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Change in Permeation Parameters and the Decontamination Efficacy of Three Chemical Protective Gloves After Repeated Exposures to Solvents and Thermal Decontaminations

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Background *Chemical protective clothing (CPC) and gloves, which provide adequate protection, are usually too expensive to be considered disposable. Repeated use of CPC without effective decontamination may result in secondary exposure and injury. However, decontamination may change the physical and/or chemical properties of the barrier material, causing variations in breakthrough time (BT) and steady-state permeation rate (SSPR).*

Methods *Glove materials including neoprene, Guardian butyl rubber, and nitrile synthetic rubber were selected for this study. Toluene and acetone were chosen as the challenge chemicals. Permeation was measured in a closed loop system using a 2.5 cm permeation cell and a MIRAN infrared analyzer (Foxboro, MA). Following the permeation test, the samples were thermally decontaminated. After each exposure/decontamination cycle, BT and SSPR were measured. A total of 260 permeation tests were conducted. Permeation test results were collected on each material/chemical combination for up to 10 exposure/decontamination cycles.*

Results *On average, changes in BT and SSPR in comparison with respect to new swatches were 11.5% and 13.7% after seven exposure/decontamination cycles. The percentages increased to 26.6% and 15.9% after 10 exposure/decontamination cycles, respectively. For at least seven cycles, the BT mean for four out of five material/chemical combinations tested (neoprene/acetone, neoprene/toluene, nitrile/acetone, and nitrile/toluene) was not significantly different from the original value of the BT for each corresponding swatch. Similarly, the SSPR mean for each of the five material/chemical combinations after at least five cycles was not significantly different from those for new swatches. The BT mean for the butyl/toluene combination, however, was significantly different from the new swatches even after the first exposure/decontamination. The SSPR mean was significantly different after five cycles compared to the new swatches.*

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Conclusions *Except for the butyl/toluene combination, thermal decontamination was an effective method in removing the solvents from the matrix of selected glove materials. Multiple reuses of some chemical protective gloves could be safe if effective decontamination methods are used and the glove materials do not have significant degradation.* Am. J. Ind. Med. 47:131–143, 2005. *Published 2005 Wiley-Liss, Inc.*[†]

KEY WORDS: chemical protective gloves; decontamination; reusability; breakthrough time; steady-state permeation rate; toluene; acetone; skin; dermal exposure

INTRODUCTION

After the implementation of engineering and administrative controls, the use of chemical protective clothing (CPC) is the primary method to prevent skin exposure to chemical hazards in the workplace. The cost of illness attributed to skin exposure was estimated to be at least $1 billion per year, and more than $800 million of protective gloves are sold in the United States annually [Boeniger and Klingner, 2002]. However, most CPC for highly toxic exposures or routine exposures is too expensive to be considered disposable. The guidelines established by the US Environmental Protection Agency's Office of Emergency and Remedial Response require Level A personal protective equipment (PPE), such as the total-encapsulating suit, during an event of terrorist attacks in which unidentified hazardous materials and unknown concentrations are encountered. Such suits are very expensive and decontamination is, therefore, essential. Although the Occupational Safety & Health Administration (OSHA) has required decontamination of protective clothing under two regulations, i.e., the general personal protective equipment (PPE) standard—29 CFR 1910.132 and the Hazardous Waste Operation and Emergency Response standard—29 CFR 1910.120, it does not define how and when CPC should be cleaned, decontaminated, or retired to ensure their effectiveness [Carroll, 1995]. In order to protect American workers, who rely on CPC and gloves for protection against various chemical hazards in the workplace, adequate evaluation of CPC decontamination efficiency has become an important issue.

It is well known that permeation parameters are of critical importance in the selection and use of CPC and gloves. Generally, permeation parameters are used to indicate how long the glove is safe to wear. Furthermore, they are important indices in evaluating decontamination efficiency of protective clothing.

Currently, two approaches are used to evaluate decontamination efficiency of CPC, i.e., direct and indirect approaches [Perkins, 1991]. Using the direct approach, effectiveness is defined as the amount of residual chemicals in the matrix of the barrier material after decontamination divided by the amount of contaminant in the matrix prior to decontamination. The amount of residual chemicals can be determined by using a gravimetric method [Berardinelli and Hall, 1988], or by using a gas chromatograph [Perkins et al., 1987; Phalen and Que Hee, 2003]. In order to determine the amount of contaminant, one must perform extraction on the specimens. Using the indirect approach, the effectiveness is defined as the change in breakthrough time (BT) when permeation tests are carried out before and after the decontamination [Vahdat and Delaney, 1989]. As pointed out by Perkins [1991], the major advantage for the direct approach is that it allows the direct determination of the amount of contaminant remaining in the polymer after decontamination, which would provide useful information for risk assessments. However, the approach requires validating extraction efficiency, which differs with different solvent–polymer pairs, thus the process can be time consuming. In contrast, the indirect approach has some advantages over the direct approach because the test procedures are well established and much simpler. Although information on the amount of residual chemicals is not available, the indirect approach is usually more relevant in the workplace, as BT for any chemical is critical for the workers protection.

Permeation parameters mainly include BT and steady-state permeation rate (SSPR). BT is the interval between time a chemical first contacts one side of a barrier material and when it is first detected on the other side. Because the sensitivity of the analytical detection method used can significantly affect the measured BT, an alternative parameter, referred to as normalized breakthrough detection time (NBT), is sometimes employed. NBT is needed to compare results from one laboratory to another, or from one analytical instrument to another. The NBT is set at 0.1 $\mu g/cm^2$/min in the United States and 1 $\mu g/cm^2$/min in Europe, respectively. However, to compare breakthrough times using the same analyzer in a single laboratory, BT is frequently used and is the approach used in this study. SSPR is the permeation rate when the amount of permeant becomes constant with respect to time, i.e., a steady state is fully developed. For a closed loop system using an analytical instrument such as a MIRAN infrared analyzer, the increase of a permeant will become linear with time in the steady-state region assuming the diffusion behavior is governed by Fick's First Law. Although five different permeation behaviors have been identified, Fickian diffusion applies to most of the chemical-material combinations [Nelson et al., 1981]. SSPR is, therefore, calculated using the slope of the linear equation and the exposed area of the barrier material.

The current study evaluated the decontamination efficiency by using the indirect approach. Instead of NBT, BT was used for the purpose of comparison because the same MIRAN infrared analyzer was employed for all the permeation tests in our laboratory. In addition, SSPR were calculated to provide more information on material permeability. The objective of this study was to investigate the changes of permeation characteristics and thus the decontamination efficacy of three chemical protective glove materials after repeated cycles of acetone or toluene exposures followed by cycles of thermal decontamination.

