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Nanoengineered Additives for Active Coatings

**by Adam M. Rawlett, Joshua A. Orlicki, John J. La Scala, Lars T. Piehler,
Nicole Zander, Pauline M. Smith, J. Derek Demaree, Wendy E. Kosik,
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and Arjan Giaya**

ARL-TN-273

April 2007

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Army Research Laboratory

Aberdeen Proving Ground, MD 21005-5069

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REPORT DOCUMENTATION PAGE				Form Approved OMB No. 0704-0188	
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1. REPORT DATE (DD-MM-YYYY) April 2007		2. REPORT TYPE Final		3. DATES COVERED (From - To) October 2005–September 2006	
4. TITLE AND SUBTITLE Nanoengineered Additives for Active Coatings				5a. CONTRACT NUMBER	
				5b. GRANT NUMBER	
				5c. PROGRAM ELEMENT NUMBER	
6. AUTHOR(S) Adam M. Rawlett, Joshua A. Orlicki, John J. La Scala, Lars T. Piehler, Nicole Zander, Pauline M. Smith, J. Derek Demaree, Wendy E. Kosik, Steven H. McKnight, Norman Rice,* Lawino Kagumba,* and Arjan Giaya*				5d. PROJECT NUMBER H76	
				5e. TASK NUMBER	
				5f. WORK UNIT NUMBER	
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) U.S. Army Research Laboratory ATTN: AMSRD-ARL-WM-MA Aberdeen Proving Ground, MD 21005-5069				8. PERFORMING ORGANIZATION REPORT NUMBER ARL-TN-273	
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)				10. SPONSOR/MONITOR'S ACRONYM(S)	
				11. SPONSOR/MONITOR'S REPORT NUMBER(S)	
12. DISTRIBUTION/AVAILABILITY STATEMENT Approved for public release; distribution is unlimited.					
13. SUPPLEMENTARY NOTES *Triton Systems Inc., 200 Turnpike Rd., Chelmsford, MA 01824					
14. ABSTRACT Novel additives for polymeric coatings have been developed based on modified hyperbranched polymers. The additives are polyfunctional and have been prepared to spontaneously segregate to the surface of the polymer in which they are dispersed. They have been used to install “active” sites in polymeric films, with little or no change in the formulation, bulk properties, or application of the coating.					
15. SUBJECT TERMS coating, additive, self-segregation, biocide, antimicrobial					
16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT UL	18. NUMBER OF PAGES 18	19a. NAME OF RESPONSIBLE PERSON Adam M. Rawlett
a. REPORT UNCLASSIFIED	b. ABSTRACT UNCLASSIFIED	c. THIS PAGE UNCLASSIFIED			19b. TELEPHONE NUMBER (Include area code) 410-306-0695

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Acknowledgments

The authors would like to thank the U.S. Army's Institute of Soldier Nanotechnologies at the Massachusetts Institute of Technology 6.2 research program for funding.

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1. Introduction

Modern coating systems are challenged to meet a large number of performance characteristics. In the commercial realm, for instance, automotive paint must provide excellent corrosion and solvent resistance while maintaining high gloss and luster. Analogs for military coatings provide similar corrosion protection while minimizing gloss and providing for camouflage schemes.

The next step in the evolution of coatings technology is introducing active sites to a painted surface. Many interesting technologies for surface modification exist in the realm of polymer chemistry, but few of them are amenable for use on a very large scale. Techniques such as chemical vapor deposition or atmospheric plasma treatment can effectively treat surfaces with exquisite control, but are often limited by substrate geometry and sample size. One notable, scaleable technique employs self-assembled monolayers, but questions arise about the robustness and the stability of the system, and, therefore, it is not suitable for real-world use.

