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16. Abstract

Modern transportation systems are subject to unintentional contamination from infected passengers, as well as deliberate contamination from criminals and political adversaries. Hydrogen peroxide has been used for years as a disinfectant in the medical community and is under consideration in the dilute vapor form as a decontaminant/disinfectant/sterilant for transportation vehicles like aircraft, buses, subway trains, ambulances, etc. Although the biological efficacy of STERIS Corporation's Vaporized Hydrogen Peroxide (VHP® a registered trademark of the STERIS Corporation, Mentor, OH.) technology has been demonstrated elsewhere, the compatibility of the process with typical aircraft materials has not been rigorously established. The present report documents a materials compatibility evaluation involving the effects of hydrogen peroxide exposure on the mechanical properties and flammability of the following commercial-grade textile materials: wool, nylon, polyester, Nomex®, and leather.

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Effects of Hydrogen Peroxide on Common Aviation Textiles

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August 2009

Final Report

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ABBREVIATIONS

As used in this report, the following abbreviations/acronyms have the meanings indicated.

Abbreviation	Meaning
ASTM	American Society for Testing and Materials
BI	Biological indicator
C	Carbon, Celsius
CFU	Colony-forming units
DI	Deionized
eV	Electron volt
FAA	Federal Aviation Administration
FAR	Federal Aviation Regulations
FTIR	Fourier transform infrared
H	Hydrogen
HPAI	Highly Pathogenic Avian Influenza
hr	Hour
HVFAA	Horizontal vertical FAA flame chamber
min	Minute
mm	Millimeter
MPa	Megapascal
N	Nitrogen
O	Oxygen
psi	Pounds per square inch
S	Sulfur
s, sec	Second(s)
SARS	Severe Acute Respiratory Syndrome
VHP	Vaporized hydrogen peroxide
XPS	X-ray photoelectron spectroscopy

Effects of Hydrogen Peroxide on Common Aviation Textiles

INTRODUCTION

Modern outbreaks of infectious disease, such as Severe Acute Respiratory Syndrome (SARS) from China to Canada in 2002, Highly Pathogenic Avian Influenza (HPAI) in Southeast Asia in 2003, as well as possible future pandemic influenza, necessitate reliable clean-up of many elements of the transportation infrastructure system to alleviate impacts to human health and mitigate negative consequences to the global economy. Among all the sanitization/decontamination technologies available, vaporized hydrogen peroxide (VHP)[1] technology is of particular interest due to rapid sterilization, easy usage, intrinsic environmental friendliness (i.e ., simple by-products composed of only water and oxygen) and compatibility with many materials and systems. VHP® technology might also be employed against deliberate biological/chemical attack, for instance, the 1995 Sarin gas attack in the Tokyo subway system (1). In fact, the VHP® process was utilized during the clean-up of buildings and vehicles in Washington, DC, after the 2001 anthrax attacks (2). VHP® is generally considered to begin sporicidal effects at concentrations above 80 ppm. Cycle times obviously vary based upon the size of the enclosed area, the capacity of the delivery system and the specific concentration desired. In general, VHP® technology shows excellent potential for application within the modern transportation system.

[1] VHP is a registered trademark of STERIS Corporation, Mentor, OH, USA.

VHP® technology has been investigated for possible usage in aircraft applications and the process has been shown to be efficacious (complete kill of 10^6 CFU of the spore forming *Geobacillus stearothermophilus*) in a narrow-body aircraft fuselage (3), as well as wide-body aircraft if the air in the cabin section was well mixed so that adequate levels of VHP® vapor were uniformly distributed (4,5). These studies used vaporized hydrogen peroxide concentrations in the range of 150 - 600 ppm and cycle times of 80 - 120 min. Maximum concentrations of hydrogen peroxide vapor were carefully controlled to avoid condensation in cool locations within the aircraft cabins. Although these previous studies did not evaluate the compatibility of the various cabin materials with exposure to vaporized hydrogen peroxide, it was noted that there were no noticeable changes to any of the cabin materials (4). OH,USA .

Many polymeric textile materials are known to be susceptible to moisture sorption and, if H_2O_2 vapor is similarly absorbed by these materials, their[2] engineering[2] properties could be seriously degraded. In considering the sanitization/decontamination of aircraft with vaporized hydrogen peroxide, cabin polymeric textiles (e.g ., wool, nylon, polyester, Nomex[2]) could be among the most vulnerable materials to potential hydrogen peroxide attack. These polymeric materials consist of long chain carbon-carbon backbones with side chain functional groups, as well as intermolecular cross-links that might be degraded by oxidation from the hydrogen peroxide. Hence, it is necessary to examine and understand the chemical stability and mechanical response

of these textiles after exposure to vaporized hydrogen peroxide. In this research, a preliminary examination of the compatibility of common airliner cabin textile materials with hydrogen peroxide has been conducted. The textile materials were composed of two basic categories: (i) natural materials, including wool and leather; and (ii) synthetic materials, including nylon, polyester, and Nomex® .

[2] Nomex is a registered trademark of E.I. du Pont de Nemours and Company, Wilmington, DE, USA.

BACKGROUND

A typical VHP® process cycle consists of an initial dehumidification step, then a conditioning phase, followed by the actual sanitization/decontamination process. Finally, an aeration phase is employed to remove residual hydrogen peroxide vapor. During the dehumidification phase, warm, dry air flows into the enclosure to lower the relative humidity to less than 10%, which allows a higher concentration of hydrogen peroxide vapor to be injected into the enclosure without condensation. Hydrogen peroxide liquid concentrate (35% liquid H_2O_2 with a pH ~ 3) is then flash vaporized and injected into[22] the enclosure during the initial conditioning phase and the sanitization/decontamination phase. The purpose of the conditioning phase is to rapidly increase the hydrogen peroxide concentration to minimize the overall cycle time. During the sanitization/decontamination phase, a steady concentration of hydrogen peroxide vapor (typically 250 - 450 ppm) is maintained to give the desired sanitization/decontamination cycle, as often measured by the 6-log kill (*i.e.*, 10^6 reduction) of a commercial biological indicator (BI) spore population of *Geobacillus stearothermophilus* . Once the sanitization/decontamination phase is completed, the enclosure is then aerated with fresh air, while any residual hydrogen peroxide vapor breaks down into environmentally benign water and oxygen.

Textile fabrics can be made from solutions (non-fibrous), directly from discrete fibers, or from yarns (6). For the construction of fabrics from yarns, *weft* yarns are interweaved over and under stretched *warp* yarns under tension, as shown in Figure 1. Since warp yarns must withstand the tension and sometimes abrasion from the weaving process, warp yarns are made stronger than weft yarns and with more twist. Warp yarns also align straighter with less crimping and bending by the interlacing mechanics than the corresponding weft yarns.

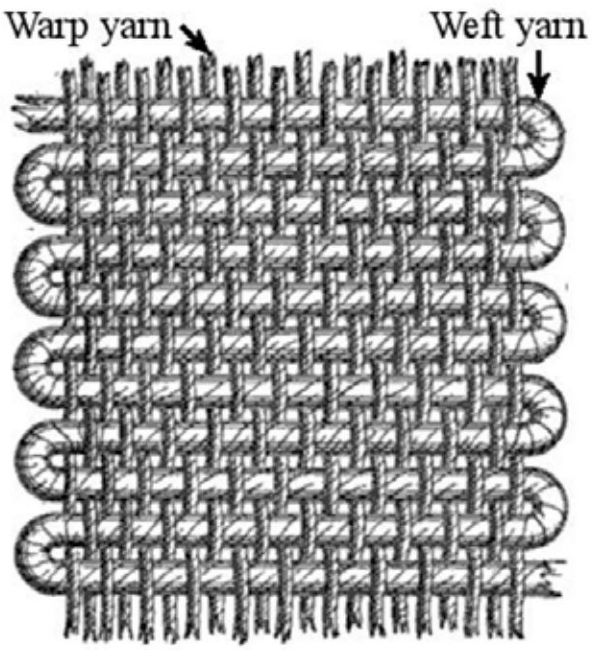


Figure 1. Distinction between the warp and weft yarns of a common textile fabric.

The basic building blocks for either natural or synthetic textile yarns are the individual fibers composed of millions of molecular chains. The micro-mechanical properties of any polymer fiber are strongly dependent on the molecular weight of the polymer, the degree of crystallinity of the polymer, and the intrinsic intermolecular forces from hydrogen and van der Waals bonding, plus covalent cross-links, when applicable. Natural fibers can be produced from either plant (cellulose structures) or animal (protein structures) sources. The natural materials used in this research included wool and leather. The synthetic (man-made) fibers used in this research included nylon, polyester, and Nomex® .

Bleaching of textiles by hydrogen peroxide solutions is often accomplished during routine processing using an alkaline 3% hydrogen peroxide solution at a temperature of 80 ~ 90 ºC (6). In an earlier study by Arifoglu et al. (7), the level of whiteness of wool after bleaching by hydrogen peroxide was found to be controlled by the exposure time, the concentration of hydrogen peroxide, the pH level, and the temperature of the treatment. Gacen and Cayuela (8) showed that when bleaching wool with an alkaline solution (pH = 9) of hydrogen peroxide (~5%) containing sodium pyrophosphate/sodium oxalate buffering medium at 55 ºC for 2 hours, the whiteness index was greater than bleaching in an acidic solution (pH = 4.7). Thus, chemical attack by hydrogen peroxide liquid concentrate was more severe in an alkaline solution than an acidic solution. In a more recent study by Cardamone et al .(9), the effect of shrinkage on wool during hydrogen peroxide bleaching was controlled by the unstable perhydroxy (HO2-) radicals. These authors found that the chemical attack of the wool disulfide cross links by hydrogen peroxide produced sulfonic acid and changed the anionic charge on the surface of wool fibers.

The net effect was to increase the hydrophobicity, decreasing the electrostatic repulsion of the fibers and causing shrinkage in the wool fabrics.

METHODS AND MATERIALS

The specific motivation for this work was to evaluate the materials compatibility of textiles representative of those typically used in commercial aircraft. Thus, laboratory-grade textile samples of wool, nylon, polyester, Nomex® (Testfabrics Inc ., West Pittston, PA, USA), and commercial-grade leather samples were acquired (McMaster Carr, Chicago, IL, USA).

Specimens were pre-cut and labeled according to the test specifications, as shown in Figure 2. Sample exposures to vaporized hydrogen peroxide were performed with a 1000ED Bio-decontamination Unit (STERIS Corporation, Mentor, OH, USA) using Vaprox[3] as the sterilant in an enclosed chamber for 1, 10, or 25 VHP cycles. Dip exposure of fabric samples to 35% liquid phase hydrogen peroxide was carried out in opaque PVC bottles (Fisher Scientific, Fair Lawn, NJ, USA) for 24 hours and 168 hours. After dip exposure, all dip specimens were rinsed in DI-water and then air-dried in an electrostatic film drying cabinet (Delta 1, CPM, Inc ., Dallas, TX) for 1 day. Sample weights were measured by an accuSeries[4] accu-124 balance (Fisher Scientific, Arvada, CO, USA) before and after hydrogen peroxide exposures.

[3] Vaprox is a registered trademark of STERIS Corporation, Mentor, OH, USA.

[4] accuSeries is a registered trademark of Fisher Scientific, Fair Lawn, NJ, USA.

