Accelerated Aging Test Method for Estimating True Shelf Life of Surgical N95 Respirator Straps

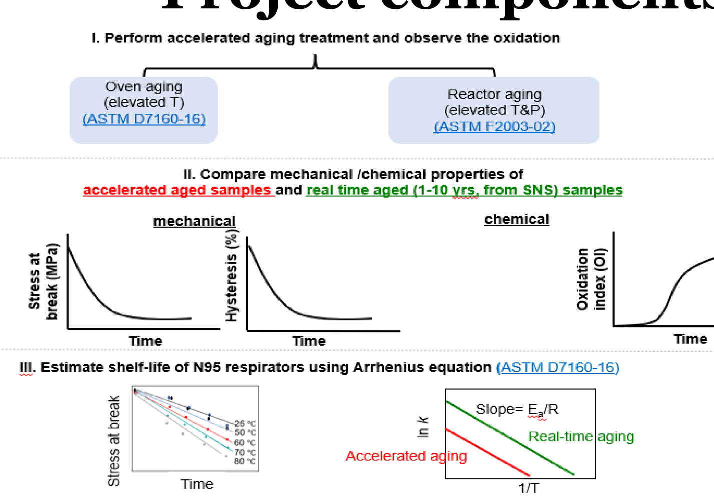
K Zin Htut, Susan Gorman, Enusha Karunasena, Anne Lucas, Katherine Vorvolakos, Tanmay Jain
DBCMS-OSEL-CDRH, US Food and Drug Administration, Silver Spring, MD
Division of Strategic National Stockpile, Administration for Strategic Preparedness and Response
Department of Health and Human Services, Washington, DC

Abstract

Personal protective equipment (PPE) in the Strategic National Stockpile (SNS), like surgical N95® respirators, is integral to public health and safety. The long-term storage of surgical N95® respirators may adversely affect their safety and integrity. Of note, polyisoprene-based elastic straps of these respirators are reported to undergo degradation and failure before the polypropylene-based filtration components. The degradation of the straps adversely impacts the fit factor and performance of the surgical N95® respirators. However, there are no standardized test methods for assessing the durability of the elastic straps and estimating their functional lifetime. This effort aims to produce an ASTM Standard Practice and/or Regulatory Science Tool (RST) which will describe an accelerated aging test method for estimating the real time shelf-life of surgical N95 respirator straps. In this work, a feasibility study was designed to assess the degradation rate of surgical N95® respirator straps at accelerated conditions. Two groups of two different N95® brands : ***Brand A*** and ***Brand B*** were subjected to accelerated aging at elevated temperature of 60 °C and 70°C and in a reaction vessel with 5 bar pressure of oxygen at 60 °C and 70°C for 8 weeks and 4 weeks, respectively. Dynamic Mechanical Analysis (DMA) and Attenuated Total Reflection Infrared Spectroscopy (AT-IR) were used to evaluate the mechanical performance and assess oxidative degradation of the respirator straps. This study assess the effectiveness of accelerated aging test methods in estimating the degradation rate of surgical N95 respirator straps. Future work includes optimizing the accelerated aging test parameters and testing of surgical N95® respirator straps from various manufacturers. To develop a correlation between the degradation rates of the accelerated aged respirators and real time aged (1-10 years) respirators obtained from the SNS, the mechanical and chemical analysis results will be compared first. Secondly, a statistical model based on the Arrhenius relationship will be used to calculate the aging factor to estimate the functional lifetime of the surgical N95® respirator straps. The outcome of this study will be beneficial to manufacturers and CDRH reviewers in assessing safety and effectiveness of surgical N95® respirators and may also help with the curation of SNS.

