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Original Article

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Evaluation of disposable protective garments against epoxy resin permeation and penetration from anti-corrosion coatings

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Abstract

Introduction: Epoxy-based resin formulations are a frequent cause of allergic and irritant contact dermatitis in the construction and painting industries. Cases of epoxy resin contact dermatitis continue to persist across many sectors and are likely attributable to the growing use of epoxy products, including epoxy-based anti-corrosion coatings and inadequate skin protection. There are no published performance data against epoxy resins for garment materials and gloves to guide proper material selection in the workplace.

Objectives: The objective of this study was to evaluate the resistance of 5 protective garment materials against permeation and penetration by bisphenol A diglycidyl ether and its higher oligomers found commonly in epoxy-based anti-corrosion coatings.

Methods: Five disposable garment materials were evaluated for resistance to bisphenol A diglycidyl ether monomers and oligomers during contact with epoxy-based anti-corrosion coatings, including latex gloves, nitrile gloves, Tyvek coveralls, polypropylene/polyethylene (PP/PE) coveralls, and a cotton T-shirt. A permeation test cell system was used to evaluate each garment material against an epoxy-based zinc-rich primer and an epoxy-based intermediate coating using a realistic application method. Glass fiber filters were used to collect permeating and penetrating epoxy resin during a 120-min test period. Bisphenol A diglycidyl ether quantification was performed with high-performance liquid chromatography–electrospray ionization-tandem mass spectrometry. Paint loading, coating thickness, and homogeneity were assessed on polytetrafluoroethylene filters sprayed in series in permeation test cells.

Results: Latex gloves provided the least resistance to permeation by BADGE in coating formulations, with a maximum cumulative permeation over the 2-h test interval of 21.7 ng cm^{-2} with the primer and 513.8 ng cm^{-2} with the intermediate coating product. Nitrile gloves were not permeated by either coating formulation. The Tyvek coveralls provided greater protection as compared to the PP/PE coveralls. The cotton T-shirt was penetrated by bisphenol A diglycidyl ether more frequently than any of the tested garment materials and resulted in a maximum cumulative penetration of 128 ng cm^{-2} with the primer and 28.0 ng cm^{-2} with the intermediate coating.

Conclusion: Although all the garment materials evaluated during this study provided sufficient protection to prevent cumulative permeation in excess of the established acceptable permeation thresholds, the use of nitrile gloves and Tyvek coverall is highly recommended to minimize skin exposure to bisphenol A diglycidyl ether. We recommend cotton T-shirts to be used under Tyvek coveralls as a secondary layer of skin protection and for added comfort, but not as a primary protection layer.

Key words: allergic contact dermatitis; BADGE; construction; epoxy coatings; penetration; permeation; skin protective clothing.

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What's Important About This Paper?

This study used a realistic laboratory system to evaluate permeation and penetration of garment and glove materials by bisphenol A diglycidyl ether. Tyvek(R) coveralls and nitrile gloves provided the greatest level of protection. Findings from this study can be used to assist with the selection of personal protective equipment to prevent skin exposure to epoxy resins.

Introduction

Epoxy resins are one of the most common causes of allergic contact dermatitis (ACD) in the workplace and have frequently been associated with the skin sensitization and skin irritation of workers in the construction and painting industries (Geier et al. 2004; Aalto Korte et al. 2014; Aalto-Korte et al. 2015; Pesonen et al. 2015; Spee et al. 2016). Despite the known sensitizing capacity of epoxy resins, cases of epoxy-related contact allergies continue to increase (Spee et al. 2016; van der Molen et al. 2016). These increases are likely attributable to the growing use of epoxy resins in the construction industry, frequent contact of epoxy paints with unprotected skin, and the incorrect selection and use of skin protective clothing (Geier et al. 2011; Spee et al. 2016).

Epoxies are reactive chemicals containing 2 or more oxirane groups, or epoxide groups, which can enter polymerization reactions with a polyamide or amine curing agent leading to resin curing (Knudsen and Forsgren 2017). The majority of commercial epoxy resins (75–95%) are mixtures based on bisphenol A diglycidyl ether (BADGE) and its respective higher oligomers (dimer, trimer, and tetramer) formed from the condensation of 2 or more monomeric units, while ~7% are based on bisphenol F diglycidyl ether (BFDGE) monomer (Niklasson et al. 2009; Aalto Korte et al. 2014; Hagvall et al. 2016). Skin contact with other epoxy resin system components, including several amine hardeners and reactive diluents, such as 1,6-hexanediol diglycidyl ether or 1,4-butanediol diglycidyl ether, has been associated with irritant contact dermatitis, whereas inhalation exposure can cause severe respiratory disorders such as occupational asthma (Kanerva et al. 2013; Aalto Korte et al. 2014; Aalto-Korte et al. 2015). To the best of our knowledge, there are currently no published occupational exposure limits for airborne exposure to epoxy resins or any skin notations.

Epoxy-based anti-corrosion coatings are frequently used in the construction industry to protect steel structures because of their strong mechanical properties, excellent adhesion, and high resistance to chemicals and water (Knudsen and Forsgren 2017). Three-coat paint systems are the generally accepted approach used to protect bridge steel in areas of moderate to severe corrosion (DOT 2015). These systems typically begin with an epoxy-based zinc-rich primer and intermediate coating, followed by an aliphatic polyurethane top-coating to prevent ultraviolet degradation (i.e. chalking) of the underlying epoxy layers. Epoxy coatings are often applied to large structures using airless spray systems for productivity (DOT 2015). These systems aerosolize and disperse the coating using a high-velocity low-pressure (HVLP) spray gun. Manual application using a brush or roller is also used for smaller areas. In 2020 there were approximately 365 300 construction and maintenance painters employed in the United States, a number that is predicted to grow by 5% in the next decade (Bureau of Labor Statistics 2022; CPWR 2022b).

Strong evidence exists to support the use of effective skin protection to minimize exposure to epoxy-based coatings and the other chemicals found in epoxy resin systems. Geier et al. (2004) identified a significantly lower incidence of hand dermatitis among patch test participants wearing protective gloves compared to those who do not wear gloves (i.e. 58% versus 91%), 38% of which were working in the construction trades (Geier et al. 2004). Additionally, Spee et al. (2016) found a positive association between cases of epoxy-related contact allergies and the use of ineffective glove materials, including leather, cotton, and latex (Spee et al. 2016). For clarity, a material breaks through a protective garment (e.g. latex glove) via 2 mechanisms, permeation and penetration. Permeation is the diffusion of chemicals through the intact material fabric. Depending on the type of garment, either one or both mechanisms may take place. Penetration refers to chemical transfer through material imperfections or openings in the material fabric (pores, holes, seams, etc.) (ASTM 2012).

There have been only a limited number of studies evaluating the efficacy of protective clothing materials against epoxy resins. Forsberg et al. (2014) found that natural rubber latex and neoprene rubber gloves provided poor resistance to pure BADGE (Forsberg et al. 2014). Butyl rubber and nitrile rubber gloves provided a greater level of protection with moderate resistance to BADGE and a breakthrough time between 1 and 4 h. In a more comprehensive study, Henriks-Eckerman et al. (2015) documented notable variability in the epoxy penetration between 2 epoxy resin formulations and various disposable glove types (rubber, latex) and clothing materials (Tyvek and cotton), despite their similar concentrations of BADGE. These results demonstrate the effects of chemical composition on facilitating material penetration and highlight the need to test each material and chemical mixture independently. Furthermore, the manner of product application such as product spraying, which deposits microscopic particles onto the fabric instead of a liquid film, the presence of nano/microparticulate additives in primer and, and different solvent formulations, likely impact the penetration rate and should be evaluated using realistic application scenarios.