MATERIALS AND METHODS

Glove Materials

Three commonly used glove materials were selected for this study, including neoprene (Stanzoil N-440), nitrile synthetic rubber (Ansell Edmont 37-155), and Guardian butyl rubber (STD 1-165). Swatches were carefully cut from the palm and the back of a glove. The material was oriented so that the outside surface was exposed to the solvents. Thicknesses of the glove materials were measured in five places for each swatch (12:00, 3:00, 6:00, 9:00, and center) using an Ames Micrometer with an accuracy of ±0.002 mm (Ames Instrument, Waltham, MA). Table I shows the mean thickness and the 95% confidence interval for the gloves. Relatively small variations were obtained because each glove material used for this study was taken from the same manufacturing lot.

Liquid Chemicals

Toluene (99.0%) and acetone (99.7%), both from Fisher Scientific (Houston, TX), were selected as challenge chemicals because both are recommended as liquid test chemicals by the American Society for Testing and Materials [1999]. Toluene was tested against all three glove materials, while acetone was used only for neoprene and nitrile synthetic rubber. BT for the acetone butyl combination has been estimated to be more than 480 min [Forsberg and Keith, 1999], therefore, test time for acetone exposure to butyl would be too long for the protocol and was eliminated in this study.

TABLE I. Thicknesses of Three Chemical Protective Glove Materials

Glove material	Thickness, mm (mean and 95% confidence interval)	Supplier
Neoprene (Stanzoil N-440)	0.758 (0.742, 0.774)	MAPA-Pioneer
Guardian butyl rubber (STD 1-165)	0.361 (0.353, 0.369)	LANX Fabric Systems
Nitrile synthetic rubber (37-155)	0.343 (0.338, 0.348)	Ansell Edmont

Permeation Test and Chemical Exposures

The Permeation test was carried out at room temperature (i.e., $23 \pm 0.5°\mathrm{C}$) with an AMK chemical permeation test cell (2.54 cm in diameter). The chemicals were added to the upper chamber of the AMK permeation cell and allowed to permeate through the glove material. The concentration for the permeant was determined using a MIRAN 1A gas analyzer in a closed loop system. Since a closed loop system has no outlet, the chemical was conserved and accumulated. As the permeant passes through the glove material it flows to the MIRAN 1A gas analyzer and then back to the glove material. The wavelengths were set at 8.5 μm for acetone and 13.9 μm for toluene. The path length was 20.25 m for both chemicals for a higher sensitivity. Chemical exposure to the material began once the chemical was added to the permeation cell and the timer started.

Data Acquisition

In addition to a chart recorder (Kipp & Zonen, Type BD112, Holland), a data acquisition system, referred to as the Permeation and Penetration Data Acquisition System (PPDAS), was coupled with the MIRAN 1A infrared analyzer to record the permeation behaviors for acetone exposures. As each experiment runs, PPDAS takes readings from the MIRAN 1A infrared analyzer at prescribed intervals, and displays the acquired data points in real-time. The readings were converted from voltage to concentration based on calibration curves. The instrument was recalibrated approximately weekly and it was found that its response did not significantly change during a period of 20 months. Figure 1a,b are typical calibration curves for acetone and toluene, respectively. The relationship between voltage and concentration was not linear so a quadratic model was applied. The quadratic model demonstrated good correlation for the data, as shown by the high values of correlation coefficients ($R^2 \geq 0.9991$). The voltage and concentration readings from the data acquisition system are saved to a database. As shown in Figure 2a,b, BT was determined as three standard deviations above the baseline in this study, and SSPR was calculated according to the following equation with units in μg/cm²/min:

$$\mathrm{SSPR} = \frac{\text{Slope of steady stage } (\mu\mathrm{g/min})}{\text{Exposed area } (\mathrm{cm}^2)} \qquad (1)$$

Decontamination of Glove Materials

After the permeation test, the permeation cell was disassembled and the swatch was air dried inside a fume hood

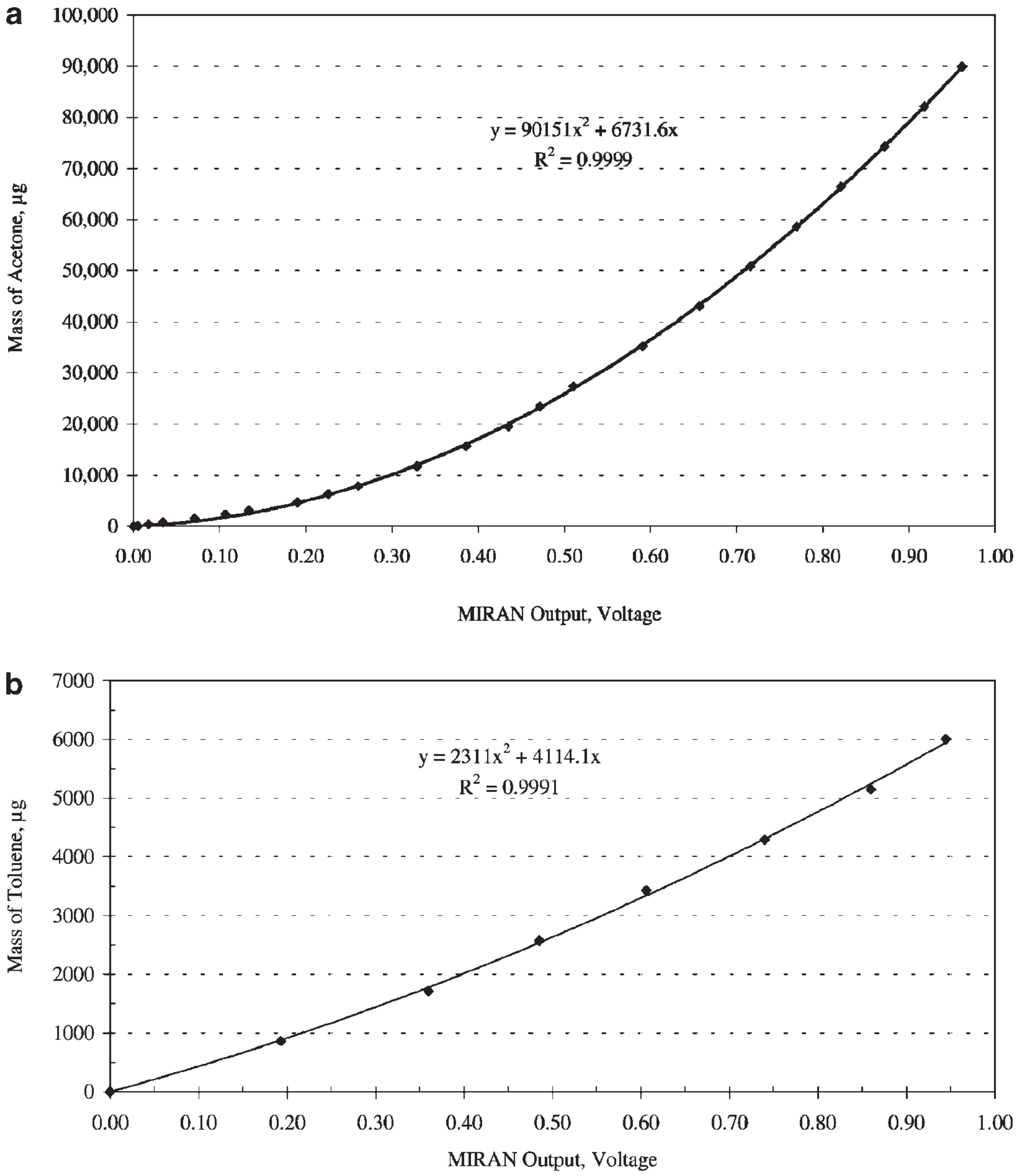

FIGURE 1. **a**: Typical calibration curve for acetone. **b**: Typical calibration curve for toluene.

for about 3 hr before placing the swatch into an incubator (Precision, Fisher Scientific). Based on a study by Vahdat and Delaney [1989], decontamination was achieved by heat extraction at 100°C for 16 hr using the incubator.

