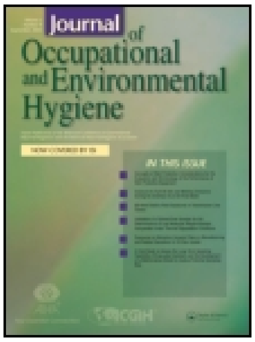

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Change in Tensile Properties of Neoprene and Nitrile Gloves After Repeated Exposures to Acetone and Thermal Decontamination

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This study investigated the change in tensile properties of neoprene and nitrile gloves after repeated cycles of exposure to acetone, followed by thermal decontamination. The glove was exposed to acetone (outer surface in contact with chemical), subjected to thermal decontamination, and tested for the tensile strength and the ultimate elongation. Thermal decontamination was carried out inside an oven for 16 hours at 100°C. The exposure/decontamination procedure was repeated for a maximum of 10 cycles. For neoprene versus acetone, the mean tensile strength consistently decreased after each exposure/decontamination cycle. Multiple comparisons indicated that the mean tensile strengths between the new swatches and each exposure/decontamination group were significantly different ($p < 0.05$). The loss of either tensile strength or ultimate elongation was less than 23% compared with new swatches after four exposure/decontamination cycles. Swatches without acetone exposure were then cycled through the oven in the same manner. It was found that both the heat used for thermal decontamination and acetone exposure significantly affected the tensile strength and ultimate elongation. For nitrile gloves exposed to acetone, the mean tensile strength remained virtually unchanged ($p > 0.05$). The mean tensile strength for the new swatches was 37.1 MPa and the mean tensile strength after nine exposure/decontamination cycles was 36.0 MPa, with a loss less than 3%. The largest single cycle loss for ultimate elongation occurred during the first exposure/decontamination cycle for both glove materials. In our previous study, decisions regarding the effectiveness of the decontamination process were based on having no discernible change in the breakthrough time and steady-state permeation rate. The results of this study indicate that the effectiveness of the decontamination process cannot be based on permeation parameters alone but must also take into account the change in physical properties.

Keywords acetone, chemical protective gloves, decontamination, degradation, tensile strength, ultimate elongation

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Prior to the availability of permeation data, information on degradation (i.e., deleterious change in one or more physical properties) was the standard for selecting chemical protective clothing (CPC). Test methods for CPC performance include resistance to degradation, penetration, and permeation. As a quick means to appraise the condition of chemical protective clothing, CPC material degradation was assumed whenever the color changed, the material wrinkled, or the material stiffened. Usually, the resistance to degradation was evaluated by noting changes in the visual appearance of the surface of a specimen after it came in contact with a chemical. Since the 1980s, several test methods have been developed for evaluating material degradation. These include ASTM D-412[(1)] based on changes in the tensile strength or elongation; ASTM D 543[(2)] and ASTM D 471[(3)] based on changes in weight, volume, or other physical property; ASTM F 1407[(4)] based on changes in weight; and ANSI/ISEA 105[(5)] based on changes in puncture resistance. According to the ANSI/ISEA standard, gloves are classified into five levels based on the change in percentage of puncture resistance (i.e., a change greater than 80%, less than 80%, less than 60%, less than 40%, and less than 20% correspond to performance levels 0, 1, 2, 3, and 4, respectively.) Changes in these parameters are used to indicate material degradation since they can be measured quantitatively.

Change in tensile property has been used as an indicator of material degradation for protective clothing and gloves under actual use conditions. For instance, Douglas et al.[(6)] evaluated changes in the tensile strength and barrier integrity of several medical gloves during hospital clinical use. Medical gloves including nonsterile vinyl, sterile vinyl, and nonsterile natural rubber latex gloves used in a clinical setting were tested for tensile strength and elongation. It was found that tensile properties of vinyl gloves did not change during use, whereas changes in latex depended on the brand evaluated. Tensile strength is one of the most common measurements of the strength of protective clothing materials.[(7)] However, tensile properties alone may not directly relate to overall performance of the product because of the wide range of specifications required for assuring product reliability in actual use.[1]

Decontamination of CPC may change the resistance of the barrier material making it more susceptible to permeation and degradation. This is because additives can be removed by either leaching due to the exposure solvent[8] or by migration/volatilization during thermal decontamination. Tensile properties are important in evaluating the protection afforded by gloves since the reusability of glove material means not only that the gloves prevent chemical permeation but also that the materials do not fail after repeated exposure/decontamination cycles. In our previous study,[9] glove materials, including neoprene and nitrile synthetic rubber, were exposed to organic solvents (which included acetone) followed by thermal decontamination. Exposure/decontamination was repeated for 10 cycles and changes in breakthrough time (BT) and steady-state permeation rate (SSPR) were measured. The results indicated that the BT and SSPR for neoprene against acetone were not significantly different from the new swatch after nine and five exposure/decontamination cycles, respectively. For nitrile exposed to acetone, the BT did not change significantly until after 7 exposure/decontamination cycles, and the SSPR did not change significantly until after 10 cycles. The purpose of this study was to investigate whether there is a correlation between the changes in permeation parameters and the changes in tensile properties after repeated exposure/decontamination cycles. Since the permeation parameters did not change significantly after repeated exposure/decon- tamination cycles, the hypothesis is that the tensile strength and ultimate elongation will not change significantly either.