Although some protective clothing manufacturers conduct chemical resistance testing to evaluate their products against select chemicals, these tests are typically limited to pure chemical substances and often do not include epoxy-based product mixtures. Additionally, the standard permeation test methods used by manufacturers [e.g. American Society for Testing and Materials (ASTM) method F-739 (2012) and International Organization for Standardization (ISO) Standard 6529 (2013)], are not suitable for polymerizing materials, since they will harden inside of the test apparatus. This limitation prevents the evaluation of polymerizing chemical mixtures in their applied state, which is necessary to evaluate the effects of the product matrix on protective clothing performance. To overcome the issues with standard permeation test methods, Ceballos et al. (2011) developed a permeation test cell system to evaluate the resistance of skin protective clothing to spray-applied polymerizing materials (Ceballos et al. 2011). This system was originally used to evaluate isocyanate permeation from automotive clear coats (Ceballos et al. 2011, 2014). In our recently published work, we utilized this system to evaluate disposable protective clothing materials against the isocyanates used in spray polyurethane foam (SPF) insulation (Mellette et al. 2018) and polyurethane anti-corrosion top coatings (Mellette et al. 2019). The effectiveness of protective gloves and coverall materials against epoxy resins used in coating and painting metal structures sites has not been documented.

In this paper, we expand upon our previous research to focus on spray-applied epoxy resin systems used as protective coatings of metal structures (Mellette et al. 2019; Bello et al. 2021; Xue et al. 2021). We tested the resistance of 5 protective clothing materials against 2 epoxy-based anti-corrosion coatings commonly used to provide corrosion protection for bridges and other metal structures (Bello et al. 2021). The main objective was to assess permeation and penetration of complex epoxy-based coatings through commonly used protective gloves and clothing using a realistic spray application technique as well as investigate the epoxy resin breakthrough in these materials over multiple time points during a 2-h period.

Methods

Glove and clothing material selection

Testing materials were selected based on field observations and discussions with industrial painters partnering in a larger investigation of reactive chemical exposures in construction (CPWR 2022a). The focus was placed on lower-cost and readily available gloves and whole-body protective clothing representative of those used in metal structure coating applications as documented by field investigations (Bello et al 2021).

Two types of *gloves* selected consisted of (i) disposable thin nitrile gloves which were most frequently observed in the field; and (ii) disposable latex gloves used less frequently at the study sites. Glove selection was also supported by results of a survey identifying the preference for lower-cost and thinner gloves due to their comfort and dexterity in a similar industry (Ceballos et al. 2014). *Coverall clothing* materials used in the field ranged from disposable hooded coveralls to typical regular clothing. Based on these observations, disposable Tyvek coveralls and polypropylene/polyethylene (PP/PE) blend coveralls were selected for testing. Both coveralls were readily available from brick-and-mortar retailers (such as the Home Depot in the United States) and recommended for painting applications. In addition, a “heavy weight” cotton T-shirt (145 g m^{-2} [6.1 oz/yd^2]) was also tested to evaluate the effectiveness of regular clothing as a protective barrier. This selection was informed by field observations indicating that on hot days painters would simply wear a cotton T-shift during painting or wear it under the PE/PP coverall. The description of each test material and their measured thicknesses are presented in Table 1. Each breathable test material was examined using a JEOL 7401F field-emission scanning electron microscope (FE-SEM) at magnification levels ranging from 55× to 10 000×. Images showing their physical structure, fiber matrix, and pores, when present, at 2000× magnification are shown in Figure 1.

Epoxy-based coating products evaluated

Two coating product types consisting of a zinc-rich epoxy primer and a fast-curing epoxy intermediate coating commonly used on steel bridges in the Northeast United States were selected as the test products. Both were part of the list approved by the Northeast Protective Coating Committee (NEPCOAT), an affiliation of Northeast states that establishes acceptance/testing criteria for highway bridge protective coating systems (NEPCOAT 2022).

Product 1*: The zinc-rich epoxy primer* is composed of 3 separately packaged components that are mixed immediately prior to product spraying. This 3-component reactive chemical system consists of Part A “paint” containing 39.6% xylene, 16.6% phenols, 11.5% polyamide, and 7% ethyl benzene; Part B “hardener” containing 39.7% epoxy polymer, 22.3% methyl ethyl ketone (MEK), 16.7% methyl n-amyl ketone, and 11.6% xylene; and Part F “zinc-dust” containing a 100% zinc additive for cathodic corrosion protection. As shown in Figure 1, the zinc dust particles appeared mostly spherical with a polydispersed range of particle sizes from 250 nm to 10 µm in diameter.

A small batch of the primer (~340 mL) was prepared for laboratory use at the ratios specified by the manufacturer (100 mL—Part A: 100 mL—Part B: 877 g—Part F) and allowed to react (“sweat-in”) for 30 min to fully compatibilized the mixture. MEK, 10 mL, was added to each batch to reduce coating viscosity for spray application. The gray/green primer color provided adequate contrast to visualize paint penetration on the white collection media.

Product 2*: The intermediate coating* was composed of 2 separately packaged components, consisting of Part A “paint” containing 27.7% titanium dioxide, 8.5% talc, 20.6% 4-chlorobenzotrifluoride, 20.5% isobutylenated methylstyrenated phenol, and 9% polyamide; and Part “B” hardener contained 50% to 75% crystalline silica, 10% to 25% epoxy polymer, ≤9.6% acetone, and ≤5% methyl isobutyl ketone (MIBK).

The intermediate coating was mixed in small batches of 200 mL at a 50:50 ratio, per the manufacturer’s recommendation. The mixture was

Table 1. Protective clothing test materials and measured thicknesses.

Material type	Manufacturer	Product #	Thickness (mm [mils])[a,b]
Disposable gloves			
Latex	HDX	432202	0.068 [2.7]
Nitrile	HDX	953849	0.073 [2.9]
Clothing			
Tyvek Coverall	Trimaco	14113	0.105 [4.1]
PE/PP[c] Coverall	3M	4540+	0.105 [4.1]
Cotton Shirt	Champion	T425	0.317 [12.5]

[a] Average thickness of all specimens measured with a dial caliper.
[b] 1 mil = 0.001 in.
[c] Polypropylene and polyethylene (PE/PP) laminate film.