Experimental Methods

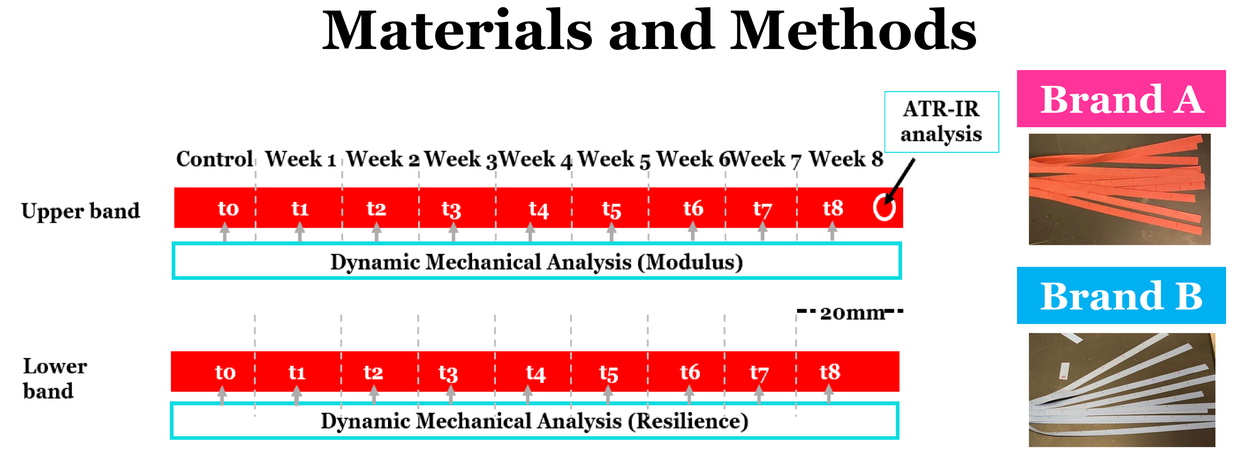
Project components



Three stages of the project. Stage I: Accelerated aging treatments like oven aging according to ASTM7160-16 (Determination of Expiration Dating for Medical Gloves) and reactor aging according to ASTM F2003-02 were performed (Standard Practice for Accelerated Aging of Ultra-High Molecular Weight Polyethylene). Stage II: Comparison of the mechanical/chemical properties of accelerated aged samples from market and real time aged samples from SNS. Stage III: Estimation of the shelf-life of the respirators using the Arrhenius equation will be performed according to ASTM D7160-16 .

In this poster, we are presenting only the result of accelerated aged samples

Materials and Methods



The straps were labeled with a 20 mm mark and were cut for mechanical and chemical testing weekly and returned to the treatment.

