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Bioinspired Surface Treatments for Improved Decontamination: Handling and Decontamination Considerations

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EXECUTIVE SUMMARY

The Center for Bio/Molecular Science and Engineering at the Naval Research Laboratory (NRL) initiated a program in January 2015 for evaluation of bioinspired treatments suitable for use as a top coat on painted surfaces with the intention of achieving improved aqueous decontamination of these materials. Funding was provided by the Defense Threat Reduction Agency (DTRA, CB10125). This report details results for variations in the methods and approaches used during the decontamination process, including use of pre-treatment with air or sorbent, increased temperature, alternative surfactants, and pressurized rinse streams. The methods were evaluated on painted aluminum coupons. Retention of the simulants paraoxon, methyl salicylate, dimethyl methylphosphate, and diisopropyl fluorophosphate following treatment of contaminated surfaces is reported.

BIOINSPIRED SURFACE TREATMENTS FOR IMPROVED DECONTAMINATION: HANDLING AND DECONTAMINATION CONSIDERATIONS

INTRODUCTION

The DoD Chemical and Biological Defense Program (CBDP) seeks to provide protection of forces in a contaminated environment including contamination avoidance, individual protection, collective protection, and decontamination. In January 2015, the Center for Bio/Molecular Science and Engineering at the Naval Research Laboratory (NRL) began an effort funded through the Defense Threat Reduction Agency (DTRA, CB10125) with a view toward evaluation and development of top-coat type treatments suitable for application to painted surfaces that would reduce retention of chemical threat agents following standard decontamination approaches. The effort sought to survey relevant and related areas of research and evaluate identified technologies under appropriate methods to determine efficacy, scalability, and durability.

During the course of the planned studies, significant variations were noted when minor variations in handling were employed during the decontamination of coupons. Further, decontamination approaches were noted to produce significantly different outcomes for evaluated top-coat type surface treatments. In conversation with members of the US Marine Corps Chemical Biological Incident Response Force (CBIRF), differences between field decontamination approaches and laboratory standard methods were identified. Based on these observations and conversations, a more careful series of evaluations was outlined in order to provide insight into the impact of laboratory and simulated field methods on decontamination outcomes. This document summarizes those results addressing a range of different decontamination approaches on the base painted aluminum coupons.

METHODS

The samples used for this study were aluminum coupons coated using a polyurethane paint system according to manufacturer specifications. For comparison, some coupons were treated with a slippery liquid infused porous surface (SLIPS) top coat. For the polymer slippery liquid infused porous surface (SLIPS) coating, [1] a 20 weight percent solution of poly(vinylidene fluoride-co-hexafluoropropylene) (PVDF-HFP; $M_w \sim 400000$, $M_n \sim 130000$, $\sim 10 \text{ mol}\%$ HFP) and DBP in acetone was prepared. A 1:2 mass ratio of PVDF-HFP: DBP was utilized; this ratio of PVDF-HFP: DBP was reported to yield a highly porous film without compromising integrity (bare spots) after extraction of DBP; and the as-cast film can be peeled off the substrate. The solution was stirred at 50°C for 1 h and aged at room temperature >1 d before casting. Polymer films were spin-cast onto polyurethane paint coated aluminum coupons. Speeds in this case were between 1000-3000 RPM; higher speeds were not possible due to weight of the square coupons and their size as compared to the available spin platform. The color of the paint is easily visible through transparent as-cast film, slightly obscured after DBP extraction, and clear again after infusion of Fomblin® Y. The coated, lubricated surfaces appear to be wet. The gel-SLIPS coating on the relatively rough painted substrates is notably less "slippery" than similar films on glass slides. The fluorinated liquid gives the appearance of being absorbed to some extent by the surface. Fomblin® Y similarly applied to paint only coupons does not produce this effect with most of the liquid flowing immediately off the surface.

Simulant exposure and evaluation methods were based on the tests developed by Edgewood Chemical Biological Center referred to as Chemical Agent Resistance Method (CARM). [2] Standard target exposures utilized a challenge level of 10 g/m^2 . The painted coupons were 0.00145 m²; the 10 g/m^2 target challenge was applied to the surfaces as three equally sized neat droplets. Following application of the target and aging, coupons were processed for decontamination. Under the CARM method, this includes an

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initial rinse with deionized water, soapy water immersion for 3 s, and a final rinse in deionized water. The coupon is then extracted using a solvent specific to the target. The standard approach at NRL is to age coupons for 1 h prior to use of a gentle stream of air to expel target from the surface. Samples were then rinsed with soapy water (0.59 g/L Alconox in deionized water), soaked in the soapy water (10 s), and rinsed with deionized water. Finally, the coupons were soaked in isopropanol for 30 min to extract remaining target; this isopropanol extract was analyzed by the appropriate chromatography method to determine target retention on the surface.