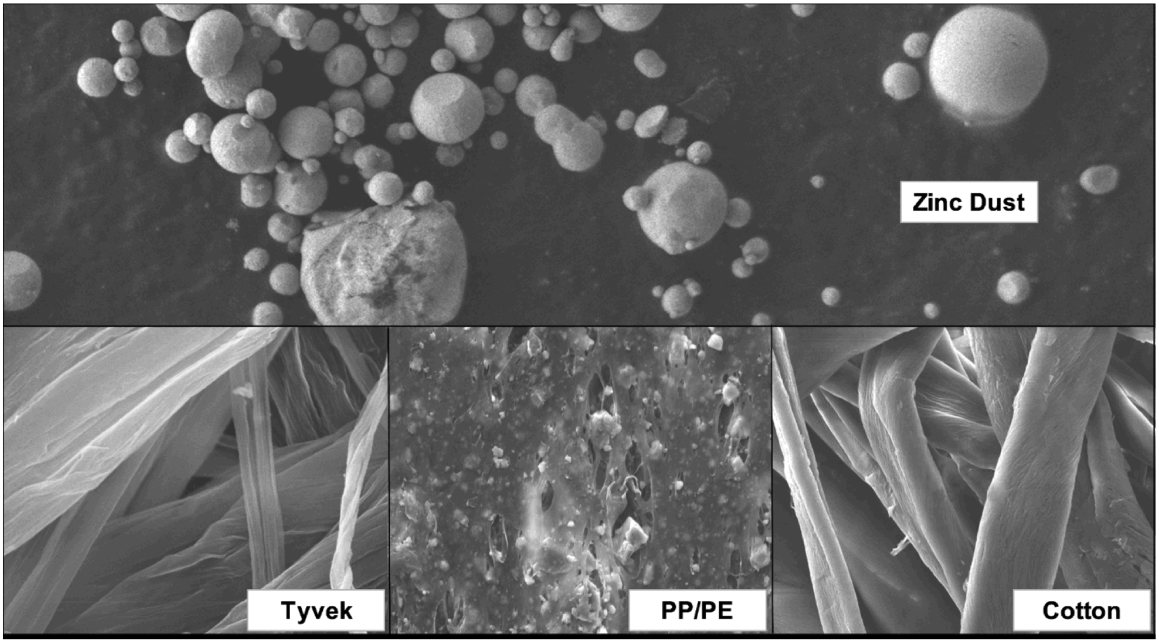

Figure 1. (Top) Photograph of zinc dust sample in the primer formulation examined by FE-SEM shown at 2000× magnification. Zinc dust particles appeared mostly spherical with a polydispersed range of particle sizes from 250 nm to 10 µm in diameter. (Bottom) FE-SEM analysis of each breathable clothing specimen at 2000× magnification. The Tyvek material is a flash-spun high-density polyethylene (PE) fiber. The PE/PP material microporous polypropylene and polyethylene laminate. The woven cotton T-shirt has a fabric weight of 145 g m^{-2} (6.1 oz yd^{-2}).

allowed to react (or "sweat-in") for 30 min, after which a compatible reducer (~20 mL) containing 60% 4-chlorobenzotrifluoride and 40% acetone was added. The intermediate coating was colored red with a compatible tint dye provided by the paint manufacturer (Real Red; SW 6868) to provide a visual indication of material penetration. Additional information such as viscosity and surface cure time of the coating products tested are provided in Table 2.

Penetration testing

Qualitatively we investigated if penetration happened by visual inspection of the color of the products selected on the opposite side of the membrane. During testing, a visual indicator (i.e. paint dye) was used to identify high levels of material penetration against the white collection media during sample collection. Besides color visualization, we also compared the ratios of BADGE monomers to higher molecular weight BADGE oligomers (e.g. trimer and tetramer) in the collected samples to those in the bulk material to discriminate permeation from penetration. This was done by comparing these ratios in each test cell sample to the ratios in direct loading samples (see "experimental design" and "epoxy mass loading" sections). The rationale for this comparison is that in the case of penetration, where fast transfer of the mixture occurs through openings on the fabric, the relative content of the BADGE monomer and dimer in the mixture will be reasonably preserved. In the case of permeation, which is a diffusion-driven process favoring the smaller and more mobile molecules passing through the membrane, the monomer content on the collected sampling media will likely be higher than that of the dimer (i.e. their ratio will be higher than in the product) and increase with time.

Experimental design

The permeation test cell system and spray application methods used in this study are described in more detail in Mellette et al. (2018, 2019) and the main setup is illustrated in Figure 2. Briefly, the coating products were sprayed over the 15 test cells of the panel equipped with the 5 protective clothing materials described above. Each test cell was mounted individually on a 15.24 × 15.24 cm single-walled corrugated cardboard panel to facilitate the efficient retrieval of the epoxy sample collection media and simplify the clean-up (ScotchBlue 2090; 3M Company, St Paul, MN, USA) to protect it from overspray.

Fifteen panel cells were loaded for each coating product and protective clothing material combination. Epoxy samples were collected in each cell at predetermined times of 5, 15, 30, 60, and 120 min, with 3 replicate samples analyzed for each time point. A total of 160 samples were collected and analyzed for epoxy resins, consisting of 150 permeation test cells (2 products × 5 test materials × 5 time points × 3 replicates/test/time point) and 10 field blanks.

Coating products were sprayed with an HVLP gravity-fed spray gun and 2-stage 5.0 psi turbine (Fuji 2203G; Fuji Industrial Spray Equipment Ltd, Toronto, Ontario, Canada) with a 1.8-mm nozzle, mounted 20 cm from the point of application in a ventilated fume hood. The product was consistently applied to each material by maintaining a constant spray interval

Table 2. Characteristics of epoxy-based coatings tested as part of this study.

Coating type	Epoxy resin[a] (% weight)	Solids[b] (% weight)	Viscosity[c] (cm/s^2)	Surface cure time[b] (min)
a) Characteristics of the tested paint formulations				
Zinc-rich primer	40	90	2.98	30
Intermediate	25	83	1.67	120

Chemical composition[d] (% w/w)	BADGE monomer	Dimer	Trimer	Tetramer	Total TEG (LC-MS/MS)[e]	TEG by IC[f]
b) Chemical composition of the epoxy prepolymer component of the paint.						
Zinc-rich primer	18.9	3.5	9.3	10.2	7.0	8.7
Intermediate	19.3	5.9	1.4	0.6	4.7	5.2

[a] Derived from manufacturer-reported product data and the mass of individual coating components.
[b] As reported by the manufacturer.
[c] Complete mixture (resin, hardener, solids, reducer) measured after sweat-in with #4 Ford viscosity cup.
[d] Chemical composition of the epoxy component measured by LC–ESI–MS/MS prior to mixing (as described in Xue et al. 2021.
[e] TEG, total epoxy group calculated as the sum of epoxy groups in the 4 species that were quantified by LC–MS/MS.
[f] TEG, total epoxy group by ion chromatography. TEG by IC tends to be higher than by LC–ESI–MS/MS by 10% to 25% due to the inclusion in IC analysis of non-chromatographable species. TEG reports only on the weight of epoxide groups.

