydrophilicity of mask construction materials and any contaminants present. To ensure complete removal of the biocidal agent, if necessary, one or more super-critical CO_2 "rinse cycles" may be employed to ensure its complete removal. And if necessary, activated charcoal may provide a convenient means of any absorbing chemical residue that cannot simply be removed by distillation as CO_2 is converted back to the gas phase.

1.5. Sterilization mechanism

We hypothesize that supercritical CO_2 may kill CIVID-19 without the assistance of biocidal additives. One potential lethality mechanism is ultra-dehydration. Water has 0.01 mole fraction of solubility in supercritical carbon dioxide under the temperature and pressure conditions contemplated.[5] Supercritical CO_2 therefore acts as very strong drying agent.[6] Complete removal of water via osmosis through the virus envelope may irreversibly damage proteins and enzymes.

Prior to extraction of water, the high partial pressure of CO_2 present should also acidify water inside the virus envelope to approximately pH = 3.[7] This is analogous to the immediate conversion of deionized water to highly carbonated water upon application of pressurized carbon dioxide. Such a precipitous drop to low pH may also be destructive towards proteins and enzymes.

Finally, unlike proposed sterilization methods based on heat, UV light, or exposure to hydrogen peroxide, the proposed super-critical CO_2 cleaning process has what we refer to as "the elution advantage". That is, virus particles can be destroyed, but they can also be simply washed away.

At the end of the CO_2 washing cycle, supercritical CO_2 containing soil, lint, virus (dead or alive), and other contaminants is re-purified by distillation to CO_2 gas. The small amount of dried foreign material left behind inside the sump trap of the CO_2 washing machine can then be aggressively sterilized by whatever means is desired (e.g. 200 C heat). The ability of supercritical CO_2 to solvate materials that might otherwise be retained within the mask material is therefore important.

On the other hand, it may be that supercritical CO_2 alone is not capable of killing the COVID-19 virus, or there may be application requirements for the destruction of other less vulnerable pathogens that necessitate the use of biocidal additives. Future work will therefore be required to explore the efficacy of candidate biocidal additives and/or surfactants to ensure adequate sterilization and soil removal, respectively. Biocidal additives currently under consideration include anhydrous materials that can preserve the ultra-dehydration properties of super-critical CO_2, and which we hypothesize will be fully soluble in super-critical CO_2. For example, hydrogen peroxide is known to be highly effective against COVID-19 and a wide variety of other

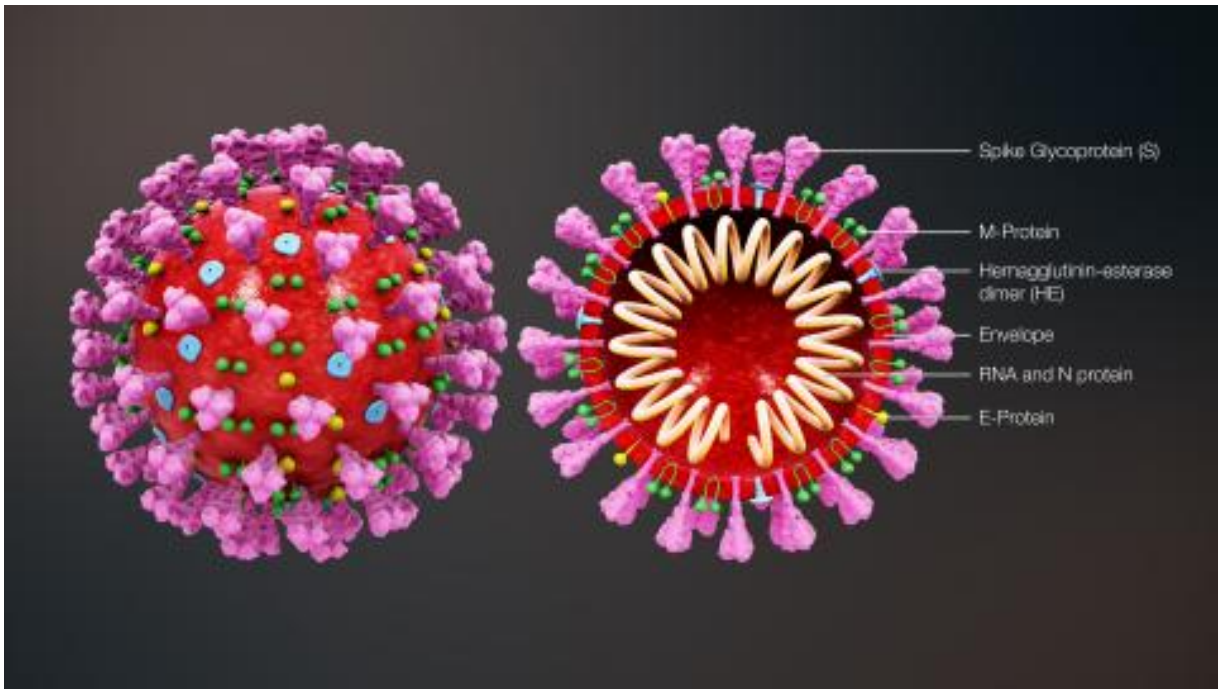

Figure 7: Anatomy of the COVID-19 virus. Source: American Society for Microbiology, https://asm.org/Articles/2020/January/2019-Novel-Coronavirus-2019-nCoV-Update-Uncoating

pathogens. It is typically used a concentration of one hundred to several hundreds of ppm. We propose to evaluate urea-hydrogen-peroxide complex (also known as "carbamide") as a means of introducing H_2O_2 while maintaining highly anhydrous conditions. Other anhydrous biocidal agents we suspect will be soluble in super-critical CO_2 include quaternary amines such as benzalkonium chloride, and sodium dichloro-s-triazenetrione, also known as dichloroisocyanuric acid. Sodium dichloro-s-triazenetrione is powerful chlorinator and oxidizer. But unlike sodium hypochlorite (bleach), chemical stability considerations do not require that it be delivered in the form of an aqueous solution. Note that a key characteristic of the three chemical reagents described above is that are widely available and can be handled by unskilled personnel.

Ultra-dehydration may not turn out to be important to lethality, however, in which case a wider variety of water-borne biocidal agents may be employed. One leading candidate is peracetic acid, which has already demonstrated high biocidal activity in supercritical CO_2. In 2006, White et al. demonstrated greater than 6 log reduction of B. stearothermophilus spores for 5%

peracetic acid in SC-CO_2.[8] This represents an extremely stringent test of biocidal activity. Accordingly, the question of what biocidal agents if any will ultimately be used in this application is not of central importance. We know supercritical CO_2 can deliver highly effective biocidal agents such as peracetic acid, and we know that supercritical CO_2 provides a convenient means of fully removing such biocidal agents at the end of the sterilization cycle. Therefore, the key questions are:

1) Can supercritical CO_2 can perform these functions without damaging any of the delicate materials used in mask construction?
2) Can we experimentally verify that CO_2 provides a vehicle for delivery of biocidal agents into the deepest recesses of such microporous structures?

2. CONSTRUCTION OF LABORATORY APPARATUS

The proposed work required the construction of two different types of apparatus to achieve two distinct goals. First, we needed a supercritical CO_2 pressure vessel large enough to treat whole masks, and ideally, many masks at a time. Second, we need an apparatus for processing of virus-inoculated mask fabric swatches to demonstrate the microporous penetration capability of the proposed sterilization methodology. Moreover, we will ultimately need to demonstrate lethality against live COVID-19 virus, even if it is already implied by lethality against more robust bio-contaminant species. Such a compact SC-CO_2 processor must fit inside a standard bio-safety cabinet, and it also must meet all requirements for sterilizability. Evaluation of this compact SC-CO_2 processor in Sandia's existing BSL-2 laboratory was an immediate objective, but a 2nd SC-CO_2 processor was constructed in the event that access to live COVID-19 virus in an offsite BSL-3 facility could be arranged (working under the assumption that such an apparatus will be not returned once it enters a BSL-3 facility). Hereafter we will refer to these as the "fabric swatch test apparatus" and the "whole mask test apparatus".

