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MMM-A-121 Federal Specification Adhesive, Bonding Vulcanized Synthetic Rubber to Steel HAP-Free Replacement

by Faye R. Toulan, Christopher Stabler, John J. La Scala, Hank Feuer, Dave Flanagan, and Paul Touchet

ARL-TR-4715 **February 2009**

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Weapons and Materials Research Directorate, ARL

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14. ABSTRACT

The U.S. Army uses numerous adhesives and sealants, among other coating materials, that contain significant amounts of hazardous air pollutants (HAPs). This work examines laboratory testing of two of the most highly used adhesives meeting MMM-A-121 specifications throughout the Army.
The scope of MMM-A-121 involves adhesives used in bonding vulcanized synthetic rubber to steel. The two most commonly used baseline products meeting the MMM-A-121 specification are 3M-1357 Scotch-Weld and 3M-1300L Scotch-Weld, which contain the following HAPs: petroleum distillate, methyl ethyl ketone, and toluene. The Sustainable Painting Operations for the Total Army program has identified 3M Scotch-Weld 847 (containing acetone, an exempt solvent) as an alternative to current adhesives conforming to MMM-A-121. Other potential materials such as 3M Fast-Bond and 30NF 3M-4491 Scotch-Grip did not have the required adhesive properties.
Various testing was done in the laboratory to determine the relative similarity of the baseline products to the potential alternative materials. This testing included solids content, rheology, dry time, and adhesion strength. Overall, this alternative material should reduce the Army HAP emissions by ~1300 lb/year and volatile organic compound emissions by ~1200 lb/year.

15. SUBJECT TERMS

adhesive, HAP-free, bonding, vulcanized rubber, strip adhesion, cohesive failure, SPOTA, rheology, non-Newtonian, VOC

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1. Introduction

The Environmental Protection Agency is in the process of mandating the Defense Land Systems and Miscellaneous Equipment National Emission Standard for Hazardous Air Pollutants (NESHAP) that will affect U.S. Army surface-coating operations (*1*). Significant quantities of adhesives and sealants containing hazardous air pollutants (HAPs) were identified as part of National Defense Center for Energy and Environment (NDCEE) Task No. 000-08, Sustainable Painting Operations for the Total Army (SPOTA), and NDCEE Task No. 325 (*1*). As part of these efforts, the NDCEE surveyed 14 Army installations that were identified as major HAP-emitting installations (*1*). The previous efforts documented all uses of these adhesives and sealants, categorized them according to their class, and highlighted areas of most concern, based on consumption volumes. Originally, the NDCEE-SPOTA team identified more than 1000 miscellaneous coatings and adhesive materials. The Army has determined that it is more cost-effective to reduce or eliminate HAP emissions from coatings operations rather than using emissions control devices to capture and treat them (*2*). Therefore, the goal of the SPOTA program is to severely reduce the amount of HAP emissions produced in coatings operations, including adhesives and sealant application and removal.

Following two down selection activities, the number of applicable coatings and adhesive materials was reduced. Once down selection activities were complete, the NDCEE researched and identified potential commercial-off-the-shelf alternative materials for the baseline materials. (Baseline is defined as the approved product currently used that meets necessary federal specifications for MMM-A-121, which can be found in the qualified products list.)

The scope of federal specification MMM-A-121 involved adhesives used in bonding vulcanized synthetic rubber to steel (*3*). The two most commonly used baseline products under the MMM-A-121 specification were 3M-1357 Scotch-Weld* Neoprene High Performance Contact Adhesive containing petroleum distillate, methyl ethyl ketone (MEK) and toluene (*4*), and 3M-1300L Scotch-Weld Neoprene High Performance Rubber and Gasket Adhesive containing petroleum distillate, acetone, MEK, toluene, and n-hexane (*5*). A possible HAP-free alternative product was identified as 3M-847 Scotch-Weld Nitrile High Performance Rubber and Gasket Adhesive (*6*) containing acetone (an exempt solvent) (*7*). Testing was needed to ensure performance, compatibility, and compliance to MMM-A-121. Two additional products were added to this series for testing based on a claim of low or HAP-free content. These products were 3M-4491 Scotch-Weld Nitrile Industrial Adhesive containing acetone and cyclohexanone (*8*) and 3M-30NF Fastbond Contact Adhesive containing primarily water (*9*). Only the two baseline adhesives claimed compliance with the requirements of MMM-A-121.

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*Scotch-Weld is a trademark of 3M.

This report summarizes the testing, performance, and compatibility of the products in table 1 to federal specification MMM-A-121.

Table 1. Products in test series.

Product Name	HAP (Weight-Percent)	VOC (g/L)
3M-1357 (baseline)	16–19	490
3M-1300L (baseline)	13–18	629
3M-847	0	0
3M-4491	0	43
3M-30NF	4–5	37

2. Experimental Method

2.1 Rheology

The viscosities of the wet adhesive samples were measured using a TA Instruments (New Castle, DE) AR2000 rheometer in steady shear flow experiments using a cross-hatched parallel plate geometry (40-mm plate) with peltier, a solvent trap containing ethanol, and a temperature of 20 °C. The purpose of the solvent trap was to keep samples from volatilizing during the experiment and skinning at the edges of the plate which would result in drag or uneven flow. The shear rate was increased from 10^{-5} s^{-1} to 1 s^{-1} and then decreased back to 10^{-5} s^{-1}, and 10 measurements were taken per decade. At a given shear rate, the shear stress was measured every 2 s. The shear rate and viscosity were recorded when the shear rate stabilized to within 5% tolerance for three consecutive intervals.