TABLE I. Thickness of the Glove Materials

Glove Material	Thickness, mm (Mean and 95% Confidence Interval)	Supplier
Neoprene (Stanzoil N-440)	0.758, (0.742, 0.774)	MAPA-Pioneer
Nitrile Synthetic Rubber (37–155)	0.343, (0.338, 0.348)	Ansell Edmont

MATERIALS AND METHODS

Neoprene (Stanzoil N-440) and nitrile synthetic rubber (Ansell Edmont 37-155) gloves were selected for this study. All of the gloves used in this study were purchased from the vendor at the same time. Circular swatches, with a diameter of 17.8 cm, were carefully cut from gloves using a cardboard pattern. Thicknesses of the glove materials were measured using an Ames micrometer (model 3W; Ames Instrument, Waltham, Mass.) with an accuracy of ±0.002 mm. Table I shows the mean thickness and the 95% confidence interval for the gloves.

Acetone (99.7%) was selected as the challenge chemical because it is one of the liquid test chemicals recommended by the American Society for Testing and Materials (ASTM).[10]

Apparatus and Procedures for Chemical Exposure

As shown in Figure 1, the test apparatus was fabricated in-house and consisted of a chamber made by holding a section of glass beaded pipe between top and bottom Teflon plates. The

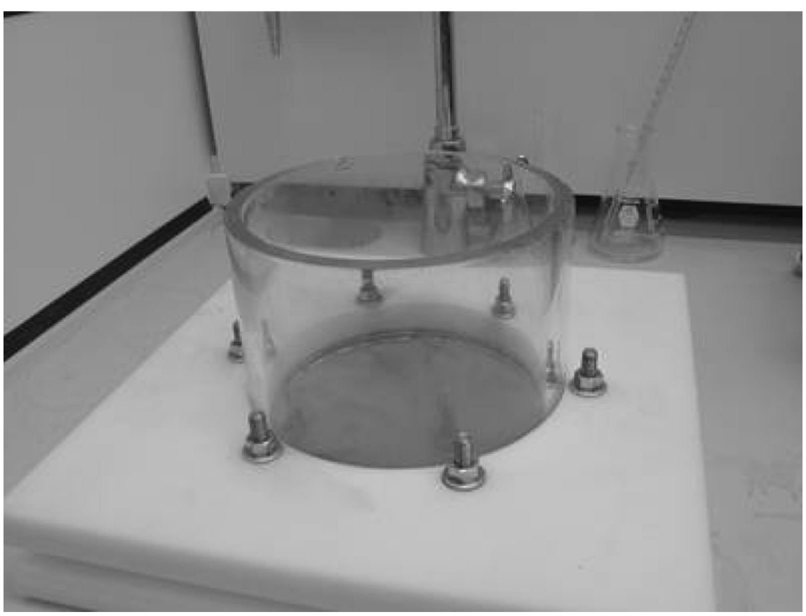

FIGURE 1. Exposure chamber

inside diameter of the chamber was 15.0 cm and the height was 12.5 cm. The outside diameter of the chamber at the base with the beaded section was 18.0 cm. Glove material, with the outside facing up for chemical exposure, was placed over GORE-TEX gasket material in the base of the exposure chamber to provide a leak-free seal. The glass beaded pipe section was placed over the glove material so that the beaded edge was facing down. The top Teflon plate was positioned over the glass beaded pipe section and the cap screws protruding from the bottom Teflon plate. It was then slid down until it rested on the bottom of the glass pipe section. The pieces for the exposure chamber were secured using hex nuts. The glove material was covered with 350 mL of acetone to a depth of approximately 2 cm. The timer was started immediately after the chemical was poured into the chamber. The top of the chamber was covered. After steady-state permeation was fully developed, which was determined by our previous permeation study,[9] acetone was poured from the chamber. The chamber was then disassembled and the swatch was removed. The material was placed on an adsorbent towel and allowed to air dry for approximately 2 hours before thermal decontamination.

Thermal Decontamination

The glove materials were then placed into an oven (Precision; Fisher Scientific, Pittsburgh, Pa.) for 16 hours at 100°C. The conditions were selected based on a study by Vahdat and Delaney[11] and were the same as our previous study.[9]

Repeated Exposure and Decontamination Cycles

To investigate the possible degradation due to increasing the number of exposure/decontamination cycles, the process was repeated for a maximum of 10 cycles.

Measurements of Tensile Strength and Ultimate Elongation

Tensile strength and ultimate elongation were measured using a materials testing instrument (model LRX; Lloyd Instruments, Fareham, England). Tensile strength is the maximum tensile stress applied in stretching a specimen to rupture; ultimate elongation is the percentage change in the length of a specimen prior to rupture. For tensile property testing, a total of 33 circular swatches with a diameter of 17.8 cm were prepared on the first day. Three of them were randomly selected and cut into 10 dumbbell specimens (3 to 4 dumbbell specimens per circular swatch) using a Type C die and an Arbor Press. Therefore, each group consisted of three circular swatches that were cut into 10 dumbbell shapes.

