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DEVCOM ARMY RESEARCH LABORATORY

Antimicrobial Coating Development

by Dawn Crawford and John Escarsega

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14. ABSTRACT
US Army coatings provide essential protection to Army assets in numerous ways, including camouflage, corrosion resistance, and chemical agent resistance. Although it has been known since WWII that military equipment is susceptible to microbial attack and deterioration, Army chemical agent resistant coatings (CARCs) do not have requirements for microbial resistance. To address this need, the US Army Combat Capabilities Development Command Army Research Laboratory initiated a program to assess the microbial resistance of water-dispersible CARCs (MIL-DTL-64159B) and the use of low concentrations of commercial biocides to minimize problems with mold, algae, and mildew growth on CARCs. This report summarizes the findings of this program and the new requirement for fungal resistance of the water-dispersible CARC recently published in MIL-DTL-64159 Rev C, March 2022.

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1. Introduction and Background

The challenge of microbial growth on military equipment is well documented in the literature. A comprehensive summary of microbial contamination of military assets has been covered in a prior report (Crawford and Escarsega 2019). Since 2014, the US Army Combat Capabilities Development Command Ground Vehicles Systems Center and Elzly Technology Corporation conducted numerous field surveys on mine-resistant ambush-protected (MRAP) vehicles in various locations including Schofield Barracks, Hawaii (Elzly Technology Corporation 2014), Joint Base Charleston, Sierra Army Depot, and outside the 402nd Army Field Support Brigade in Kuwait (Elzly Technology Corporation 2015). Although the initial purpose of the field surveys was to assess vehicles for corrosion, problems with mold were evident in almost every vehicle by sight and smell. Mold test kits placed in the vehicles confirmed the presence of various molds in every vehicle (Porter 2019). Mold test kits and laboratory testing confirmed that numerous fungus types were present in the vehicles, including *Aspergillus*, *Cladosporium*, *Penicillium*, *Sterile*, *Verticillium*, *Acremonium*, *Epicoccum*, *Alternaria*, *Humicola*, *Cleoycete*, *Engyodontium*, *and Botryosporium*, confirming the need for antimicrobial solutions for Army coatings.

Many technical approaches for microbial resistance of coatings and other materials can be found in the literature. Numerous antimicrobial technologies were reviewed in Crawford and Escarsega (2019). Understandably, with the onset of the global pandemic in early 2020 there has been a great interest in new antimicrobial technologies for coatings and disinfectants to protect against the spread of disease. Providing antimicrobial properties to chemical agent resistant coatings (CARCs) will reduce the presence of molds and other microbes on vehicles and equipment, reducing the sustainability and maintenance burden required by recurrent cleaning, thereby improving readiness and a safer working environment for Soldiers and military personnel.

2. Experimental

The use of CARC is mandated by Army Regulation AR-750-1 (HDQA 2017) for all tactical Army platforms and is also used by the Marine Corps, which includes ground, aviation, and related support assets. Tactical items (i.e., used in or near a combat zone) include obvious military equipment such as tanks, trucks, missiles, ammunition, aircraft, and communication transports but also hardware such as generators, water-purification units, and high-mobility material-handling forklifts. The coatings must provide the standard characteristics of any protective finish,

including corrosion resistance, durability, and identification marking. In the special case of tactical equipment, these coatings must also provide resistance to chemical warfare agents (CWAs), resistance to agent decontamination procedures, and optical characteristics such as camouflage. To implement these requirements the Army uses CARCs, which are defined and cited in MIL-DTL-64159B (2015) and MIL-DTL-53039E (2018).