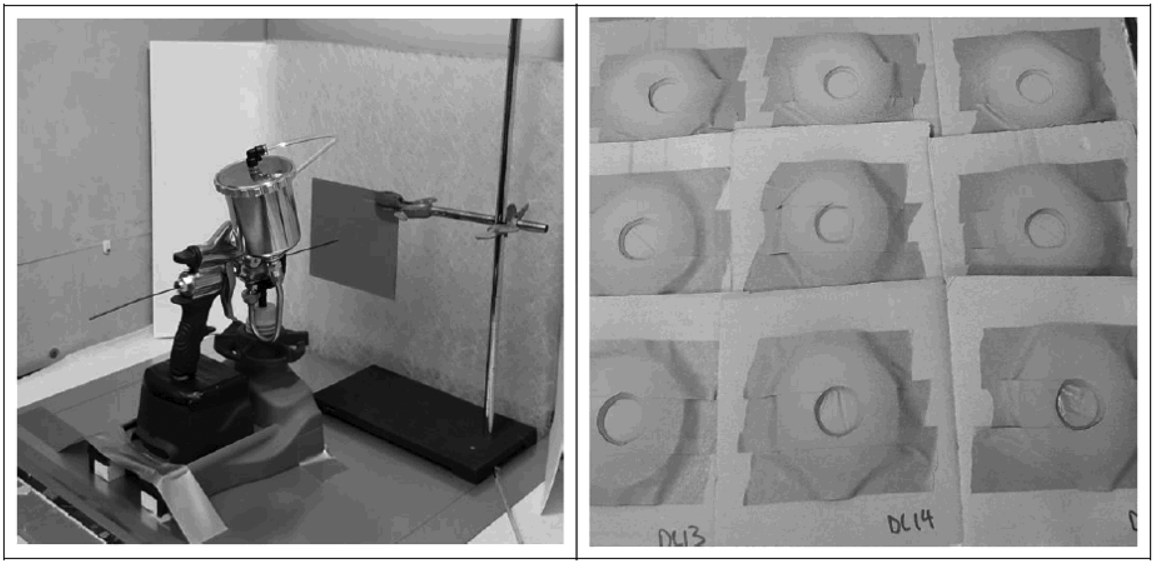

Figure 2. Test system setup. Left panel: A fixed-position spray gun was used to spray epoxy-based paints (a zinc-rich primer or an intermediate coat) over an 8-second spray interval to minimize loading variability from spray gun stroke speed, number of passes, and nozzle distance. Right panel: Spray cells were sprayed in batches of 15 test cells. The photograph shows cells following the application of zinc-rich primer.

of 8 s and fixing the spray gun adjustments (i.e. airflow, fluid volume, and spray pattern) throughout each batch of test cells. Prior to each test session, the spray system was visually calibrated using aluminum blanks to achieve the desired coverage. A product thickness of ~0.08 mm [3.0 mils (1 mil = 0.001 in)] was found to provide consistent coverage without excessive run-off with both coatings. A computer-based timer with an audible alarm was used to indicate the beginning and end of each spray interval and to monitor elapsed time for solid media collection.

Epoxy mass loading on test media at time zero

A total of 10 PTFE filters (5 for each coating product) were sprayed directly with each product to determine the amount of BADGE and its higher oligomers deposited onto the outer surface of each garment. Filters were transferred immediately post-application (~ 8 s spraying) to 5 mL DMF solution for subsequent chemical analysis (see 'epoxy sampling and chemical analysis' sections). These values were used as surface loading data at time t_0.

Homogeneity of coating application

The homogeneity of the applied coating film was assessed by measuring the amount of dried paint deposited on test filters via gravimetric analysis and by measuring dried film thickness on aluminum blanks (Part no.: L400021-RD-1000; 25.4 mm × 0.81 mm; Rose Metal Products Inc., Springfield, MO, USA) as described earlier (Mellette et al. 2019) and briefly described here. Eight 37-mm polytetrafluoroethylene (PTFE) filters (Toyo Roshi International Inc., Los Angeles, CA, USA) were sprayed for each batch of product/test material combination. After spraying, PTFE filters were cured at room temperature for 7 days in accordance with the coating cure times, followed by their weighing inside a temperature and relative humidity-controlled environmental chamber using an analytical balance (Model XP26; Mettler-Toledo, LLC, Columbus, OH, USA) with a 0.001 mg resolution.

The dry paint mass was determined as the difference between the 2 filter mass measurements (post-spray—pre-spray mass) ($n = 8$). Film thickness was determined with a dial caliper as the difference in thickness between aluminum blank with the dry film coating on it and the bare aluminum blank ($n = 8$) as described in our earlier work (Mellette et al. 2019).

Epoxy sampling and chemical analysis

Epoxy samples were collected on 25-mm glass fiber filters attached to each cell of the panel, that post-collection was transferred into 5 mL of dimethyl formamide (DMF) to inhibit the polymerization reaction. BADGE-d10 (100 ng) was added to each sample as an internal standard. Samples, when necessary, were diluted 10 times with methanol to ensure that the concentration of the sample was within the linear range of the standard curve. Field blanks were analyzed to assess the level of contamination introduced through the test method and laboratory blanks were analyzed with each sample preparation batch to ensure contamination was not introduced during the analytical process.

The separation and quantification of BADGE (epoxy monomer) and its oligomers (dimer, trimmer, tetramer) was performed with high-performance liquid chromatography—electrospray ionization—tandem mass spectrometry (LC-ESI-MS/MS) according to the method developed in our laboratory [see Supplementary Information Fig. S1] as described in detail in Xue et al. (2021). The limits of detection (LOD) for epoxy oligomers were as follows: BADGE, 2.5 pg injection on column; 0.25 ng cm^{-2} filter/fabric; dimer, 10 pg on column; 1 ng cm^{-2}; Trimer, 18 pg on column; 1.8 ng cm^{-2}.

Theoretical protection factor calculation

The theoretical protection factors were calculated for each of the 2 coating formulations (primer versus intermediate) and the 5 protective clothing material combinations. Theoretical protection factors (PFs) describe the ratio between epoxy surface loading on the outer surface of the garment under the test conditions and the expected amount of epoxy permeation or penetration on the inside of each garment. Theoretical PFs were calculated using the following equation: PF = L_o/L_i; where PF is the theoretical protection factor (unitless); L_o is the expected epoxy surface loading based on the average values for direct loading; L_i is the maximum cumulative permeation or penetration (ng cm^{-2}) at 0.08 mm (3.0 mils) coating thickness (ng cm^{-2}). We established the minimum protection factors (MPF) for each coating product tested using direct loading data and the cumulative permeation/penetration threshold as described in 'Permeation data analysis' section.

Permeation data analysis

Epoxy permeation was quantified as the amount of epoxy species accumulated on the glass fiber filter positioned under the protective clothing material. The amount was normalized by the cross-sectional area of the filter (5.1 cm^2) and reported as ng cm^{-2}. The maximum BADGE penetration was defined as the highest amount measured across the 5 different time periods and the corresponding time was reported as the time when the maximum cumulative permeation was measured.

Furthermore, cumulative permeation (cumulative mass per unit surface area) was compared with the acceptable recommended threshold for protective clothing materials. Although there are currently no occupational exposure limits for skin exposure to epoxy resins, a cumulative permeation limit of 2 µg cm^{-2} for glove materials and 10 µg cm^{-2} for loose fitting clothing was used as the threshold for cumulative permeation (Henriks-Eckerman et al. 2015). These limits are based on the local lymph node assays (LLNA) for BADGE and diamine hardeners expressed as an "EC_3" value. EC_3 is the estimated skin concentration of a compound required to induce a 3-fold increase in lymph node cell proliferation as compared to a control. The EC_3 for BADGE has been reported between 1.24% and 1.50% w/v or 310–375 µg BADGE cm^{-2} skin (Warbrick et al. 2001; O'Boyle et al. 2014; Henriks-Eckerman et al. 2015). The recommended BADGE garment permeation threshold (that would match the EC_3-equivalent skin sensitization threshold) was compared with the maximum BADGE permeation in our experiments and reported as a ratio of maximum permeation/recommended permeation threshold (%).

When steady state permeation occurred (the same mass measured for 2 consecutive times), simple linear regression analysis was conducted to determine the rate of permeation as described in Mellette et al. (2019). Permeation rates were compared with the ASTM F-739 and IS Standard 6529 normalized rate of breakthrough (0.1 µg cm^{-2} min^{-2}) as a measure of threshold for minimum acceptable permeation resistance.