Tensile testing was performed according to ASTM D412-98a Method[1] for these specimens that represent the values for the new materials. Parameters, including the length and thickness of the material, were entered into the instrument's software so that the tensile strength and ultimate elongation could be calculated. The remaining 30 circular swatches were exposed to acetone using the exposure chamber and then thermally decontaminated as described above. After that, 3 out of the 30 circular swatches were randomly selected and cut into 10 dumbbell specimens. Tensile properties were measured that represent the values for the materials after the first exposure/decontamination cycle.

The exposure/decontamination cycle was continued for the remaining 27 circular swatches. Three swatches were randomly selected again for the next day and the procedures continued. Exposure/decontamination cycling and tensile testing were continued in this manner, that is, three of the remaining circular swatches were selected at random for tensile testing and the rest of the swatches were continued for exposure/decontamination for a maximum of 10 cycles. Nitrile gloves underwent only nine cycles because the oven was out of control during the last thermal decontamination.

When in use, the internal surfaces of the gloves will be wet from perspiration; therefore, a comparison of tensile strengths between dry and wet gloves was made. For the wet condition, the inside of the glove was placed on a wet paper towel for at least 20 min before the testing. Twenty dumbbell specimens for each of the glove materials were measured ($n = 20$). Each dumbbell specimen was measured only once because the test was destructive.

Since our preliminary study found that the changes in tensile properties for neoprene gloves were significant, neoprene swatches without exposure to acetone were heated at 100°C for 16 hours inside the oven prior to measuring the tensile strength and ultimate elongation. The testing was also conducted up to 10 cycles to determine whether the changes in tensile properties were caused by the heat, acetone, or both.

Statistical Method

One-way analysis of variance (ANOVA, S-PLUS 6 for Windows, Insightful Corporation, Seattle, Wash.) was used to determine whether the exposure/decontamination cycles had an effect on tensile strength and ultimate elongation. Multiple comparisons at a 95% confidence level using the Dunnett method[12] were also employed for the data analyses. Two-factor ANOVA was conducted to determine whether the changes in tensile properties, if any, were attributed to the thermal decontamination (i.e., the heat) as well. Diagnostic residual plots were first made for each tensile strength and ultimate elongation means in order to determine if data transformation was required. Data transformation was not required since the plots indicated that the measurements did not significantly deviate from normality.

RESULTS

Comparison of Tensile Strength and Ultimate Elongation Between Dry and Wet Gloves

Figure 2 is a comparison of the tensile strength between dry and wet for both glove materials. The comparison of ultimate elongation is shown in Figure 3. The error bars in the figures represent 95% confidence intervals of the means. As shown in Table II, there was no significant difference between dry

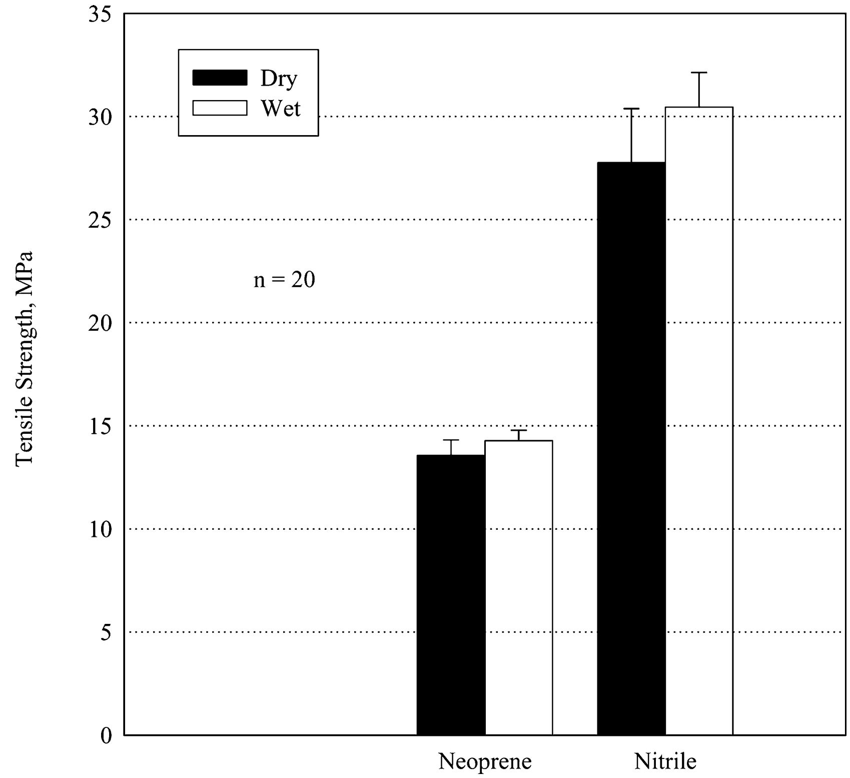

FIGURE 2. Comparison of tensile strengths between wet and dry gloves (n = 20)