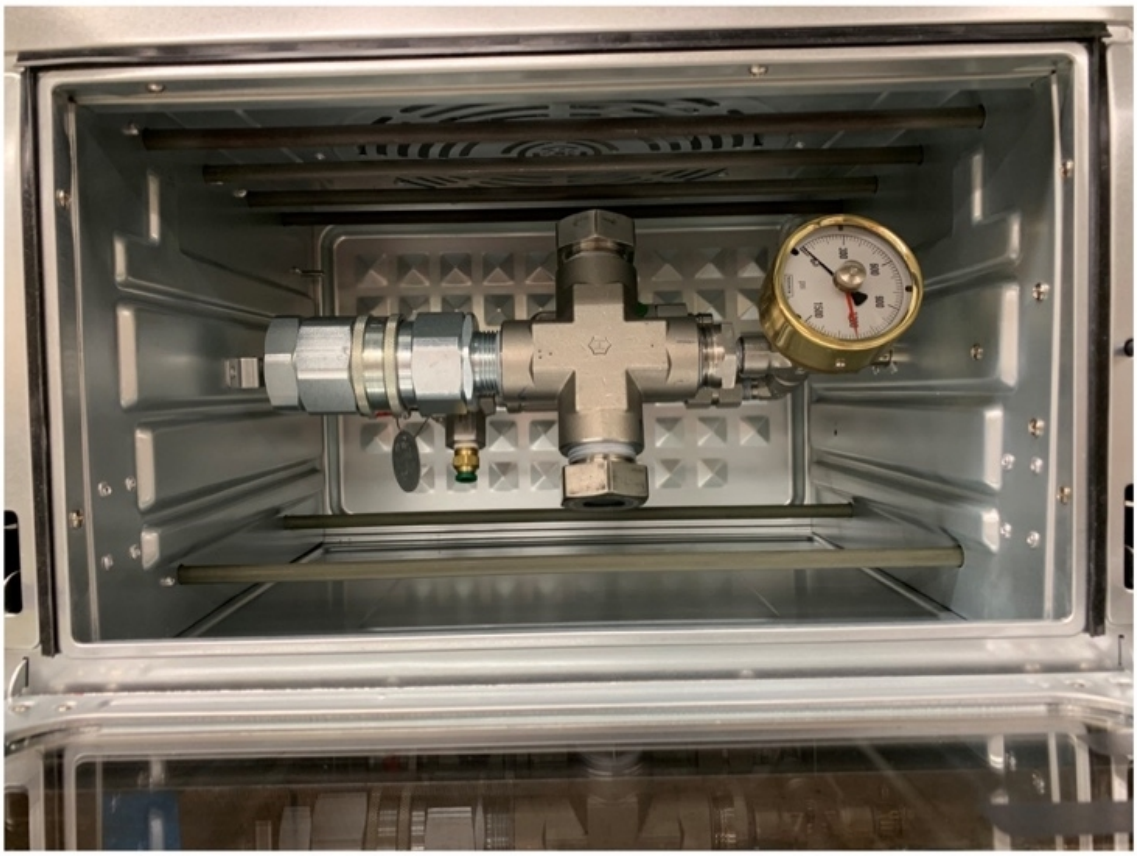

Figure 8: Fabric swatch test apparatus inside warming chamber with heating elements, convection fan, and rotisserie drive visible.

Referring to Figure 8, the fabric swatch apparatus is a high-pressure cell that is filled with liquid carbon dioxide (CO_2) at room temperature, and then placed in a 37 C temperature-controlled

chamber to affect the liquid-to-super-critical phase transition of CO_2. An operating temperature of 37 C was selected because it is above the supercritical temperature of carbon dioxide (31 C) and it ensures that mask materials will not experience temperature excursions in excess of what is normally encountered in the field. But, it's very likely that supercritical CO_2 processing at higher temperatures (e.g. 50 C) will prove to be permissible. This has important practical implications because as discussed later, accurate temperature control during vessel pressurization and depressurization presents some challenges.

The fabric swatch apparatus shown in Figure 8 also requires a CO_2 filling/venting apparatus. Early versions comprised a Size 1A CO_2 gas cylinder equipped with an internal syphon tube for dispensing liquid, and a custom-built gas handling system for dispensing of CO_2 liquid and venting of CO_2 gas. Later versions employed a much smaller 5 lb. CO_2 cylinder to allow the entire experimental apparatus to reside within the confines of a bio-safety cabinet.