Results

Homogeneity of coating application

A total of 80 gravimetric and 80 coating thickness samples were obtained to assess the homogeneity of paint loading and average dry coating thickness for each batch of test cells. The average loading across all batches of the zinc-rich primer was 160.47 mg (21.8% CV, coefficient of variation) with an average dry coating thickness of 0.086 mm (3.40 mils). The average dry paint mass loading across all intermediate coating samples was 69.22 mg (16.1% CV) with an average dry coating thickness of 0.095 mm (3.7 mils). A complete summary of average loading and thickness data has been provided in Table 3. Although the homogeneity of coating was more variable than in our previous study with polyurethane anti-corrosion coatings (Mellette et al. 2019), the range of observed variability was consistent with the level of variability expected from manual spraying (typically 20% CV; Ceballos et al. 2011).

Epoxy permeation/penetration through disposable gloves

A summary of permeation and penetration data for each clothing type for both coating products tested is shown in Table 4. The 2 types of disposable gloves, the thin latex [0.068 mm (2.7 mils)] and thin nitrile [0.073 mm (2.9 mils)], showed significantly different permeation profiles.

Table 3. Average dry paint mass loading (mg) and dry film thickness (mm) stratified by garment type and coating formulation (*n* = 8/cell).

Garment Type	Material	Zinc-rich primer: Average mass loading of the dried film coating mg (% CV)[a]	Zinc-rich primer: Average thickness of the dried film coating mm (mils)	Intermediate coating: Average mass loading of the dried film coating mg (% CV)	Intermediate coating: Average thickness of the dried film coating mm (mils)
Glove	Latex	213.28 (10.8)	0.068 (2.7)	54.08 (14.2)	0.100 (3.9)
Glove	Nitrile	143.49 (9.1)	0.069 (2.7)	66.35 (8.9)	0.094 (3.7)
Coverall	Tyvek	147.95 (9.2)	0.094 (3.7)	68.63 (11.6)	0.086 (3.4)
Coverall	PP[b]	168.49 (17.3)	0.091 (3.6)	81.43 (6.5)	0.103 (4.0)
Shirt	Cotton	129.12 (10.4)	0.070 (2.8)	75.62 (5.4)	0.092 (3.6)
Total averages by coating type		160.47 (21.8)	0.086 (3.4)	69.22 (16.1)	0.095 (3.7)

[a] % CV: coefficient of variation (%). [b] PP, polypropylene.

Table 4. Maximum penetration of BADGE, % of cumulative penetration threshold, and theoretical protection factors for each coating and protective clothing material combination.

Garment	Zinc-rich primer					Intermediate coating				
Type	Maximum BADGE permeation[a]	Collection time	Detectable Samples (%)	% of penetration threshold[d]	Theoretical protection factor[e]	Maximum BADGE permeation[a]	Collection time	Detectable Samples	Penetration threshold	Theoretical protection factor[e]
	(ng cm^{-2})	(min)[b]	(%)[c]			(ng cm^{-2})	(min)[b]	(%) [c]	(%)[d]	
Disposable gloves										
Latex	21.8	120	13.3	1.1	12620	514	60	40	25.5	310
Nitrile	<0.1	…[f]	…[f]	…[f]	2.7E+6[g]	<0.1	…[f]	…[f]	…[f]	1.6E+6[g]
Clothing										
Tyvek	<0.1	…[f]	…[f]	…[f]	2.7E+6[g]	6.5	15	6.7	0.1	23850
PE/PP[h]	5.0	15	6.7	0.1	54750	603	30	13.3	6.0	265
Cotton Shirt	128.2	5	100	1.3	2150	28.1	120	66.7	0.3	5600

[a] Refers to permeation through highly impermeable materials (i.e. gloves) and penetration through porous materials (i.e. breathable clothing).
[b] Time for which the maximum cumulative permeation was observed.
[c] Number of detectable samples in each batch ($n = 15$) for a coating and protective clothing material combination.
[d] % of recommended penetration threshold by protective clothing type: 2 $\mu\text{g cm}^{-2}$ for gloves; 10 $\mu\text{g cm}^{-2}$ for clothing (Henricks et al., 2015). Percent was calculated as the ratio of maximum BADGE permeation/recommended permeation threshold ×100.
[e] Theoretical protection factor = BADGE direct loading concentration (ng cm^{-2})/maximum cumulative penetration (ng cm^{-2}).
[f] All results were below the BADGE limit of detection (LOD, 0.25 ng/cm^{-2}).
[g] Theoretical protection factor was calculated using the BADGE LOD for the maximum cumulative penetration.
[h] PP/PE = polypropylene/polyethylene coverall.

The *nitrile gloves* did not allow permeation of BADGE or higher oligomers *for both products*, since every single sample across the whole timeframe of tests was not detectable (BADGE LOD, 2.5 pg injected on the column; 0.25 ng cm^{-2}).

Latex gloves were permeated by BADGE for both coating products, although the permeation was higher and time-dependent only for the intermediate coat. In the case of *zinc-rich primer,* BADGE permeation through latex gloves was measured only for 2 out of 15 individual cells, at 15 min (3.9 ng cm^{-2}) and 120 min (21.8 ng cm^{-2}) (Fig. 3A). The BADGE *dimer* was detectable only in one out of 15 individual cells of the zinc-rich primer (at 120 min, 0.08 ng cm^{-2}). The other

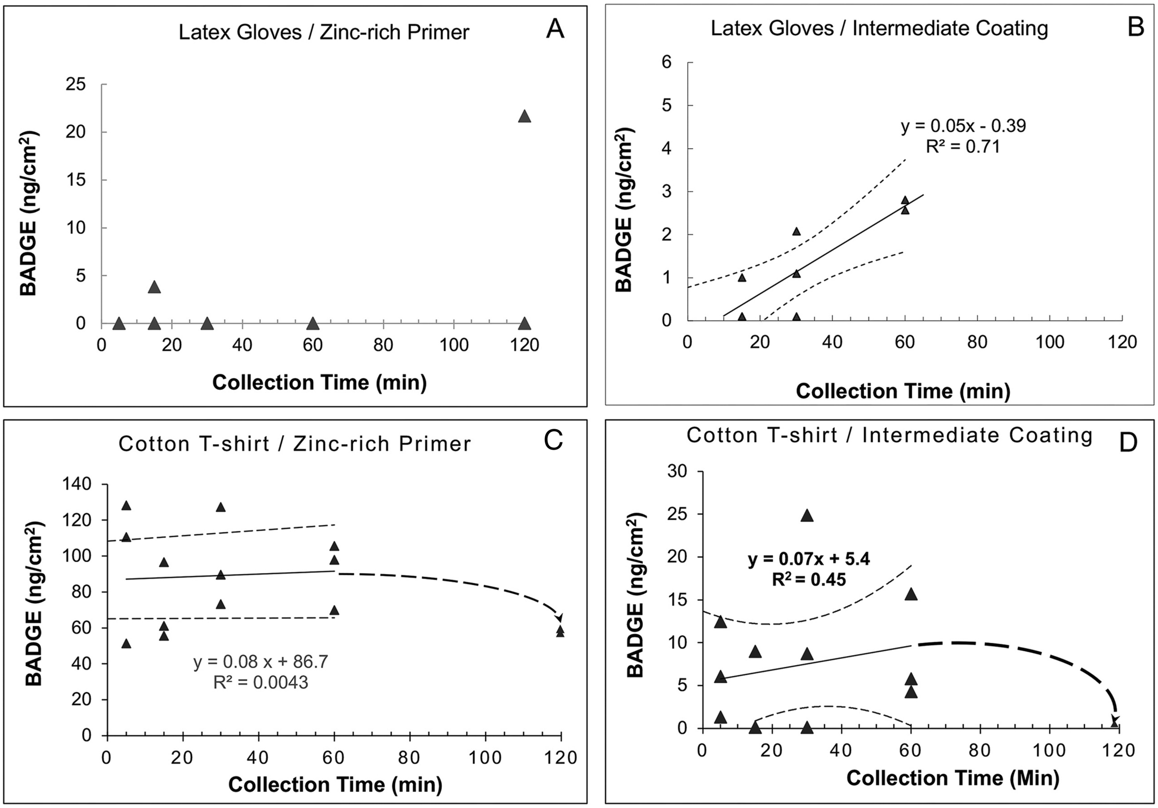

Figure 3. BADGE permeation rates through latex gloves (A, B) and cotton shirt (C, D) sprayed with a NEPCOAT-approved zinc-rich primer (A, C) and an epoxy-intermediate coating product (B, D). Regression lines for quantifiable data points up to 60 min and its 95% confidence interval are also shown. Dashed line represents extrapolation of data between 60 and 120 min.