Water-dispersible polyurethane CARC (MIL-DTL-64159B [2015], Tan 686) was selected for modification with a commercially available biocide, zinc pyrithione (ZPT) and iodopropynyl butyl carbamate (IPBC). IPBC was used as a blend with ZPT in very low concentrations to determine if it showed additional fungal resistance compared to ZPT alone. ZPT, an antifungal agent, is well known and has been used for decades in paint and other materials (Baldrian 2003, 2010; Hochmannova and Vytrasova 2010; Gaylarde et al. 2011; Bellotti et al. 2013; Ustaogluiyigundogdu et al. 2014; Tornero 2018; Kumar et al. 2019). ZPT has been used for 50 years to treat dandruff and seborrheic dermatitis (Schwartz 2016). ZPT is not a skin sensitizer and is FDA approved at concentrations up to 2% in rinse-off hair products (FDA 2018). Because of the long-term use and testing on humans, ZPT was chosen as the primary biocide in the formulations.

MIL-DTL-64159B (2015) formulations with and without ZPT dispersion were spray applied on pretreated and primed panels, dry cured for 7 days at ambient temperature and post cured at 122 °F for an additional 7 days. The coated panels were then evaluated for coating properties according to the military specification. Pencil hardness, color, and gloss were measured before and after immersion in deionized water and multipurpose diesel fuel (JP-8). MIL-DTL-64159B coatings with and without biocides underwent accelerated weathering to determine the stability of the additives. Fungal resistance tests were performed by Army Test and Evaluation Command (ATEC) at Aberdeen Proving Ground, Maryland, according to ASTM D5590 (2017). To evaluate fungal susceptibility or resistance in the actual tropic environment, the coated panels were also evaluated after 18 months' outdoor exposure at the Tropic Regions Test Center (TRTC) in both Panama and Suriname.

2.1 Materials

MIL-DTL-64159B (2015) samples were prepared according to the specification guidelines. To prepare the mixtures of the base paints with the commercial off-the-shelf biocides, the two-component paint system was admixed and 400-g aliquots were then used to make the antimicrobial paints with the concentrations shown in Table 1. This process ensured that each paint had identical stock material. Two commercially available biocides were acquired from Lonza: Zinc Omadine ZOE

dispersion and Omacide IPBC. For laboratory tests, the coatings were sprayed onto cold-rolled steel panels that were pretreated with Bonderite 952 zinc phosphate coating and sealed with Parcolene 60 chrome sealer (in accordance with TT-C-490 [2018]) and primed with MIL-DTL-53022E Type IV (2017). The panels were 0.032 inch thick. Panels were purchased from ACT Test Panels LLC. The coated panels were air dried for 7 days at 50% humidity and 72 °F and then post-cured in a convection oven for 7 days at 122 °F to ensure optimum cure. Sample preparation was the same for outdoor exposure evaluation at TRTC with the exception that the panels were 2024 T3 aluminum.

Table 1 Matrix of paint biocide concentrations

Base coating	Antimicrobial Additive	Additive (wt%)	Active ZPT (wt%)	Code name
MIL-DTL-64159 Waterborne polyurethane exterior CARC	Zinc Omadine ZOE dispersion	0	0	64-1
		0.4	0.15	64-2
		3.0	1.11	64-3
	ZOE dispersion IPBC blend	0.3 0.3	0.11	64-4
MIL-DTL-64159 Weatherometer exposure 500 h (W500)	ZOE Dispersion	0	0	W500 64-1
		0.4	0.15	W500 64-2
		3.0	1.11	W500 64-3
	ZOE dispersion IPBC blend	0.3 0.3	0.11	W500 64-4

2.2 Accelerated Weathering

An Atlas Ci-5000 Xenon Arc weatherometer was used for accelerated weathering in accordance with ASTM G155 (2013) Method 1 for 500 h. The xenon arc lamp was fitted with an inner and outer borosilicate filter and operated continuously with a light irradiance of 0.35 W/m² at 340 nm. The chamber conditions were set at 145 °F black panel temperature and 50% relative humidity. The method involves a 120-min cycle of continuous light at 108 °F. The first 102 min (light only) was followed by 18 min with water spray (light plus water spray) directed at the front of the sample panels. The unit for color change is noted by Delta E (dE) and the change in gloss values are reported at the angle of reference d20, d60, and d85.