Repeated Exposure and Decontamination Cycles

In order to investigate permeation behaviors after repeated exposure/decontamination cycles fully, each swatch was exposed at least eight times and decontaminated at least seven times. BT and SSPR were measured after each exposure/decontamination cycle. The first exposure established a baseline for a new swatch and then subsequent exposures simulated the material being reused several times. A total of 260 permeation tests were conducted for the five materials/chemical combinations.

Statistics

One-way analysis of variance (ANOVA, S-plus 6 for Windows, Insightful Corporation, Seattle, WA) was used to determine if the exposure/decontamination cycles had an effect on breakthrough times and steady-state permeation rates. Multiple comparisons at a 95% confidence level using the Dunnett method [Dunnett, 1964] were also employed for the data analyses. Diagnostic residual plots were first made for each BT and SSPR means in order to determine if data

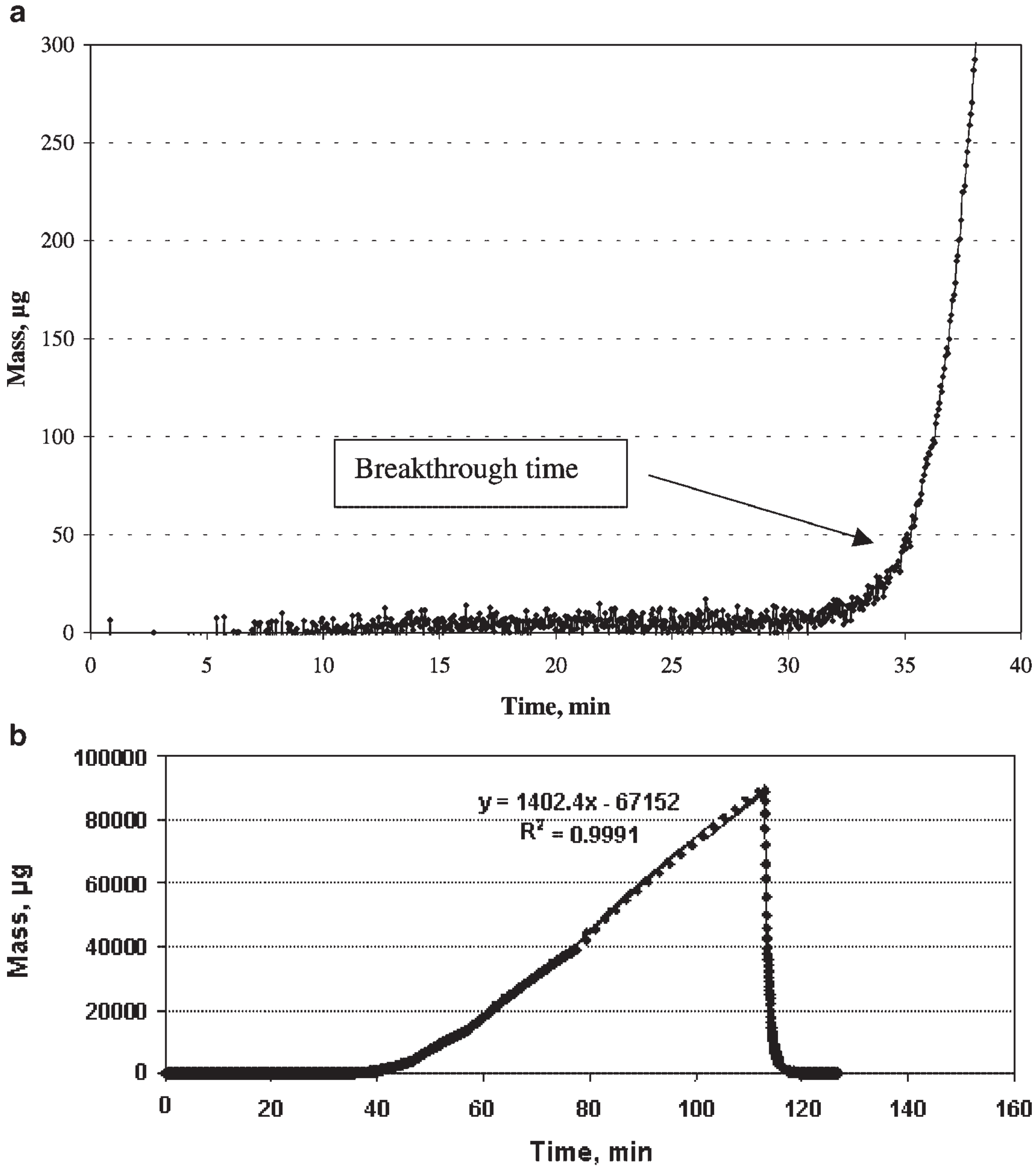

FIGURE 2. **a**: Determination of breakthrough time based on the permeation curve (neoprene vs. acetone). **b**: Typical steady-state permeation rate (SSPR) calculated using Equation 1. Here slope = 1402.4 µg/min, area = 5.07 cm^2, therefore SSPR = 276.6 µg/cm^2/min.

transformation was required. The plots indicated that the measurements exhibited no serious departure from normality.