Changes in the various fabrics after exposures to either 35% liquid hydrogen peroxide or vaporized hydrogen peroxide were initially examined via straightforward physical observation (i.e ., appearance to the unaided

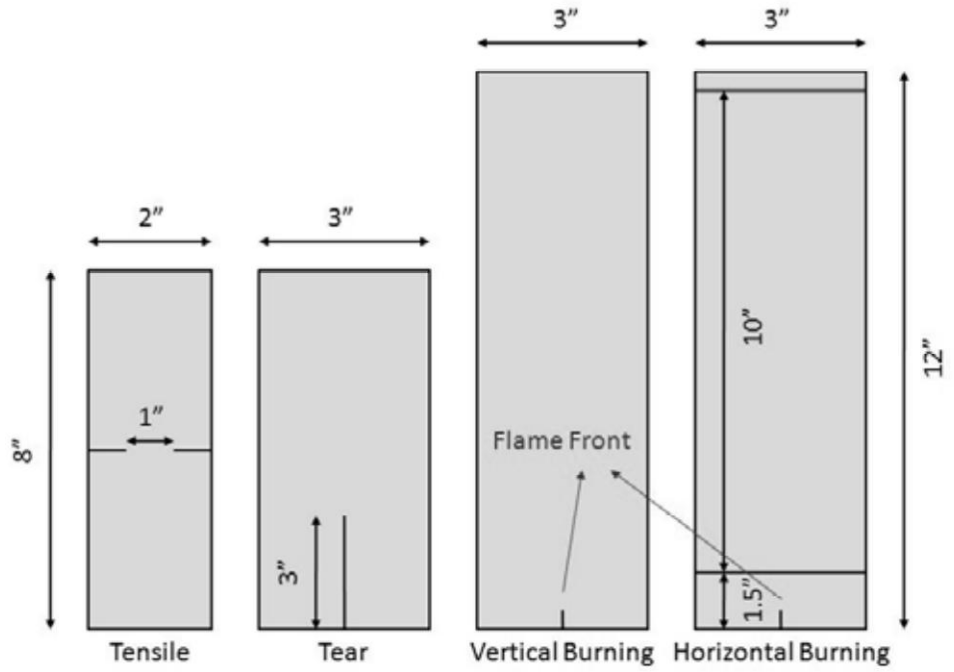


Figure 2. Physical dimensions of textile specimens for tensile testing and tear testing as well as vertical and horizontal flammability tests.

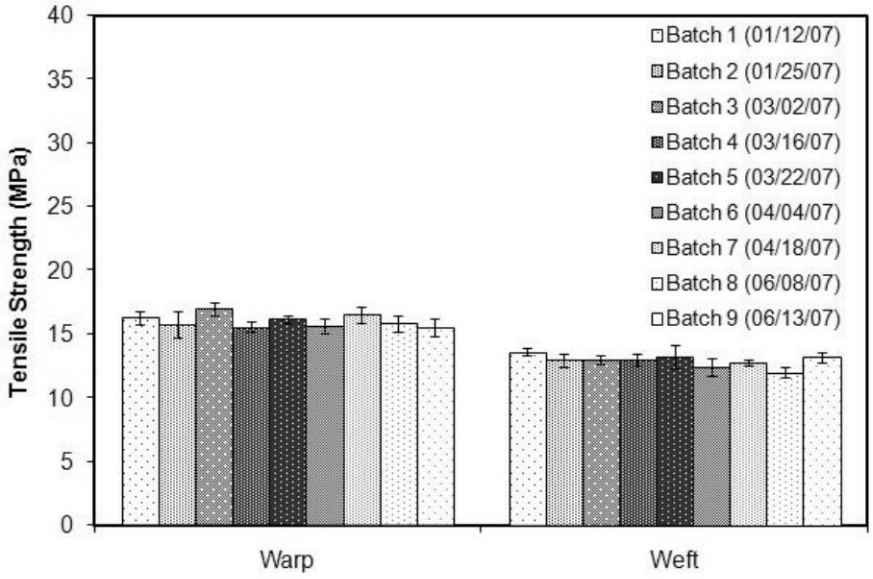
eyes and residual post-exposure aroma), weight change, and observation via optical and scanning electron microscopy.

After fabric specimens were properly exposed and aged, mechanical testing was performed in an environmentally conditioned laboratory (21 ± 1º C, 65 ± 2% relative humidity) using a screw-driven Instron 4400R (Instron Inc ., Canton, MA, USA) universal materials testing machine in accordance with ASTM standards D 5034 – 95 (10) and D 2261 – 96 (11). Control specimens were performed regularly to confirm the stability of the materials, machine, and testing procedures and conditions. A calibrated 200-lb load cell was employed, and a 12 in/min crosshead speed was used for both tensile and tear testing. Stress-strain curves were plotted according to the measurement of load and displacement.

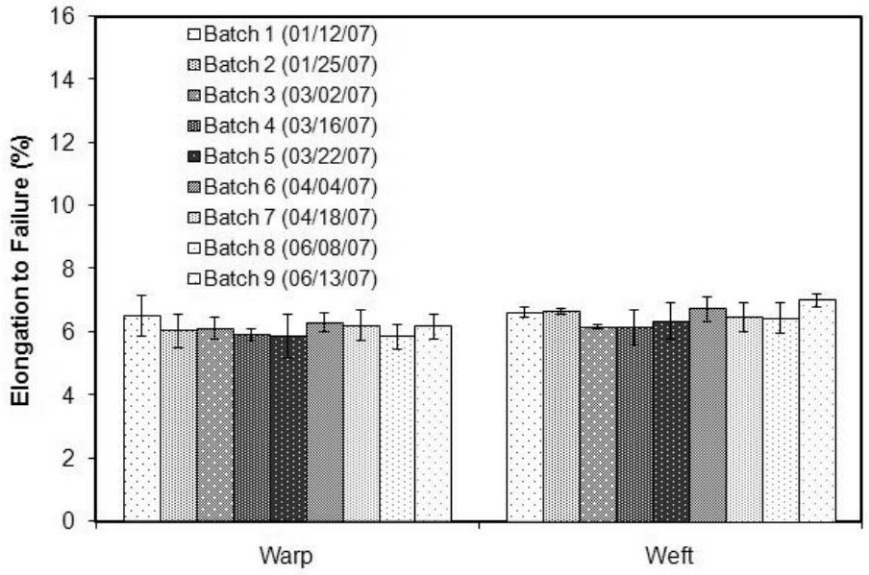
As noted above, high-quality laboratory grade textile samples were procured for this study. Nevertheless, textiles invariably exhibit some variability of weave and anisotropy from batch-to-batch of sample material. These issues, when combined with less than exact sample alignment in the mechanical testing machine relative to the textile's warp and weft directions, complicate the statistical interpretation of mechanical behavior due to the exposure conditions. A mechanical property variability study was conducted using wool (most variability in mechanical behavior, as discussed below) and Nomex® (least variability in mechanical behavior, discussed below). The results of this study are shown in Figures 3 and 4 for wool and Nomex®, respectively. It is clear that the synergistic influence of these experimental variabilities was small.

Flammability tests were performed in vertical and horizontal configurations in a HVFAA horizontal vertical flame chamber (ATLAS Materials Testing Technology, Chicago, IL, USA) in accordance with the Aircraft Materials Fire Test Handbook (12). Specimens were pre-cut and labeled according to the test specifications (Figure 2). Prior to flammability testing, specimens were conditioned in a textile conditioning unit (Parameter Generation & Control, Black Mountain, NC, USA) at 21 ± 3 ºC and 50 ± 5 % relative humidity for at least 1 day. A 99% pure methane gas was used for burner fuel with an inlet pressure of 2½ ± ¼ psi, and the flame height was calibrated for 1½ inch using a flame height indicator. A 12-second ignition time (length of time the flame is applied to the test specimen) was employed for vertical burning tests, whereas a 15-second ignition time was employed for horizontal burning tests.

Chemical analyses were performed using Fourier Transform Infrared Spectroscopy (FTIR) using an IR Prestige – 21 (Shimadzu Scientific Instruments, Columbia, MD, USA) spectrometer on pellets made of a mixture of the ground textile fibers and potassium bromide, over a scan range from 400 cm^{-1} to 4000 cm^{-1} . Raman spectroscopy

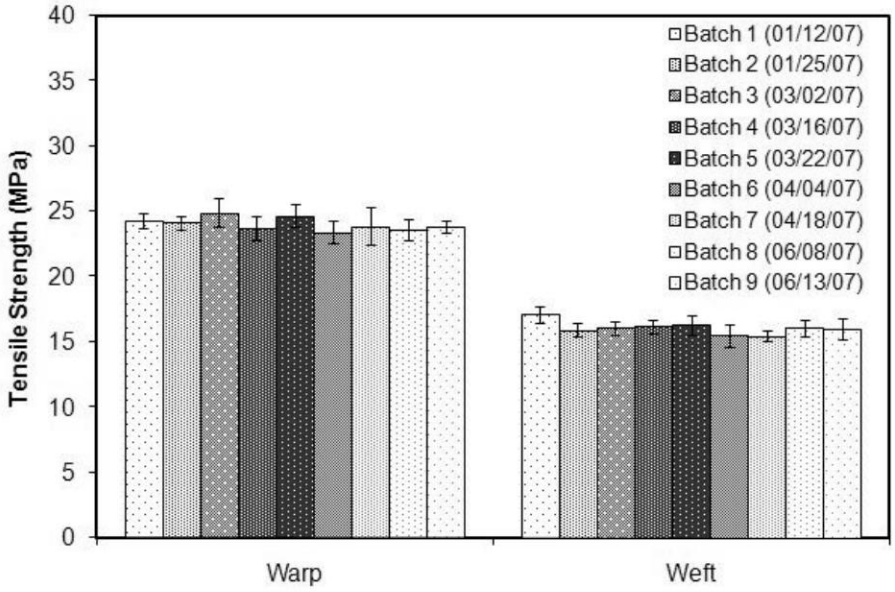


(a)

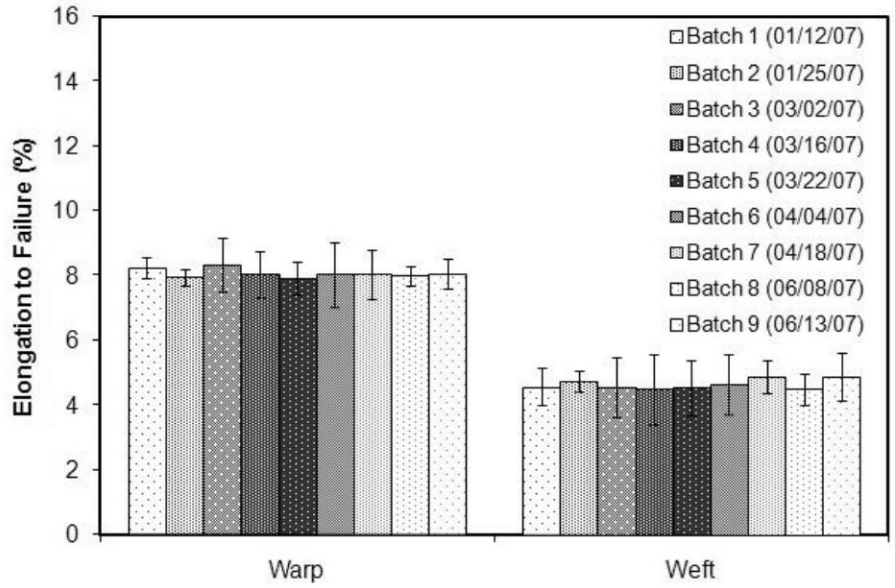


(b)

Figure 3. Results of batch-to-batch and day-to-day variability study of measured tensile strength and elongation to failure of as-received wool (13).