2.3 Coated Panel Tests

2.3.1 Immersion Tests

Coated panels were partially immersed in deionized water and JP-8 jet fuel for 168 h at room temperature (77 ± 2 °F) as described in MIL-DTL-64159B (2015). After 168 h of immersion, the panels were removed and inspected for color change, gloss change, visible wrinkling, blistering, or delamination.

2.3.2 Pencil Hardness

Pencil hardness tests were performed on the coated panels in accordance with ASTM D3363 (2005) before and after immersion testing. The specification requirement states that the coating shall not soften more than two hardness units from the original value following immersion tests.

2.3.3 Color and Specular Gloss

Color and color difference was measured using HunterLab UltraScanPRO spectrophotometer with EasyMatchQC software version 3.72.00 with a D65 light source (according to ASTM E1164 [2017]). The color difference was calculated according to ASTM D2244 (2016). Gloss was measured using a BYK Gardner micro-TRI-gloss instrument at angles 20°, 60°, and 85° according to ASTM D523 (2018).

2.4 Fungal Testing

Fungal tests were performed by ATEC, Applied Science Test Division, at Aberdeen Proving Ground, Maryland, according to ASTM D5590 (2012). Triplicate samples for each spore suspension were tested. Three species of test spores were used according to the test method. *Aspergillus niger* and *Penicillium pinophilum* were applied as a mixed spore suspension and *Aureobasidium pullulans* was maintained separately. Potato dextrose agar was used as the test medium. Agar plates were inoculated with the appropriate test spores and incubated for 28 days at 82 ± 2 °F and 85%–90% relative humidity. Complete details of the fungal testing can be found in the ATEC final report (Aberdeen Test Center 2019).

Outdoor exposure testing was performed at the US Army TRTC (Horoko, Panama, and Afobaka, Suriname). The coated panels were observed for fungal growth at 1-month intervals for 18 months. Images were taken on site each month to record visual progression of mold growth. At the end of 18 months the exposed samples were returned to DEVCOM Army Research Laboratory for visual inspection and microbial analysis was initiated.

3. Results and Discussion

Weathering results for MIL-DTL-64159B (2015) panels indicated that for low-level loading of ZPT and the blend of ZPT and IPBC, the additives provided a noticeable improvement in color stability (Table 2). While zinc may assist protection of the coating by inhibiting photons from damaging the binder system, it is notable that this photo-protective effect did not increase with increasing ZPT concentration. In fact, the lowest ZPT concentration in the blend showed the greatest improvement. The coating with 0.4% Zinc Omadine ZOE dispersion (0.15% active ZPT) had a color change of 1.15 dE and the Zinc Omadine ZOE dispersion/IPBC (0.3%/0.3%) blend exhibited a color change of only 0.71 dE compared to the control (no additive) which showed a color change of 2.03 dE. The highest weight % of Zinc Omadine ZOE dispersion (1.11 % active ZPT) showed a modest improvement in color stability of 1.80 dE compared to the control. There was virtually no change in the Tan 686 gloss values compared to the control (no additive). The 85° gloss values increased nominally; however, they were similar for all loading levels (including the control), and gloss values of roughly 0.3 are typical for that viewing angle and weathering exposure. Thus, the biocidal additives did not have a detrimental effect on the weathering performance.

Table 2 Accelerated weathering results

MIL-DTL-64159 Tan 686 + biocide	Biocide (wt%)	Color change (dE)	Gloss change (d20°)	Gloss change (d60°)	Gloss change (d85°)
None	0	2.03	0.06	-0.01	0.33
Zinc Omadine ZOE dispersion	0.4	1.15	0.00	0.00	0.31
Zinc Omadine ZOE dispersion	3.0	1.80	0.00	-0.06	0.30
ZOE dispersion/IPBC blend	0.3/0.3	0.71	0.00	0.09	0.26

Pencil hardness values did not change as a result of immersion in deionized water or JP-8 for all loading levels (Table 3). Values of 3H and 4H were measured for all samples.