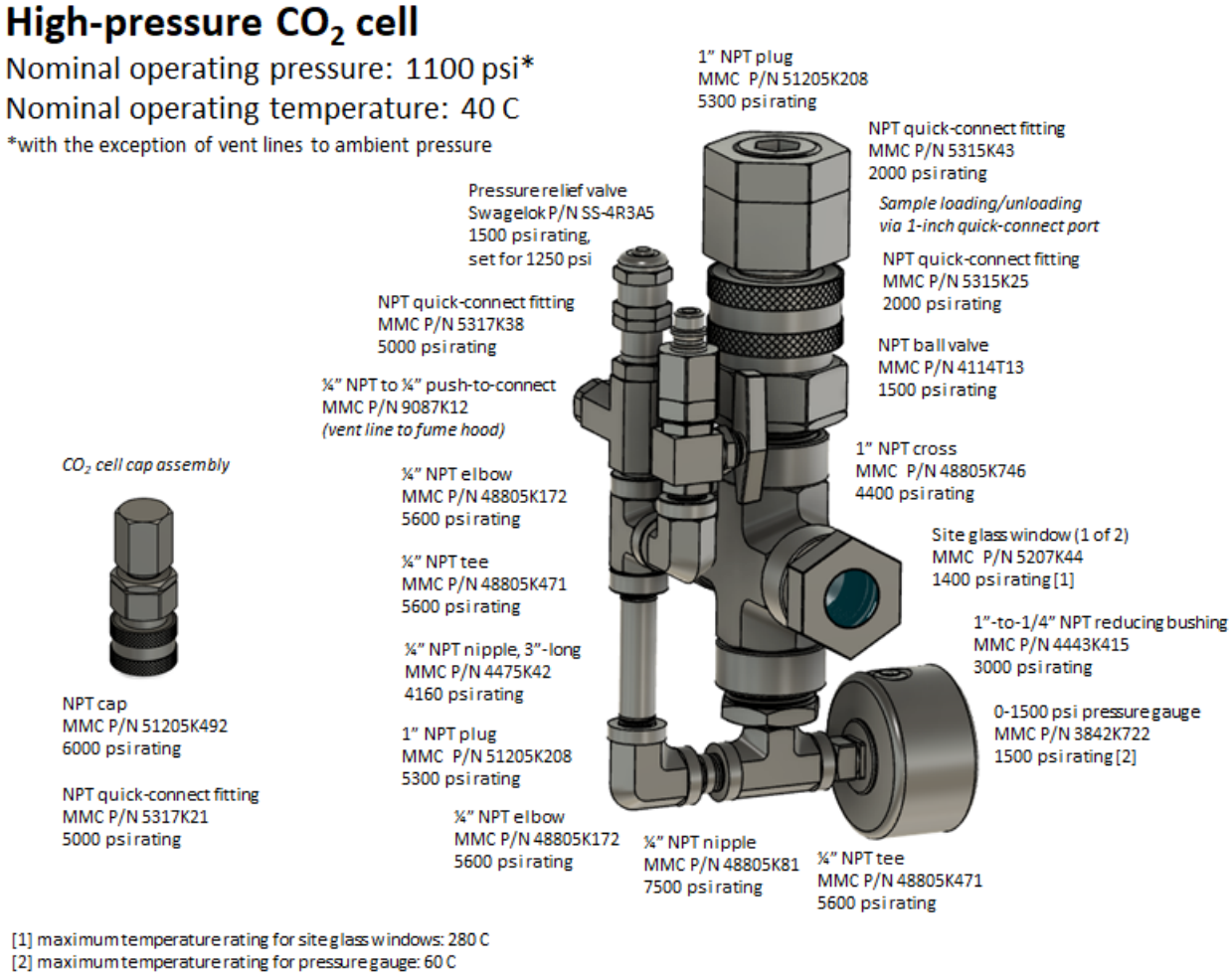

Figure 9: Design details for fabric swatch high-pressure cell

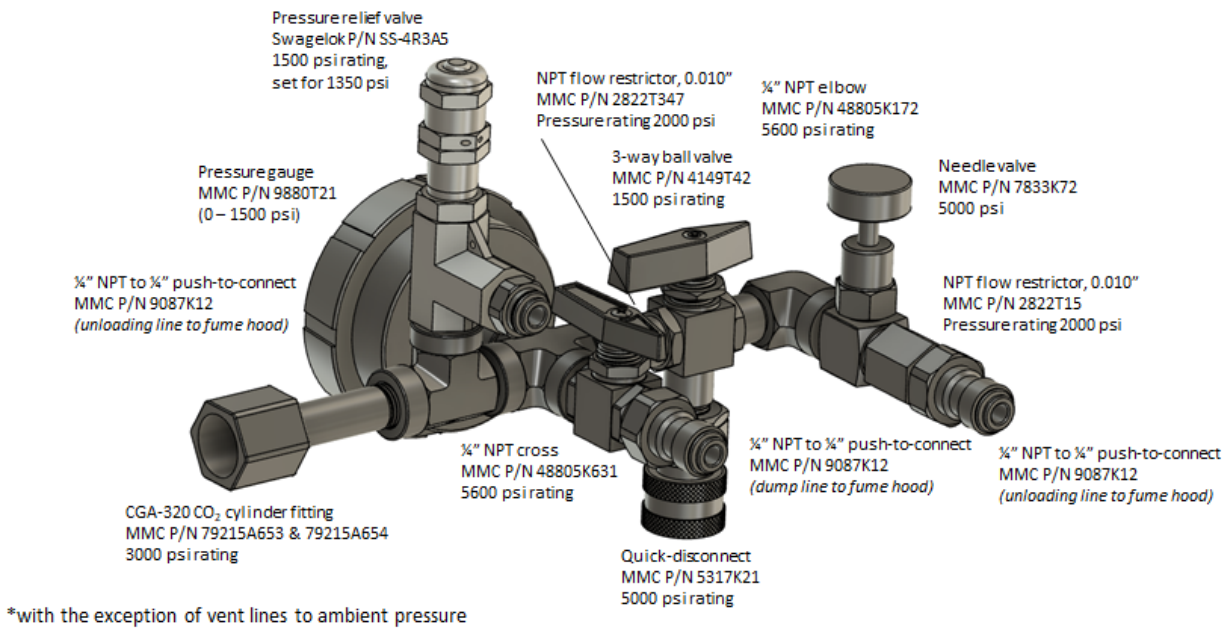

Figure 10: Design details of CO_2 loading/unloading apparatus

The high-pressure cell used for processing of fabric swatches is constructed entirely from commercial, off-the-shelf, pressure-rated, components procured from McMaster-Carr and Swagelok. There are no custom-made components used anywhere in high-pressure cell, or anywhere in the associated CO_2 filling/venting apparatus. The internal volume of the gas cell is 180 ml. A volume of approximately of 115 ml of liquified carbon dioxide added to the cell during the filling operation. The 4-port pressure cell is constructed from a 1-inch NPT cross. Port 1 is used for filling and venting of carbon dioxide. Ports 2 and 4 accommodate sight glass windows. This allows the level of CO_2 liquid to be determined by visual inspection, and visual confirmation of the liquid-to-super-critical phase transition once the high-pressure cell is placed in the warming chamber. Port 3 is used for sample loading and unloading. The system component with lowest pressure rating (1400 psig) is the site glass, McMaster-Carr P/N 5207K44. The pressure ratings of other components range from 1500 to 7500 psig, as documented in the figures below. Both the CO_2 filling/venting apparatus and the high-pressure cell have independent pressure gauges and pressure relief valves. Under normal operating conditions, after the sample (e.g. a 1 cm^2 swatch of N95 mask fabric) is loaded into the pressure cell, liquified CO_2 is added to the cell, and the pressure cell is placed in a warming chamber that is maintained at 37 C.

Operating procedure:

Loading of the CO_2 into the high pressure cell

1) Start with all valves in their closed/blocked positions.
2) Connect CGA 320 fitting to CO_2 cylinder equipped with syphon tube
3) Connect CO_2 gas cell to fill plumbing via quick-connect coupling.
4) Rotate 3-way valve gas delivery position.
5) Open ball valve on gas cell to permit filling.
6) Crack gas cylinder to begin filling cell.
7) Close ball valve on gas valve when desired liquid level is reached.
8) Close gas cylinder valve.
9) Rotate 3-way valve to venting position.
10) Crack needle valve to bleed off excess CO_2.
11) Disconnect CO_2 gas cell to fill plumbing via quick-connect coupling.
12) Rotate 3-way valve back to blocked position.
13) Install quick-connect cap to blank off gas cell fill port.

Unloading of CO_2 from the high pressure cell

14) Reconnect CO_2 gas cell to fill plumbing via quick-connect coupling.
15) Rotate 3-way valve to venting position.
16) Open ball valve on gas cell to permit venting.
17) Crack needle valve to slowly bleed off excess CO_2.
18) Rotate 3-way valve back to blocked position.

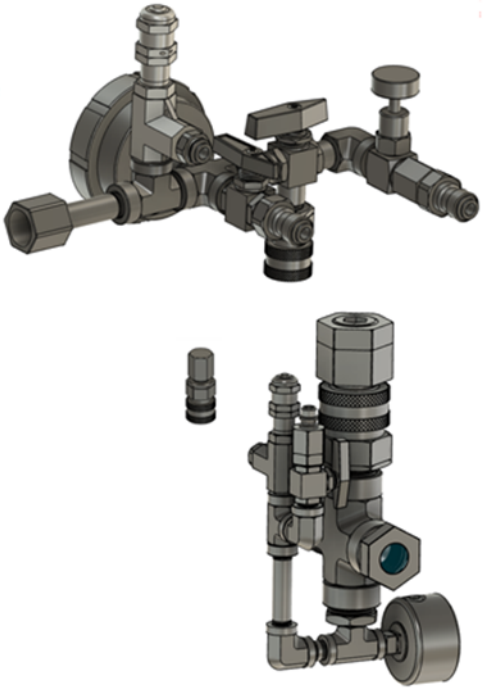

Figure 11: Operating procedure for loading/unloading CO_2 into fabric swatch test apparatus

The supercritical point for CO_2 is 31 C and 1080 psia. As noted earlier, we operate the cell at a temperature of 37 C and a pressure of 1200 psig to ensure that supercritical conditions are maintained throughout the duration of the disinfection cycle, which is nominally 1 hour. The warming chamber features a light to aid in visual detection of the liquid-to-super-critical phase transition, and both a circulating fan and a 3-rpm pressure cell rotisserie to promote isothermal conditions. Cell rotation is also desirable from the standpoint of mixing candidate biocidal agents. To ensure compatibility with candidate biocidal agents, all pressure cell components are stainless steel, with the exception of the sight glass windows which are borosilicate glass.