(a)



(b)

Figure 4. Results of batch-to-batch and day-to-day variability study of measured tensile strength and elongation to failure of as-received Nomex® (13).

was employed to complement the infrared spectroscopy. Raman spectra were acquired using an Invia Confocal Raman Microscope (Renishaw, Wotton-under-Edge, Gloucestershire, UK) using a 514.5 nm wavelength and 1 mW laser excitation source. Spectra were recorded using a 50X objective lens that generated a 1μm laser spot.

RESULTS AND DISCUSSION

Changes in Physical and Mechanical Properties

Warp specimens of wool, nylon, polyester, and Nomex® intended for tear testing were weighed before and after hydrogen peroxide exposure, and the net weight change data are shown in Figure 5 (13). As seen in Figure 5, the weight changes observed for the polyester samples exhibited no statistically significant differences for the conditions investigated. The wool, nylon, and Nomex® samples all gained weight after the VHP® treatments followed by aging in laboratory air. The wool samples exhibited the largest weight gains, with approximately 1% gain after 1 VHP® treatment and 1 day of laboratory air aging, and more than 8% weight gain after 10 VHP® cycles and 14 days aging in laboratory air. The weight gains of nylon and Nomex® were approximately half those of wool.

However, quite different results were seen for samples aged in a vacuum dessicator after exposure to hydrogen peroxide vapor. All samples exhibited weight losses after a single VHP® treatment, followed by aging in a vacuum dessicator for either 1 or 14 days. Aging samples in a vacuum dessicator after 10 VHP treatments showed no statistically significant change in weight for polyester and Nomex®, whereas nylon and wool exhibited reduced increases in weight of only about 1% and 2%, respectively. A significant part of the weight gains found after laboratory air aging were probably due to the presence of residual moisture in the fibers due to (i) absorption of water vapor directly from the environment and/or (ii) decomposition of absorbed hydrogen peroxide to water and oxygen.

Decreases in weight after a single VHP® treatment followed by vacuum aging are consistent with the weight gain being primarily absorbed moisture that is subsequently removed by the vacuum treatment. Net increases in the weight of wool and nylon after 10 VHP® treatments followed by vacuum aging are consistent with chemical oxidation of the polymers. In addition, the appearance of the wool samples after 10 VHP® cycles exhibited a slight discoloration from the hydrogen peroxide treatment.

Examination of the surface morphology of the synthetic fibers in the scanning electron microscope revealed no significant qualitative changes in surface morphology after VHP® and aging treatments. However, the surfaces of wool fibers displayed significant evidence of the hydrogen peroxide exposure. Secondary electron images of wool fiber morphology after 10 VHP® cycles are shown in Figure 6 (13). The outer cuticle layer exhibited some separation from the exterior para-cell and ortho-cell cortex after 10 VHP® cycles.

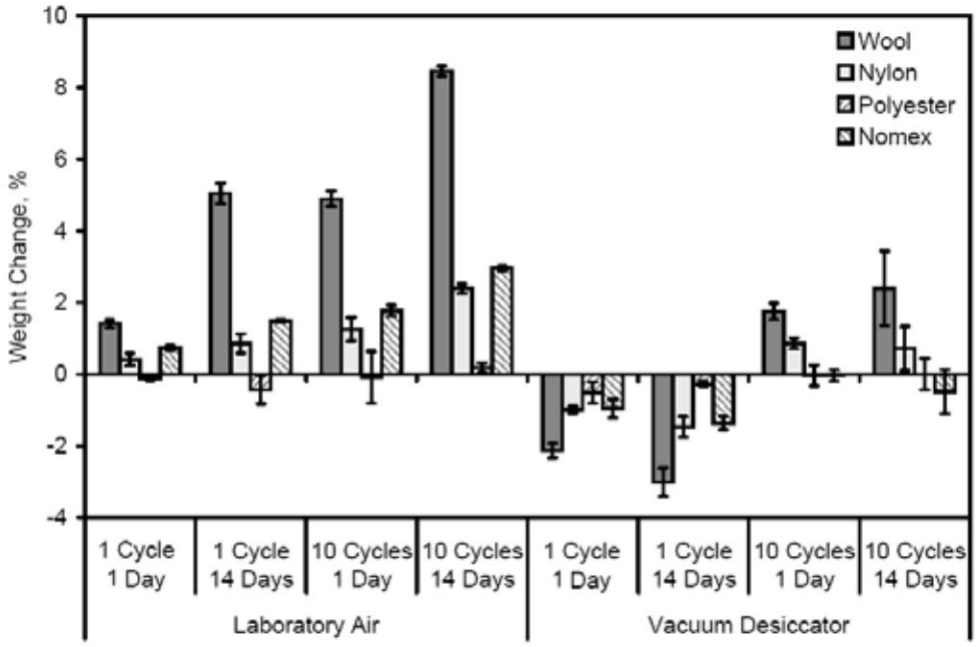


Figure 5. Percentage change in weight for wool, nylon, polyester, and Nomex® after 1 or 10 VHP® cycles and 1 or 14 day post-exposure aging times in laboratory air or vacuum (13).

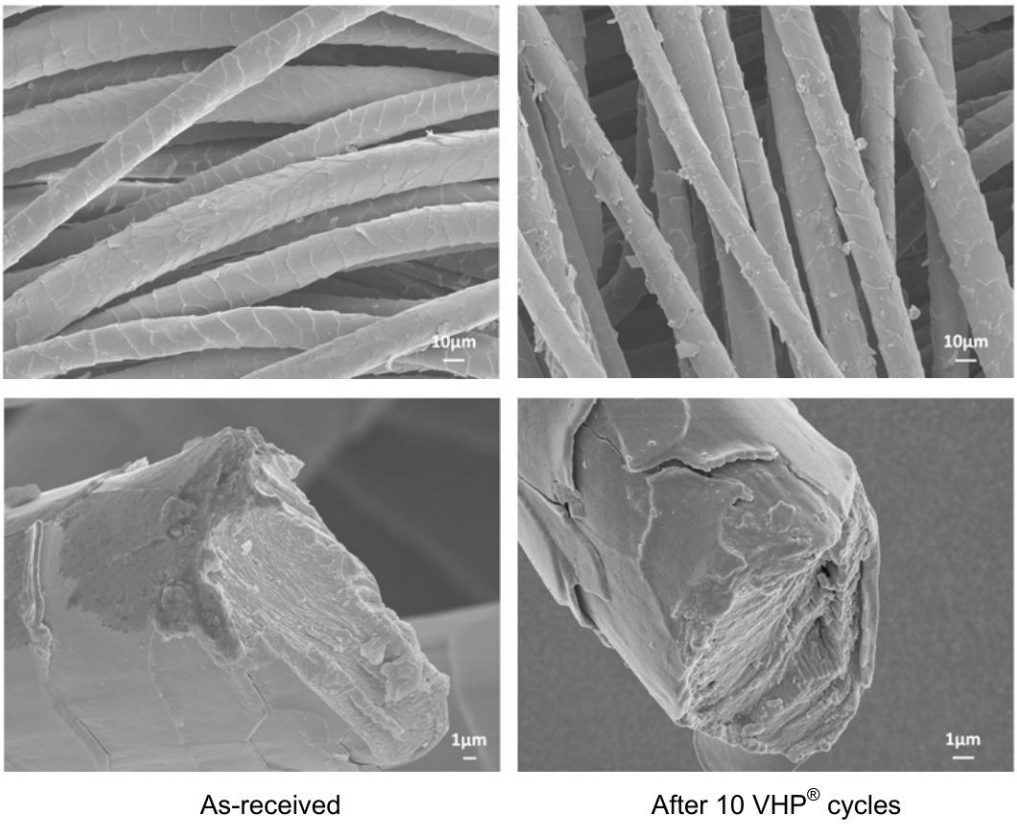


Figure 6. Secondary electron images for wool fibers (13).

The effects of VHP® exposure on the tensile strength and elongation to failure for samples oriented to tensile test either the warp or the weft fibers are shown in Figures 7-10 for wool, nylon, polyester and Nomex®, respectively (13). With only laboratory aging in air, increasing VHP® exposures lead to decreases in the tensile strength and increases in the elongation to failure for both orientations of wool samples. However, aging treatments in the vacuum dessicator after VHP® exposure made the wool results less conclusive. Mechanical test data for the nylon samples (Figure 6) yielded slight decreases in tensile strength (~5-10%) for almost all test conditions and inconclusive results for changes in the elongation to failure with no statistically significant trends. The polyester samples generally exhibited decreases in tensile strength (~10-20%), as well as decreases in elongation to failure (~10-20%) for most test conditions. Finally, the Nomex® samples exhibited no consistent changes in tensile strength for all test conditions and slight increases in the elongation to failure (~2-10%) for most of the test conditions.

The effect of exposure of the various textiles to 35% liquid hydrogen peroxide was investigated by leaving samples in the concentrated hydrogen peroxide solutions for periods of 1 day or 7 days (168 hours). These tests were undertaken because of the possibility of experiencing condensation of liquid hydrogen peroxide in cool sections of large, complex enclosures during VHP® treatment. The percentage changes in tensile strength and elongation to failure for the various textile materials (warp and weft orientations) are shown in Figures 11 and 12, respectively (13). The polyester samples exhibited minimal changes in tensile strength and essentially no changes in elongation to failure after exposure to the liquid hydrogen peroxide. Except for the 24-hour exposure with 1 day air aging treatment, the nylon samples also exhibited minimal changes in tensile strength but slight increases in elongation to failure for most of the test conditions after exposure to the liquid hydrogen peroxide. The reason for the large decreases in tensile strength for the nylon samples subjected to a 24-hour exposure and 1 day air aging treatment is unclear. Wool samples generally exhibited large decreases in tensile strength. . Although the Nomex® samples generally exhibited large decreases in tensile strength, the Nomex® samples exhibited inconsistent elongation to failure results for these test conditions.