2 replicate samples at these respective time points were non-detectable. It is likely that latex penetration by BADGE in these 2 zinc-rich primer cells may reflect the disintegration of the membrane rather than permeation since the BADGE values for the 2 other replicates at each of those 2-time points were non-detectable, and there is no clear pattern. The BADGE permeation rate for the primer could not be calculated because the steady state was not reached.

In the case of *intermediate coating*, BADGE permeation through latex gloves reached a steady state between 15 and 60 min, at a rate of 0.05 ng cm^{-2} min^{-1} (Fig. 3B, Supplementary Fig. S2.A). The BADGE permeation rate of 0.05 ng cm^{-2} min^{-1} is over 1000× below the established permeation threshold of 0.1 µg cm^{-2} min^{-1}. No detectable BADGE was present in collected samples after 60 min, indicating that the BADGE was fully reacted (i.e. consumed due to polymerization reactions) on the collection media underneath the garment/membrane and/or exterior surface of the membrane beyond 60 min. The maximum value of 513.8 ng cm^{-2} (1 of 6 detectable samples) was excluded from the regression analysis as an outlier. We do not have an explanation for this outlier value, but we hypothesize that material imperfections may be one possible explanation. Product formulation, especially the presence of solvents and viscosity, matters greatly when permeation and penetration are concerned. BADGE *dimer* was quantified in only one sample (at 60 min, 2.8 ng cm^{-2}; LOD of dimer, 1 ng cm^{-2}). No trimer was detected in any sample, at any time point (LOD for trimer, 1.8 ng cm^{-2}).

Visual penetration of the coating tint was not observed on any of the nitrile and latex glove collection media suggesting no significant penetration through these glove materials. These findings were further substantiated by comparisons of the BADGE monomer to dimer ratios in permeation samples where both analytes were quantified relative to their corresponding bulk products. The BADGE monomer/dimer ratio was 5.4 (18.9/3.4; % w/w) in the bulk zinc-rich primer and 3.3 (19.3/5.9; % w/w) in the bulk intermediate coating (Table 2b). These ratios were much higher in the permeated latex glove samples than in the bulk material. For the latex glove—zinc-rich primer combination this ratio could be calculated only for one sample (at

120 min) and was much higher than the corresponding bulk (37.7 versus 5.4). The BADGE/dimer ratio for latex glove-intermediate coating was 132 (at 60 min) which is much higher than the expected value of 3.3 for the bulk. These limited datapoints do reinforce the notion that permeation is the predominant mechanism of BADGE breakthrough in latex gloves, especially for the intermediate coating.

Epoxy permeation/penetration through protective clothing

Three breathable clothing materials that were tested against both coating products during this study consisted of Tyvek coveralls [0.105 mm (4.1 mils)], PP/PE coveralls [0.105 mm (4.1 mils)], and a heavy weight cotton T-shirt [0.317 mm (12.5 mils)].

The Tyvek coveralls provided superior resistance to BADGE from both coatings—the zinc-rich primer and the intermediate coating, followed by PE/PP, which also provided excellent protection (Table 4). No BADGE was detected in any of the samples from Tyvek material sprayed with the zinc-rich primer. Only one sample from the Tyvek material sprayed with the intermediate coating contained BADGE at 6.5 ng cm^{-2}, which represents only 0.1% of the suggested penetration threshold for these garments. *The PP/PE coveralls* also showed a high level of resistance to BADGE, with only 1 detectable sample when the zinc-rich primer was used (5.0 ng cm^{-2}) and 2 detectable samples with the intermediate coating: 1.34 ng cm^{-2} and 600 ng cm^{-2}, the later value representing ~6.0% of the recommended permeation threshold. Due to the limited number of quantifiable BADGE samples, the penetration time profile, permeation rate and cumulative permeation for Tyvek could not be established. The data however establish convincingly that Tyvek and PP/PE coveralls provide excellent protection against BADGE penetration in the tested zinc-rich primer and the intermediate coating products.

The cotton T-shirt was penetrated by BADGE and its higher oligomers (dimer and trimer) in 100% of zinc-rich primer samples and in 66% of intermediate coating samples (10 out of 15 samples) (Supplementary Table S1 and S2). With both coatings, penetration of BADGE and other oligomers through the cotton fabric occurred rapidly during the first 5 min of contact, with a maximum cumulative penetration of 128.2 ng cm^{-2} (BADGE) with the zinc-rich primer and 28.1 ng cm^{-2} (BADGE) with the intermediate coating (Fig. 3C and D).

Visual penetration of green zinc-rich primer was observed on the collection media when testing the cotton T-shirt, but not with the coverall materials. All primer samples had quantifiable dimer and trimer. The ratio of BADGE monomer/ dimmer (4.9, Supplementary Table S1 versus 5.4 in bulk, Table 2) and BADGE/trimer in T-shirt samples (1.9, Supplementary Table S1 versus 2.0 in bulk, Table 2) was nearly identical to the raw material, supporting the argument that penetration was the primary mechanism of chemical transfer through the cotton T-shirt for zinc-rich primers (Fig. 4A, Supplementary Table S1).

Red tint from the intermediate coating was not observed on any collection media, confirming no major penetration. For 67% of samples for which dimer and trimer were quantified, we could calculate the BADGE/dimer and BADGE/trimer ratios. The high dimer and trimer content in permeated samples and the consistent pattern suggests considerable penetration through the porous cotton fabric by the intermediate coating aerosol droplets. The much higher BADGE/dimer ratio in samples relative to the intermediate coating product (6.5 versus 3.3 in bulk) suggests that permeation is also an important mechanism. Taken together, the data suggest that both penetration and permeation mechanisms contributed to the breakthrough of cotton T-shirt by the intermediate coating (Fig. 4B, Supplementary Table S2).

Theoretical protection factors

PTFE filters were sprayed directly with each coating product followed by chemical analysis immediately post-application and gravimetric analysis after the product was cured (Table 4). The average film thickness of the dry zinc-rich primer was 0.070 mm [2.8 mils (1 mil = 0.001 inch)]. The zinc-rich primer contained an average BADGE loading of 273.8 μg cm^{-2} (range 258.2 to 309.0 μg cm^{-2}). The average dry intermediate coating thickness was 0.092 mm (3.6 mils). The intermediate coating samples contained an average BADGE loading of 157.4 μg cm^{-2} with a range of 149.6 to 161.2 μg cm^{-2}.