One potentially practical route to developing modified coating systems employs self-segregating additives, as illustrated in figure 1. The image details the difference in distribution between an additive in bulk and one concentrated at the surface. Surface segregation is attractive for coatings, as it reduces the total quantity required to achieve high concentration at the surface and minimizes the impact of the additive on the bulk properties of the coating. The additives detailed herein have been applied to a range of model polymer and real-world coating systems and demonstrate the formation of an “active” surface using standard application techniques.

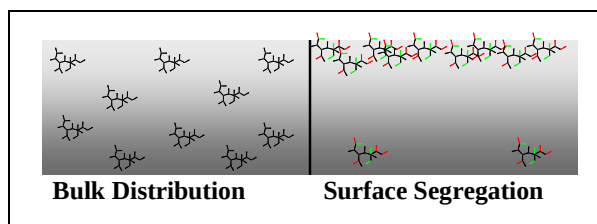


Figure 1. Illustration of bulk and surface distributions.

2. Experimental Details

Standard chemicals were obtained from Alfa Aesar and were used as received, unless otherwise noted. BASF Corp. provided the hyperbranched polyethyleneimine (PEI), and Perstorp, Inc. provided the hyperbranched polyester (PE). The perfluorinated components were either donated by DuPont or purchased from Exfluor Research. Resolution Performance Products provided the aliphatic epoxy components. Noveon provided thermoplastic polyurethane (TPU).

2.1 Representative Synthetic Procedure

(Note that alternative routes were employed for the PE and PEI systems, and only one route is described for illustration). A PEI-hyperbranched-polymer (HBP) core (~46 g, 418.2-mmol 1° amines) was suspended in dry tetrahydrofuran (THF) (150 mL) in a 500-mL round bottom flask under dry nitrogen. Freshly prepared perfluorooctanoyl chloride (26.9 g, 62.3 mmol, prepared from perfluorooctanoic acid) was added via syringe over several minutes. Within 15 min, the milky turbid suspension of PEI had formed a clear yellow solution, to which aliphatic epoxy was added (34.74 g, 120.3 mmol). The reaction was stirred for 16 h at room temperature, and the solvents were removed by rotary evaporation. The resultant viscous polymer was dissolved in methanol, transferred to a tared container, and dried under heat (50 °C) and vacuum. This polymer began with an equivalent weight of 1° amines of ~110 g/mol; the final product contained residual 1° amines with an equivalent weight of ~454 g/mol. To prepare the quaternary ammonium salt analog, a two-step procedure was used. The polymer was first treated with a bromo-alkyl acid chloride (room temp, dry THF) to install a halo-alkyl chain end via an amide linking group. The second reaction was performed in dry DMAc by combining the polymer with N,N-dimethyldecyl amine, at 80 °C for 48 hr. The two steps proceeded in quantitative yield and resulted in a glassy hygroscopic material with good solubility in methanol.

2.2 Film Preparation and Coating Analysis

To test the additive performance in polymer systems, a TPU was chosen as a proxy for commercial coatings. Model formulations were obtained by combining a solution of the TPU with a solution of the additive. The formulations were then cast on a substrate (typically a glass microscope coverslip [18 × 18 mm] or a half-microscope slide) and dried in a controlled environment. Dried films were evaluated using contact-angle analysis. Near surface compositional depth profiling was performed using the Kratos Ultra* x-ray photoelectron spectroscopy (XPS) system, equipped with a hemispherical analyzer. A 100-W monochromatic Al_{Kα} (1486.7-eV) beam irradiated a 1- × 0.5-mm sampling area. Survey scans were taken at pass energy = 80 eV. Elemental high resolution scans for C1s, O1s, N1s, and F1s were taken at pass energy = 20 eV. Kratos Ultra software was utilized for all data analysis—linear background subtraction, curve fitting, peak integration, and charge compensation.

The activity of modified coating systems was evaluated using techniques according to ASTM E 2180¹, a method developed for examining hydrophobic coatings. In this method, a quantity of microorganisms (~10⁶ organisms) is loaded onto a substrate (e.g., TPU-coated half-microscope slide) with an agar slurry as the inoculum vehicle. The antimicrobial coatings are prepared at 1% by weight in a TPU polymer matrix (Noveon X-1150). The ASTM E 2180 test is run in triplicate for the 24-hr time points for controls and test samples and is run in duplicate for the 0-hr controls.