Table 3 Pencil hardness

Coating	Zinc Omadine ZOE dispersion (wt%)	JP8	Water	Original
64-1	0	4H	4H	4H
64-2	0.40	4H	4H	4H
64-3	3.00	4H	4H	4H
64-4	0.3/0.3	4H	4H	4H

Note: Pencil hardness scale: 6B-5B-4B-3B-2B-B-HB-F-H-2H-3H-4H-5H-6H; softest to hardest.

MIL-DTL-64159B (2015) gloss readings remained the same for all biocide loading levels and after immersion in both fluids (Table 4).

Table 4 Color and gloss

Immersion fluid	Coating	Zinc Omadine ZOE (wt%)	Color change	Gloss change			
			dE	Original gloss 60	d60	Original gloss 85	d85
Deionized Water	64-1	0	0.41	1.3	0.1	1.3	0.1
	64-2	0.40	0.44	1.4	0	1.4	0
	64-3	3.00	0.72	1.6	0	1.4	0
	64-4	0.3/0.3	0.19	1.5	0	1.4	0
JP8	64-1	0	0.15	1.3	0.1	1.3	0.1
	64-2	0.40	0.09	1.4	0	1.3	0
	64-3	3.00	0.30	1.4	0	1.4	0
	64-4	0.3 ZOE/ 0.3 IPBC	0.10	1.4	0	1.4	–0.1

Note: Initial gloss requirements for MIL-DTL-64159B, TAN 686; 60° ≤ 1.6 units and 85° ≤ 4.0 units. No change in gloss allowed after immersion for deionized water or JP8. MIL-DTL-64159 was updated after this testing was performed to replace JP-8 with F-24 for fuel immersion tests.

Fungal tests were performed by ATEC (Aberdeen Test Center 2019) and evaluated for the 28-day incubation period. Fungal growth on test coupons were rated according to the guide shown in Table 5.

Table 5 ASTM D5590 (2017) guide for rating observed fungal growth on specimens

Growth amount	Grade	Organic substrates
None	0	Substrate is devoid of microbial growth
Trace	1	Traces of growth (<10%)
Light	2	Light growth (10%–30%)
Moderate	3	Moderate growth (30%–60%)
Heavy	4	Heavy growth (60% to complete coverage)

Reference: Aberdeen Test Center (2019).

The water-dispersible polyurethane exterior CARC coating (MIL-DTL-64159B 2015), both baseline and weathered controls (no antimicrobial additive), were susceptible to fungal growth and exhibited light to moderate growth as shown in Table 6 and Fig. 1. Generally, MIL-DTL-64159B coatings that contained antimicrobial additives allowed only trace growth, which was limited to the edge of the samples (Table 6 and Fig. 2) suggesting antifungal activity.

Table 6 Observed fungal growth *(A. pullulans)* by week

Code name	Sample	Rating (Week 1)	Rating (Week 2)	Rating (Week 3)	Rating (Week 4)
64-1	1	2	3	3	3
	2	1–2	2	2	2
	3	2	3	3	3
64-2	1	1	1	1	1
	2	1	1	1	1
	3	0	0	0	0
64-3	1	1	1	1	1
	2	1	1	1	1
	3	0	0	0	0
64-4	1	0	0	0	0
	2	0	0	1	1
	3	1	1	1	1
W500 64-1	1	1–2	2	2–3	2–3
	2	2–3	3	3	3
	3	2–3	3	3	3
W500 64-2	1	1	1	1	1
	2	1	1	1	1
	3	1	1–2	1–2	1–2
W500 64-3	1	0	1	1	1
	2	0	1	1	1
	3	0	1	1	1
W500 64-4	1	0	1	1	1
	2	0	1	1	1
	3	1	1	1	1

Reference: Aberdeen Test Center (2019).