Results

Morphological analysis

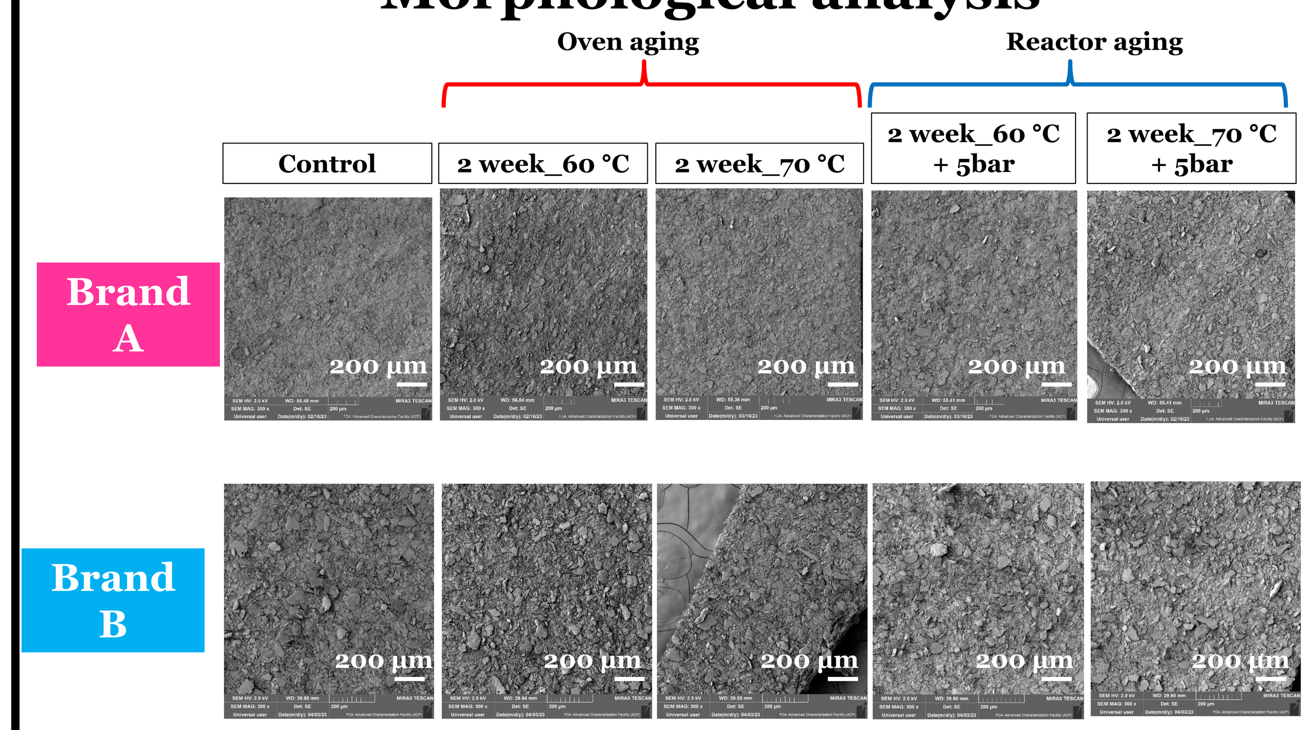


Figure 1. SEM images of un-aged (control), and 2 weeks aged samples of Brand A and brand B using oven aging treatment (60 °C and 70 °C) and reactor aging treatment (60 °C+ 5 bar, and 70 ° C+ 5bar).

The control samples seem to have a smoother surface than two weeks-aged samples which show visible small flakes.

Mechanical analysis (Modulus)