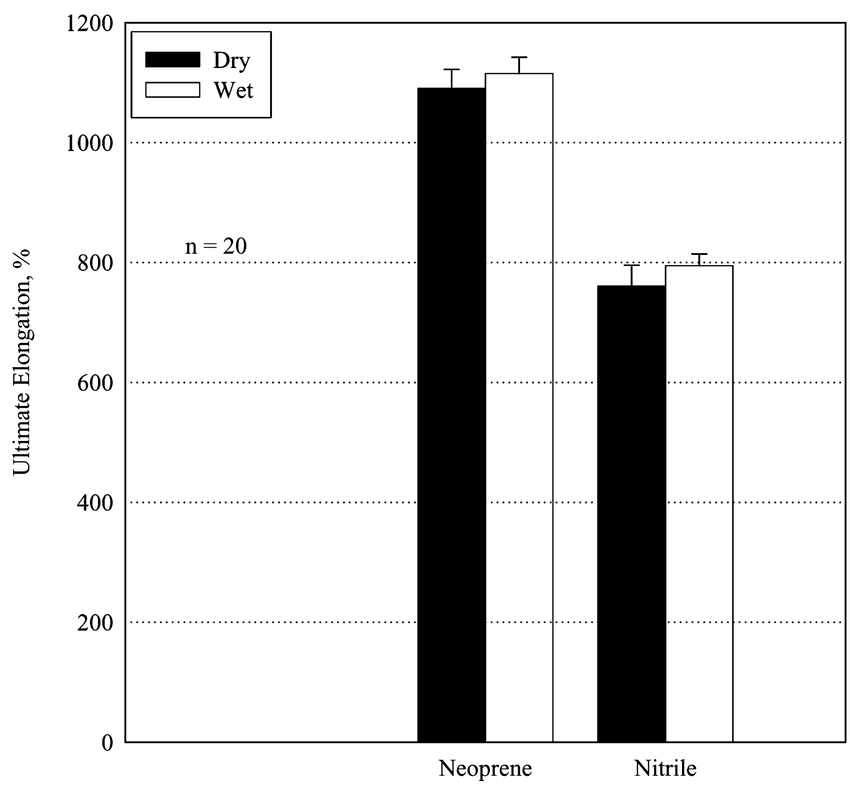

FIGURE 3. Comparison of ultimate elongations between wet and dry gloves (n = 20)

TABLE II. Comparison of Tensile Properties Between Dry and Wet Swatches

Material	Nitrile				Neoprene			
	Tensile Strength, MPa		Elongation (%)		Tensile Strength, MPa		Elongation (%)	
Parameter	Dry	Wet	Dry	Wet	Dry	Wet	Dry	Wet
Mean	27.8	30.5	760.6	794.8	13.6	14.3	1090	1115
Standard deviation	5.6	3.6	74.4	41.5	1.6	1.1	68.6	58.2
P-value	0.04		0.04		0.05		0.11	

and wet gloves for neoprene but the difference was significant for nitrile. Due to the relatively small p values for all the statistics, the repeated exposure/decontamination tests on both glove types were performed using wet samples to mimic the in-use situation where the internal surfaces of the gloves will be wet from perspiration.

Nitrile versus Acetone

Figures 4a and 4b show the changes in tensile strength and ultimate elongation for nitrile gloves against acetone after repeated exposure/decontamination cycles. The error bars in the figures represent 95% confidence intervals ($n = 10$). For the new swatches and for the swatches after four and nine exposure/decontamination cycles, the mean tensile strengths were 37.1, 37.3, and 36.0 MPa, respectively. On the other hand, for the new swatches and after four and nine exposure/decontamination cycles, the mean ultimate elongation was 732%, 658%, and 615%, respectively. For all the exposure/decontamination cycles, the coefficient of variation (CV) ranged from 4.8% to 15.0% for both tensile strength and elongation tests. The pooled CVs were 10.3%, and 7.9% for tensile strength and elongation, respectively.

The percentages of tensile strength and ultimate elongation retained after each exposure/decontamination cycle compared with new nitrile glove swatches were then calculated. Following nine exposure/decontamination cycles, the average loss was 5.3% for the tensile strength and 14.7% for the ultimate elongation. The loss of these tensile properties was essentially due to the first exposure/decontamination cycle. The tensile properties remained virtually unchanged during the subsequent exposure/decontamination cycles.

Neoprene versus Acetone

Figures 5a and 5b show the changes in tensile strength and ultimate elongation for neoprene against acetone after repeated exposure/decontamination cycles. Again, the error bars represent 95% confidence intervals of the means ($n = 10$). The mean tensile strengths were 15.0 MPa for the new swatches, 11.7 and 8.3 MPa for the swatches after four and seven exposure/decontamination cycles, respectively. The measurement consistently decreased to 8.1 MPa after 10 exposure/decontamination cycles. Correspondingly, for the new swatches and after four and seven exposure/decontamination cycles, the mean elongation was 1087%, 838%, and 640%, respectively. For the elongation, the measurement CVs ranged from 2.6% to 15.6%. The variations for tensile strength were greater than that for ultimate elongation (pooled CV 10.7% versus 7.2%).