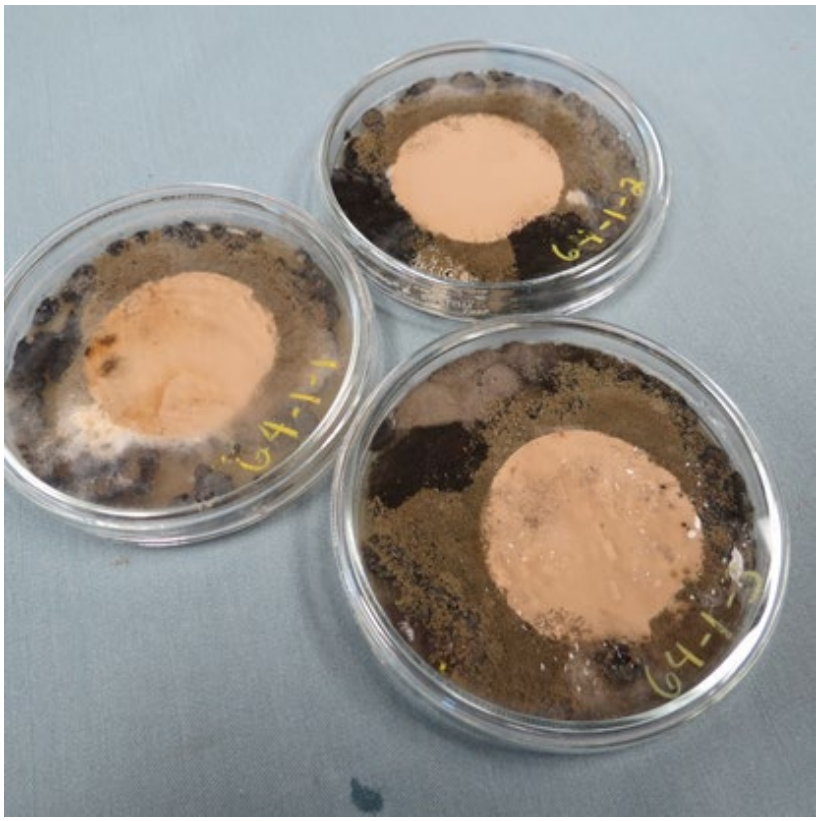

Fig. 1 MIL-DTL-64159B (2015) control coatings (without biocide additive). Trace to light growth was observed, ASTM D5590 (2017) (Aberdeen Test Center [2019]).

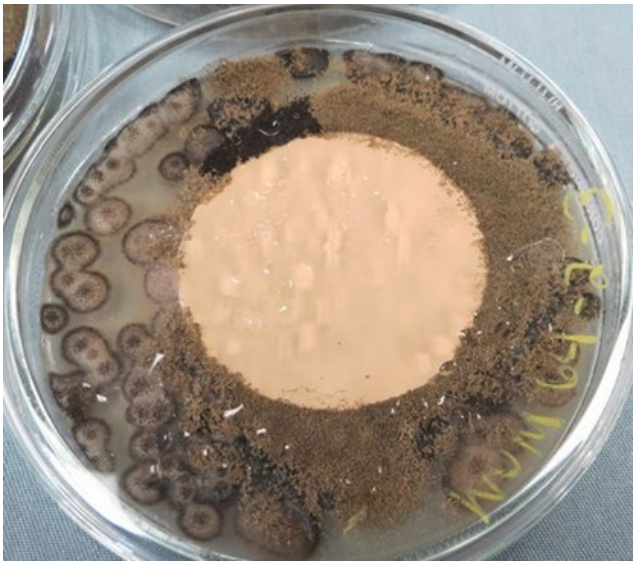

Fig. 2 Light-scattered *Aspergillus* growth over the surface of weathered MIL-DTL-64159B (2015) CARC-coated sample with a 0.4% ZOE dispersion (0.15% active ZPT), ASTM D5590 (2017) (Aberdeen Test Center [2019])

The ASTM D5590 (2017) 28-day fungal test method allows a minimally acceptable incubation time to observe visual fungal growth on CARCs. This could be due to the chemical composition of CARC that may provide some initial microbial resistance necessitating longer fungal exposure times and/or modified test conditions to allow sufficient fungal growth to screen materials. This observation has also been documented in the literature on military coatings similar to CARC (Lavoie et al. 1997; Abbott 2012). Little et al. (2000) reported positive results after 100 days of fungal exposure, and Abbott (2012) was able to accelerate growth over 30 days by increasing relative humidity to 100%. New or modified fungal test methods that accelerate mold growth are needed to provide definitive results in a 28-day test period.