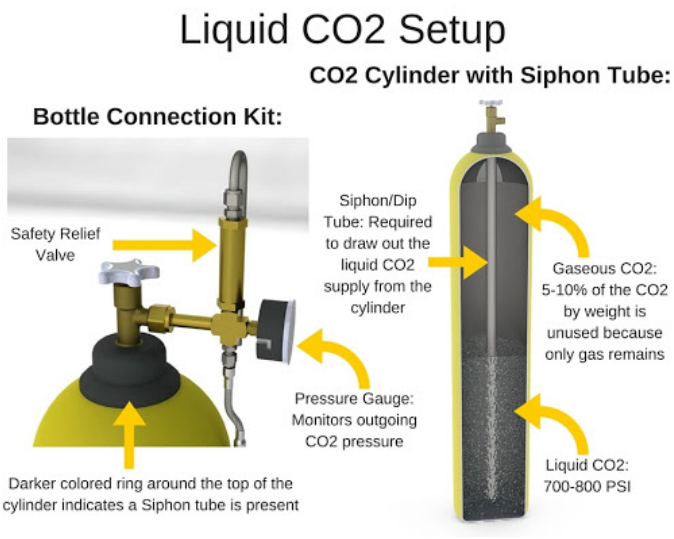

Figure 12: Anatomy of a siphon-tube CO_2 gas cylinder

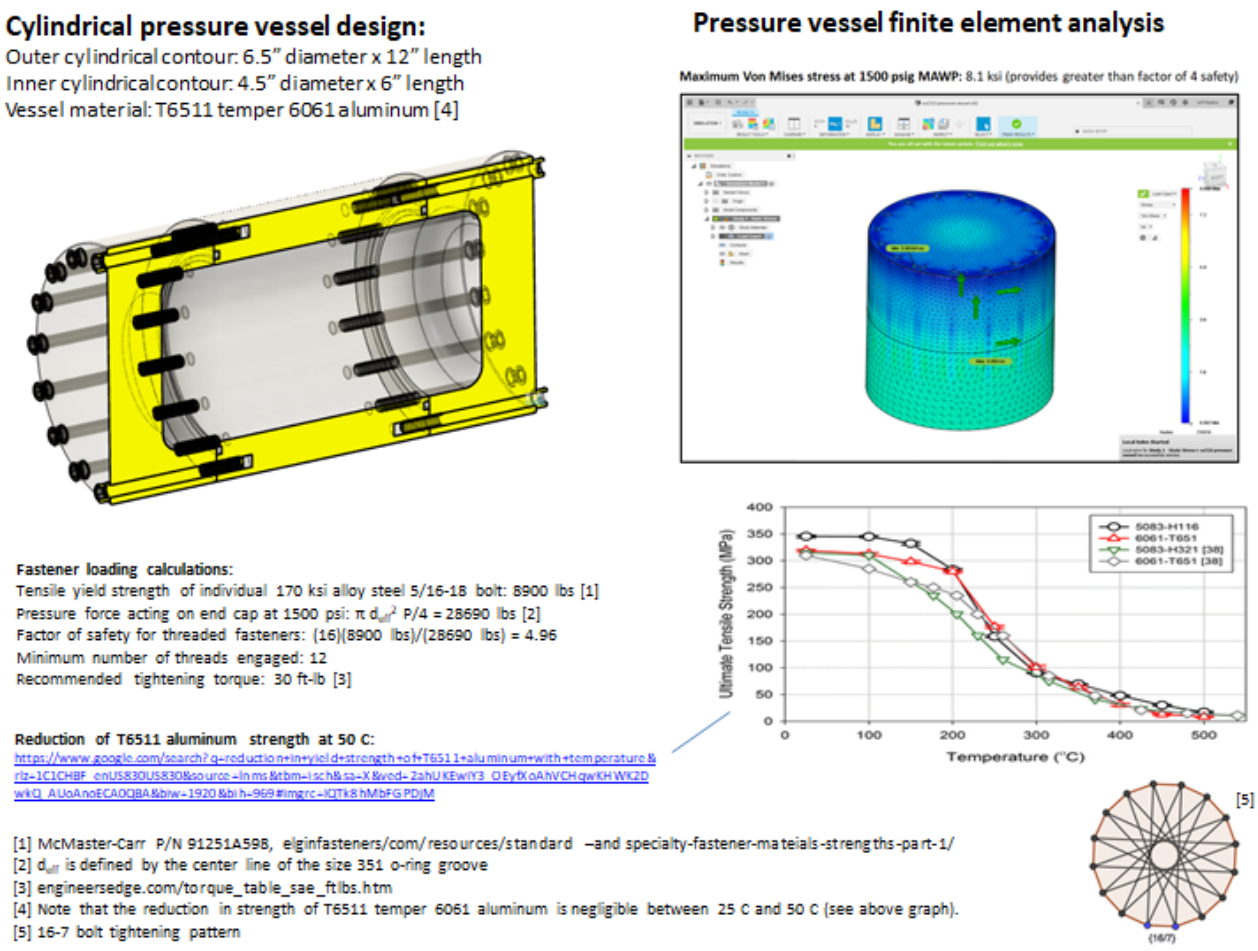

Figure 7: Summary of pressure vessel design for whole mask apparatus

The system component with the lowest maximum temperature rating (60 C) is the pressure gauge of the pressure cell (McMaster-Carr P/N 3842K722). The warming chamber employs a 35 W resistive heater that is powered by a fixed voltage (36 VDC) power supply. This low-power, voltage-limited, resistive heater is incapable of raising the oven temperature more than 15 C above ambient temperature. The use of an intrinsically power-limited heating element prevents the possibility of overheating due to user error or controller malfunction. Protection from overheating in the unlikely event of an ac/dc converter overvoltage malfunction is provided by 400 mA cartridge fuse at the line voltage input of the dc power supply. If somehow overheating were to occur, the pressure relief valve of the pressure cell would gradually vent CO_2 into the warming chamber, which is a vented enclosure that resides inside the bio safety cabinet. Implementation of redundant engineering controls to prevent over-pressurization by over overheating was deemed prudent in the unlikely event that the pressure relief valve malfunctions. The lack of temperature adjustment on the warming chamber also prevents a potential source of operator error.

Figure 4: Thick-walled aluminum pressure vessel constructed for whole mask testing.

Maximum Von Mises stress at 1500 psig MAWP: 8.1 ksi (provides greater than factor of 4 safety)