Theoretical protection factors in Table 4 quantify the expected level of protection provided by each garment material from BADGE permeation or penetration. Protection factors were not quantified for the nitrile gloves since no permeation was detected. The nitrile gloves were the most protective garment material tested with both coating formulations. With the latex gloves, the protection factors were >12 600 with the zinc-rich primer and >300 with the intermediate coating. Theoretical protection factors for the clothing materials with the zinc-rich primer ranged from no penetration with the Tyvek coverall (highest value, 2.7E+6) to a high penetration with the cotton shirt (lowest value, 2150). Clothing protection factors with the intermediate coating ranged from no penetration with the Tyvek coverall (23850) to a high penetration with PP/PE coverall (265).

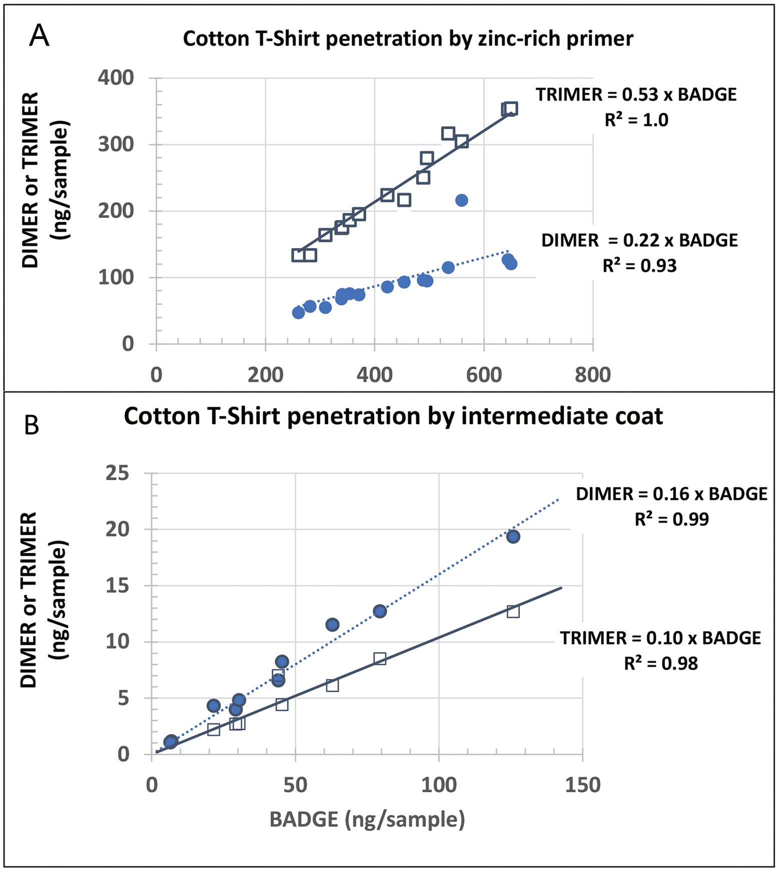

Figure 4. Penetration of cotton T-shirt by (A) zinc-rich primer and (B) intermediate coat. In the zinc-rich primer, BADGE, and its higher oligomers (dimer, trimer) maintain the same ratio as in the bulk product, providing strong evidence that this is likely due to fabric penetration, not permeation. In B, the ratios are different from the product, suggesting a mix of penetration and permeation processes.

Discussion

Permeation of disposable gloves

The objective of this study was to assess the effectiveness of common protective garment materials to prevent skin exposure to an epoxy-based zinc-rich primer and an epoxy-based intermediate coating. Nitrile gloves demonstrated superior barrier performance and provided a highly effective barrier against both coatings when compared to the latex gloves, which was shown by the lack of measurable penetration of BADGE through the nitrile material with either the zinc-rich primer or intermediate coating.

Although the latex gloves were less protective than the nitrile material, they did provide sufficient resistance to maintain the cumulative permeation of BADGE well below the recommended cumulative permeation threshold of 2 µg cm^{-2} with both coatings (primer: 1.1% of threshold; intermediate coating: 25.5% of threshold). Additionally, although steady-state BADGE permeation occurred with the latex gloves and intermediate coating (Fig. 3), the permeation rate remained well below the ASTM F-739 standardized rate of breakthrough of 0.1 µg cm^{-2} min^{-1} (observed permeation rate: 0.05 ng cm^{-2} min^{-1}). Furthermore, the frequency and cumulative amount of BADGE permeation through the latex gloves were significantly higher with the intermediate coating than with the zinc-rich primer (cumulative permeation: 513.8 ng cm^{-2} versus 21.7 ng cm^{-2}). These differences in BADGE permeation between the 2 formulations could be related to the carrier solvent composition of the intermediate coating, which contained several solvents, including 4-chlorobenzotrifluoride and MIBK, that have been shown to rapidly degrade natural rubber latex (Ansell Healthcare 2022). The higher viscosity and solids content of the zinc-rich primer further favors lower permeation/penetration rates compared to the intermediate coating.

There is one highly relevant publication on the penetration of epoxy formulations through gloves and garment materials. Henriks-Eckerman et al. tested a selection of similar protective clothing materials to evaluate BADGE penetration from epoxy-based pipe relining and floor coating systems (Henriks-Eckerman et al. 2015). As they demonstrated, there was no detectable BADGE permeation through the disposable nitrile gloves with either epoxy-based test formulation. Conversely, the disposable latex gloves were readily permeated by BADGE from the floor coating system. Our results of BADGE permeation through nitrile and latex gloves agree with the Henriks-Eckerman et al. (2015) data supporting a preference for nitrile gloves used when working with similar epoxy-based systems. Similarly, our observation that penetration data are specific to the garment/paint formulation also agrees with Henriks-Eckerman's study and points to the need for more systematic investigations of larger selections of glove materials, from various manufacturers and batches within each manufacturer, with realistic formulations.

Permeation and penetration of protective clothing

The Tyvek coverall provided the best resistance to both epoxy coating formulations. BADGE penetration ranged from non-detectable with the zinc-rich primer, to only one detectable sample with the intermediate coating (6.5 ng cm^{-2}). The PP/PE coverall also provides high resistance to the zinc-rich coating and was only penetrated by BADGE on one sample (5.0 ng cm^{-2}). The PP/PE membrane was less resistant to the intermediate coating, and although BADGE penetration was only detected on 2 samples, 1 sample was relatively high (603 ng cm^{-2}), possibly due to variability in the membrane pore structure. Despite these differences, both coverall materials provided sufficient protection to maintain the maximum cumulative BADGE permeation well below a recommended cumulative permeation threshold of 2 µg cm^{-2} with both coatings (Henriks-Eckerman et al. 2015). Although the PP/PE coverall provided the least amount of resistance of the 2 types of coverall material, it still only reached 6.0% of the recommended cumulative permeation threshold. Henriks-Eckerman et al (2015) reported higher penetration of BADGE in pipe relining (18 µg cm^{-2}) and floor coating systems (163 µg cm^{-2}) than in our study. The observed differences between the 2 studies can be attributed to a combination of factors, especially differences in product composition (viscosity, solvent content, and presence of particulate additives) and the different application methods (spraying over the whole membrane surface versus droplet deposition).

BADGE penetration through both coverall materials was higher when the intermediate coating was applied, despite its lower BADGE content. This higher rate of penetration is believed to be related to the lower viscosity of the intermediate coating versus the zinc-rich primer (1.67 versus 2.98 cm^2 s^{-1}) and a slower cure time (120 versus 30 min). FE-SEM analysis of the fabric microstructure also shows that the Tyvek's high resistance may have been related to its high-density flash spun fiber structure. Unlike the Tyvek, the PP/PE membrane showed high variability in the distribution of pore sizes that may have accounted for the greater frequency and concentration of BADGE penetration. Additionally, some of these pores were greater than 10 µm in diameter and may have allowed particulate from both coatings to pass through more readily than the tightly woven Tyvek fiber matrix.