To verify the antifungal properties of ZPT-modified CARC, the panels were also evaluated at the US Army TRTC in Panama and Suriname (Fig. 3) at 1-month intervals for a total of 18 months. Even in the severe tropical environment, approximately 8 months of exposure was required to visually discern mold growth on the CARC panels. Visual appearance of mold was very apparent after 10 months of exposure of the CARC control panels as shown in Fig. 4.

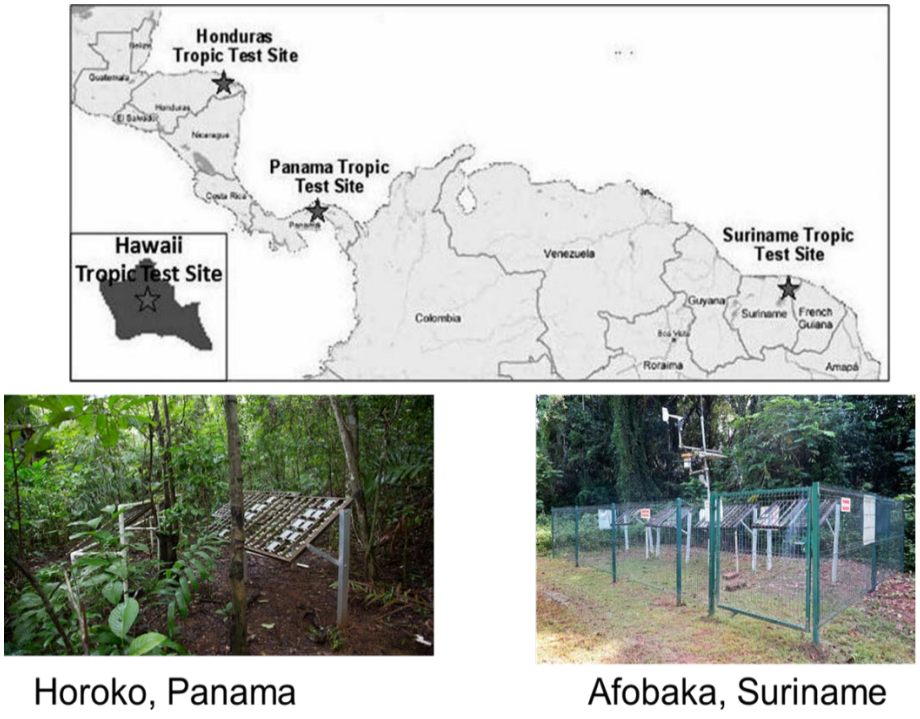

Fig. 3 TRTC outdoor test sites in Horoko, Panama, and Afobaka, Suriname

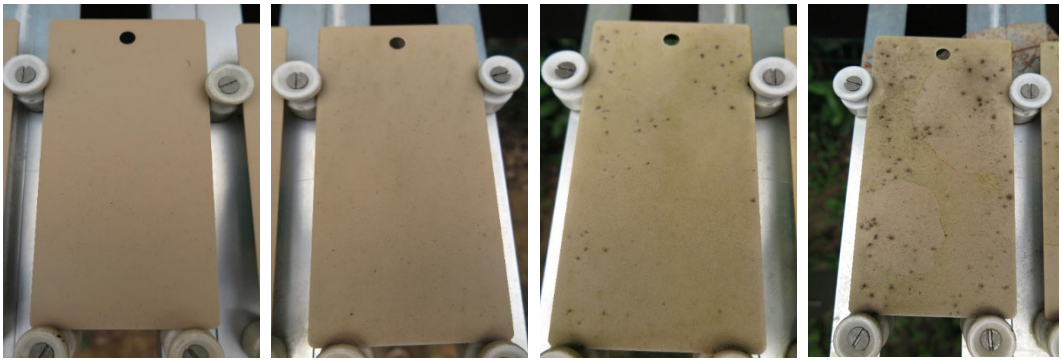

Fig. 4 MIL-DTL-64159B (2015) control (without ZPT) panels after 2, 5, 8 and 10 months (left to right) of outdoor exposure in Afobaka, Suriname

Figures 5–7 show ZPT-modified CARCs after 18 months exposure in Panama and Suriname. The images clearly show that the CARC with the 1.11% active ZPT exhibited significantly improved fungal resistance at both sites. The CARCs with the lower ZPT percentages and