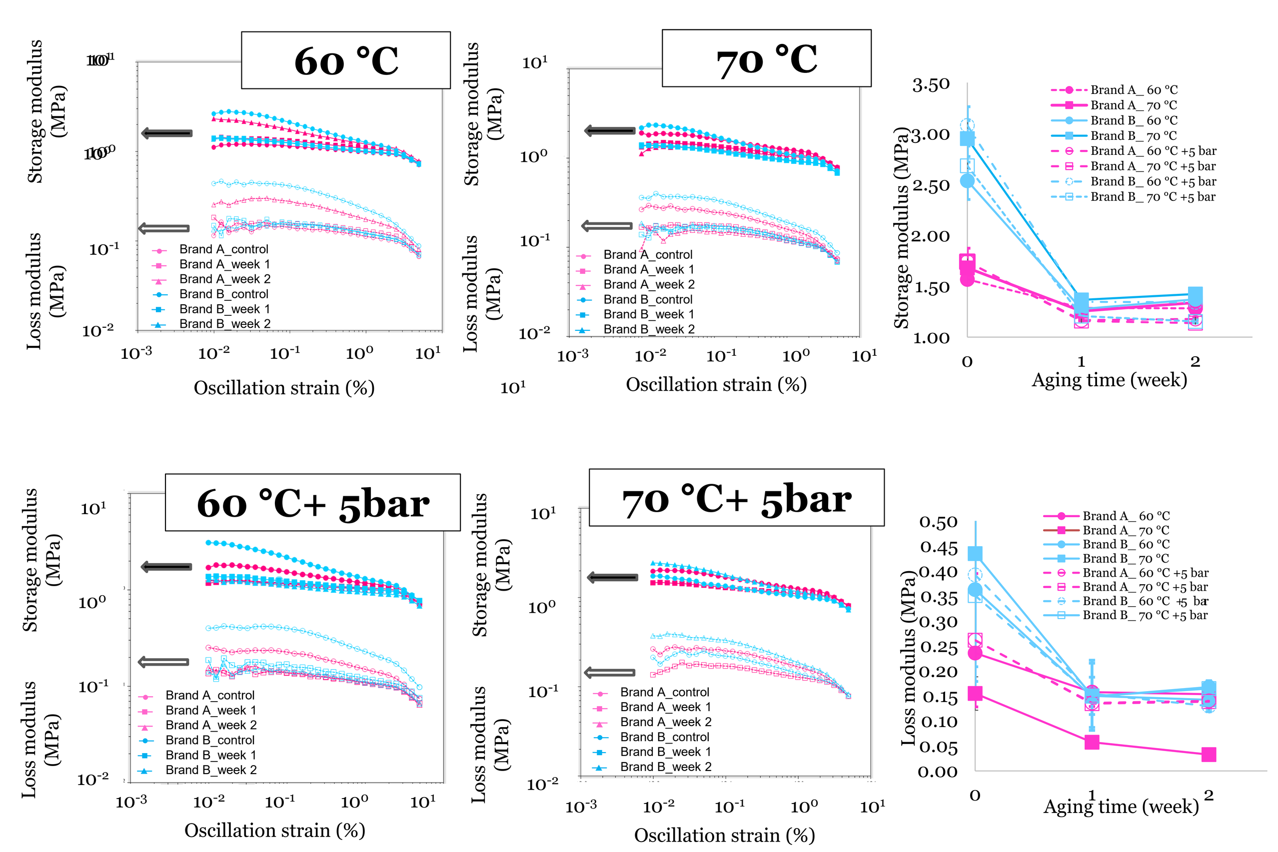


Figure 2. Storage modulus and loss modulus data of un-aged (control), 1-week and 2-week aged samples of ***Brand A and Brand B*** using oven aging (60 °C and 70 °C) and reactor aging (60 °C+ 5bar, and 70 ° C+ 5bar in O_2 atmosphere). Solid symbols represent storage modulus and void symbols represent loss modulus. Averaged modulus data with aging time is plotted on the right.

Storage and loss modulus decreases after one week of accelerated aging in the oven and reactor. No additional change in modulus values was observed after the first week.

Mechanical analysis (Resilience)