RESULTS

Neoprene vs. Acetone

Figure 3a,b combine a total of 66 permeation tests, and illustrates the changes in BT and SSPR for the neoprene/acetone combination after repeated exposure/decontamination cycles. Mean breakthrough times were 34.0, 35.3, and 37.6 min for the new swatches, after 5 and 10 cycles, respectively. Mean steady-state permeation rates were 259.8, 279.6, and 287.1 µg/cm^2/min for the new swatches, after 5 and 10 cycles, respectively. The increase of either BT or SSPR was less than 11% after 10 exposure/decontamination cycles. Variations in the data for replicated samples were relatively small for both the BT and SSPR. The results were very comparable with other investigators, who reported a BT of 36 min and a SSPR of 295 µg/cm^2/min for new swatches [Forsberg and Keith, 1999]. Multiple comparisons by the

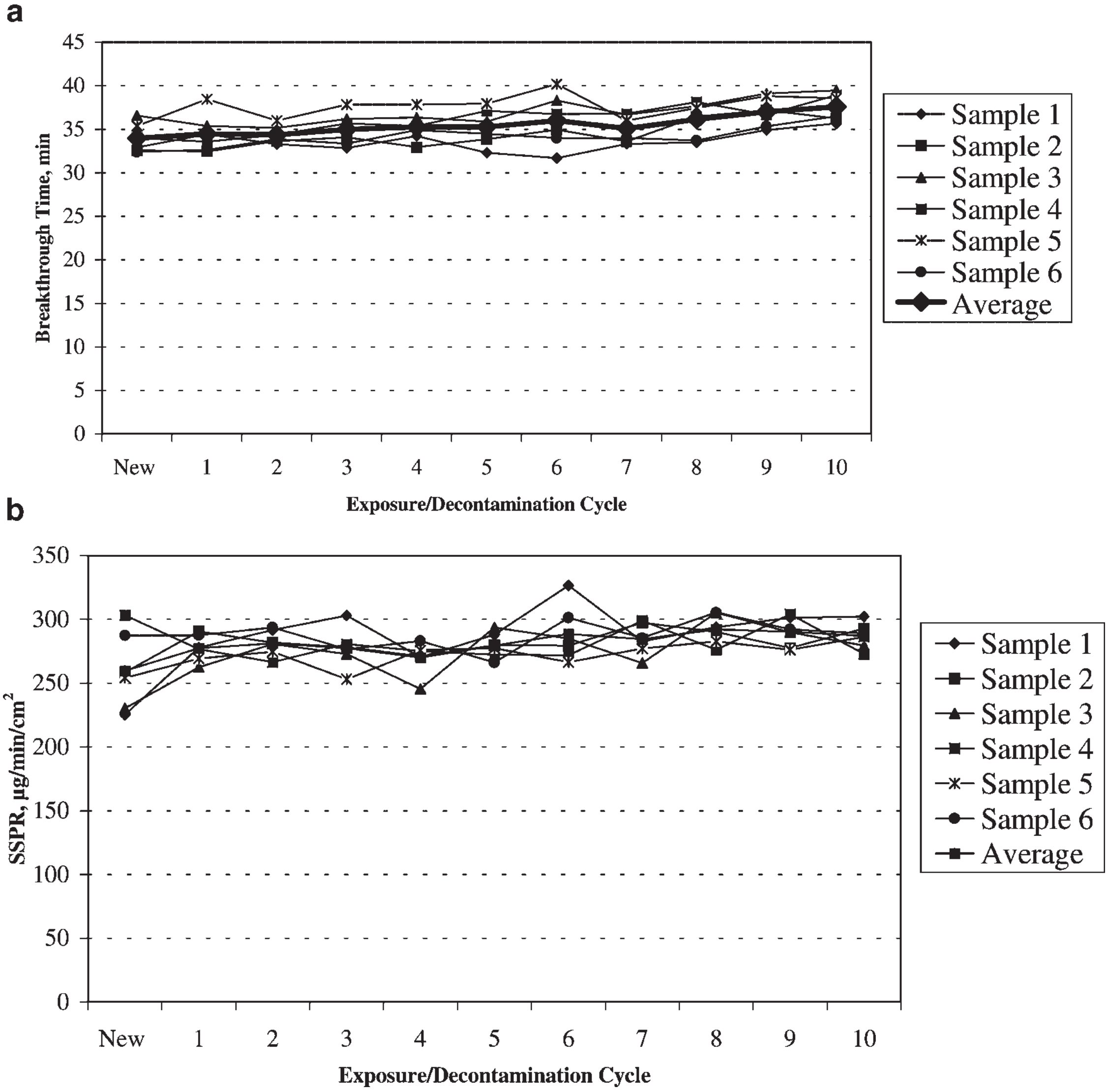

FIGURE 3. **a**: BT for the neoprene/acetone combination. **b**: SSPR for the neoprene/acetone combination.

Dunnett method indicated that the BT mean, after nine exposure/decontamination cycles, and the SSPR mean, after five exposure/decontamination cycles, were not significantly different from the new swatch.

Neoprene vs. Toluene

Figure 4a,b shows the changes in BT and SSPR for neoprene against toluene. The experiment had to be stopped after the seventh exposure/decontamination cycle because the O-ring for the permeation cell punched a hole in the swatch. In general, BT increased while SSPR decreased with increasing exposure/decontamination cycle. The mean breakthrough times were 19.9 min for the new swatches, 20.3 and 20.7 min for the swatches after four and seven exposure/decontamination cycles, respectively. The mean steady-state permeation rates were 395.4 µg/cm^2/min for the new swatches, 333.9 and 305.1 µg/cm^2/min for the swatches after four and seven exposure/decontamination cycles, respectively. Changes in BT and SSPR were less than 16% and 30% during the seven repeated cycles. The reproducibility of the BT test results was less consistent after the swatch had undergone several cycles. Changes in BT and SSPR were 1.1% and 25.3% after seven exposure/decontamination cycles. Multiple comparisons indicated that the BT, before seven cycles, and the SSPR, before six cycles, were not significantly different from those for the new swatches.

Nitrile Synthetic Rubber vs. Acetone

Figure 5a,b shows the changes in the BT and SSPR for nitrile synthetic rubber against acetone. The mean