* Ultra is a trademark of Kratos Analytical.

¹ASTM E 2180. Standard Test Method for Determining the Activity of Incorporated Antimicrobial Agent(s) in Polymeric or Hydrophobic Materials. *Annu. Book ASTM Stand.* **2001**.

3. Results and Discussions

Several HBP systems were evaluated to see if they were suitable for the modification technology developed by the U.S. Army Research Laboratory (ARL). Two routes of modification have previously been identified by ARL: route 1 involves nucleophilic substitution of amines via Michael addition and epoxide-opening chemistry, and route 2 involves melt condensation of an acid and alcohol. After the initial functionalization, the HBPs were further functionalized using acid chlorides. This was followed by quaternization using N,N-dimethyl alkyl amines of varying lengths, with chain lengths of hexyl (C_6), decyl (C_{10}), and hexadecyl (C_{16}) under evaluation. One other route attached a biguanide to the periphery of the HBP. The secondary modification of the HBP cores is shown as route 3 in figure 2. Compositions for the prepared compounds can be found in table 1, where extent of modification is expressed as a fraction of chain ends consumed by the indicated functional group. The bis-biguanido hexane used in this study is shown in figure 3.

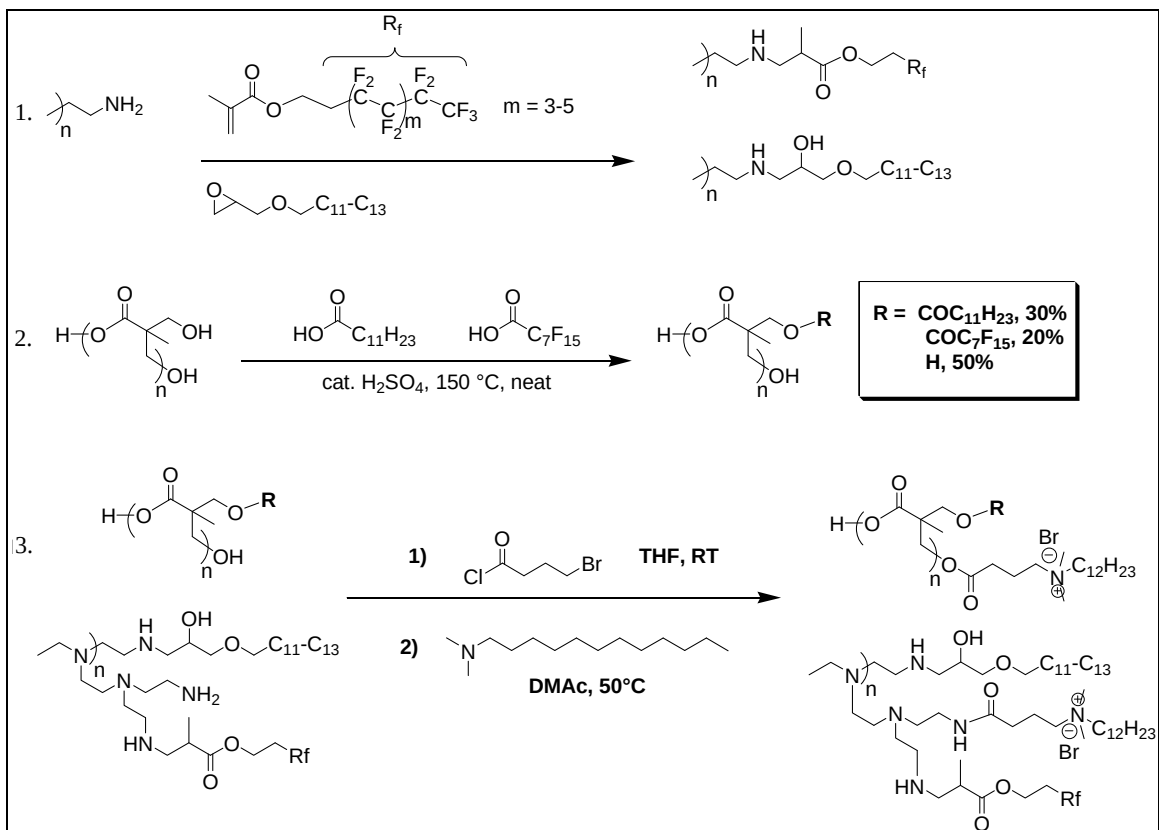


Figure 2. Synthetic routes for HBP modification.

Table 1. End group composition of HBP-based additives.

Sample	Base HBP	Mole Fract Perfluoro	Mole Fract Alkyl	Mole Fract Ammonium or Biguanide
ARL 1	PEI	0.2	0.4	0.0
ARL 2	PE	0.3	0.2	0.0
TSI 1	PEI (ARL 1)	0.2	0.4	0.4 (C ₁₀ quat)
TSI 2	PEI (ARL 1)	0.2	0.4	0.4 (biguanide)
TSI 3	PE (ARL 2)	0.3	0.2	0.5 (C ₁₀ quat)

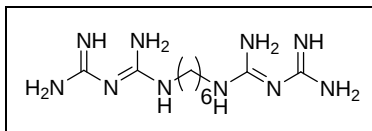


Figure 3. Bis-biguanide with C₆.