Similarly, percentage changes in tensile strength and ultimate elongation were calculated for comparison. The decreases in tensile strength and ultimate elongation due to the first exposure/decontamination cycle were 13.2% and 12.2%, respectively. The total losses during the ten cycles were 31.5% for tensile strength and for ultimate elongation. The average loss of the tensile properties was only approximately 2% for each of the nine remaining exposure/decontamination cycles. Compared with the new swatches, retained percentages for the tensile strength and ultimate elongation were about 80% after three exposure/decontamination cycles. According to ANSI/ISEA standard 105 this is classified as the highest performance level.[5]

Effect of Heat Alone on the Changes in Tensile Properties for Neoprene Gloves

Figures 6a and 6b illustrate the changes in tensile strength and ultimate elongation for neoprene gloves that were cycled through the oven at 100°C for 16 hours. The error bars represent 95% confidence intervals ($n = 10$). The mean tensile strength was 13.7 MPa for the new swatches, somewhat different from the mean presented in Figure 5a, probably due to a different glove lot. The decreases in tensile strength and ultimate elongation caused by the first exposure/decontamination cycle were 5.9% and 10.6%, respectively. The total losses of tensile strength and ultimate elongation for the 10 cycles were 30.1% and 25.3%, respectively. It is apparent that the losses were all smaller than those with acetone exposure/thermal decontamination.

Statistical Analyses

For neoprene challenged by acetone and heat, multiple comparisons indicated that the mean tensile strengths and elongation between the new swatches and each exposure/decontamination cycle were significantly different ($p < 0.05$). The two-factor ANOVA indicated that both the heat during thermal decontamination and acetone exposure significantly affected the tensile strength and ultimate elongation. The combination of acetone exposure and the heat had a more averse effect on the changes in tensile properties. For nitrile, the differences in tensile strength were not significant ($p > 0.05$). The difference