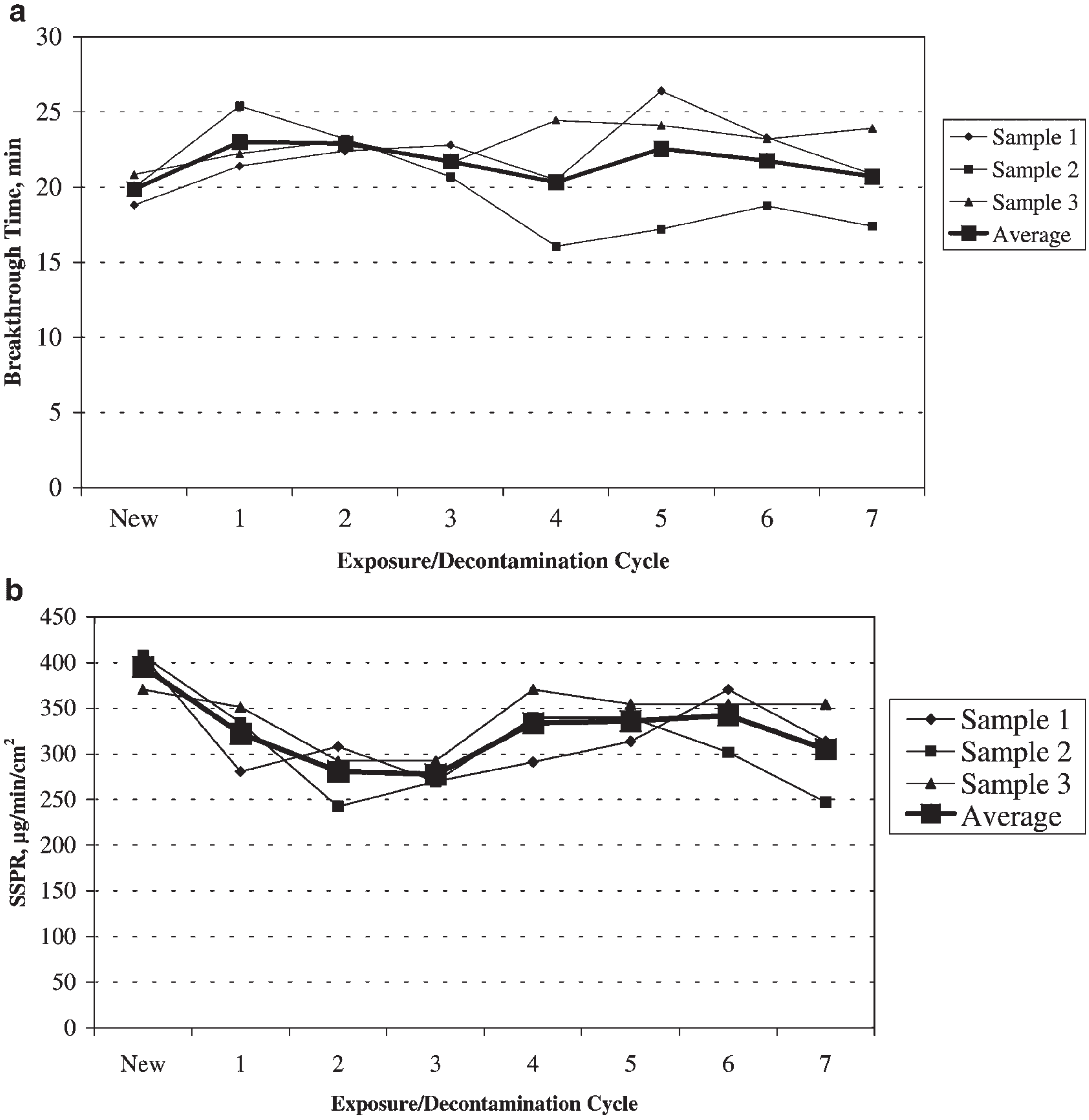

FIGURE 4. **a**: BT for the neoprene/toluene combination. **b**: SSPR for the neoprene/toluene combination.

breakthrough times changed less than 12%. The mean BTs were 5.5 min for the new swatches, 5.8 min for the swatches after 5 cycles, and 6.1 min after 10 exposure/decontamination cycles, respectively. The mean steady-state permeation rates were 2871.6, 2872.6, and 2702.9 µg/cm^2/min for the new swatches, after 5 cycles, and after 10 exposure/decontamination cycles. Note that following samples 10, 13, and 16 were used instead of samples 11, 12, 14, and 15 so that the material thickness would be similar. Selecting any of the four swatches (samples 11, 12, 14, or 15) would result in an increase in the variations for the BT and SSPR among the replicated measurements due to the increase in thicknesses. The measurements for the BT and SSPR were comparable with published literature, i.e., a BT of 5 min and a SSPR of 2000 µg/cm^2/min for new swatches [Forsberg and Keith, 1999]. Multiple comparisons revealed that the BT had not changed significantly until after 7 exposure/decontamination cycles and the SSPR had not changed significantly until after 10 cycles, respectively.

Nitrile Synthetic Rubber vs. Toluene

Figure 6a,b shows the changes in the BT and SSPR for nitrile synthetic rubber against toluene. As can be seen from Figure 6, the mean breakthrough times were 19.5 min for the new swatches, 20.2 and 21.3 min for the swatches after five and seven exposure/decontamination cycles, respectively. The mean steady-state permeation rates were 261.9 µg/cm^2/min for the new swatches and increased to 306.4 µg/cm^2/min for the swatches after seven exposure/decontamination

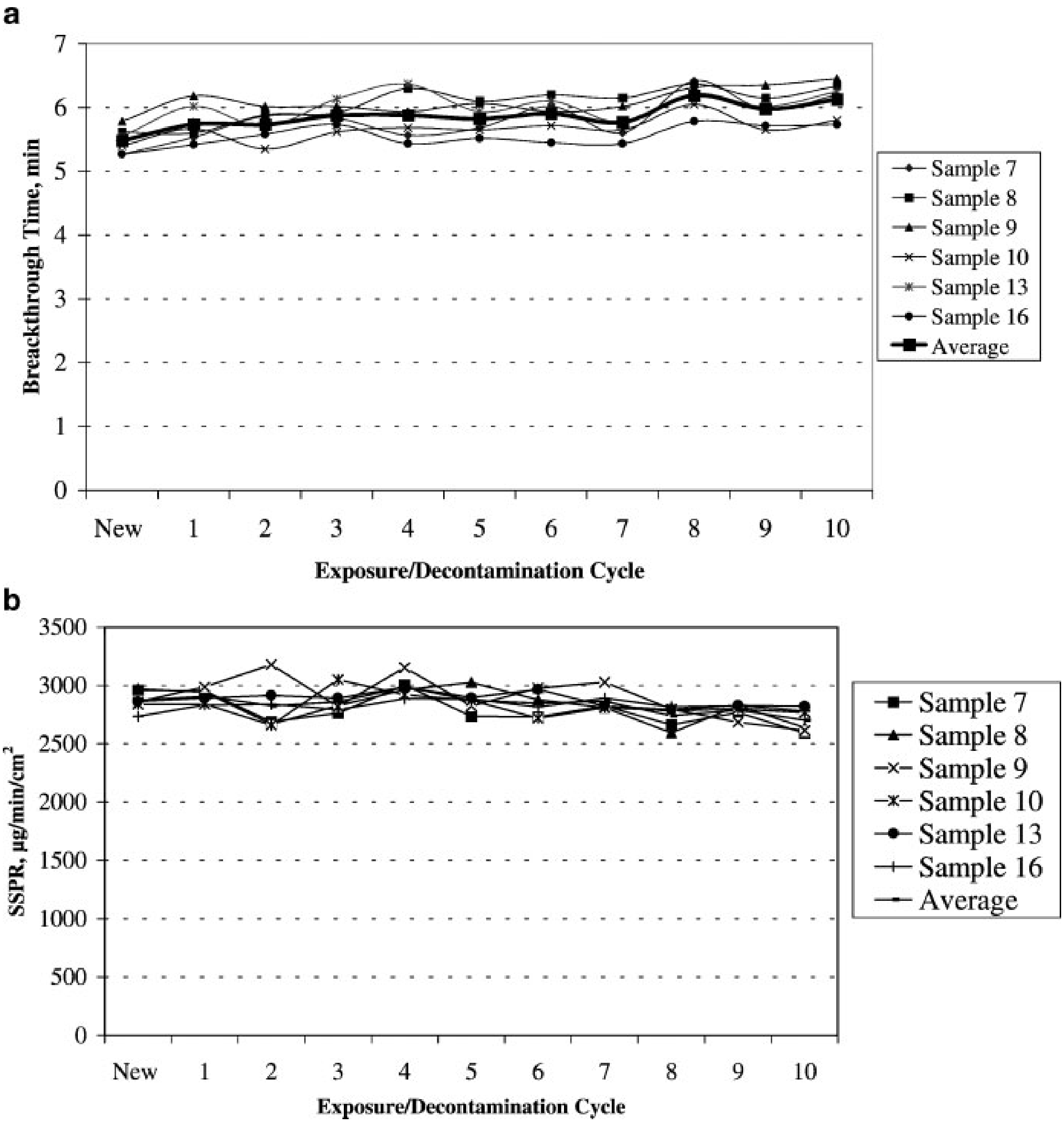

FIGURE 5. **a**: BT for the nitrile/acetone combination. **b**: SSPR for the nitrile/acetone combination.