For paraoxon analysis, a Shimadzu High Performance Liquid Chromatography (HPLC) system with dual-plunger parallel flow solvent delivery modules (LC-20AD) and an auto-sampler (SIL-20AC; 40 μ L injection volume) coupled to a photodiode array detector (SPD-M20A; 277 nm) was used. The stationary phase was a C18 stainless steel analytical column (Luna, 150 mm x 4.6 mm, 3 μ m diameter; Phenomenex, Torrance, CA) with an isocratic 45:55 acetonitrile: 1% aqueous acetic acid mobile phase (1.2 mL/min). [3] For analysis of methyl salicylate (MES), diisopropyl fluorophosphate (DFP), and dimethyl methylphosphonate (DMMP), gas chromatography-mass spectrometry (GC-MS) was accomplished using a Shimadzu GCMS-QP2010 with AOC-20 auto-injector equipped with a Restex Rtx-5 (30 m x 0.25 mm ID x 0.25 μ m df) cross bond 5% diphenyl 95% dimethyl polysiloxane column. A GC injection temperature of 200°C was used with a 1:1 split ratio at a flow rate of 3.6 mL/min at 69.4 kPa. The oven gradient ramped from 50°C (1 min hold time) to 180°C at 15°C/min and then to 300°C at 20°C/min where it was held for 5 min.

RESULTS

The solution used as soapy water in the NRL evaluations is comprised of water and Alconox. The solution used under the CARM method is based on a nonylphenol polyethoxylate nonionic emulsifier/wetting agent. Field applications often rely on materials at hand or those easily acquired, Dawn® dishwashing liquid (detergent), for example. Laboratory experiments frequently use deionized water while field applications will likely be using municipal water sources (tap water) or even surface water. The CARM method indicates use of a stream of rinse solution; however, it does not indicate use of elevated temperature or pressure during the rinse step. The Joint Service Transportable Decontamination System (M26) produces a stream of soapy water at up to 870 psi with temperatures of 50 or 80°C. The following evaluations were intended to consider the impact of these variations on the removal of simulant targets from the painted coupons.

Alconox and Tergitol were selected to represent the conditions used by NRL and the Edgewood Chemical Biological Center (ECBC) CARM approaches, respectively. Dawn dishwashing liquid was used as an example of easily obtained materials. The material used under the ECBC CARM protocol is 7930-00-282-9699 marketed as a non-solvent degreaser under the names Ability-One and Tergitol NP-9. This material is labeled with the signal word Danger and with pictograms for corrosion (GHS05), acute toxicity (GHS07), and environment (GHS09). Alconox is also labeled with the signal word Danger and pictograms for corrosion (GHS05) and acute toxicity (GHS07). Hazard codes are fully described in the appendix. Dawn dishwashing liquid is labeled with the signal word Warning and with no pictograms; eye irritation is the only noted exposure risk.

Figure 1 presents the results for comparison of three soapy water solutions prepared using both deionized water and tap water. Here, the CARM approach is used for decontamination: the coupon is rinsed with water, immersed in soapy water for less than 10 s, and rinsed with water. DMMP is fully soluble in water; retention of this target tends to be low for even a simple water wash. At room temperature in deionized water, Tergitol tends to produce the best results with coupons showing the lowest levels of target retention. The use of tap water rather than deionized water tends to reduce retention of the simulants slightly when

either Alconox or Dawn is used in the wash solution. Tap water and deionized water produced similar results when used in combination with Tergitol. The impact of temperature was also considered across these variations in wash solution (Figure 1). The increased temperature (80°C) significantly reduced retention when using the Alconox solution. Retention of MES and paraoxon was also reduced for the Tergitol and Dawn solutions at elevated temperature. The results are summarized in Table 1.



Fig. 1 — Target retention by coupons following treatment with a low pressure soapy water rinse followed by 10 s soak and low pressure water rinse: (A) room temperature, (B) 80°C. Here, the use of deionized water and tap water in preparation of rinse solutions is also compared.