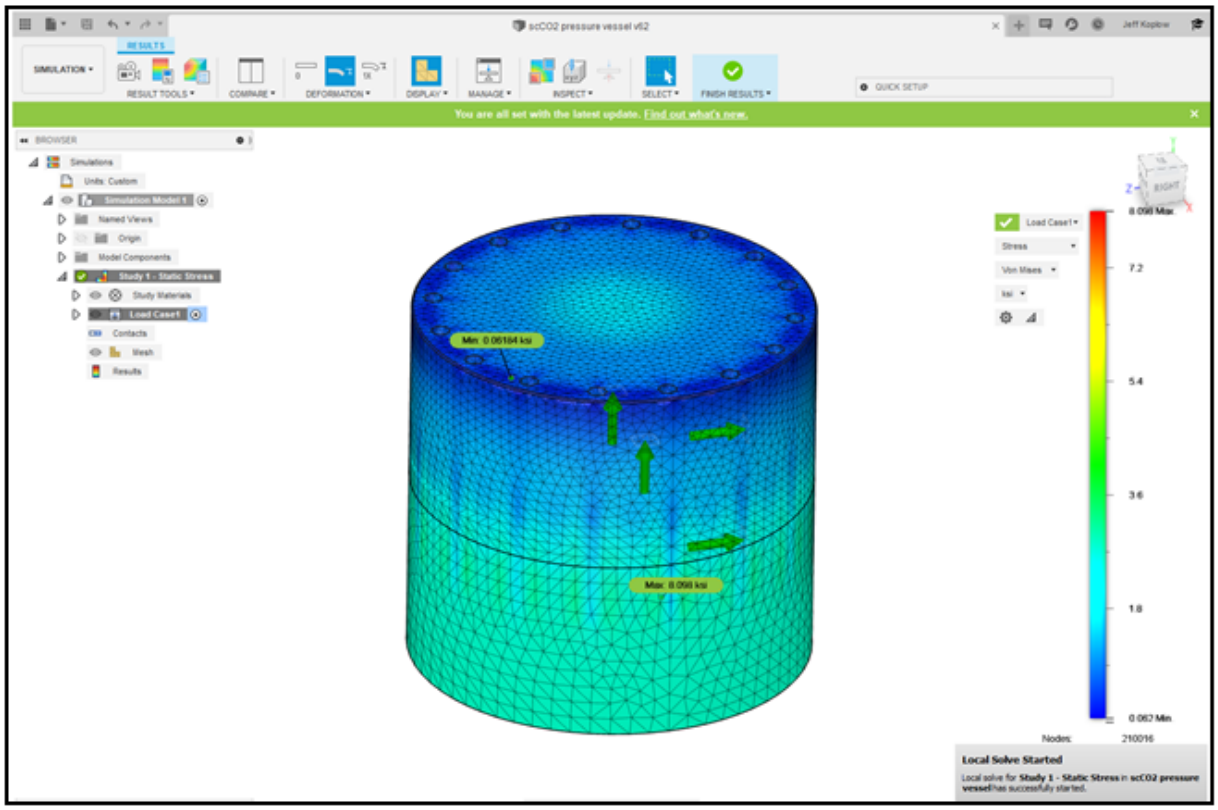

Figure 15: Finite element mechanical stress analysis of whole mask apparatus pressure vessel

Development of the whole mask test apparatus required construction of a much larger pressure vessel. Referring now to the cylindrical pressure vessel illustrated in Figures 13, 14, and 15, an internal diameter of 4.5” was required to allow 1860 masks to be loaded into the chamber with minimal folding or bending. The internal length of the cylindrical pressure vessel is 9.0”. This is large enough to allow eight 1860 masks to be treated at one time. The whole mask pressure vessel is constructed from T5611 temper alloy 6061 aluminum having a minimum wall thickness of 1.0”. The chamber comprises a central cylindrical tube and two end caps, each of which is sealed by EPDM o-rings and mechanically secured be a circular array of sixteen, size 5/16”-18 high-strength steel alloy bolts. Both the pressure vessel and bolts arrays used to construct this apparatus are engineered to have a factor of safety of approximately 5. Finite element analysis was used to calculate the stress field in the chamber walls and minimum factor of safety. This custom-built vessel was then hydrostatically tested at 2300 psi for 5 minutes prior to commissioning.

3. DEVELOPMENT OF EXPERIMENTAL PROTOCOLS

We initially provided team member Oscar Negrete with samples of 8210 N95 masks that could be diced into samples and inoculated with the surrogate virus. Although the same anti-viral test protocol was eventually be applied to 3M 1860 masks, for initial testing we opted to conserve our very limited supply of 1860 masks for the time being. The 8210 N95 masks are however very similar in construction.

Figure 16: 8210 mask material used in early tests

Small swatches of N95 mask material were cut into 1 cm^2 squares and surface contaminated on the exterior with vesicular stomatitis virus (VSV), Indiana serotype strain. 10 μl of the virus suspension was used which equates to a starting concentration of 10^6 plaque forming units (pfu). Following one hour of air drying in a biosafety cabinet, one of test swatches was placed inside the super critical CO_2 chamber and the other in a petri dish as a control for the involvement of temperature and time in VSV decay.

Following application of supercritical CO_2 processing, virus was eluted from the mask material by soaking the mask square in 1 ml of virus culture medium. After 10 minutes of soaking, the liquid was centrifuged at 300 g for 2 minutes to concentrate the media at the bottom of the tube. 1 ml of media was also used to rinse the tubing used for the CO_2 gas vent line through which CO2 is released at the conclusion of the one-hour treatment cycle. A series of 10-fold dilutions were used to measure virus viability using a standard plaque assay on Vero cells. 48 hours after infection, plaques were counted and viral titers were calculated.

Prior to running the experiment with live surrogate virus, the team conducted two dry run experiments under the observation of bio-safety officer Natalie Jouravel and pressure-safety officer George Buffleben. The sequence of movements and operations performed by the team was carefully scrutinized to detect any potential opportunity for inadvertent release of surrogate virus. It was determined that the series of sample insertion, gas filling, gas venting, etc. operations required to perform the experiment precludes the possibility of virus release, is fully consistent with all pressure safety requirements, and that slowly vented CO_2 gas does not disturb the laminar flow field inside the bio safety cabinet.

The experimental first dry run did however reveal that the pressure relief valves used on the supercritical CO_2 fabric swatch cell were unreliable for this application. Upon actuation, the cooling effect of super-critical CO_2 flash evaporating to CO_2 gas caused enough localized to cooling to prevent proper resealing of the o-ring inside the pressure relief valve. For this reason, we replaced our original pressure release valves (Swagelok P/N SS-4A3R5) with Circle Seal P/N

5180B-2MP-1400 pressure relief valves rated for operation down to a temperature of -100F. This solved the pressure relief valve malfunction.

4. EXPERIMENTAL RESULTS

To evaluate the question of whether the construction materials of the N95 are altered or harmed in any way during supercritical CO_2 processing, early in this project we consulted with polymer chemist LeRoy Whinnery to compile a list required test criteria.

1) Pressure/flow-rate curve for air flow through N95 mask.
2) Glass transition temperature measurement of the fibrous polymeric material (melt-blown, non-woven, polypropylene fiber) used to construct the mask via differential scanning calorimetry.
3) Measurement of compression set and durometer hardness for elastomeric components.
4) Microscopy of cross-sectioned material samples to discern changes in polymeric fiber morphology.
5) Determination of the effect of supercritical CO_2 exposure to the electrostatic filtering properties of the mask.

Shortly thereafter, NIOSH, the government agency responsible for certification of respirator masks and other occupational safety equipment stood up a mask testing program. Under this new NIOSH program, research institutions working on candidate N95 sterilization techniques can send treated masks to NIOSH to verify that they still function properly. The battery of tests established by NIOSH and the specialized equipment on which such tests are run are exactly those used to certify N95 masks made by various manufactures before they can be placed on the market. To obtain adequate statistics, NIOSH requires participating institutions to submit 20 masks, 15 of which are given the proposed sterilization treatment, and 5 of which are used as control samples. These tests cover all aspects of mask performance such as pressure/flow characteristics, particle filtration efficiency, and questions surrounding proper fitting of masks to standardized mannequin test fixtures.

It will be recalled that the custom-built pressure chamber for whole mask testing was constructed with an internal diameter of 4.5” and an internal length of 9.0”. This is large enough to allow eight 1860 masks nested on inside another to be treated at one time. The set of fifteen 3M 1860 masks was processed in two batches, each subjected to the same cleaning protocol. In each cycle, the CO_2 pressure was gradually ramped to 1200 psi over a period of 15 minutes, maintained at 1200 psi (at a temperature of 37 C) for 60 minutes, and then ramped down to atmospheric pressure over a period of 15 minutes. This cleaning cycle was repeated 10 times to simulate the effect of multiple reuse. The ability to process multiple masks at one time helped reduce time and labor, but for follow on work we strongly recommend automation of the above cycle including closed loop pressure and temperature control. Conducting these operations manually was tedious and very time consuming.

Before the above batch processing of masks was executed, we conducted a variety of one-mask/one-cycle experiments to check for the following potential problems.

1) Warpage or deformation
2) Loss of mechanical strength
3) Loss of mechanical flexibility (e.g. due to fabric dehydration)
4) Chemical incompatibility with SC-CO_2 (e.g. the adhesive used to secure the foam nose cushion strip)
5) Damage to the elastomeric band used to hold the firmly mask in place

The results of such a test are shown in "before and after" figure below. The 1860 mask did not suffer from any of the above five problems. Although the CO_2 cleaning process did cause fading of its stenciled label on the 1860 mask, treated and untreated 1860 masks were otherwise indistinguishable.