Stress-strain curves for representative wool samples are shown in Figure 13. These curves are typical of viscoelastic materials like wool and exhibit an initial low modulus region, followed by linear elastic deformation and then yielding where the modulus drops significantly. It can be seen that the load-carrying capacity and the

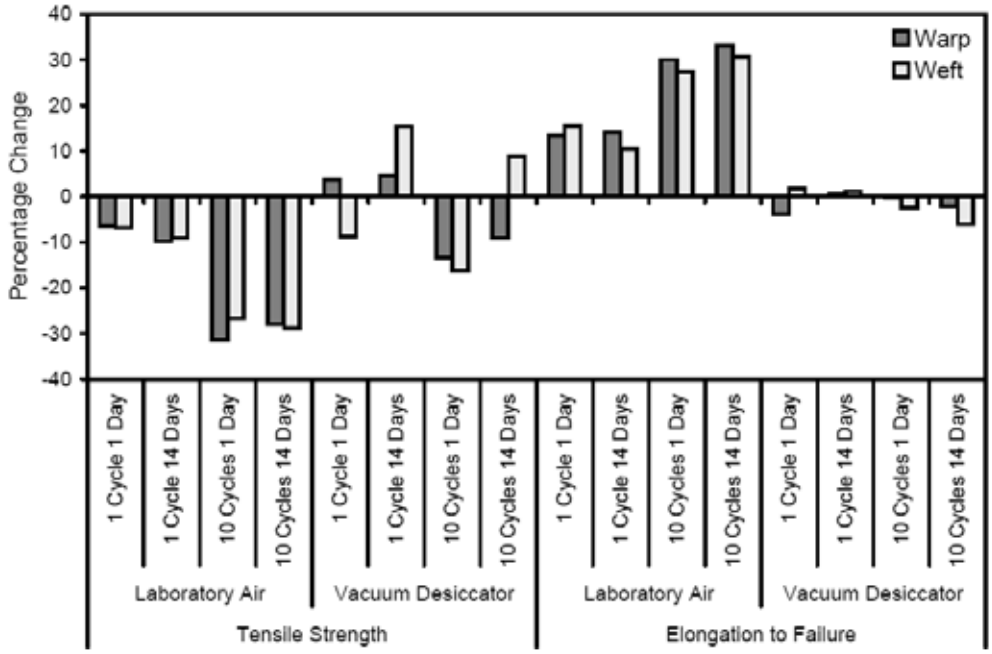


Figure 7. Percentage change in tensile strength and elongation to failure of wool after the indicated exposure conditions (13).

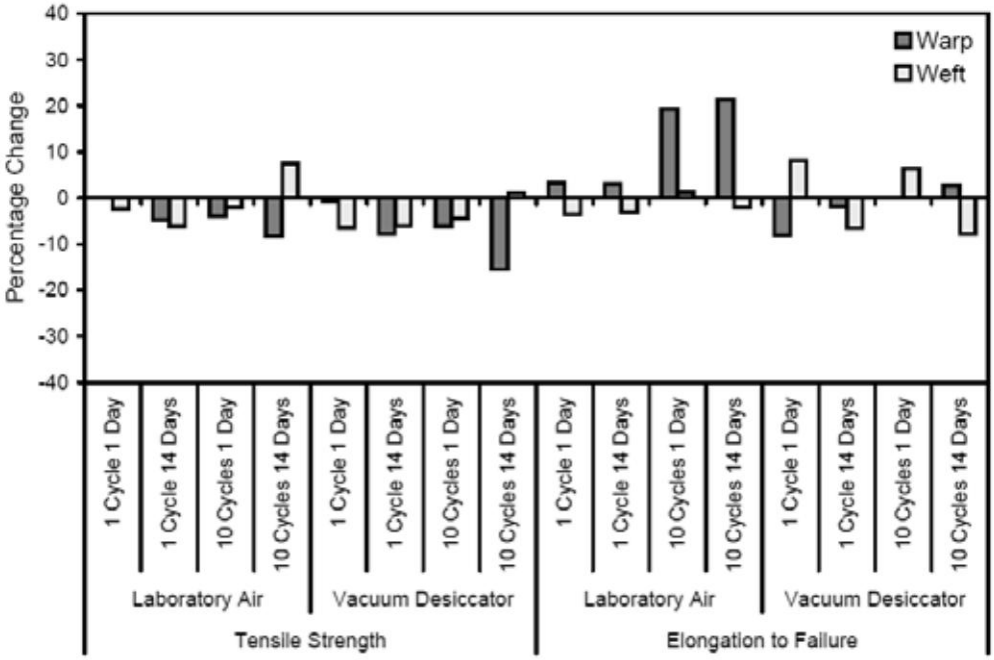


Figure 8. Percentage change in tensile strength and elongation to failure of nylon after the indicated exposure conditions (13).

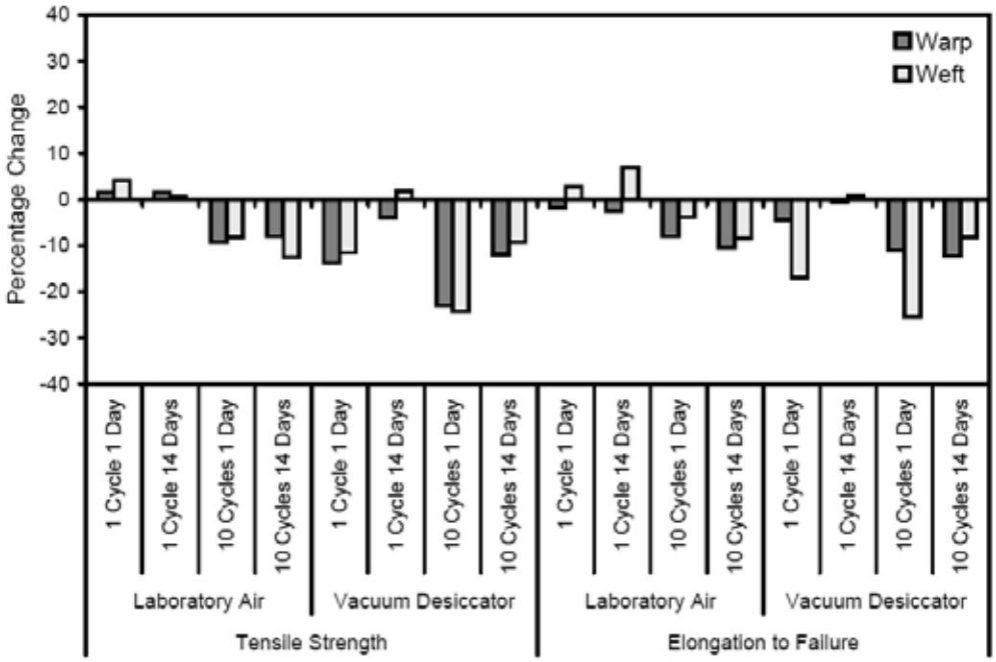


Figure 9. Percentage change in tensile strength and elongation to failure of polyester after the indicated exposure conditions (13).

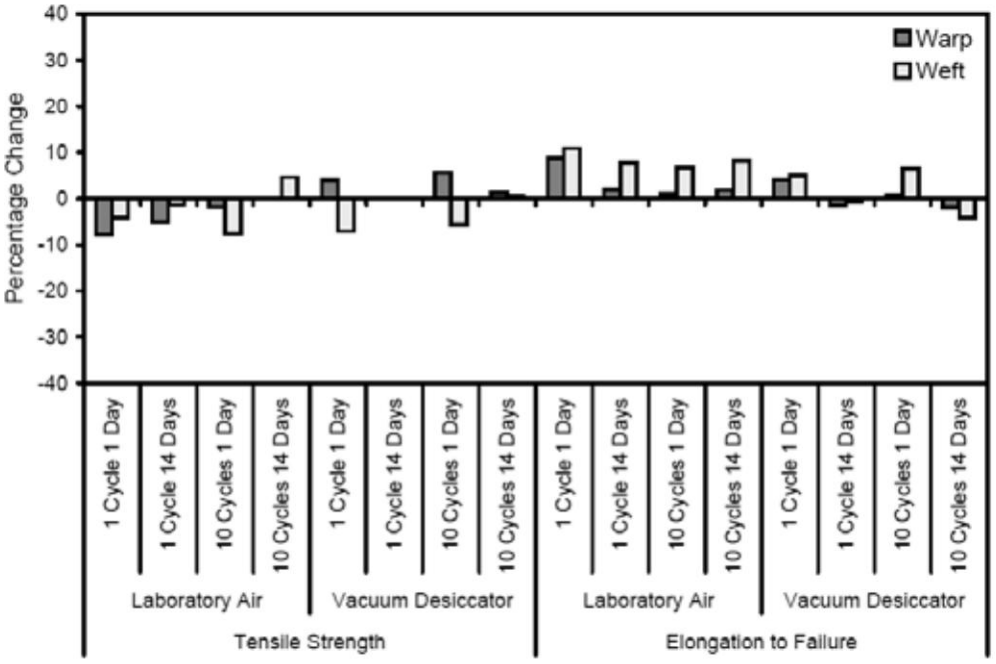


Figure 10. Percentage change in tensile strength and elongation to failure of Nomex® after the indicated exposure conditions (13).

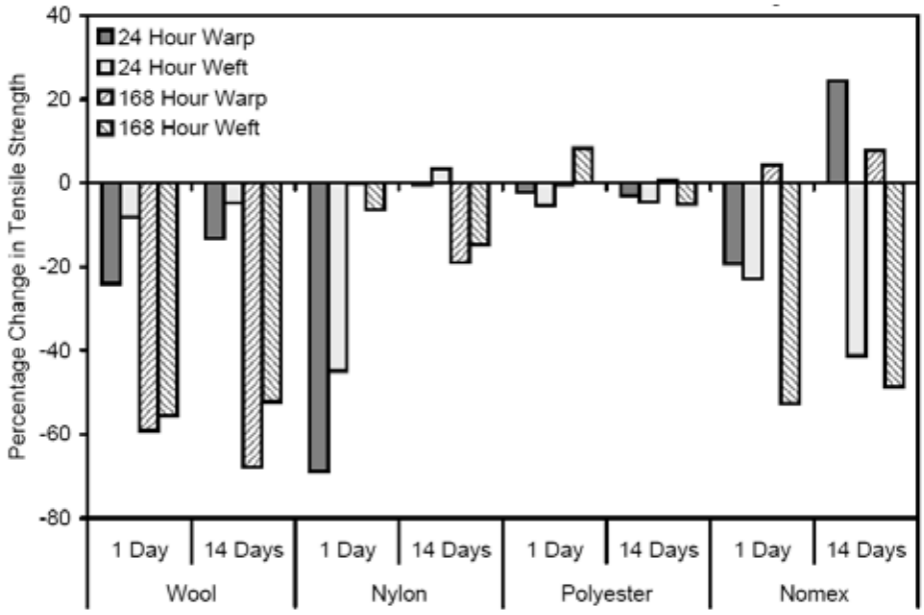


Figure 11. Percentage change in tensile strength of wool, nylon, polyester and Nomex® after 24- or 168-hour exposures to 35% liquid hydrogen peroxide for the indicated post-exposure laboratory air aging conditions (13).