These materials were characterized by Fourier Transform infrared spectroscopy (observed consumption of the methacrylate functionality by ~10 wavenumber shifts in carbonyl absorption) and ¹H/¹³C nuclear magnetic resonance (NMR). NMR confirmed the ring opening of the epoxide groups, and ¹³C NMR clearly showed the installation of the amide chain ends. Unfortunately, NMR was not well suited for identifying the perfluoro chains—the low mole fraction incorporated in the HBPs and the splitting of ¹H and ¹³C by the fluorine made interpretation of spectra difficult.

The base HBPs (ARL 1 and 2) demonstrated good overall solubility in common organic solvents. The quaternary ammonium salts maintained fair solubility in most solvents tested and exhibited better water solubility than the corresponding core polymer. The biguanide-containing material required more polar solvents to achieve full solvation, dissolving most easily in polar protic solvents (e.g., water, methanol).

The HBPs were tested for their ability to influence polymeric film surface properties using a model TPU system. The only “curing” mechanism involved in a TPU film formation is the gradual loss of solvent as it evaporates. The model systems, though, allow for careful study of film properties in the absence of competitive reactions, with fewer questions about kinetics. The model systems were used to demonstrate the surface migration properties of the additives by preparing 1% additive formulations in dilute solution, using industrially applicable solvents (e.g., 2-butanone or methyl-ethyl ketone [MEK]). The films were cast onto glass substrates and dried under a solvent-saturated atmosphere, which lengthened drying times and generated thick films with marginal application defects.

The surface properties of the films were surveyed using contact-angle analysis, a technique that characterizes the hydrophobicity or hydrophilicity of a surface based on its wetting characteristics. Several solvents are suitable for contact-angle analysis, but the most widely employed is pure water. A graph of contact-angle measurements is shown in figure 4,

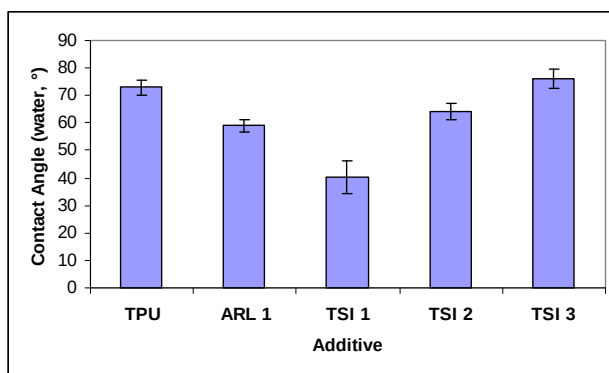


Figure 4. Contact angle as function of additive, 1% loading.