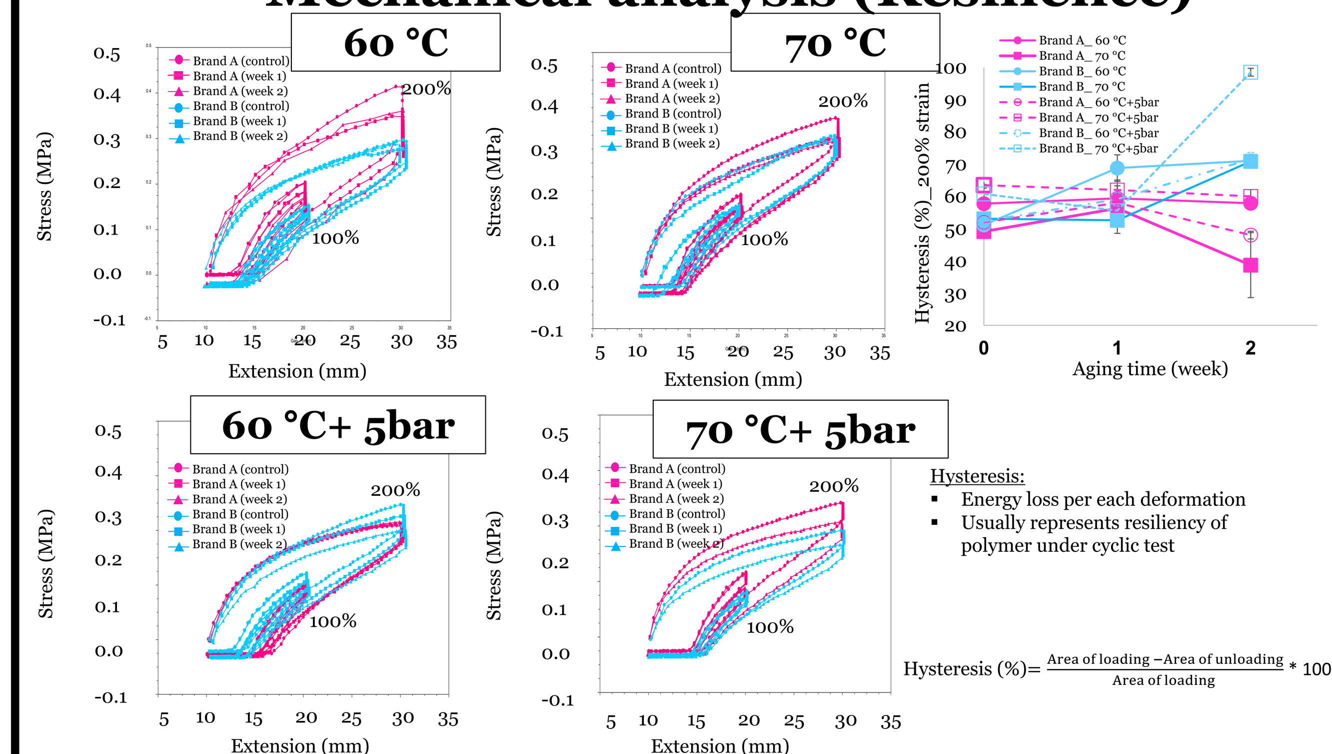


Figure 3. Resilience test of un-aged (control), 1-week and 2-week aged samples of ***Brand A*** and ***Brand B*** using oven aging (60 °C and 70 °C) and reactor aging (60°C+ 5bar, and 70°C+ 5bar in O2 atmosphere). Hysteresis (%) was calculated using stress vs extension test at 200% strain, with respect to the original length, and is plotted with aging time.

Brand A loses its resiliency whilst Brand B shows random deformation with aging time.

Chemical analysis (Attenuated reflected infrared spectroscopy)