Table 1 – Low pressure soap rinse at indicated temperature followed by 10 s soak and low pressure water rinse at room temperature. Average target retention (g/m^2) following 10 g/m² challenge and 1 h aging.

Conditions	Paraoxon		MES		DMMP		DFP	
	23°C	80°C	23°C	80°C	23°C	80°C	23°C	80°C
		Deic	nized Wa	ter				
Alconox in Deionized Water	3.65	2.47	8.10	4.04	0.08	0.07	2.84	0.47
Tergitol in Deionized Water	3.61	1.43	5.98	2.97	0.02	0.03	0.19	0.56
Dawn in Deionized Water	4.14	3.30	6.68	2.21	0.07	0.03	0.73	1.10
		Т	ap Water					
Alconox in Tap Water	3.53	3.03	5.31	4.61	0.10	0.13	2.00	0.64
Tergitol in Tap Water	3.67	2.09	5.89	3.06	ND	0.06	0.20	0.67
Dawn in Tap Water	1.97	2.25	4.60	3.46	0.02	0.13	1.50	1.16

ND = not detected

The standard protocol used by NRL incorporates an air stream as the first step for removal of excess target on the surface prior to beginning the rinsing process. This was meant to be the laboratory equivalent of driving a vehicle for some distance prior to decontamination. As shown in Figure 2 (Table 2), this step has little impact on retention of paraoxon, but significantly reduces retention of MES and DFP by the surfaces. The CARM method uses a water rinse as the first step rather than the air stream or the initial soapy water rinse used above. When this process is followed (water, soap, water rather than soap, soap, water), little impact is seen on retention of paroxon or MES, while DFP retention by the surfaces is greater than when soapy water is used for the initial rinse step (Figure 2; Table 2). The effect this series of evaluations was intended to capture was initially observed when working with a gel SLIPS top-coat formulation; shaking the samples prior to processing produced lower retention of MES. For that treatment, retention of MES was 3.91 g/m² when treated with water initially, 2.87 g/m² when treated with soapy water initially, and 0.57 g/m² when treated with the air stream prior to processing. This is likely the impact of a partitioning effect. While the solubility of MES and DFP in the SLIPS polymer and lubricating oils is low,

addition of water to the surfaces drives partitioning of the target into the polymer/oil layer rather than rinsing them from the surface as intended.



Fig. 2 — Target retention by coupons following treatment: (A) with and without an initial air stream followed by a low pressure soapy water rinse, 10 s soak, and low pressure water rinse, room temperature and (B) with and without the use of an initial rinse with water only followed by a soapy water rinse, a 10 s soapy water soak, and low pressure water rinse, room temperature.

Table 2 – Comparison of initial treatment with air stream, soapy water, or deionized water. Average target retention (g/m²) following 10 g/m² challenge and 1 h aging. "Water" indicates a water rinse followed by soapy water rinse and soak and a water rinse. "Soapy water" indicates a soapy water rinse followed by soapy water soak and a water rinse. "Air" indicates treatment with air followed by soapy water rinse, soapy water soak, and a water rinse. All steps completed at room temperature.

Approach	Paraoxon	MES	DMMP	DFP
Extract Only	9.84	9.54	9.90	7.39
Water	3.31	4.57	0.03	1.76
Soapy Water	3.65	8.10	0.08	2.84
Air	3.91	4.30	0.02	1.71

Table 3 provides results for target retention following only the air treatment as compared to no treatment (extract only) and compared to the full process used by NRL including air pretreatment, rinsing with soapy water, 10 s soak, and rinsing with water. An additional variation was also evaluated, the use of a dry adsorbent applied prior to processing. In this case, wood dust was rubbed onto the contaminated aged sample and immediately removed with a stream of air. As shown in Figure 3 (and Table 3), the wood dust significantly reduced retention of all four targets considered.



Fig. 3 — Target retention by coupons following treatment. Here, coupons were either treated with an initial air stream followed by a low pressure soapy water rinse, 10 s soak, and low pressure water rinse (room temperature) or by application of wood dust followed by the same procedure.

Table 3 – Comparison of target removal with and without air and adsorbent pre-treatment. Average target retention (g/m²) following 10 g/m² challenge and 1 h aging. Here, Decon Procedure indicates a soapy water rinse (Alconox), followed by soapy water soak and water rinse all at room temperature.