where the influence of the additives is manifested as change in the observed hydrophilicity. This behavior is not observed in instances where the HBP does not migrate to the surface, as the properties of the TPU continue to dominate interactions. Because the base material ARL 2 can cause dewetting attributable to its strong influence on interfacial properties, that data is not presented. In alternative TPU systems, though, it demonstrates a significant influence over observed surface characteristics.

In addition to the contact-angle results, XPS analysis was used to profile the surface compositional profile of the films. Increases in surface levels of fluorine and nitrogen are typically observed if the PEI-based systems have migrated to the surface, while fluorine is the only indicator for the PE systems. Table 2 illustrates the compositional profiles observed. The levels of fluorine observed at the surface are quite high, considering the bulk quantities of fluorine in the films.

Table 2. Surface composition of films.

System	C	F	N	O
TPU	70.97	0.00	5.50	23.53
ARL 1	68.24	6.95	9.93	14.89
TSI 1	72.98	2.80	7.14	15.85
TSI 2	74.98	0.10	5.51	19.33
TSI 3	74.40	3.09	4.05	18.17

The model TPU systems were also examined with respect to their resistance to microbial challenges. Three organisms were selected to represent gram positive (*S. aureus*), gram negative (*E. coli*), and fungi (*C. albicans*), which provided a profile of broad spectrum activity. The results of this survey are provided in table 3, and they demonstrate that the additives maintain substantial activity when incorporated into the TPU at 1% loading based on total solids.

Table 3. Efficacy of 1% additive in TPU matrix based on 6-log loading of organism.

HBP	Reduction C. Albicans	Reduction E. Coli	Reduction S. Aureus
ARL 1	—	<1 log	<1 log
ARL 2	—	1 log	<1 log
TSI 1	6 log	4 log	6 log
TSI 2	<1 log	3 log	6 log
TSI 3	—	2 log	<1 log

While the results obtained in the TPU system were encouraging, there remained questions about the source of antimicrobial activity. One key attribute for preparing a persistent active surface is preventing leaching of the active component. Several commercial additives may be incorporated into a coating to grant it antimicrobial activity, but almost all rely on gradual release of the active component from the coating, leading to eventual depletion of the film. Small TPU samples were evaluated using a Kirby-Bauer test to probe for leaching of the active components. In the test, a sample of the film is placed on a mat of microbes (*E. coli* and *S. aureus*), and as the antimicrobial component diffuses from the substrate, it generates a zone of clearing around the film via microbial death. For a TPU sample containing a small molecule quaternary ammonium salt, a zone of clearing of a couple of millimeters was observed. There was no observed leaching of the HBP-based additives.

While the model systems just described have been informative in evaluating the HBP-based additives, the TPU-based materials are not likely to yield commercially useful or viable systems. True sealants or coating systems employ reactive constituents, which cure or undergo spontaneous reactions to generate robust finish. One point of concern for the HBP-based additives has been the potential to react prematurely with the coating, preventing migration or resulting in tremendous shifts in the performance of the coatings.

To test the influence of the additives on viable commercial coating systems, the HBPs were incorporated into formulations based on polyurethanes (PU), polyureas (PUr), and epoxy systems. They were formulated to provide an ultimate composition of 1% additive, based on the total solids content. The additives were dissolved in a solvent (generally, methanol or MEK) prior to mixing to enable their dispersal in the coating formulation. Samples were prepared by spray or draw-down application to give a dry final film thickness of 1–2 mil (~25–50 μm). The films were cured at room temperature and evaluated using contact-angle analysis and XPS. The film properties of the resultant coatings were also investigated.