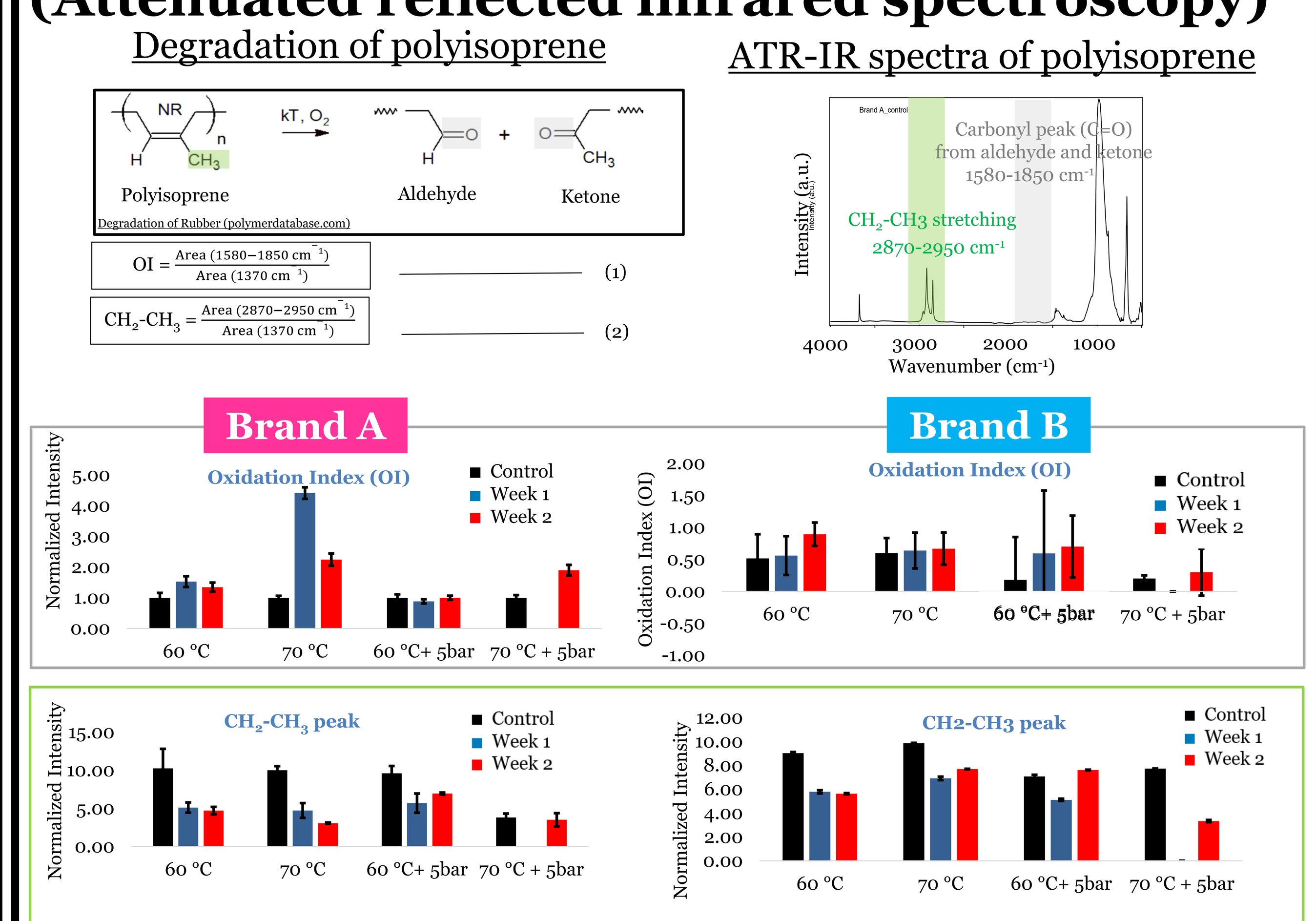


Figure 4. ATR-IR spectra of un-aged (control), 1-week and 2-week accelerated aged samples of Brand A and Brand B using oven aging (60 °C and 70 °C) and reactor aging (60 °C+ 5bar, and 70 ° C+ 5bar in O_2 atmosphere). Oxidative index and CH_2-CH_3 groups were calculated using equation (1) and equation (2) ,respectively from IR spectra (omitted here). The values are plotted with aging time to observe oxidation with aging time.

No change in oxidative index was observed after aging while the peaks representing the CH2-CH3 functional groups decreased with aging time.

Discussion

- Due to their potential negative impact on respirator fit and performance, the degradation rate of polyisoprene-based elastic straps in respirators was studied using accelerated aging test methods.
- Both Brand A and Brand B surgical N95 respirator straps showed minor morphological changes at the surface after accelerated aging via oven and reactor treatments (i.e. 60 °C and 70 °C vs 60 °C + 5bar, and 70 ° C + 5bar in O_2 atmosphere).
- However, a reduction in storage modulus and loss modulus after one week of aging and no further change in modulus after one additional week might suggest the presence of diffusion limited oxidation (DLO) phenomenon, which results in limited degradation in polymer bulk. Contradictorily, real-time shelf aging has been shown to result in polymer bulk degradation.
- Hysteresis data also showed no significant change after the first week of aging.
- Considering the overlapping characteristic IR peaks in CH_2-CH_3 region where two peaks correspond to CH3 asymmetrical and symmetrical stretching (2870 cm^{-1}, 2950 cm^{-1}) and 1 peak to CH_2 stretching (2920 cm-1), the area could be dominated by CH3 end group vibration, and hence, be considered to be representative of number of polymer chain end-groups. ATR-IR result showed accelerated aging caused a decrease in CH_2-CH_3 end groups, likely due to hydrogen depletion during high temperature and pressure treatments. There is more than one location the –CH_3 can reside along the chain so the most prevalent reaction pathway for degradation will be determined in future work.

Conclusions

- In this study, we observed the change in morphological, mechanical and chemical characteristics of of surgical N95 respirator straps after accelerated aging.
- At present, we are planning to complete the remaining accelerated aging data set for a complete study: samples aged for 4 weeks by oven aging and samples aged for 8 weeks by reactor aging.
- In order to relate the degradation processes to a well-known trend like the Arrhenius equation, we will need to optimize the measurement of both bulk and surface measurements over proper intervals in order to capture the degradation time and location.
- Overall, our findings provide some insights into heterogeneous process of surgical N95 respirator straps due to DLO. Current findings will inform development of aging methods for estimating shelf life of respiratory protective equipment.

Acknowledgement: This work is funded by the CDRH OSEL Emergency Preparedness Program, led by Dorn Carranza and Jenna Osborn. We thank Michael Eppihimer and Ed Margerrison for their support.

Disclaimer: The findings and conclusions in this poster have not been formally disseminated by the Food and Drug Administration and should not be construed to represent any agency determination or policy. The mention of commercial products, their sources, or their use in connection with material reported herein is not to be construed as either an actual or implied endorsement of such products by Department of Health and Human Services.