Approach	Paraoxon	MES	DMMP	DFP
Extract Only	9.84	9.54	9.90	7.39
Air Only	6.47	8.43	3.39	1.91
Decon Procedure	3.65	8.10	0.08	2.84
Air + Decon Procedure	3.91	4.30	0.02	1.71
Wood Dust + Decon Procedure	1.24	3.01	0.02	0.70

Additional results provided in the appendix.

Finally, the impact of pressure on the decontamination procedure was evaluated. Here, a paint gun was used to deliver a pressurized stream of soapy water followed by a pressurized stream of deionized water. The paint gun provided 50 psi and delivered 15 mL/min. The angle of impact for the stream on the surface was varied to provide additional information on any impact this may have during decontamination: 20°, 45°, 90° (perpendicular to the surface). The 45° impact angle was also evaluated using a soapy water solution heated to 80°C. The use of the pressurized stream, regardless of impact angle significantly reduced target retention over the NRL laboratory procedure and the other approaches evaluated here (Table 4, Figure 4). No trends or significant differences were noted with respect to the angle of incidence used in the pressurized decontamination process (Figure 4, Table 4). Increasing the temperature of the decontamination solution had little impact on the result (Figure 5, Table 5).



Fig. 4 — Target retention by coupons following treatment: (A) comparison of NRL procedure (initial air stream followed by a low pressure soapy water rinse, 10 s soak, and low pressure water rinse, room temperature) and the pressurized soapy water wash (45°) followed by pressurized water rinse; (B) comparison of results for pressurized decontamination at 20° and 90° incidence angles.

Table 4 – Comparison of target removal with and without use of pressurized delivery. Average target retention (g/m²) following 10 g/m² challenge and 1 h aging. Here, a pressurized soapy water rinse (Alconox) is followed by a pressurized deionized water rinse at room temperature. Values for the NRL procedure including an air stream followed by soapy water rinse (Alconox), soapy water soak, and water rinse at room temperature are included for comparison.

Approach	Paraoxon	MES	DMMP	DFP
NRL Procedure	3.91	4.30	0.02	1.71
20° Pressurized Rinse	0.44	1.17	0.73	0.03
45° Pressurized Rinse	0.64	1.18	0.05	0.52
90° Pressurized Rinse	0.16	0.69	0.53	0.04

Additional results provided in the appendix.



Fig. 5 — Target retention by coupons following treatment. Shown here is a comparison of the 45° incidence pressurized rinses at room temperature and at 80°C.

Table 5 – Comparison of target removal at room and elevated temperature with pressurized delivery. Average target retention (g/m^2) following 10 g/m² challenge and 1 h aging. Here, a pressurized soapy water rinse (45°) is followed by a pressurized deionized water rinse.

Soap	Temperature	Paraoxon	MES	DMMP	DFP
Alconox	23°C	0.64	1.18	0.05	0.52
	80°C	0.61	0.86	0.07	0.06
Tergitol	23°C	0.40	0.85	0.15	0.04
	80°C	0.62	0.45	0.19	0.05
Dawn	23°C	0.45	1.11	0.59	0.04
	80°C	0.85	1.43	0.40	0.04

CONCLUSIONS

Under the CARM protocol, all of the soaps considered perform similarly; however, even a minor change, such as changing the initial rinse to a soap solution, produces a significant change in outcomes (Appendix Table A-2). If the impact of pressure is excluded, tap water tends to produce slightly less target retention than deionized water, and warm water produces slightly less retention than room temperature water. The use of air has a positive impact on the total MES and DFP retained; these are the less water soluble of the targets considered. The use of an adsorbent pre-treatment (wood dust) also had a positive impact on target retention. The use of a pressurized rinse stream had more impact on target retention than any of the other

variations considered under this study, regardless of the angle of incidence used with the decontamination stream.

The samples used for this study were aluminum coupons coated using a polyurethane paint system applied according to the manufacturer's specifications. All of the coupons had been aged and cured identically before the evaluations completed here. Though only briefly mentioned, differences in the coating can produce significant differences in performance under the range of conditions evaluated. There is little difference in retention for coupons treated with the CARM and NRL protocols; however, these methods produce dramatically different results on a SLIPS coupon. As changes to current paint formulations and treatments are considered, it would be of benefit to also consider how those materials perform under the decontamination conditions used in the field. As noted in the introduction, use conditions can vary significantly from the ideal protocol.