Figure 5 illustrates the influence of the additive on the surface properties (wetting characteristics) of the resultant films. As was observed in the TPU systems, the coatings exhibited a general decrease in contact angle with water, though the magnitude was much smaller for the crosslinked systems than for a low T_g thermoplastic matrix. While the increased hydrophilicity at the film surfaces suggests the migration of the HBPs, XPS can provide complementary compositional information.

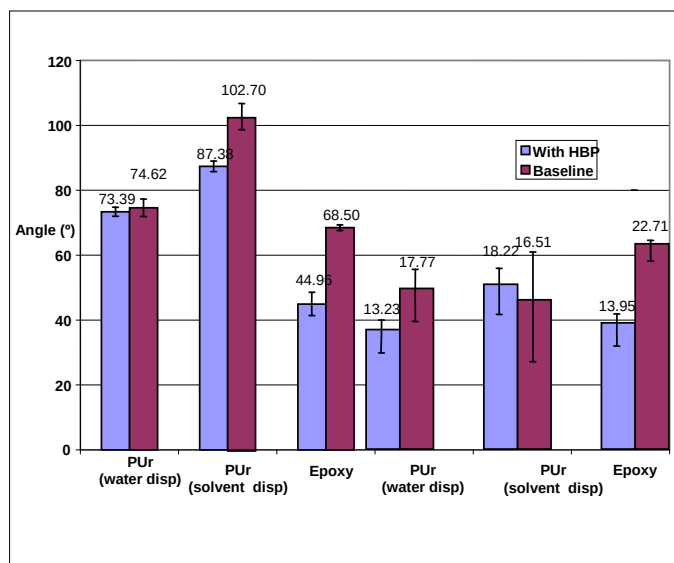


Figure 5. The advancing and receding contact angle for samples with (1 weight-percent) and without PEI-quat HBPs.

Table 4 shows the measured elemental compositions of the formulations. The increase in fluorine levels at the film surfaces is interpreted as evidence of migration of the additives to the surface of the films and correlates well with the observed contact-angle results.

Table 4. XPS results showing the elemental atomic percent of chemicals at the surface for the various military coatings with and without HBP.

Sample	Composition (atomic-percent)			
	C	O	N	F
Epoxy baseline	82.05	14.36	3.29	0.30
Epoxy TSI 1	74.69	16.37	5.55	3.39
Epoxy TSI 3	77.15	16.12	3.62	3.11
PUr (water dispersible) baseline	72.54	19.95	5.24	ND
PUr (water dispersible) TSI 3	70.32	16.20	7.04	6.45
PUr (solvent dispersible) baseline	78.79	12.28	7.41	ND
PUr (solvent dispersible) TSI 3	71.53	14.79	5.15	8.53

Note: ND = not detected.

The motivating factor behind the use of self-segregating additives to modify coating systems was to minimize the impact they have on the bulk properties of the matrix. Several physical characteristics of the cured coating systems were investigated, including T_g and loss modulus.

The differential scanning calorimetry (DSC) traces used to determine T_g values are presented in figure 6. These demonstrated that there was little influence of the additive on the thermal properties of the coating. This was anticipated—HBPs typically exhibit very small T_g

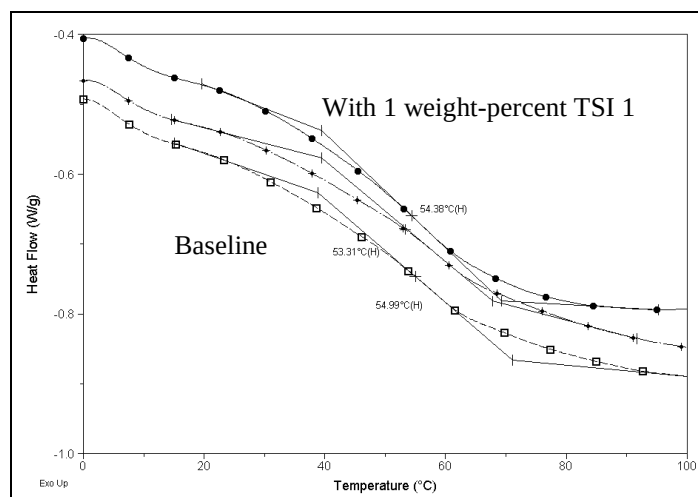


Figure 6. The DSC traces for PUr (water dispersible) with and without HBP.

transitions, so observing the additive's contribution to the thermal profile would be very difficult. The alternative would have been to overall lower the matrix T_g , if the additive acted as a plasticizer.