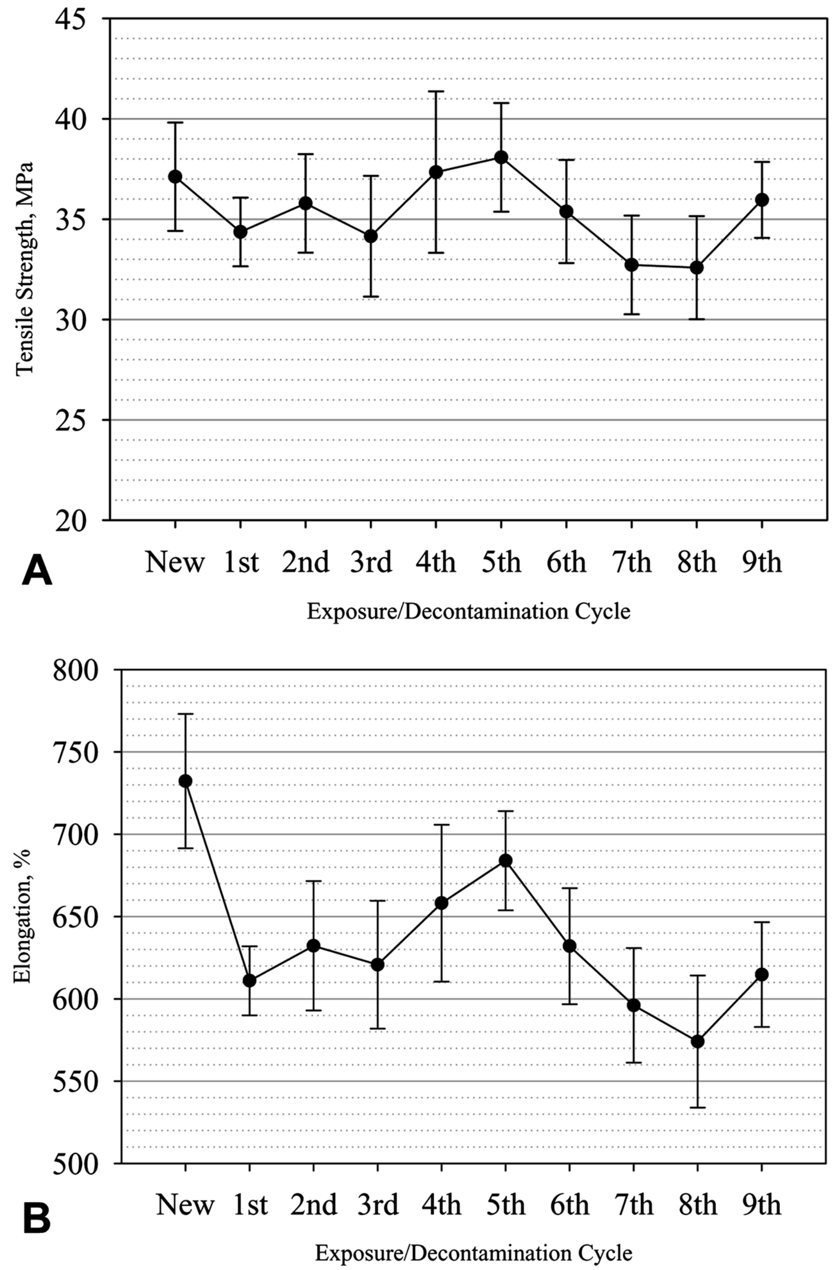

FIGURE 4. (A) Changes in tensile strength for nitrile gloves with acetone and thermal decontamination. The error bars represent 95% confidence intervals (n = 10). (B) Changes in ultimate elongation for nitrile gloves with acetone and thermal decontamination. The error bars represent 95% confidence intervals (n = 10).

in ultimate elongation was significant after the first cycle. However, the ultimate elongation remained statistically unchanged with subsequent cycling.

DISCUSSION

In the past, degradation testing was based on change in thickness or percentage swelling. However, the accuracy and variations were generally unacceptable. For instance, one interlaboratory evaluation of five protective clothing materials against five liquid chemicals showed a CV within-lab to be as high as 4957% when using thickness measurements.[13] In contrast, the highest CV in our current study was 15.6%; similar variations were obtained by another study[6] using tensile properties. Therefore, a more appropriate test for degradation would evaluate physical properties that could be more accurately and precisely quantified, such as changes in tensile strength, elongation, puncture resistance, cut resistance, or tear resistance.

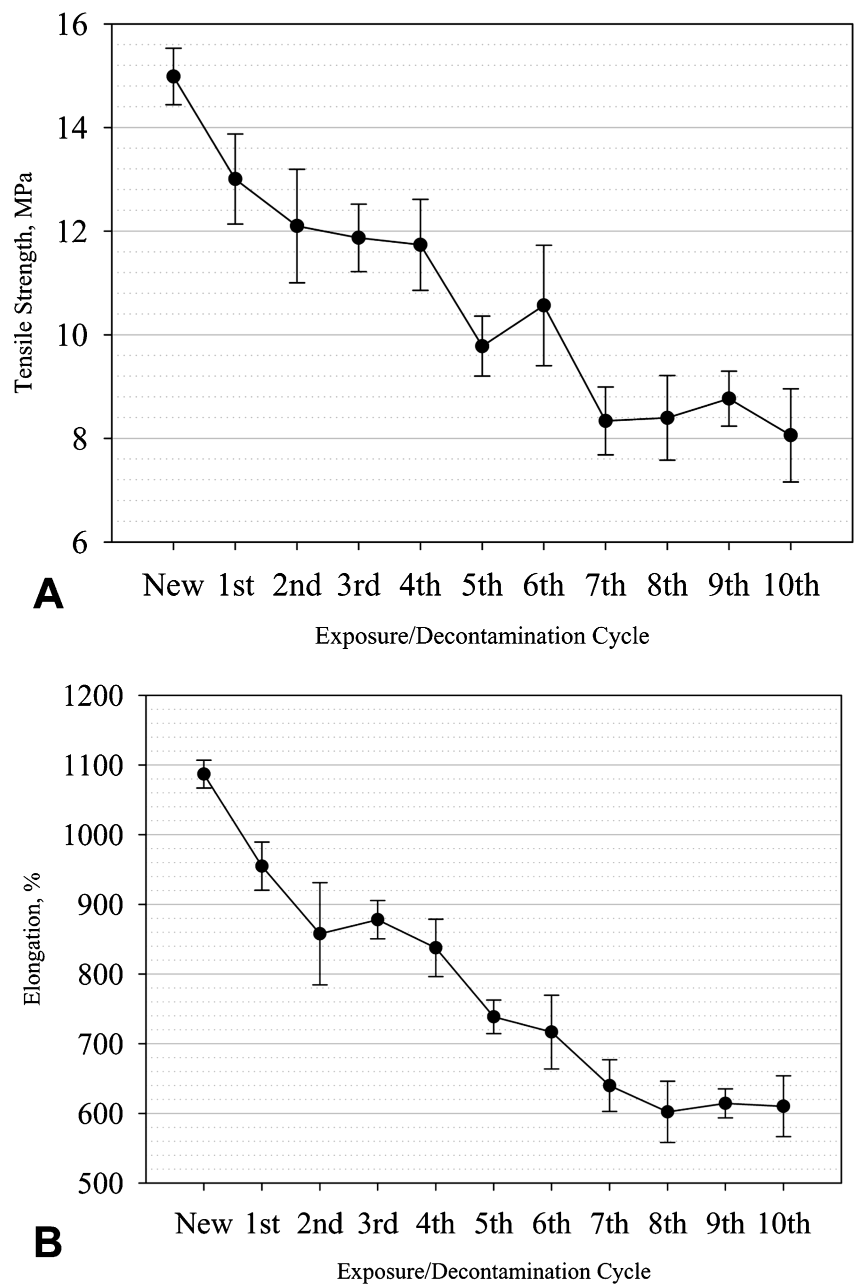

FIGURE 5. (A) Changes in tensile strength for neoprene gloves with acetone and thermal decontamination. The error bars represent 95% confidence intervals (n = 10). (B) Changes in ultimate elongation for neoprene gloves with acetone and thermal decontamination. The error bars represent 95% confidence intervals (n = 10).