the ZPT/IPBC blend showed a slight improvement in fungal resistance compared to the control. The outdoor tropical exposure testing provided confirmation of the antimicrobial properties of ZPT (1.11% active)-modified CARC in a real-world environment. A requirement for antimicrobial CARC has been established in the MIL-DTL-64159C (2022) specification, providing an option for applications where antifungal protection is needed in the field.

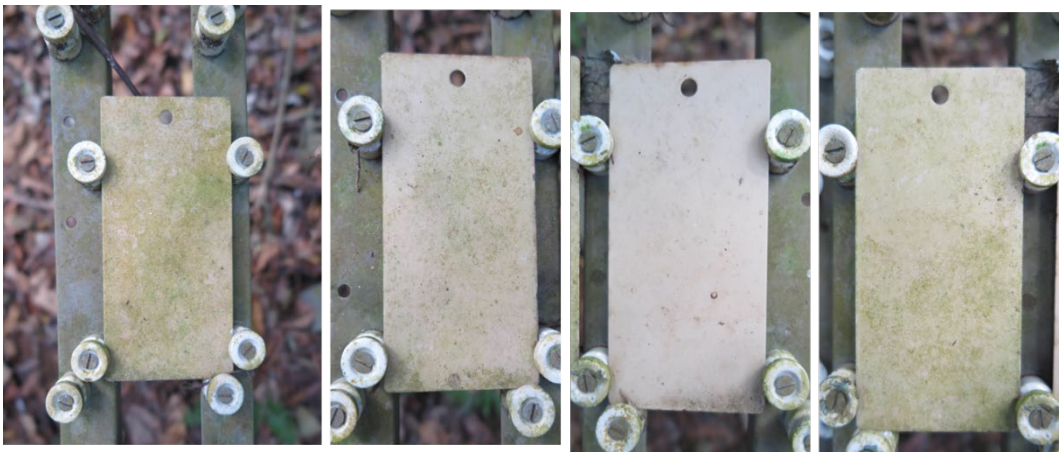

Fig. 5 MIL-DTL-64159B (2015) after 18 months' outdoor exposure in Horoko, Panama. (Left to right) Control 0% ZPT, 0.15% active ZPT, 1.11% active ZPT, 0.11% active ZPT/0.285% IPBC blend.

* All weight percentages are for active ZPT

Fig. 6 MIL-DTL-64159B (2015) full rack of panels after 18 months' outdoor exposure in Afobaka, Suriname

Fig. 7 MIL-DTL-64159B (2015) panels after 18 months' exposure in Afobaka, Suriname. (Left to right) Control 0% ZPT, 0.15% active ZPT, 1.11% active ZPT, 0.11% active ZPT/0.285% IPBC blend.