cycles. An increase of SSPR indicates that toluene and/or heat adversely affects the glove material. BT and SSPR for sample 3 after its 2nd exposure/decontamination cycle were not obtained because the permeation cell broke during the testing. BT and SSPR increased 9.0% and 17.0% after seven exposure/decontamination cycles, respectively. However, multiple comparisons indicated that neither the BT nor SSPR had changed significantly at a 95% confidence level until after seven exposure/decontamination cycles.

Butyl vs. Toluene

As shown in Figure 7a,b, the mean breakthrough time for the butyl toluene combination was 5.8 min for the new swatches, and it consistently decreased to 2.5 min after 10 exposure/decontamination cycles, a decrease of 57.5%. The mean steady-state permeation rate was 790.8 µg/cm^2/min for the new swatches and increased to 1038.7 µg/cm^2/min for the swatches after 10 exposure/decontamination cycles, an increase of 31.3%. Multiple comparisons indicated that the BT consistently decreased with exposure/decontamination cycle and that the differences were significant. Although the SSPR before five exposure/decontamination cycles was not significantly different from those for the new swatch, there was a trend showing an increase in SSPR values; indicating that toluene and/or heat adversely affects butyl material.

Table II summarizes the statistical significance of changes in the permeation parameters due to repeated

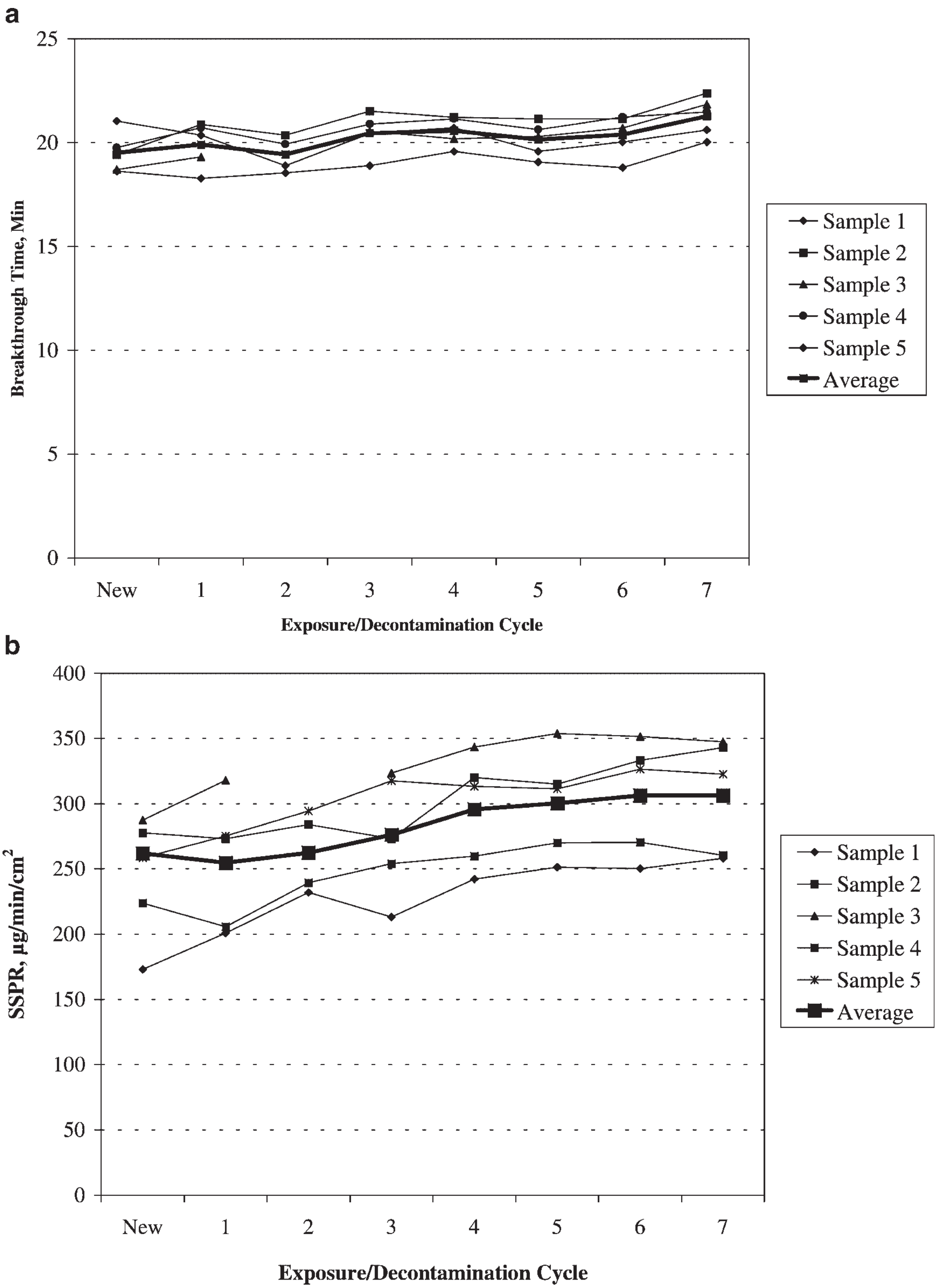

FIGURE 6. **a**: BT for the nitrile/toluene combination. **b**: SSPR for the nitrile/toluene combination.