A difference in physical properties was found between a wet glove and a dry glove. Interestingly, the results shown in Table II indicate that wet gloves possessed an even higher value for either tensile strength or ultimate elongation. The difference was significant for nitrile gloves. This phenomenon needs further investigation to determine the cause for the increased values of tensile properties for wet gloves. However, since the inner surfaces of a glove will be wet while being used, physical property testing under wet or in-use condition is suggested.

The results showed that nitrile, although its thickness was approximately half of that for neoprene, is significantly stronger than neoprene when both are new (37.1 versus 15.0 MPa). When comparing flexibility or elasticity, neoprene was superior to nitrile gloves (1087% versus 732%). However, neoprene loses its elasticity gradually after each exposure/

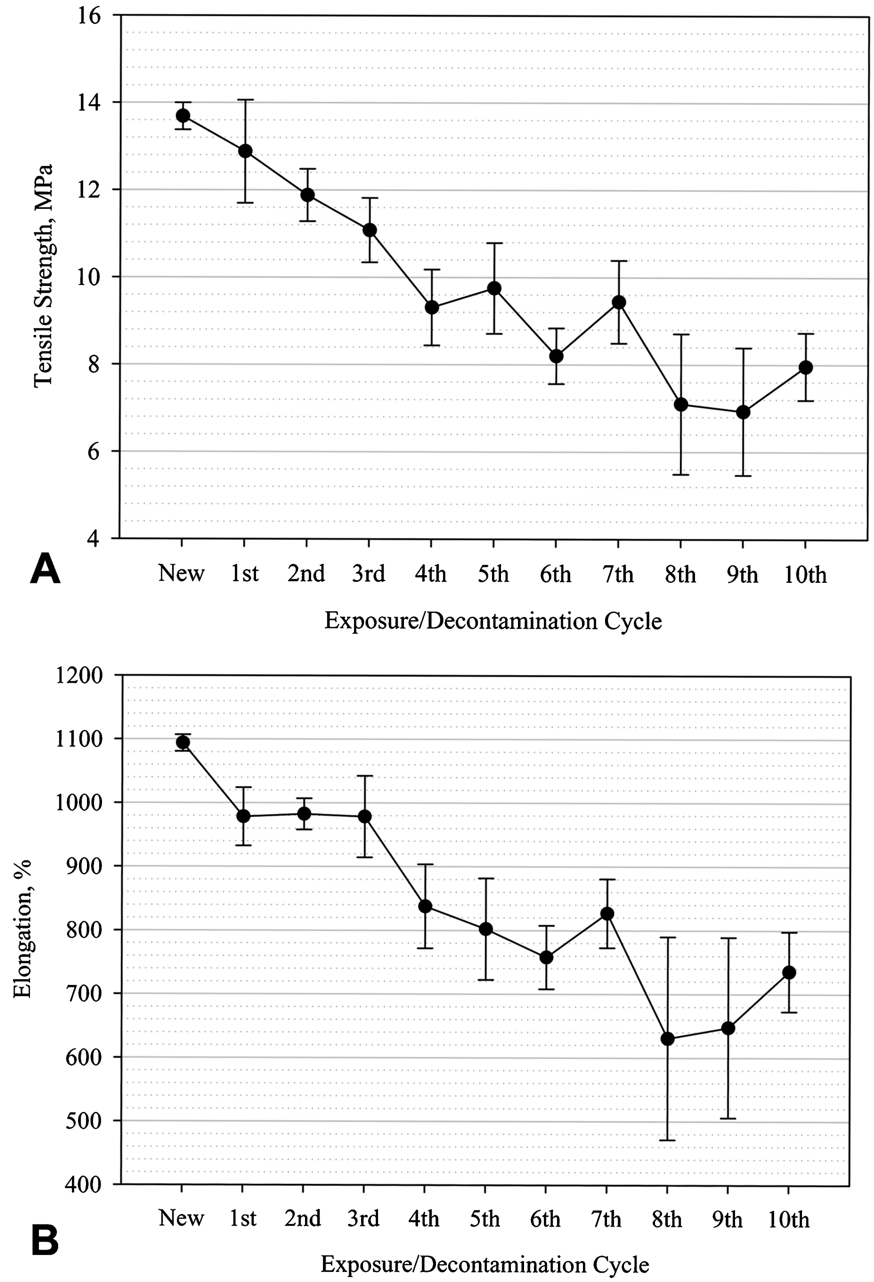

FIGURE 6. (A) Changes in tensile strength for neoprene gloves with heat only. The error bars represent 95% confidence intervals (n = 10). (B) Changes in ultimate elongation for neoprene gloves with heat only. The error bars represent 95% confidence intervals (n = 10).

decontamination cycle so that the ultimate elongation was about the same as that for nitrile after nine repeated cycles (614% for each).