The influence of the additives on the overall film appears slight (measured using glass transition temperature and loss modulus [figure 7]). The films were also evaluated for their efficacy as biocidal surfaces using the ASTM E 2180 technique. In these experiments, summarized in table 5, the additives are less effective in cross-linked systems for the overall biocidal activity. This eventuality was not completely unforeseen—the cross-linked nature of the PU/PUr/epoxy films leads to decreased mobility for the additives at the film surface.

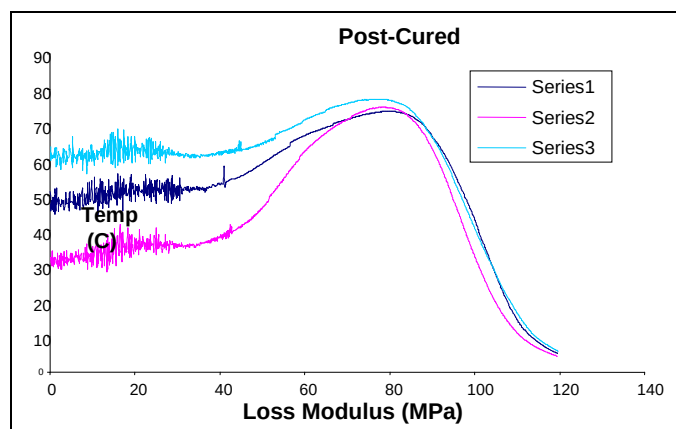


Figure 7. The loss modulus for PUr (solvent dispersible) for the baseline (blue), 1 weight-percent TSI 1 (pink), and 1 weight-percent TSI 3 (light blue).

Table 5. Summary of 24-hr ASTM E 2180 tests with 1 weight-percent additive in PUr (solvent dispersible) based on 6-log loading of organism.

HBP	Reduction C. Albicans	Reduction E. Coli	Reduction S. Aureus
TSI 3	2 log	1 log	6 log

4. Conclusions

There is a tremendous need to improve coating systems by incorporating reactive moieties that can actively decontaminate various environmental pathogens. The objective of this effort was to develop affordable nanotechnology-based solutions that can be directly integrated into polyurethanes used by the Army while maintaining all military-specified properties. This was accomplished through tailored modification of the surface of the coating by self-assembling nanoparticulate additives resulting in local changes in surface properties. The Army presently spends over \$100 million on chemical-agent-resistant-coating topcoats annually. Improved performance and reduced coating costs will have widespread and significant impact on Army materiel.

The technology presented here represents a significant advancement over known systems in that it (1) combines the polyfunctional nature of nonentangling additives, such as HBPs, to provide a tailored material that (2) is soluble or dispersible within a paint system, (3) migrates to the air interface, (4) transports active antimicrobial or decontaminating agents to that interface, (5) chemically reacts with the polymeric binder of the coating to prevent leaching of the active agent, and (6) is useable in various types of coatings, including epoxy primers, polyurethane topcoats, and latex systems. A strong potential exists for using traditional coatings materials with a small quantity of additive providing substantial antimicrobial and decontaminating activity. Furthermore, these results indicate that self-segregating additives have the ability to transport a myriad of active materials to the air/polymer surface of a coating; thus, acting as a “universal transport vehicle.” These materials may also have potential use in coated fabrics, latex paints, etc. for military and civilian applications.

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