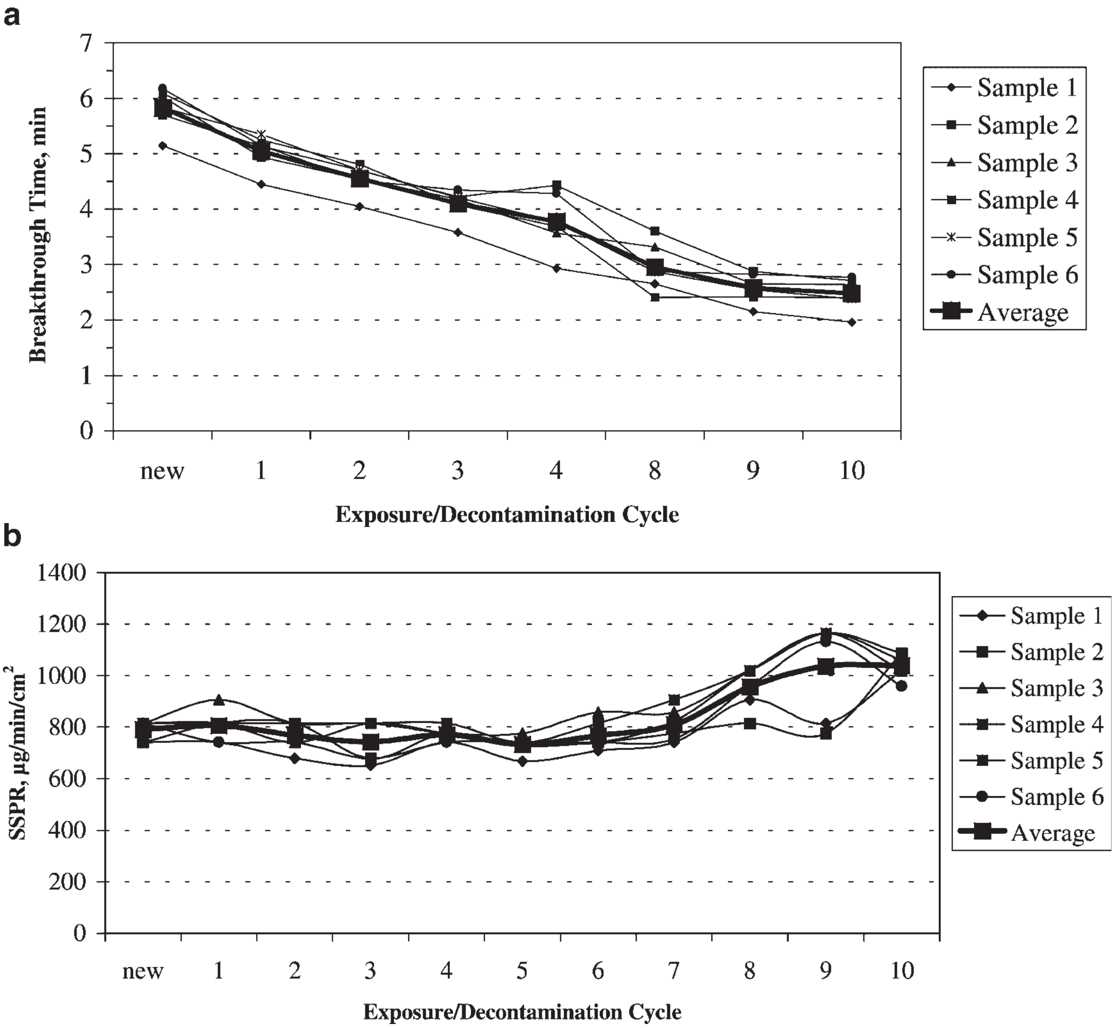

FIGURE 7. **a**: BT for the butyl/toluene combination. **b**: SSPR for the butyl/toluene combination.

exposure/decontamination cycles for all of the tested material/chemical combinations. Figure 8a,b illustrates the changes in percentage with respect to new swatches for BT and SSPR, respectively. It can be seen that the changes in permeation parameters were within 15% for most of the cases. On average, BT did not exceed 13% and SSPR did not exceed 11% after seven exposure/decontamination cycles for all the tested material/chemical combinations. The changes did not exceed 27% for BT and 16% for SSPR after 10 exposure/decontamination cycles. To represent a worst-case, the average changes in percentages were calculated by using the absolute values.

TABLE II. Significance of Changes in Permeation Parameters of Three Chemical Protective Glove Materials at 95% Simultaneous Confidence Intervals

	NEOPRENE		NITRILE		BUTYL	
	BT	SSPR	BT	SSPR	BT	SSPR
Acetone	9[a]	5	7	10	Not tested	
Toluene	7	6	7	7	1[b]	5

[a] No significant difference before nine exposure/decontamination cycles.
[b] Significant difference for all exposure/decontamination cycles.

DISCUSSION

Chemical protective clothing has been marketed as disposable (use one time and discard), limited-reusable (use several times prior to disposal), or reusable (multiple uses, decontaminations, repairs, and reuses prior to disposal). As stated earlier, CPC for highly toxic exposures, routine exposures, or emergency response are reusable items; therefore, information about glove material performance following decontamination is needed. Over the past decades, researchers and industrial hygienists attempted to determine