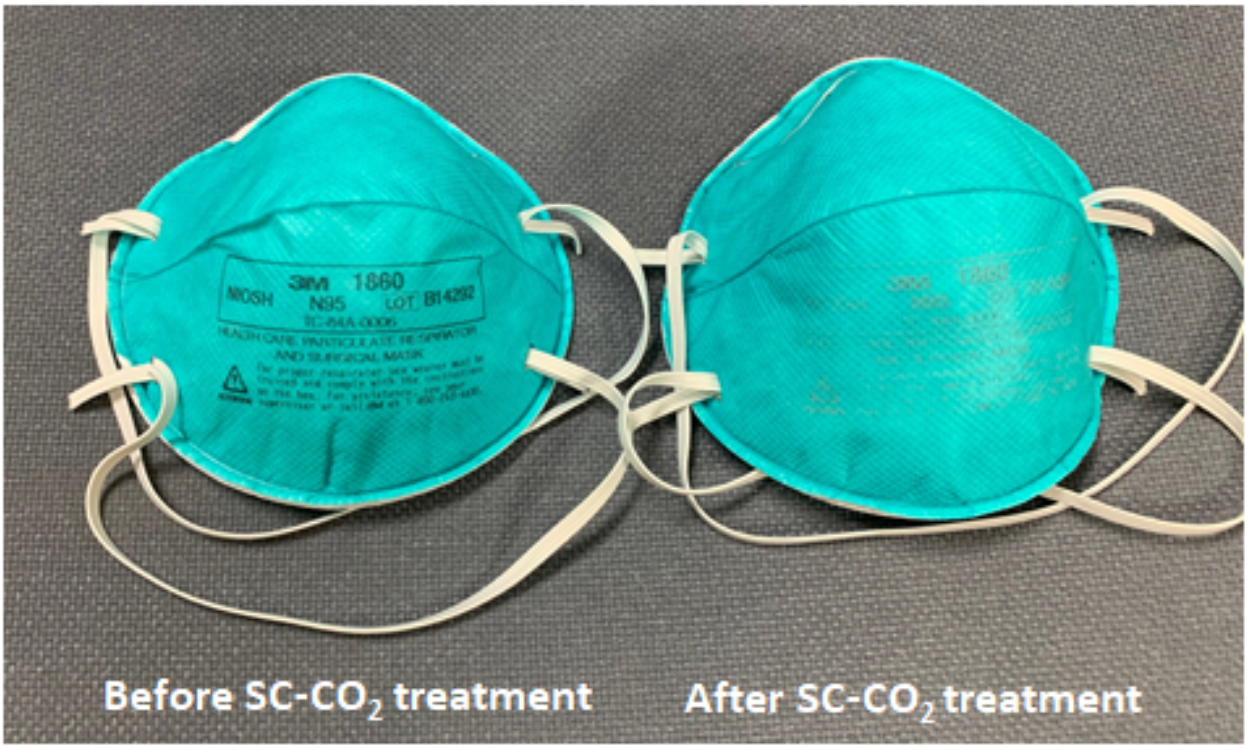

Figure 17: Before and after photos of a 3M type 1860 N95 mask subjected to one 60-minute supercritical CO_2 treatment cycle.

Figure 18: Triplicate viral assays indicate that super-critical carbon dioxide alone does not exhibit biocidal activity against the VSV surrogate virus. This suggests that a biocidal additive such as peracetic acid will be required for N95 mask sterilization.

We also verified that a black sharpie marker could be used for colorfast labelling of the masks, and we tested a variety of RFID tags to demonstrate their compatibility with supercritical CO_2 cleaning. The ability to return masks to their original owners is important from the standpoint of successfully encouraging reuse.

Evaluation of Decontaminated N95 Respirators

NPPTL
National Personal Protective Technology Laboratory

Organization: Sandia National Laboratories

Date Tested: 7/15/2020 – 7/16/2020

Respirator Model(s): 3M 1860

Tests: Filtration with NaCl (modified version of STP-0059), Manikin Fit Factor with Static Advanced Headform, and Strap Integrity with Tensile Testing

Decontamination Method: New, never-used FFRs are treated with liquid-to-vapor supercritical CO_2 at a temperature of 37°C and a pressure of 1100 psig for 1 hour

Decontamination Cycles: 10 cycles

While decontamination and reuse of FFRs are not consistent with standard and approved usage, these options may need to be considered when FFR shortages exist. This assessment was developed to quantify the filtration efficiency and manikin fit factor[1] of an N95 respirator that has been decontaminated. This assessment is not to determine the effectiveness of the decontamination procedure at killing pathogenic microorganisms. The results provided in this report are specific to the subset of samples that were provided to NPPTL for evaluation. These results may be used to update the CDC guidance for Crisis Capacity Strategies (during known shortages).

20 respirators that were unworn and not subjected to any pathogenic microorganisms were submitted for evaluation. This included 15 respirators that were subjected to 10 cycles of the supercritical CO_2 decontamination process and an additional 5 respirators that served as controls. Figure 1 photos document the procedures used. Photos of the samples and information received are shown in Figures 2 and 3. The samples were tested using a modified version of the NIOSH Standard Test Procedure (STP) TEB-APR-STP-0059 to determine particulate filtration efficiency. The TSI, Inc. model 8130 using sodium chloride aerosol was used for the filtration evaluation. For the laboratory fit evaluation, a static manikin headform was used to quantify changes in manikin fit factor. The TSI, Inc. PortaCount® PRO+ 8038 in "N95 Enabled" mode was used for this evaluation. Additionally, tensile strength testing of the straps was performed to determine changes in strap integrity. The Instron® 5943 Tensile Tester was used for this evaluation. The full assessment plan can be found here.

Filtration Efficiency Results: None of the treated respirators measured more than 95%. See Table 1.

Manikin Fit Factor Results: The manikin fit factor showed passing fit factors (greater than 100) for 4 of the 5 respirators evaluated. See Table 2.

Strap Integrity Results: No visual degradation of the straps was observed. The top strap showed a less than 1.00% decrease in recorded force and the bottom strap showed a 2.18% increase in force. See Table 3.

Other notes: The respirator information on the front of the mask was discolored (turned from black to white). The inner surface of the respirators was tinted green in some places. See Figure 1.

Figure 19: Summary documentation for NIOSH tests conducted on fifteen SC-CO_2 treated 3M 1860 N95 masks and five control samples.

Figure 20: Left-hand panel: SC-CO_2 treated masks affixed to planar flange for flow resistance measurements. Center panel: SC-CO_2 treated masks tested for proper fit under standardized pressure/flow conditions. Right-hand panel: Discoloration resulting from transfer of dye from an adjacent mask during immersion in SC-CO_2. The use of a SC-CO_2 colorfast dye would be desirable to prevent such staining, because aesthetics has a bearing on the willingness of health care workers to reuse masks.

Referring now to Figure 18, test results from the virology laboratory indicate that SC-CO2 alone does not exhibit biocidal activity with respect to the with vesicular stomatitis virus (VSV) Indiana serotype strain surrogate virus. This is an indication that a biocidal agent such as peracetic acid will most likely be required. Lack of access to a BSL-3 facility precluded the same experiment with live corona virus, however.

The results of the standardized NIOSH mask tests are presented in Figures 19-23. The “Manikin Fit Factor” test data shown in Figure 21 collected suggest that:

(a) 10 cycles of SC-CO_2 treatment will likely not compromise mask fit in the intended application, but

(b) bending of the N95 masks to accommodate the small size of our SC-CO_2 test chamber measurably chamber degraded the fit factor of the masks.

Indeed it was apparent upon removal from the chamber that the mask fabric had taken a set to the bent geometry, and in particular that the bottom most mask in the stack of 8 masks (experiment #1) and 7 masks (experiment #2) was deformed more severely (splayed out) than the masks located above it. We suspect this accounts for sample 15 having an anomalously low fit factor, because samples 8 and 15 were the bottom-most masks in the experimental runs 1 and 2 respectively. Moreover, no such deformation was observed when a single N95 masks was

processed in the chamber, presumably because the mask didn't need to be subjected to significant bending to make it fit within the chamber.