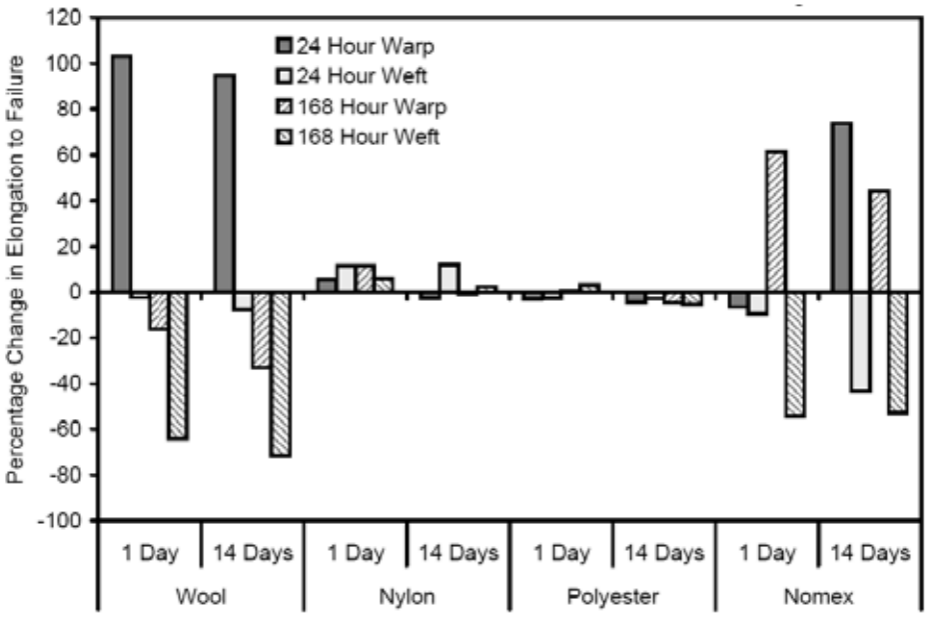


Figure 12. Percentage change in elongation to failure of wool, nylon, polyester and Nomex® after 24 or 168 hour exposures to 35% liquid hydrogen peroxide for the indicated post-exposure laboratory air aging conditions (13).

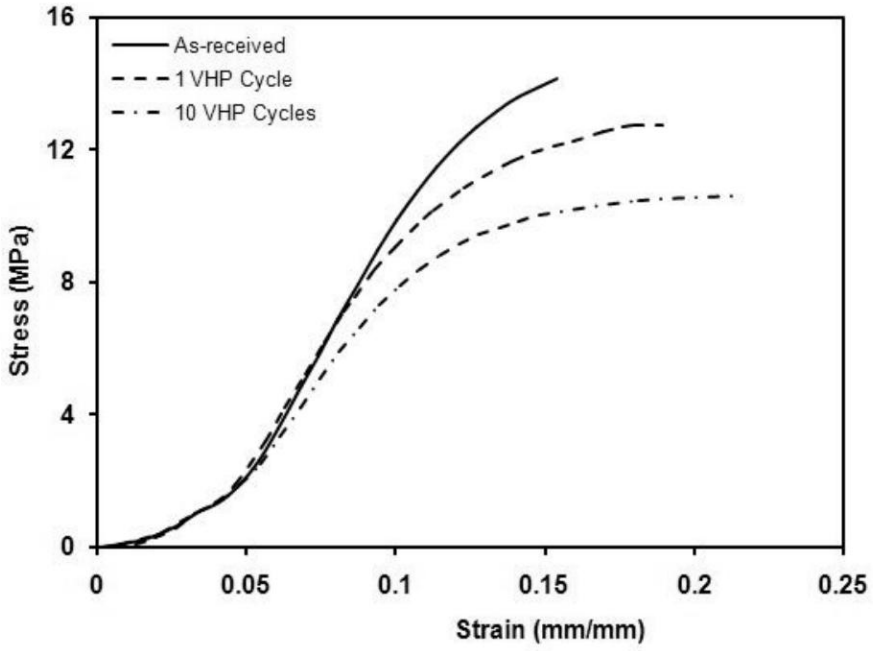


Figure 13. Tensile stress vs. strain curves for wool (a) as-received, (b) subjected to 1 VHP cycle and (c) 10 VHP cycles.

elastic modulus of the wool materials decreased after 1 VHP® cycle and then decreased again after 10 VHP® cycles. The strain at which the yielding began for each sample decreased from about 0.13 in the as-received wool sample to around 0.10 for the sample subjected to 10 cycles of VHP® .

Stress-strain curves for typical leather samples are shown in Figure 14. The tensile strength of the leather samples was seen to decrease from 22 MPa in the as-re-ceived condition to 20 MPa after 1 VHP® treatment to 9 MPa after 10 VHP® treatments to 7 MPa for a 10-min dip in 35% liquid H_2O_2 . The elongation to failure was also seen to decrease[2] from[2] 0.6 in the as-received condition to 0.35 after 1 VHP® treatment to 0.30 after 10 VHP® treatments. Interestingly, the 10-min dip in 35% liquid H_2O_2 caused the measured elongation to failure to increase[2] to[2] 0.8.

Changes in Flammability Behavior

Note: These tests were designed simply to evaluate possible changes in the intrinsic flammability behavior of the various basic textile materials due to H_2O_2 exposures and were not intended to assess compliance[22] with any specific flammability requirement.

The effects of exposures to hydrogen peroxide in either the vapor phase (1 and 10 cycles) or liquid phase (24 or 168 hour dips) on flammability of nylon, polyester, and Nomex® are shown in Figures 15, 16 and 17, respectively. Changes in flammability test results in the vertical and horizontal configurations and for the warp and weft textile orientations due to hydrogen peroxide

exposure are presented. The changes in flammability results for wool due to hydrogen peroxide exposure are shown in Figures 18 and 19.

Exposures of nylon (Figure 15) to 10 cycles of vaporized hydrogen peroxide did not produce significant changes in the vertical or horizontal flammability behavior in either the warp or weft directions. The 24-hour dip exposures significantly increased all measured flammability parameters for the both vertical and horizontal orientations of the warp weave. In contrast, 168-hour dip exposures produced no statistically significant changes in flammability behavior.

Polyester (Figure 16) exhibited significant increases in vertical flammability parameters for the weft weave during vertical flammability testing, whereas the warp weave showed no significant changes during vertical flammability testing (vapor or liquid exposures). Horizontal flammability testing of polyester yielded increases in burn length and burn time for the warp weave after exposure to 1 or 10 cycles of vaporized hydrogen peroxide; results for the weft weave were inconclusive. Dip testing of polyester tended to decrease the burn lengths and burn times during horizontal flammability testing.

Exposures of Nomex® (Figure 17) to either the vaporized or the concentrated liquid hydrogen peroxide did not appear to influence the measured flammability parameters of the material in either the vertical or horizontal flammability tests for either the warp weave or the weft weave.

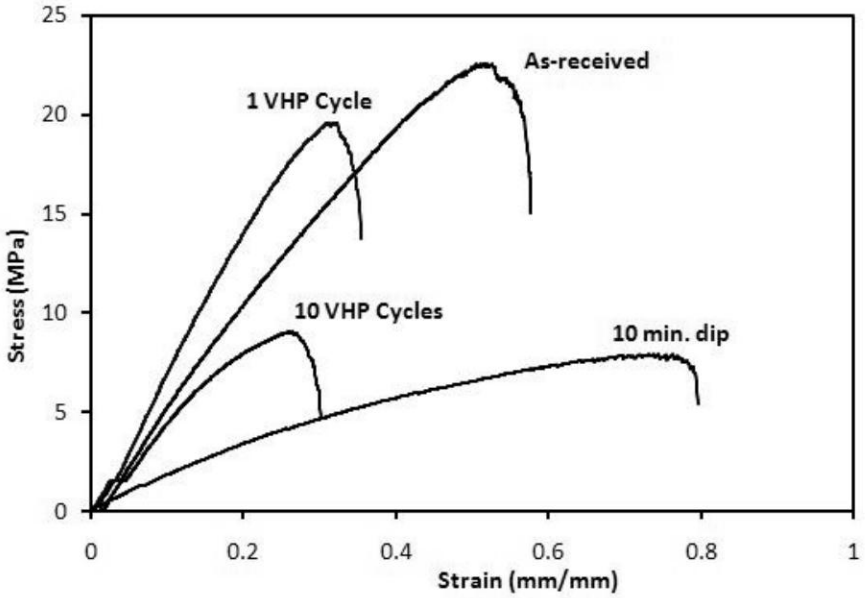


Figure 14. Tensile stress vs. strain curves for leather subjected to 1 VHP cycle, 10 VHP cycles and 10-min dip in 35% liquid H2O2.