Our previous permeation testing[(9)] indicated that the mean BT values for neoprene/acetone and nitrile/acetone combinations were not significantly different from the original values after nine and seven cycles, respectively. The mean SSPR values for these two material/chemical combinations were not significantly different from those for new swatches after 5 (neoprene) and 10 (nitrile) cycles. The fact that the tensile strength for nitrile did not significantly change with increasing exposure/decontamination cycles is consistent with the change in their permeation parameters.

For neoprene, tensile strength and elongation consistently decreased with increasing exposure/decontamination cycles and the differences were all significant. Therefore, changes in permeation resistance and physical property are not correlated. The lack of observed correlation between the changes in permeation parameters and the losses of the tensile properties for neoprene may indicate that the loss of the plasticizers due to the acetone exposure and/or the thermal decontamination had a considerably greater impact on the tensile properties than on the changes in chemical resistance. Obviously, for this study, the tensile properties were able to detect this effect before changes in permeation parameters occurred. If ANSI/ISEA's glove performance classification[5] as described earlier is adopted, the neoprene can be reused up to four exposure/decontamination cycles while remaining at the highest performance level—Level 4.

The largest single cycle loss of the ultimate elongation during the first exposure/decontamination cycle could be attributed to the loss of plasticizers—the agents added to increase the polymer's flexibility. Plasticizers have much higher solubility in a solvent (such as acetone during exposure in this study), and they evaporate much easier during thermal extraction due to their low molecular weights when compared with the barrier materials. Since the plasticizers are not covalently bonded with the polymer, dissolution or evaporation of the plasticizers occurs mainly during the first exposure/decontamination cycle; thus a greater loss of the elongation is observed. Another possible cause for the loss in elongation is that the polymer undergoes additional crosslinking due to the thermal decontamination process.

The fact that heat also significantly reduced the tensile strength and ultimate elongation of neoprene gloves may indicate that the temperature used for the decontamination was too high or the duration was too long. Further studies are needed to optimize the thermal decontamination conditions or to develop a better decontamination method for this material/chemical combination.

Chemical resistance and CPC integrity are two important criteria regarding reusability. For determining whether CPC can be reused, we suggest that both the permeation and tensile properties be evaluated to ensure that CPC not only functions correctly for adequate protection but also does not fail to avoid immediate risks of experiencing a harmful exposure. The permeation properties measure only how well the glove protects from chemical permeation. There may be no indication that the glove's integrity has changed. For checking the glove's integrity, the tensile properties ensure that the glove will have the acceptable strength and stretch.

Although a correlation between tensile properties and permeation measurements does not necessarily exist as found in the present study, a correlation could exist if the chemical permeating through the glove affected the chemistry of the glove material in such a manner that it affected both the permeation resistance and the tensile properties. For example, a chemical could be soluble within the glove matrix, resulting in the removal of the plasticizer. This would cause a reduction in the stretchability for the glove, thus reducing the ultimate elongation. Our permeation testing results showed that if the permeation parameters did not change during the first exposure/decontamination cycle, they usually remained virtually unchanged for the next several cycles.[9] Similarly, the largest single loss for ultimate elongation occurred during the first exposure/decontamination cycle. These are good indicators as to whether the CPC can be reused. That is, if a piece of CPC can be effectively decontaminated after its first service, most likely it can be reused several more times using the same decontamination method. However, further studies are needed to determine if the largest loss in permeation and tensile properties occurs mainly after the first decontamination cycle for other material/chemical combinations.

SUMMARY AND CONCLUSIONS

This study investigated the changes in tensile strength and ultimate elongation of two commonly used glove materials after repeated acetone exposure and thermal decontamination.

1. For neoprene exposed to acetone, tensile strength and ultimate elongation consistently decreased with increasing exposure/decontamination cycles. The losses were less than 23% after four exposure/decontamination cycles. Both the heat and acetone significantly affected the tensile properties. The combination of the two factors had a more aversive effect on the changes in the tensile properties when compared with heat alone.
2. For nitrile exposed to acetone, tensile strength and ultimate elongation remained virtually unchanged after the first exposure/decontamination cycle. The average loss of tensile strength and ultimate elongation through the nine exposure/decontamination cycles was 5.3% and 14.7% respectively.
3. The biggest single cycle loss for the ultimate elongation was observed during the first exposure/decontamination cycle for both glove materials.
4. When compared with our permeation testing for these material/chemical combinations, we found that a correlation between the changes in permeation parameters and the changes in tensile properties after repeated exposure/decontamination cycles does not necessarily exist. Constant breakthrough time and steady-state permeation rate do not ensure constant tensile properties. Therefore, both permeation behavior and material degradation should be carefully investigated for determining the reusability of the CPC.

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