Table 2. Manikin Fit Evaluation

Manikin Fit Factor of Decontaminated N95s					
Respirator Model, Decon Method, # of cycles	**Treated Sample #**	**mFF Normal Breathing 1**	**mFF Deep Breathing**	**mFF Normal Breathing 2**	**Overall Manikin Fit Factor**
3M 1860, Supercritical CO2, 10 Cycles Static Advanced Medium Headform (Hanson Robotics)	**11**	173	56	165	101
	12	200+	77	180	127
	13	191	56	152	102
	14	200+	70	163	118
	15	70	43	55	**54**
	Control 4	200+	200+	200+	200+
	Control 5	200+	200+	200+	200+

Notes:

- Per OSHA 1910.134(f)(7), if the fit factor as determined through an OSHA-accepted quantitative fit testing protocol is equal to or greater than 100 for tight-fitting half facepieces, then the fit test has been passed for that respirator.
- This assessment does not include fit testing of people and only uses two exercises (normal and deep breathing) on a manikin headform.
- This assessment is a laboratory evaluation using a manikin headform and varies greatly from the OSHA individual fit test. This headform testing only includes normal breathing and deep breathing on a stationary (non-moving) headform; therefore, fit results from this assessment cannot be directly translated to using the standard OSHA-accepted test. Instead, this testing provides an indication of the change in fit performance (if any) associated with the decontamination of respirators.
- **BOLD** overall manikin fit factors < 100.

Figure 21: The "Manikin Fit Factor" test data collected strongly suggest that 10 cycles of SC-CO_2 treatment will not compromise mask fit. The small size of our SC-CO_2 test chamber forced us to bend the masks to some degree, and it was apparent that the mask fabric took a set to this bent geometry over time. This likely accounts for sample 15 having a low fit factor. In an actual SC-CO_2 washing machine, there would be no need to bend/compress the masks to fit them into the pressure chamber.

The remainder of the mask fit data is consistent with this interpretation as well. The mask fit scores for samples 11, 12, 13, and 14 were 101, 127, 102, and 118 respectively. When referenced to the control samples (4 and 5), this data is consistent with the adverse effect of mask bending, but less dramatic than sample 15 which had a fit score of only 54.

In aggregate these data suggest (but do not prove) that ten successive treatments in SC-CO_2 will not impair mask fit if a suitably sized chamber that does not subject the mask to mechanical deformation is used. In addition, the data shown in Figure 22 strongly indicate that ten successive one-hour cycles of supercritical carbon dioxide do not adversely effect on elastomeric strap integrity.

At first glance, the data on mask filtration efficiency (Figure 23) looks problematic because the measured filtration efficiency had a mean value of 81% with a standard deviation of 7%. This is well below the 95% threshold for a properly functioning N95 mask. If this is due to de-poling of the electrostatically poled fibers in the mask material, and/or microscopic changes in fabric morphology, super-critical carbon dioxide cannot be used in this application.

Table 3. Strap Integrity Evaluation

Tensile Force in Respirator Straps of Decontaminated N95s (recorded force values are at 150% strain)			
Respirator Model, Decon Method, # of cycles	**Straps from Treated Sample #**	**Force in Top Strap (N)**	**Force in Bottom Strap (N)**
3M 1860, Supercritical CO2, 10 Cycles	**1**	2.676	3.020
	2	2.696	2.947
	3	2.751	2.903
	Decontaminated Strap Average	2.708	2.957
	Control 1	2.670	2.850
	Control 2	2.794	2.937
	Control Strap Average	2.732	2.894
	% Change ((Deconned - Controls) / Controls)	-0.88%	2.18%

Figure 8: 10X SC-CO_2 treatment of the 3M 1860 N95 masks caused no significant degradation of the elastomeric strap used to achieve proper fit.

But further inspection of the numerical data indicates that reductions in filtration efficiency are entirely attributable to leakage around the edges of the mask, and that this is true for all 10 ten mask samples; filtration efficiency percentage is equal to 100 percent minus maximum leakage percent in all table entries. This indicates that if SC-CO_2 treatment adversely effects fabric microstructure, the magnitude of the effect is insignificant.

It is regrettable that the very limited time and budget of this project precluded the construction of a larger chamber. But it is also fair to say that had we known how important the avoidance of mask bending effects would be to unambiguous test results, we would have diverted more resources towards chamber fabrication. Accordingly, we strongly recommend that the above testing of SC-CO_2 treatment in conjunction with NIOSH be repeated without the confounding variable of mask deformation. We further recommend that the loading/unloading of SC-CO_2 into the treatment chamber be automated to reduce labor and to ensure that conditions for formation of the liquid CO_2 phase is avoided. According to thermocouple data, the difficulty of controlling upward and downward temperature excursions during pressurization and depressurization via manual valves likely resulted in frequent instances of liquid CO_2 formation. In this regard the tests performed were harsher than conditions encountered in a commercial SC-CO_2 washing machine.

Finally, in a separate set of experiments, the SC-CO_2 whole mask test chamber was used to evaluate the effect of SC-CO_2 exposure on various components used in hospital ventilator machines. In particular, there is interest in exploiting the gas-like volumetric-space-filling property of SC-CO_2 to sterilize long lengths of corrugated ventilator tubing. Much like N95 masks, such tubing was originally intended to be disposable. It was therefore not designed to survive thermal sterilization protocols such as autoclaving. The use of liquid sterilization agents is problematic because the extreme aspect ratio and highly corrugated shape of the tubing makes it difficult to ensure that all internal surfaces are fully wetted and fully rinsed. With the assistance of David Chandler, we obtained a complete set of what are normally considered one-time use ventilator components from Stanford University Hospital.

Table 1. Filter Efficiency Evaluation

Respirator Model, Decon Method, # of cycles	Treated Sample #	Flow Rate (Lpm)	Initial Filter Resistance (mmH_2O)	Initial Percent Leakage (%)	Maximum Percent Leakage (%)	Filter Efficiency (%)
3M 1860, Supercritical CO_2, 10 Cycles Min Fil Eff: 66.90% Max Fil Eff: 89.80%	**1**	85	7.2	11.9	15.5	**84.50**
	2	85	7.5	11.0	13.8	**86.20**
	3	85	8.4	9.59	12.0	**88.00**
	4	85	7.7	8.43	10.2	**89.80**
	5	85	7.5	16.0	23.6	**76.40**
	6	85	7.5	13.7	16.9	**83.10**
	7	85	7.4	11.7	14.5	**85.50**
	8	85	7.0	23.7	27.4	**72.60**
	9	85	7.5	28.6	33.1	**66.90**
	10	85	7.9	17.8	21.8	**78.20**
	Control 1	85	8.0	0.503	0.953	99.05
	Control 2	85	7.4	0.640	1.69	98.31
	Control 3	85	8.6	0.442	0.788	99.21

Notes:

- The test method utilized in this assessment is not the NIOSH standard test procedure that is used for certification of respirators. Respirators assessed to this modified test plan do not necessarily meet the requirements of STP-0059, and therefore cannot be considered equivalent to N95 respirators that were tested to STP-0059.
- **BOLD** filter efficiencies < 95%.

Figure 23: Inspection of the above numerical data for mask filtration efficiency indicates that reductions in filtration efficiency observed for SC-CO_2 treated masks are entirely attributable to leakage around the edges of the mask, and that this is true for all 10 ten mask samples. This strongly implies that if SC-CO_2 treatment adversely effects fabric microstructure, the magnitude of the effect is insignificant.

Referring now to Figure 24, this includes tubing, connectors, valves, and sensors.
The corrugated blue tubing along with its non-removable fittings did not exhibit any change or degradation during extended exposure to SC-CO_2. It was initially feared that leaching of SC-CO_2-soluable plasticizers might cause the corrugated flexible tubing to become brittle. But there was in fact no discernible change in the appearance, feel, or flexibility of the tubing after SC-CO_2 treatment. The PPE garment material did not exhibit any change or degradation either.