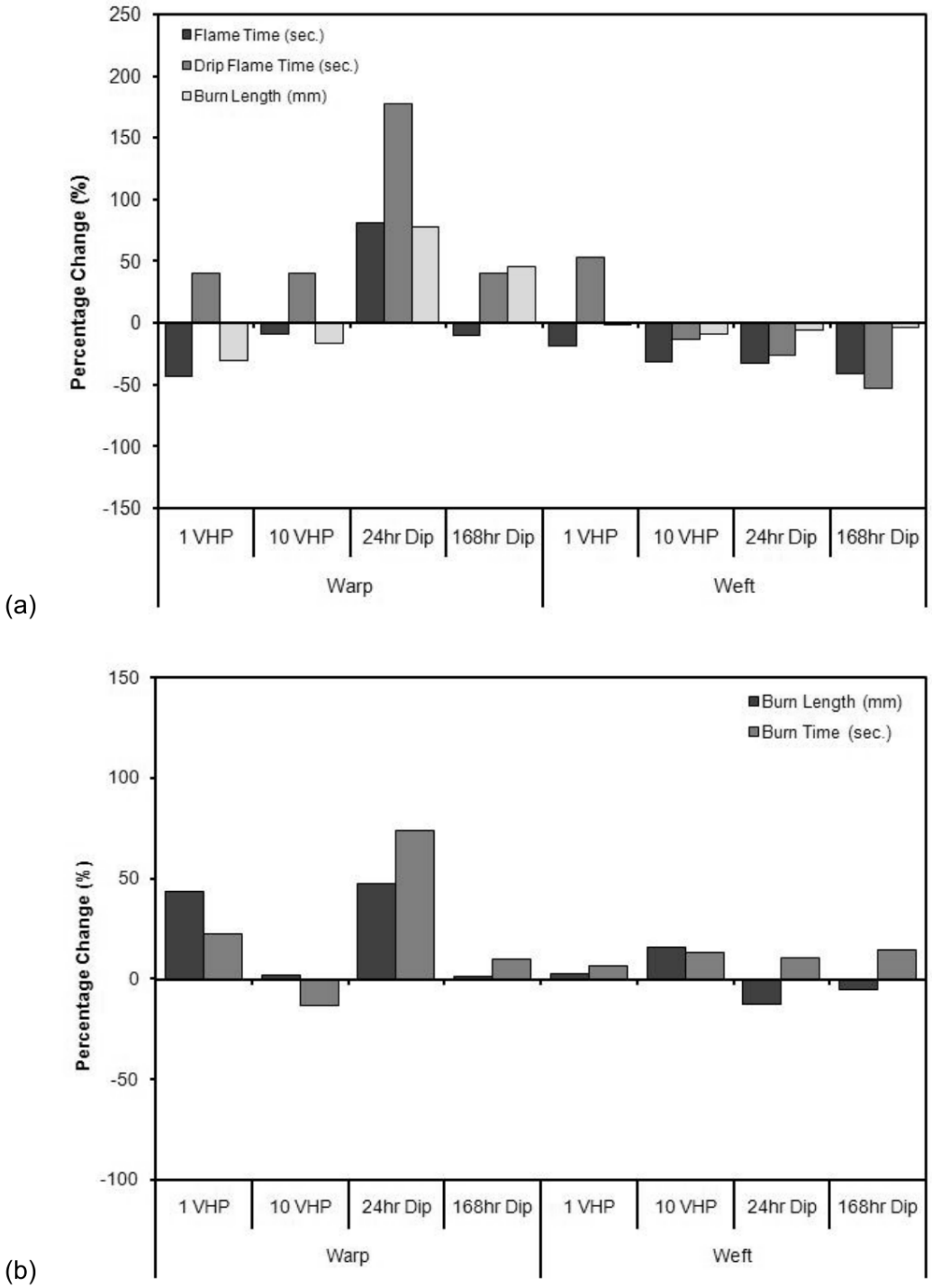
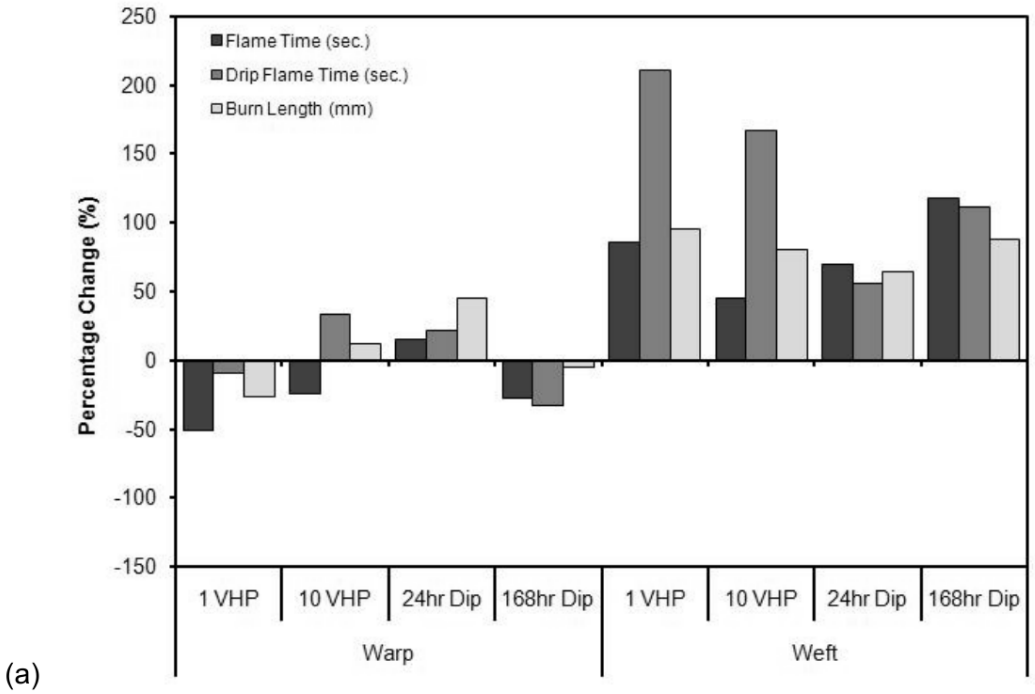
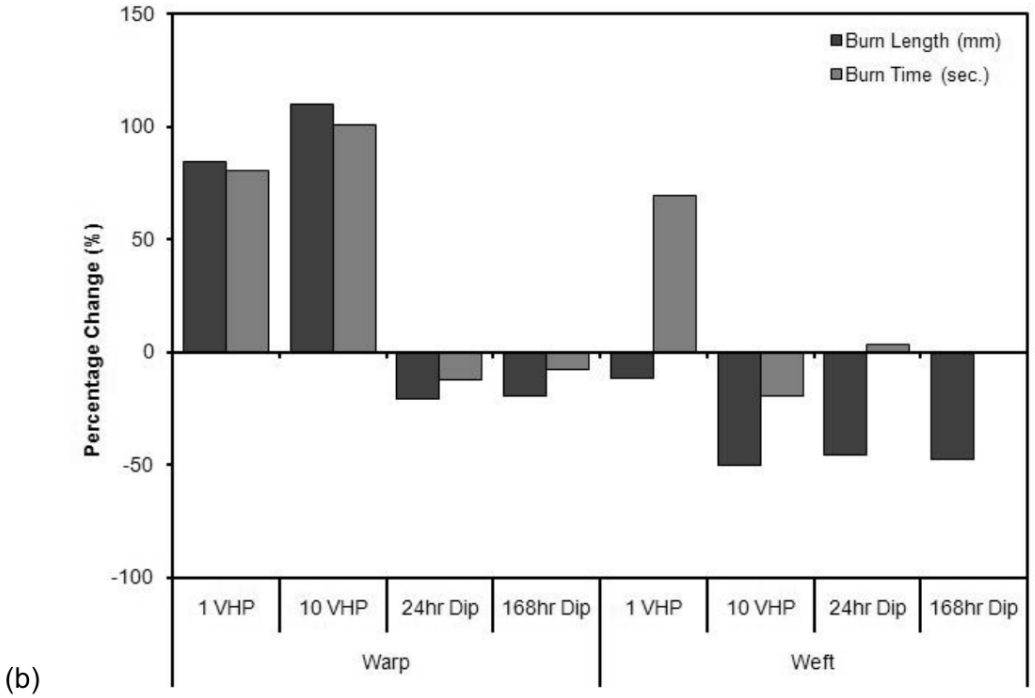


Figure 15. (a) Vertical and (b) horizontal flammability test results for nylon.

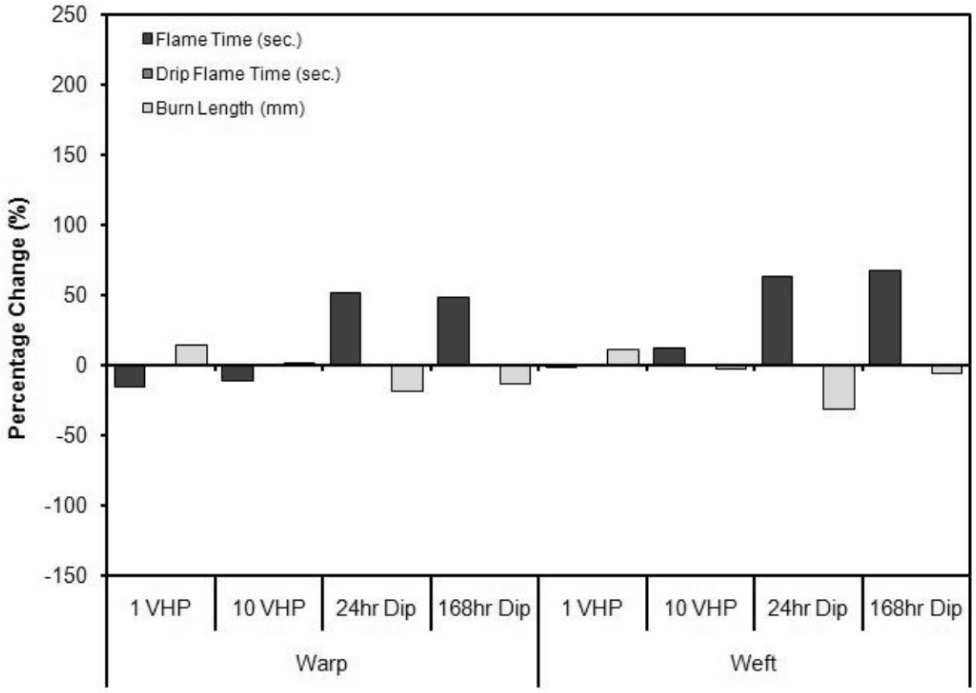


(a)

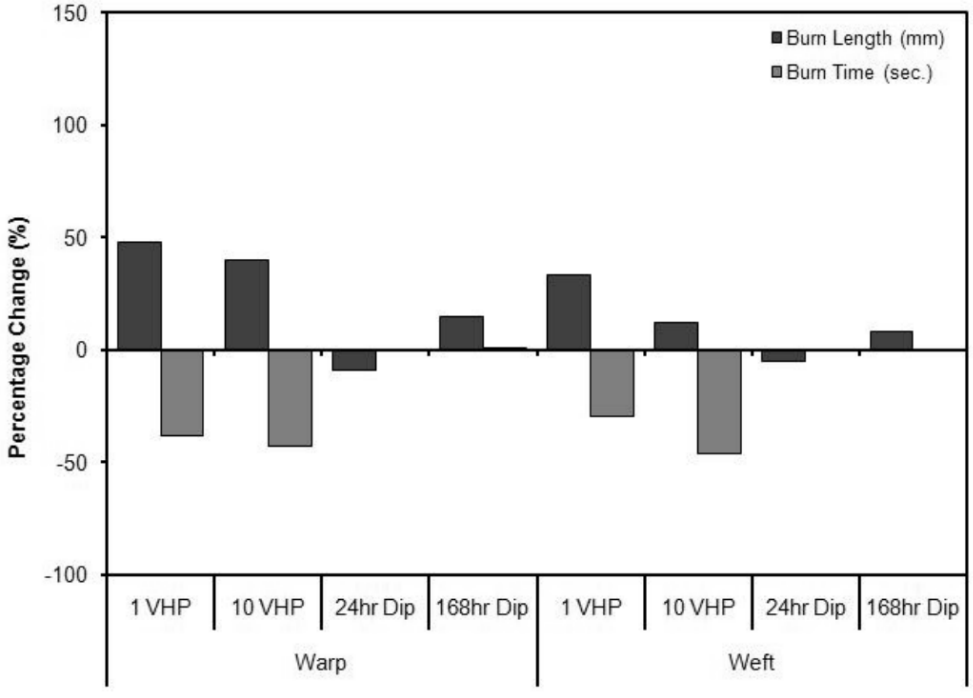


(b)

Figure 16. (a) Vertical and (b) horizontal flammability test results for polyester.

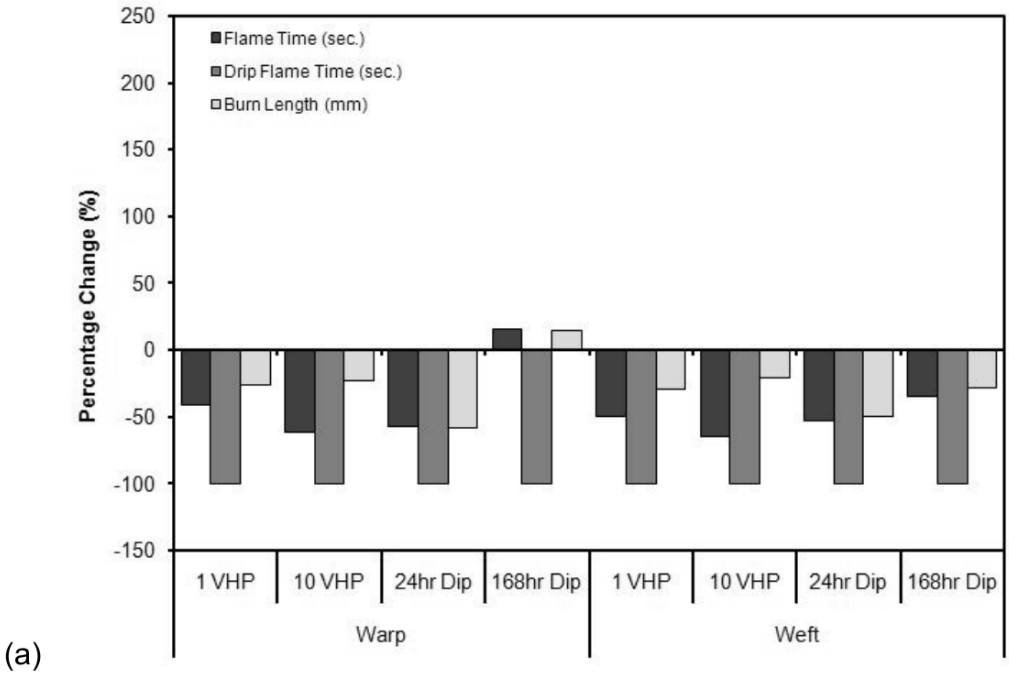


(a)