The Environmental Protection Agency (EPA) registration document, registration number 6836-413, dated November 21, 2019, for Lonza's Zinc Omadine ZOE product used for this study, states on page 4 that for dry film preservation of industrial and non-marine paints, up to 12,760 parts per million (ppm) of the product can be used to inhibit "algae, bacterial slime, mildew and other fungi" (EPA 2019). The 12,760-ppm ZOE dispersion correlates to 4721 ppm of active ZPT for this product. The results described in this DEVCOM ARL study show data for a

low concentration (approximately 1100 ppm) and a higher concentration (approximately 11,000 ppm) of active ZPT in the modified MIL-DTL-64159B (2015) CARC. While the high concentration showed excellent mold resistance, the lower concentration can be increased over four times its current amount and stay within the EPA guideline. It is the view of the authors that active ZPT levels up to the EPA guideline (approximately 5000 ppm) will provide adequate resistance to mold and related microorganisms for MIL-DTL-64159C (2022) CARC.

4. Specification Revision

MIL-DTL-64159C was published on 24 March 2022 with revisions including an antimicrobial requirement. The antimicrobial classification is defined in Section 1.25 Method 2. Antimicrobial requirements are described in Sections 3.4.1.2 Antimicrobial Additive and 3.6.16, Antimicrobial Resistance as follows:

> 3.4.1.2 Antimicrobial Additive:
>
> The additive shall be Zinc Pyrithione and shall not exceed Environmental Protection Agency (EPA) guidelines for non-marine paint and coatings registered uses and shall not exceed 5000 ppm by weight of total paint.
>
> 3.6.16 Antimicrobial Resistance;
>
> When tested as specified in Section 4.4.27, films of the coating shall show no checking, cracking, or appreciable film deterioration. There shall be no more than light microbial growth with a rating of 1 or less in IAW ASTM D5590. The color shall show no excessive change in value and chroma and no change in hue. After removal of any chalking and microbial growth that has occurred the original color shall be substantially restored and the washed area shall show no more than slight fading or darkening. The color shall not exceed 2.5 Delta e when compared to an unexposed sample of the same batch using L*a*b* color coordinates after being cleaned.

The placement of the requirement will enable vendors to submit samples to DEVCOM ARL and pursue verification and validation of their material to be considered for CARC applications where fungal resistance is desired.

5. Conclusions

The use of ZPT (Zinc Omadine ZOE Dispersion) as an antifungal additive to MIL-DTL-64159C clearly demonstrates microbial resistance based on 18 months of outdoor exposure at the TRTC in Central America. This enhancement to MIL-DTL-64159C provides essential protection against mold, algae, and mildew growth on CARC. The use of low-level and relatively benign additives such as ZPT in CARC will greatly add to the availability and implementation of antifungal CARC products for the DOD.

The use of the zinc-based additive also provided additional color stability and durability at low-level concentrations that may provide enhancements to key requirements such as chemical agent and corrosion resistance.

The ability to minimize growth of mold and algae will be beneficial to reduce the labor and upkeep of Army vehicles and may reduce the need for caustic cleaning solutions currently used to mitigate mold, algae, and mildew growth and enable the use of more benign cleaners.

6. Future Work

MIL-DTL-53039E (2018), a non-water-based chemical agent resistant topcoat and MIL-PRF-22750G (2019), an interior epoxy coating, are excellent choices to further expand the use of antimicrobial-based coatings. MIL-DTL-53039E is used extensively throughout the DOD and MIL-PRF-22750G is the primary interior coating for all Army and US Marine Corps assets. Mold, mildew, and algae have been documented on exterior and interior CARC. Further work to revise these specifications would provide comprehensive fungal resistance on CARC assets. Additionally, a field demonstration of antimicrobial CARC technology is being planned.

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List of Symbols, Abbreviations, and Acronyms

ARL	Army Research Laboratory
ATEC	Army Test and Evaluation Command
CARC	chemical agent resistant coating
CCDC	US Army Combat Capabilities Development Command
CWAs	chemical warfare agents
dE	Delta E
EPA	Environmental Protection Agency
gm	gram
IPBC	iodopropynyl butyl carbamate
MRAP	mine-resistant ambush-protected
TRTC	Tropic Regions Test Center
ZPT	zinc pyrithione

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