The solvent composition may have also (likely) affected the degree of penetration with both coverall materials, but because of their similar polyolefin composition, this is not believed to be the primary reason for the observed differences in BADGE penetration between the 2 materials. Although chemical resistance data was not available from either garment manufacturer, a review of polypropylene and polyethylene fabric chemical compatibility data provided by the vendor did show that both polyolefin materials may be degraded by many of the solvents found in both coatings.

The cotton T-shirt provided the least consistent protection of any of the clothing materials tested. During testing with both coating formulations, cotton fiber penetration occurred rapidly over the first 5 min of contact. Penetration of BADGE occurred more consistently and at higher concentrations with the zinc-rich primer (max BADGE concentration, 128.2 ng cm^{-2}) than with the intermediate coating (max BADGE concentration, 28.1 ng cm^{-2}). Unlike BADGE in the zinc-rich primer which penetrated the cotton fabric in all samples, only 10 of 15 samples from the intermediate coating penetrated the cotton fabric. These differences in BADGE penetration through the cotton fabric between the 2 formulations are counter intuitive given the higher viscosity and the faster curing rate of the zinc-rich primer. It is likely that the higher epoxy content in the zinc-rich primer (40% versus 25%) may be a possible explanation. Despite the visible voids in the cotton fiber matrix, the cotton T-shirt also provided sufficient protection to remain well below the recommended cumulative permeation threshold with both coating formulations (primer: 1.3% of cumulative penetration threshold; intermediate coating: 0.3% of cumulative penetration threshold, Table 4).

Similar to Henriks-Eckerman et al (2015), our study demonstrated cotton clothing were readily penetrated by each of the coating mixtures, although the magnitude of penetration was significantly higher with the pipe relining (326 µg cm^{-2}) and floor coating systems (69 µg cm^{-2}) compared to corrosion control coatings

(0.128 μg cm^{-2} for the primer). The differences between the 2 study results may be in part due to differences between test method application techniques (i.e. spraying a thinner film over a larger surface area of the test fabric material versus a single droplet deposition by pipette over a much smaller surface area of the fabric), the higher concentrations of BADGE found in the pipe relining (>51% BADGE) and floor coating (64%) test mixtures (Henriks-Eckerman et al., 2015) versus ≤40% in primer/intermediate coatings, and differences in solvents types/concentration and product viscosity. Other differences between Henriks-Eckerman's study and ours included significantly thicker glove and cotton materials in their study, a lower volume of applied test chemical (50 μL versus ~70 μL), and shorter exposure times (30 min versus 120 min). However, the overall conclusion from both studies are similar, and this is an important finding.

Study limitations and future improvements in the test system

To accurately measure the cumulative permeation or penetration of an epoxy resin permeating a barrier material, ideally the polymerization reaction between BADGE/epoxies and (amine) hardeners must be stopped upon first contact with the collection media. In principle, this can be accomplished in 2 ways: by using a derivatizing reagent that will convert epoxy functionalities into a uniquely recognizable and stable compound, or by inhibiting the polymerization reaction. Sampling for airborne epoxy-containing aerosols is typically conducted using a liquid collection medium with an impinger or centrifugal aerosol sampler. These established methods use a polar aprotic solvent such as dimethyl formamide (DMF) to inhibit the epoxy-amine curing reaction and allow for accurate quantification of epoxy content (and other component of the mixture, such as amines) at the point of sample collection (Xue et al. 2021). During this study, impregnation of solid media with DMF was not possible, since DMF and other polar aprotic solvents would have significantly affected permeation through the garment materials. This is a recognized major limitation of our study and other similar studies to date.

Due to the inability to stop the epoxy-amine reaction on contact with the collection media, the data obtained during this study may have underestimated the permeation and penetration of BADGE during the later collection time points between 60 and 120 min, due to potential curing of accumulated epoxy resin and (other components of the mixture) on the filter media. This has also demonstrated in the Supplementary Fig. S2, which shows an average BADGE consumption of 25% between 30 and 120 min with the zinc-rich primer and 100% between 60 and 120 min with the intermediate coating. The consumption of BADGE for both coatings corresponds directly with their manufacturer published surface cure times. New generations of sampling methods should incorporate filter coating with nonvolatile derivatizing reagents that stabilize epoxies as stable derivatives using, for example, a similar principle to that of isocyanate sampling. Such an approach will further enable co-analysis of other chemicals of interest in the mixture, especially amine hardeners, by providing a more stable chemical milieu. We did not measure amine hardeners in this experiment, in part because sensitive and validated methods for doing so were not available at that time. Future work will aim at quantifying simultaneously multiple components of the reaction mixture to better understand and document penetration phenomena for epoxy coating systems.

We acknowledge that our test system is more demanding to set up and execute than the published models. This is in a way the price we must pay for reproducing relevant application techniques and product-protective fabric combinations. The manual one-at-a-time mode of application is also recognized as a limitation for the current study. However, additional improvements in the test system are possible. The use of a robotic arm for spraying will improve reproducibility and sample throughput. Spraying over larger surface areas of the same fabric or multiple fabrics simultaneously and simultaneous co-monitoring for penetration of multiple analytes in the mixture can significantly increase data density, which can make this test system more attractive for routine testing.

The technique of visual inspection of the color tint on the collecting membrane for penetration, although informative, is susceptible to high variability due to inherited variations in color perception by the naked human eye, as well as low sensitivity. The application of automated scanners, now readily available in a cell phone, can further strengthen the reproducibility and sensitivity of this additional monitoring endpoint.

Conclusion

All of the garment materials evaluated during this study, including the heavy-weight cotton T-shirt, provided sufficient resistance to prevent cumulative BADGE permeation and penetration in excess of the established cumulative thresholds. The evaluation of garment materials showed that the nitrile gloves and Tyvek coveralls provided the greatest level of protection against epoxy resin permeation from both the zinc-rich primer and intermediate coating. It should be noted that there is considerable variability between coating formulations in use, therefore these results should only be applied to similar, if not identical formulations. Latex gloves did provide some level of protection; however, due to concerns with their degradation and their limited resistance to BADGE permeation and other mixture ingredients, their use is not recommended with epoxy resin systems. The PP/PE coveralls provide some resistance to BADGE penetration; however, the high variability of their pore structure does not appear to provide sufficient consistency to effectively resist penetration and therefore they are not recommended. Tyvek® coveralls and nitrile gloves, especially the thicker gloves (i.e. >0.76 mm [5 mil]) that offer greater resistance to wear and tear, are recommended for use with epoxy coatings to maintain skin exposure to these potent sensitizers at the lowest possible levels. The cotton T-shirt provided only moderate protection and is not recommended as a standalone skin protection barrier. However, it should be considered for use under Tyvek coveralls to provide an additional layer of skin protection and comfort relative to wearing such coveralls on bare skin, as observed occasionally in the field. Most importantly, users should avoid bare skin areas (such as hands, forearms, neck, and face) when they use such products. Additional testing of other garments/membranes with realistic formulations is recommended.

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Conflicts of Interest

All authors declare that they have no conflicts of interest.

Data availability

The data that support the findings of this study are available upon reasonable request from the corresponding author.

SUPPLEMENTARY DATA

Supplementary data are available at *Annals of Work Exposures and Health* online.

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