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Appendix

ADDITIONAL DATA AND SUPPORTING INFORMATION

Pictograms	Hazard Code	Hazard class and category	Soaps	
		Corrosive to metals, category 1		
A CONTRACTOR	GHS05	Skin corrosion, categories 1A, 1B, 1C	Tergitol and	
		Serious eye damage, category 1	Alconox	
		Acute toxicity (oral, dermal, inhalation),		
		category 4	Tergitol and	
\Diamond	GHS07	Skin irritation, category 2		
		Eye irritation, category 2		
		Skin sensitization, category 1		
		Specific target organ toxicity – single		
		exposure, category 3		
		Hazardous to the aquatic environment		
₩ <u></u>	GHS09	-Acute hazard, category 1	Tergitol	
		-Chronic hazard, categories 1, 2		

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* The category provides information on the degree of hazard presented by the product. Category 1 presents the greatest threat; category 2 within the same hazard class is more hazardous than category 3, and so on.

Table A-2 – Comparison of initial treatment with air stream, soapy water, or deionized water. Average target retention (g/m²) following 10 g/m² challenge and 1 h aging. Water indicates a water rinse followed by soapy water rinse and soak and a water rinse. Soapy water indicates a soapy water rinse followed by soapy water soak and a water rinse. Air indicates treatment with air followed by soapy water rinse, soapy water soak, and a water rinse. All steps completed at room temperature.

Solution	Target	Water	Soapy Water	Air
	Paraoxon	3.31	3.65	3.91
Alconov in Deionized Water	MES	4.57	8.10	4.30
Alconox in Defonized water	DMMP	0.03	0.08	0.02
	DFP	1.76	2.84	1.71
	Paraoxon	3.20	3.61	3.61
Targital in Deignized Water	MES	4.78	5.98	4.08
Tergitor in Defonized water	DMMP	ND	0.02	0.03
	DFP	1.87	0.19	1.42
	Paraoxon	3.94	4.14	4.13
Dawn in Deionized Water	MES	4.18	6.68	2.09
	DMMP	0.02	0.07	0.03
	DFP	1.89	0.73	0.39

	Approach	Paraoxon	MES	DMMP	DFP
	Extract Only	9.84	9.54	9.90	7.39
	Air Only	6.47	8.43	3.39	1.91
Alconox	Decon Procedure	3.65	8.10	0.08	2.84
	Air + Decon Procedure	3.91	4.30	0.02	1.71
	Wood Dust + Decon Procedure	1.24	3.01	0.02	0.70
Tergitol	Decon Procedure	3.61	4.08	0.03	1.42
	Air + Decon Procedure	3.61	5.98	0.02	0.19
	Wood Dust + Decon Procedure	0.86	2.80	0.02	0.22
Dawn	Decon Procedure	4.13	2.09	0.03	0.39
	Air + Decon Procedure	4.14	6.68	0.07	0.73
	Wood Dust + Decon Procedure	0.48	4.44	0.03	0.56

Table A-3 – Comparison of target removal with and without air and adsorbent pre-treatment. Average target retention (g/m²) following 10 g/m² challenge and 1 h aging. Here, Decon Procedure indicates a soapy water rinse, followed by soapy water soak and water rinse all at room temperature.

Table A-4 – Comparison of target removal with and without use of pressurized delivery. Average target retention (g/m²) following 10 g/m² challenge and 1 h aging. Here, a pressurized soapy water rinse is followed by a pressurized deionized water rinse at room temperature. Values for the NRL procedure including an air stream followed by soapy water rinse, soapy water soak, and water rinse at room temperature are included for comparison.

	Approach	Paraoxon	MES	DMMP	DFP
Alconox	NRL Procedure	3.91	4.30	0.02	1.71
	20° Pressurized Rinse	0.44	1.17	0.73	0.03
	45° Pressurized Rinse	0.64	1.18	0.05	0.52
	90° Pressurized Rinse	0.16	0.69	0.53	0.04
Tergitol	NRL Procedure	3.61	5.98	0.02	0.19
	20° Pressurized Rinse	0.77	1.54	0.08	ND
	45° Pressurized Rinse	0.40	0.85	0.15	0.04
	90° Pressurized Rinse	0.29	0.97	0.27	0.03
Dawn	NRL Procedure	4.14	6.68	0.07	0.73
	20° Pressurized Rinse	0.43	1.62	0.39	0.04
	45° Pressurized Rinse	0.45	1.11	0.59	0.04
	90° Pressurized Rinse	0.24	1.33	0.38	0.06

*ND indicates not detected