The only incompatibility observed was swelling/blistering of an elastomeric seal material used in component #1 of Figure 24. It is well known that the non-polar solvent properties of SC-CO_2 allow it infiltrate certain elastomers. An extreme example is Buna N rubber, which exhibits volumetric swelling greater than 50% after 1 hour of exposure to SC-CO_2 at 1200 psig when subsequently returned to atmospheric pressure. After a few days, the Buna N sample returns to its original size as CO_2 gradually escapes the elastomeric matrix. A subset of the elastomers vulnerable to SC-CO_2 infiltration will exhibit blistering if depressurization is not carried out very slowly. There are, however, other inexpensive engineering polymers that do not exhibit any of the above effects. For example, EPDM rubber o-rings swell by only 2 to 3% and are not vulnerable to blistering. Neoprene rubber is even better, swelling less than 1% and immune to blistering effects. This implies that substitution of neoprene rubber for elastomeric seals would render all ventilator components suitable for SC-CO_2 sterilization.

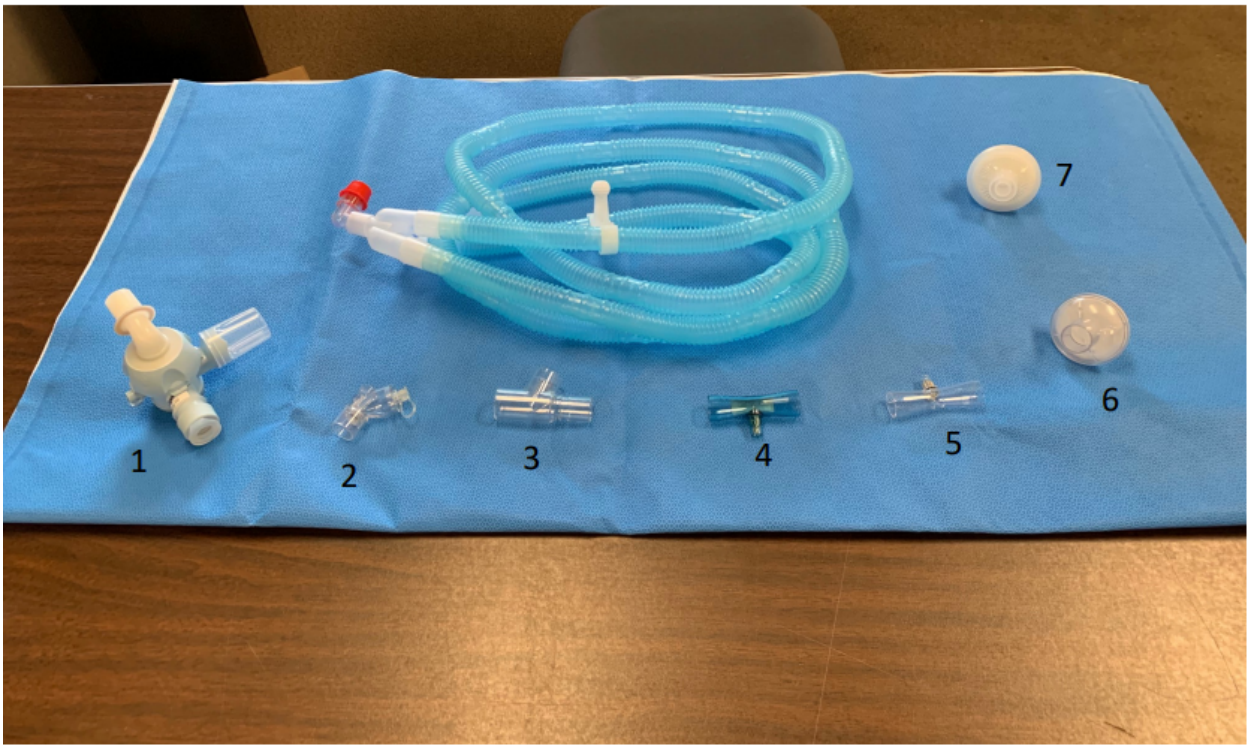

Figure 24: One-time use ventilator components laid out on top of a one-time use PPE garment, all of which were subjected to SC-CO_2 treatment.

5. FOLLOW ON WORK AND REAL-WORLD DEPLOYEMENT

There are several objectives that were beyond the scope of our $54k project budget, even though numerous Sandians generously donated their time and subject matter expertise to help this project achieve its goals. If follow-on funding can be obtained, we recommend that the following work be prioritized.

1) Construction of larger SC-CO_2 pressure vessel will enable the NIOSH tests to be repeated without the confounding variable of mask bending. This will allow us to measure even a small reduction in mask filtration efficiency due to alteration of the fabric microstructure due to exposure to SC-CO_2 (if there is any). This larger pressure vessel should accommodate fifteen 3M 1860 N95 masks without any bending or compression of the mask material. It also be equipped with automated proportional valves that allow the addition and removal of CO_2 from the pressure vessel in a prescribed manner that does not result in uncontrolled temperature excursions.
2) Viral assay of mask fabric swatches contaminated with a non-pathogenic, BSL-2, surrogate virus, subject to the SC-CO_2 + peracetic acid protocol described by White in reference 7 should be undertaken to demonstrate the micro-penetration capabilities of super-critical carbon dioxide when used as a vehicle to deliver peracetic acid.
3) An analogous experiment in a BSL-3 facility with live Corona virus would be desirable as well to directly demonstrate sterilization efficacy against COVID-19.

The remaining challenges pertain to real world deployment. As discussed earlier, there is existing infrastructure in the dry-cleaning industry that is presumably well suited to this application. But it also must be understood that:

a) The dry-cleaning industry is still in the process of transitioning from perchloroethylene to supercritical carbon dioxide. The ROI period for switching to eco-friendly supercritical carbon dioxide dry cleaning equipment is 2 to 4 years. As a result, in the dry-cleaning industry there are early adopters, those awaiting the results of early adaption, and hold outs that will continue using perchloroethylene for the foreseeable future.
b) Even if it could be proven that the dry-cleaning industry could safely handle some of the burden of N95 mask sterilization, public perception, concerns about potential liability, and politics would likely preclude this deployment scenario.

Accordingly, we envision working with the manufacturers of such SC-CO_2 dry cleaning equipment towards deployment dedicated machines for sterilization applications in the health care industry. The European Union has been far more aggressive than the U.S. in pushing towards environmentally friendly industrial processes. The EU may therefore be a good place to look for candidate vendors. See for example the campaign launched by international compressed gas distributor Linde:

https://americandrycleaner.com/articles/linde-launches-co2-brand-throughout-europe

We would also propose to conduct a technoeconomic analysis of the proposed N95 mask reuse technology. But at the outset, it should be understood that constructing a super-critical CO_2 cleaning apparatus is fairly simple. We just built a small unit in the span of a few days using off-the-shelf components and without a great deal of prior experience. To build a large capacity machine, the most difficult item to get a hold of is the large steel pressure vessel (picture something the size of a large hot water heater). But there are companies who make such vessels for a wide variety of purposes, and the balance of other fabrication work relies on commonly available COTS parts and is fairly generic in nature.

The following two videos illustrate how quickly and cost effectively such a high-strength steel pressure vessel can be fabricated by industry. The following video is from Spuncast Inc. of Watertown, WI:

Spin casting thick-walled steel pipe: https://www.youtube.com/watch?v=OmApwBP2m4c

And here is a video from MJC engineering of Huntington Beach, CA that illustrate how end caps would be incorporated into such a spun cast steel pipe:

Forming of pressure vessel end caps: https://www.youtube.com/watch?v=DTJ_n6_4xwA

It will also be understood that spin-casting can be adopted to the fabrication of thick-walled steel pressure vessels with integral flanges, such as that required for the pressure vessel door.

Such a techno-economic study would also examine the cost savings associated with procurement of several hundred identical SC-CO_2 washing machines, whereas such machines are typically purchased individually by dry cleaning operations.

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