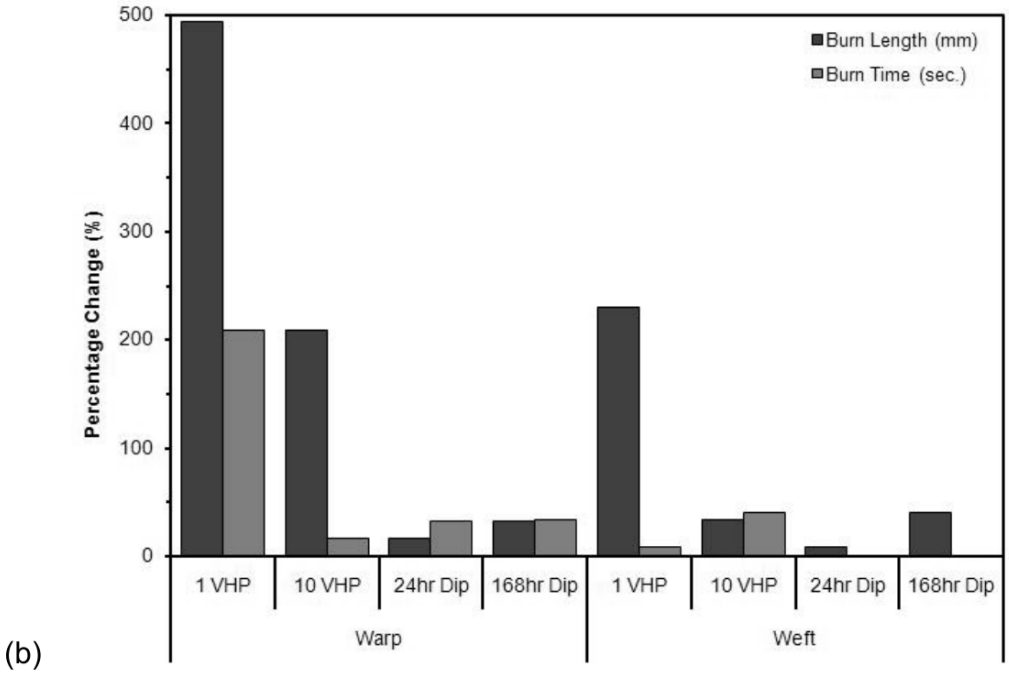


(b)

Figure 17. (a) Vertical and (b) horizontal flammability test results for Nomex.



(a)



(b)

Figure 18. (a) Vertical and (b) horizontal flammability test results for wool.

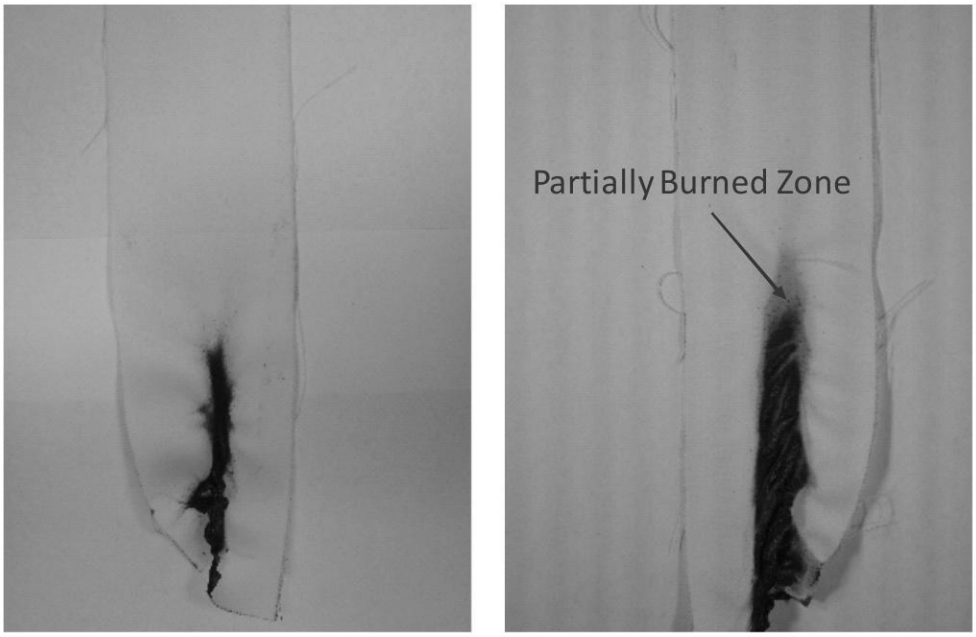


Figure 19. Appearance of wool after vertical flammability testing.

Wool (Figure 18) exhibited a consistent trend towards decreases in vertical flammability parameters for all conditions (vapor exposures, dip exposures, warp and weft weaves). Figure 19 shows samples of wool (as-received and VHP® treated) after vertical flammability testing. An expanded brownish region was observed between the flame decomposition product region and the unburned wool for the VHP® exposed specimens. During horizontal flammability testing of wool, VHP® treatments usually produced large increases in burn length and burn time, while dip treatments only produced minor increases in burn length and burn time. This can be associated with sample weight increases due to moisture sorption in the VHP® process, as well as in the conditioning unit, thus resulting in incomplete combustion.

As noted above, these tests were not intended to evaluate the compliance of any material with the materials flammability requirements, as outlined in Title 14 of the Code of Federal Regulations Part 25 § 853 for aircraft compartment interiors of transport category airplanes. None of these materials had any flame retardants applied.

Nevertheless, it is interesting to note that all flammability results for Nomex® — both as-received as well as after all exposures to hydrogen peroxide — were within the limits of §25.853 for both vertical and horizontal flammability testing.

The horizontal burn rates for nylon and polyester, as-received as well as after all exposures to hydrogen peroxide, met the requirements of § 25.853. The horizontal burn rates for wool (as-received and after all exposures to hydrogen peroxide) did not meet §25.853 limits.

Additionally, although the vertical flammability data for wool, nylon and polyester (as received and as exposed to hydrogen peroxide) met the limits of §25.853 for drip flame time, the limits of §25.853 were not met for these materials with respect to flame time. The vertical flammability data for nylon and polyester generally met the requirements of §25.853 with respect to burn length. The vertical flammability results regarding burn length for wool showed no consistent trend with respect to meeting the limits of §25.853 .(12).

Six representative samples of aviation grade, blended wool textiles and six samples of aviation grade leathers — all presumably with flame retardants applied — were acquired from aviation suppliers. The wool blends contained either polyamide (5 or 9% by weight) or nylon (8 or 10% by weight) with the balance natural wool fibers. Any specific flame retardant(s) utilized were not known for these preliminary tests. Vertical flammability testing was performed on an as-received sample from each supplier. A second sample of each was then exposed to 10 cycles of VHP® prior to vertical flammability testing. Weight changes due to exposure to the vaporized hydrogen peroxide were noted. The wool blends averaged a 3.6% gain in weight, whereas the leather samples averaged a 1.9% decrease in weight. The results of this preliminary vertical flammability testing are shown in Table I. The flammability of the wool blends exhibited increases in burn length from 78 to 154 mm and increases in flame time from 1.6 to 19.6 s. The flammability of the leather samples exhibited increases in burn length from 46 to 105 mm and increases in flame time from 8.8 to 13.4 s. The Aircraft Materials Fire Test Handbook (12) notes that the average flame time will not exceed 15 seconds, and the average burn length will not exceed 203 mm for the 12-s ignition time vertical test. The 10 cycles of VHP® exposures caused the wool blends to exceed the limits of average flame time.

Changes in Chemical Composition and Structure

FTIR and Raman spectroscopy were used to examine the chemical structure of all the fabrics tested. The analysis showed that only wool exhibited any measurably significant chemical structural change and the data for nylon, polyester, and Nomex® are not presented. Figure 20 shows the FTIR spectra of wool in the as-received condition and wool treated with either 1 or 10 VHP® cycles, followed by either 1 or 14 days in either laboratory air or a vacuum dessicator. The usual characteristic bands observed in the IR spectra of wool include the amide I (C=O stretching), amide II (N-H stretching), and amide III (N-H in-plane bending and C-N stretching) modes at 1630-1650 cm^{-1}, 1500-1550 cm^{-1} and 1200-1250 cm^{-1}, respectively. A distinctive new peak at around 1035 cm^{-1} is seen for the VHP® -treated wool specimen. Although barely discernable after a single VHP® treatment, the new peak grows significantly and is easily seen after 10 VHP®

Table 1. 12-Second Vertical Flammability Testing of Aviation-Grade Textiles.

Treatment	Wool Blends: Burn Length (mm)	Wool Blends: Flame Time (s)	Leathers: Burn Length (mm)	Leathers: Flame Time (s)
As-received	78	1.6	46	8.8

Treatment	Wool Blends: Burn Length (mm)	Wool Blends: Flame Time (s)	Leathers: Burn Length (mm)	Leathers: Flame Time (s)
After 10 VHP® Cycles	154	19.6	105	13.4

Note: The Aircraft Materials Fire Test Handbook (DOT/FAA/AR-00/12, 2000) notes limits of 15 s for average flame times and 203 mm for average burn lengths for the 12-s vertical flammability test.

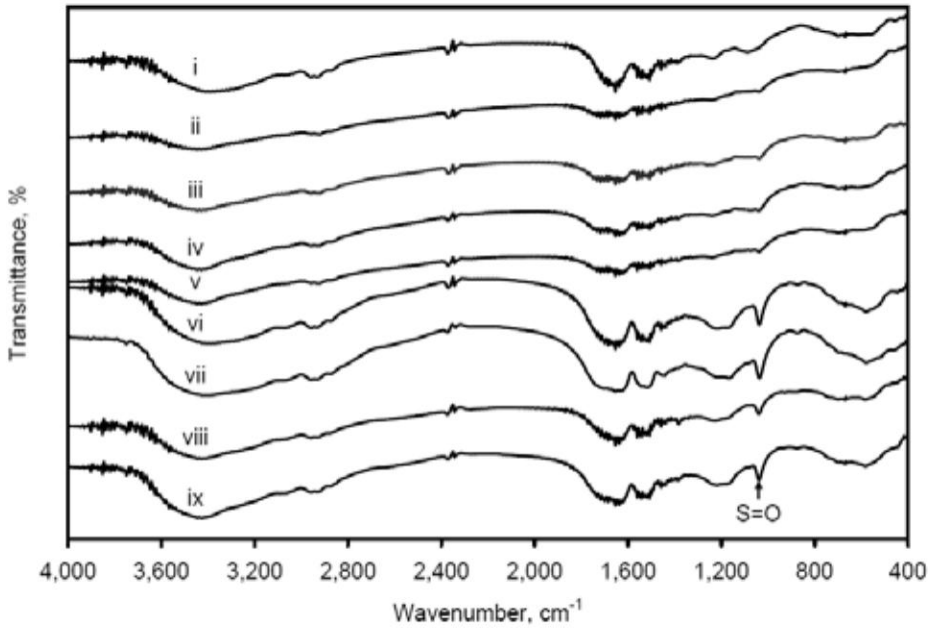


Figure 20. FTIR spectra of wool after exposure to vaporized hydrogen peroxide: (i) as-received; (ii) 1 VHP cycle + 1 day vacuum desiccator; (iii) 1 VHP cycle + 1 day laboratory air; (iv) 1 VHP cycle + 14 days vacuum desiccator; (v) 1 VHP cycle + 14 days laboratory air; (vi) 10 VHP cycles + 1 day vacuum desiccator; (vii) 10 VHP cycles + 1 day laboratory air; (viii) 10 VHP cycles + 14 days vacuum; (ix) 10 VHP cycles + 14 days laboratory air.

cycles. This new peak can be assigned to S=O bonds from the formation of sulfinite (SO_3^{2-}) and/or sulfanite (SO_4^{2-}). Similar results were seen for the wool samples exposed to concentrated liquid hydrogen peroxide by dip testing.