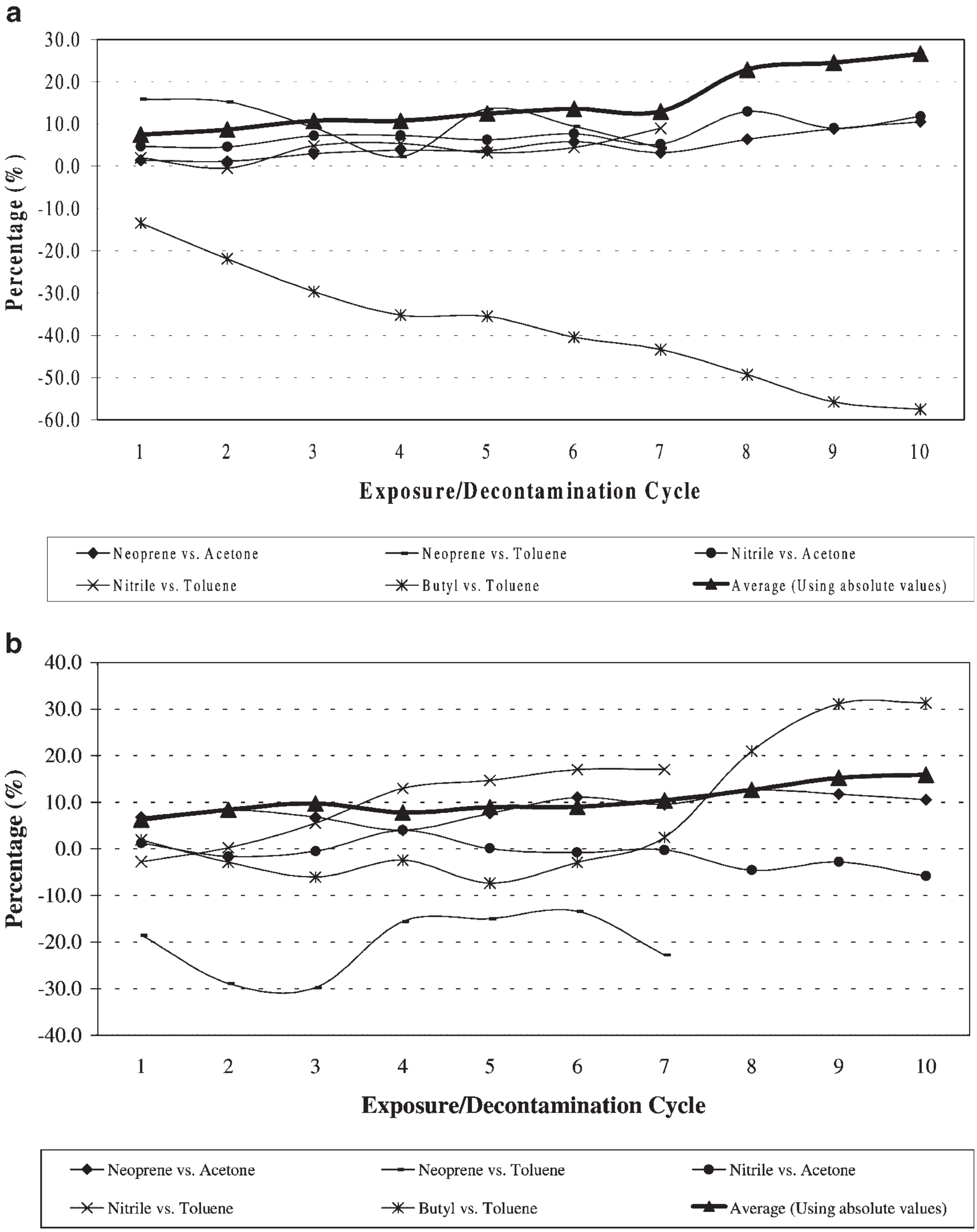

FIGURE 8. **a**: Change in BT with respect to new swatches for all the material-chemical combinations. **b**: Change in SSPR with respect to new swatches for all the material-chemical combinations.

how best to remove low volatility chemicals from the matrix of protective clothing materials so that the CPC could be reused safely. Several methods have been commonly used for CPC matrix decontamination, e.g., heated air, hot water, cleaning agents, agitation techniques, and liquid laundry detergent with water. Volatile chemicals, such as organic solvents, can be decontaminated effectively by using thermal energies, including heated air, heated water, and other means of thermal extraction [Garland and Torrence, 1987; Perkins et al., 1987; Vahdat and Delaney, 1989; Perkins, 1991; Mansdorf, 1992]. Decontamination efficacy for one exposure/decontamination cycle is available from the published literature, but hardly any information is available for more than one cycle.

Our study is the first report on decontamination efficacy for several exposure/decontamination cycles. Continually observing the change in the permeation parameters provides valuable information for the end-users when considering reusing CPC. While this study evaluates the change in permeation parameters to ensure that the glove materials function correctly after repeated exposure/decontamination cycles, we have also investigated the change in tensile property to make sure that the materials do not fail as well. As shown by Gao et al. [2004], after repeated exposures to acetone and thermal decontaminations, BT did not significantly change in up to nine cycles for neoprene and seven cycles for nitrile synthetic rubber; the tensile properties were reduced up to 20% after seven and four cycles, respectively. Thus, investigation of the change in permeation parameters was not conducted further, as reusability of CPC can not be based on permeation parameters alone, but must also take into account the change in physical properties.

The significant finding of this study, as shown in Figures 3–6, is that, if the permeation parameters remain virtually unchanged after the first exposure/decontamination cycle, they usually did not change for the following several repeated cycles as well. Furthermore, if BT did not change appreciably, SSPR would stay constant, and vice versa. One possible explanation is that the thermal decontamination at the selected temperature and duration was able to successfully remove the residual chemicals from the matrix and did not change the properties of the barrier materials noticeably. However, as shown in Figure 4a, the reproducibility of the test results was less consistent after the swatch had undergone several cycles. This may indicate that homogeneity in chemical resistance among the tested swatches declined after some exposure/decontamination cycles as a result of different magnitudes in either chemical or thermal degradation. In addition, there was a tendency for BT to slightly increase with increasing exposure/decontamination cycle for some material chemical combinations, as indicated in Figures 3a, 5a, and 6a. This may be that the barrier materials were not cured correctly, as it is a heat/time phenomenon. Therefore, a little heat may cause more cure and crosslinking of the materials, becoming more resistant to permeation. Similar phenomenon was reported by Berardinelli and Hall [1988] when using solvent to decontaminate *n*-butyl acetate from the matrix of neoprene gloves. In contrast, as shown in Figure 7 for the butyl/toluene combination, the BT consistently decreased after each exposure/decontamination cycle. The SSPR slightly decreased during the first four cycles and consistently increased afterwards. This phenomenon might indicate that there was swelling of the barrier materials due to the heat during the first four cycles. The following increase in SSPR could be associated with the plasticizers that were added to the polymer being either dissolved by the toluene, or vaporized by heating. In general, when BT decreases and SSPR increases, the challenge chemical or decontamination method adversely affected the CPC materials; thus weakening its resistance to permeation. As addressed earlier, change in tensile properties after repeated exposure/decontamination cycles, including tensile strength and ultimate elongation, has been investigated in our laboratory to test this possibility.

Vahdat and Delaney [1989] conducted thermal decontamination for several glove materials against different organic solvents. They found that heat at 100°C for 16 hr provided the best result when compared to various temperatures including 70, 125, and 150°C for neoprene, nitrile, butyl, and PVC. Although the red inner liner of the neoprene gloves turned slightly darker after thermal extraction in our study (this can be attributed to slight degradation in the material's components), we confirmed that the selected temperature and duration for thermally decontaminating most of the glove/chemical combinations in our study were appropriate. Thermal decontamination has several advantages over other methods, including hot water, cleaning agents, agitation techniques, and liquid laundry detergent with water, because no water or waste solvent is involved. It also does not introduce any solvents as new contaminants to the barrier materials and the processing is environmentally compatible. However, the main disadvantage of thermal decontamination is that heat increases material degradation; due to (a) volatilization of plasticizers, (b) increase in pore size, or (c) hardening effects on the matrix, or (d) a combination of all these effects. Such degradation would happen even at room temperature over time [Goydan et al., 1990]. Change in material property based on tensile strength and elongation for these gloves after repeated exposure and thermal decontamination cycles has also been investigated and the results will be published separately.

In conclusion, this study demonstrates that multiple reuses of some chemical protective gloves could be safe if effective decontamination methods are used. The relatively small changes in permeation parameters are encouraging for the reusability of CPC and gloves. Cost of thermal decontamination is insignificant in comparison to the replacement costs of new gloves. For example, neoprene gloves cost nearly $15 a pair and Viton gloves can cost over $100, while some garments can cost more than $3,500. Although this study utilized swatches taken from protective gloves, the results can be generalized to suits and other pieces of protective clothing, because they are often constructed from the same base materials. For other chemical/material combinations, repeated exposure/thermal decontamination may significantly change the material's permeation characteristics, resulting in skin injury. Our future studies will focus on more effective decontamination techniques for various CPC material-chemical combinations and computer modeling to predict decontamination and degradation effects.

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