Raman spectroscopy was then used to examine the protein structure of wool (i.e ., amide I, II, and III). It can be seen from Figure 21 that the amide I and III bands corresponding to wavenumbers at 1630~1650 cm^{-1} and 1200~1250 cm^{-1} do not display any shift or change after 10 VHP® exposure cycles. The distinct peak at 1035cm^{-1} (due to the formation of S=O bonds) was also observed in the Raman spectra. In addition, peaks at 1450cm^{-1} were assigned to C-H bending and peaks at 890~960 cm^{-1} correspond with a C-C backbone vibration. A high wavenumber Raman spectrum of wool, shown in the inset of Figure 21, clearly shows C-H stretching.

To further investigate the oxidation of wool keratin with vapor phase hydrogen peroxide, X-ray photoelectron spectroscopy (XPS) was employed. Figure 22 (a) shows an overall scan for as-

received wool, as well as VHP® treated wool. The binding energies for carbon, nitrogen, and oxygen atoms remain the same. A detailed scan for sulfur atoms around the sulfur *2p* region is shown in Fig. 22 (b). The binding energy for sulfur *2p* in the as-received wool keratin was found to be 164 eV (as-received data, broad diffuse peak) whereas the binding energy for hydrogen peroxide-treated wool keratin was shifted to 168.7 eV (H_2O_2 exposed data, broad diffuse peak). The shift in binding energy[22] indicates a structural change from disulfide crosslink to sulfonic acid in the formation of sulfanite - SO_4^{2-} .(14)

CONCLUSIONS

1. Exposures of wool, nylon, polyester and Nomex® to vaporized or liquid phase hydrogen peroxide caused increases in weight, presumably due to absorption of water vapor from the treatment as well as decomposition of H_2O_2 to oxygen and water vapor. Aging samples in a vacuum[22] dessicator removed the absorbed water and produced net weight decreases for samples exposed to a single VHP® cycle. Net weight gains remained after 10 VHP® cycles, followed by vacuum aging. Preliminary data with leather samples indicated a net loss of weight after exposure to vaporized hydrogen peroxide. 2. The tensile strength of wool was moderately degraded (~20-30% loss), and the elongation to failure was increased by exposure to vaporized hydrogen peroxide. 3. The tensile strength and the elongation to failure of polyester were slightly degraded (~10% loss) by exposure to vaporized hydrogen peroxide. 4. The tensile strength of nylon was minimally degraded (<10% loss) by exposure to vaporized hydrogen peroxide. Results for the elongation to failure were inconclusive. 5. The tensile strength and the elongation to failure of Nomex® were unchanged by exposure to vaporized hydrogen peroxide. 6. The tensile strength and the elongation to failure of leather were severely degraded (~50% loss) by exposure to vaporized hydrogen peroxide.

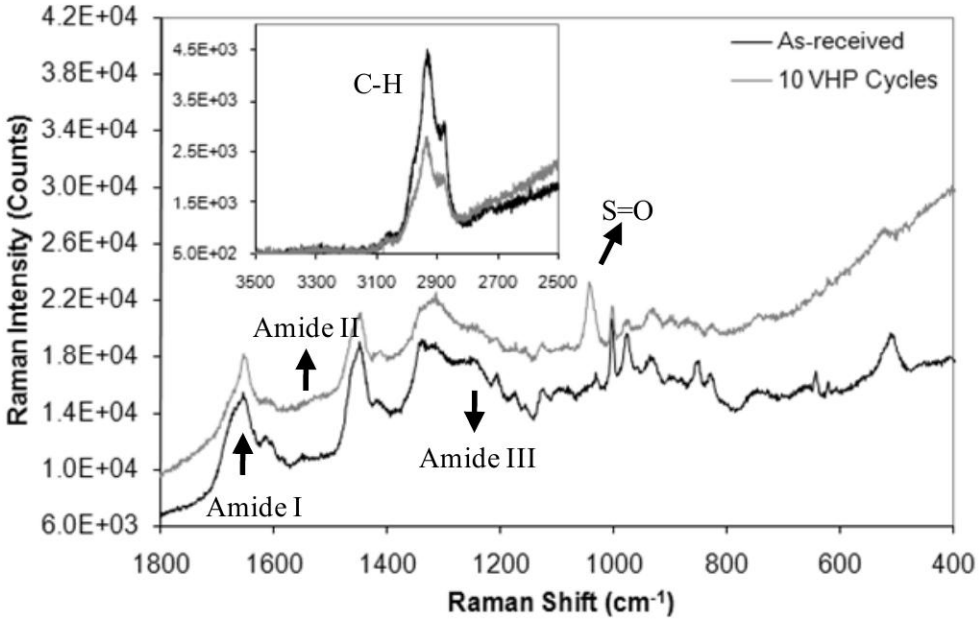
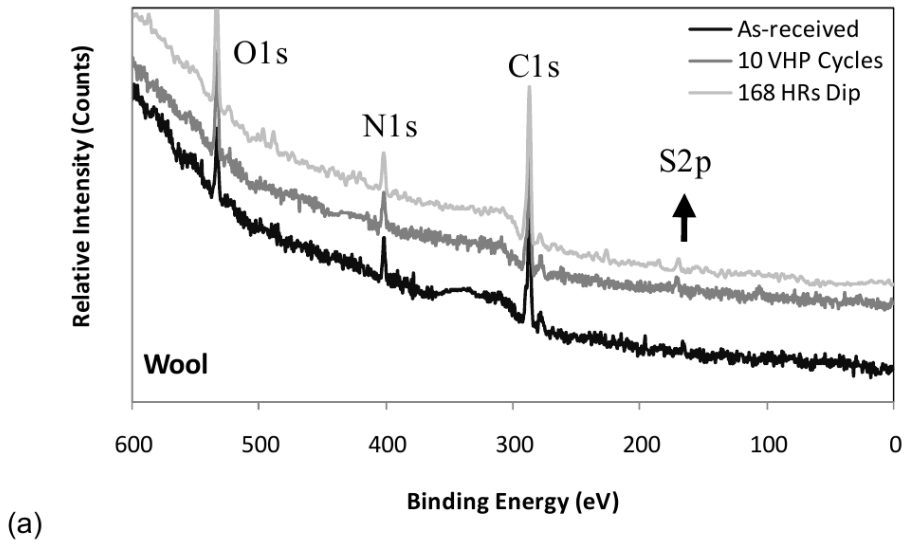
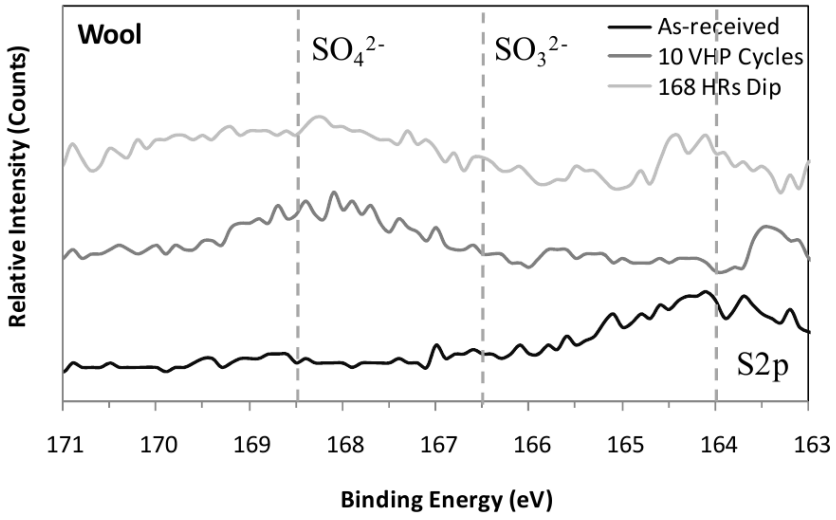


Figure 21. Raman spectra for as-received wool and VHP-treated wool. Insert shows high wavenumber spectra for examination of C-H stretching.



(a)



(b)

Figure 22. XPS spectra for wool in the conditions indicated: (a) overview scan and (b) detailed scan in the sulfur atom region.

7. The tensile strength of nylon and polyester generally were only slightly degraded (<10% loss) by exposures to 35% concentrated liquid hydrogen peroxide.

8. The tensile strength of wool and Nomex® were severely degraded (~50% loss) by exposures to 35% concentrated liquid hydrogen peroxide.

9. All preliminary flammability results for Nomex® — both as-received as well as after all exposures to hydrogen peroxide — were within the limits of §25.853 for both vertical and horizontal flammability testing.

10. The preliminary flammability testing of wool, nylon, and polyester indicated complex effects on flame/burn times, burn lengths and horizontal burn rates, presumably due to the presence of absorbed water vapor from the hydrogen peroxide exposures and the pre-flammability testing conditioning treatments. Additional research into these effects is necessary to fully understand and rigorously characterize this important behavior.

11. The preliminary flammability testing of aviation-grade wool blends indicated that 10 cycles of VHP exposures would cause unacceptable increases in flame times. Additional research into the effects of VHP exposure to aviation flame retardants is necessary to characterize these effects.

12. FTIR and Raman spectroscopy indicate that the disulphide cross-link bonds in wool fibers are oxidized to sulfonic acid in the formation of sulfanite — SO_4^{2-} by exposure to hydrogen peroxide. Apparently, the disulfide crosslinks that connect the wool's α-helix with the keratinassociated proteins were oxidized by hydrogen peroxide vapor, forming the newly observed S=O bonds.

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ERRATA

The corrections and notes below were made when this report was prepared for republication through the National Transportation Library. Minor typographical, spelling, and punctuation errors were also corrected and are not listed individually.

Corrections Made in This Edition

1. Results and Conclusions: chemical formulas repaired where subscripts had shifted to adjacent words: “sulfinite (SO_3^{2-}) and/or sulfanite (SO_4^{2-})” and “sulfanite — SO_4^{2-}”; missing subscripts restored in “H_2O_2.”

2. Figure 20 caption, item (v): “1 VHP cycle + days laboratory air” corrected to “1 VHP cycle + 14 days laboratory air.”

3. Figure 13 appeared twice on consecutive pages of the original (identical chart and caption); it appears once in this edition.

4. Results: “strongly dependant on” corrected to “dependent on”; reference author “Nu ń ez” corrected to “Nuñez.”

Discrepancies Noted but Not Changed

5. Results and Conclusions: the terms “sulfanite” and “sulfinite” are retained as originally published; they may be intended as “sulfonate” and “sulfite.”