2.2 Nonvolatile Content (Solids)

A suitable container was weighed, and ~10 g of thoroughly mixed adhesive was poured into the tared container, covered, and weighed. After removing the cover, the container was placed in an oven at 70 ± 1.1 °C (158 ± 2 °F) until the sample reached a constant weight. The covered container with the sample was cooled to 23 ± 1.1 °C (73.5 ± 2 °F) before weighing. Each sample was run in duplicate (*10*).

2.3 Dry Time - ASTM D 1640 03

Adhesive was applied on a glass plate to a uniform thickness of 4 mil at room temperature (*11*). The tackiness of the samples was then measured periodically with a wooden dowel using an industry standard “touch-test” as a function of time until the sample was no longer tacky. The time required for the sample to become tack-free was recorded as a range rather than a single value. Note that when the product does not transfer to the dowel and does not deform the film, the product is dry or tack-free (*10*).

2.4 Strip Adhesion - MMM-A-121

The rubber gasket materials used for this test were prepared as specified in MMM-A-121 (*3*). Three classes of rubber substrates (1-neoprene, 2-SBR, and 3-nitrile) were formulated and prepared by the Multifunctional Materials Branch. Due to raw material limitations accounting for the fact that the MMM-A-121 specification is over 40 years old, the rubber compounds specified in MMM-A-121 (*3*) were slightly reformulated. The dimensions of the molded rubber substrates were 1 × 6 × 1/4 in. All of the rubber substrates were hand sanded using a course grit paper and cleaned with acetone prior to adhesive application. This method was the pivotal performance test for compliance with MMM-A-121.

The requirements for this federal specification were tested as follows (*3*). The sheet steel panels to which the rubber strips were bonded were cold-rolled, commercial quality. The strips of rubber material previously described were bonded to steel panels, 3 × 6 × 0.032 inches in dimensions (figure 1). The steel panels were cleaned with acetone immediately prior to bonding. When the cleaning solvent had evaporated completely, one brush coat of the adhesive material was applied to the prepared surfaces of the rubber strips and panels. Release tape (*10*) was used to create a consistent start point of ~2 in for all the strip adhesion panels. The adhesive was allowed to dry according to the MMM-A-121 requirements listed in table 2. Immediately after the strips were bonded to the panels, they were rolled down with six single passes of a 10-lb roller, 2 in wide, requiring about 2 s per pass. The panels with the bonded strips were conditioned and tested, as shown in table 3. Strip adhesion tests were conducted in triplicate on specimens prepared from rubber gasket materials from classes 1–3 for each of the following test conditions:

1. Wet adhesion before and after aging the adhesive for 2 weeks at 49 ± 1.1 °C (120 ± 2 °F).
2. Initial adhesion.
3. Adhesion after immersion in salt water solution.
4. Adhesion at 60 ± 1.1 °C (140 ± 2 °F).

The rubber strips were used only once for the adhesion tests.

Table 2. Rubber compounds for MMM-A-121.

Ingredients, PPHR	Neoprene (Class 1)	SBR (Class 2)	Nitrile (Class 3)
Neoprene WRT or Bayprene 110 polychloroprene rubber	100.00	—	—
DSM Copo 1500 SBR	—	100.00	—
Paracril B rubber or Nipol 30-8, 30% ACN nitrile rubber	—	—	100.00
Magnesium oxide, Maglite D or Elastomag 170	2.00	—	—
Zinc oxide	5.00	5.00	5.00
Stearic acid	2.00	2.00	1.00
Wingstay 100AZ, mixed diaryl p-phenylenediamine antioxidant	2.00	—	—
Agerite resin D, antioxidant DQ, (2,2,4-trimethyl-1,2 dihydroquinoline antioxidant)	—	2.00	2.00
N-990 carbon black	10.00	20.00	91.50
Statex B black replaced with N-330 carbon black	—	20.00	—
Atomite whiting, Hubercarb Q325, calcium carbonate	60.00	70.00	27.70
Dixie clay, HC-100 clay	45.00	—	—
Paraffin wax, IGI 1231A	2.00	—	—
Solid petrolatum, SR-172 petro	2.00	—	—
Akrosorb 19627 HC resin P10 (72%)	—	—	28.00
Dibutyl phthalate, DBP plasticizer	—	—	10.00
Sundex 790 or Circo light process oil	15.00	25.00	—
Spider sulfur MC-98	—	2.00	1.50
ETU – 75%, ethylene thiourea accelerator	0.75	—	—
Methyl tuads, tetramethylthiuram disulfide accelerator	—	0.40	0.80
Formula Weight	245.75	246.40	267.50
Cure time at °F, min (sheets/ross)	40/45 @ 320 °F	15/20 @ 310 °F	10/15 @ 310 °F
Monsanto rheom.2000E, max torque, lb-in @ 310 °F	10.53	9.1	8.53
Scorch ts2, min	4.98	4.49	3.99
Mh95, lb-in	10.02	8.67	8.15
Mfin., lb-in	10.52	8.89	8.5
Torque difference, Mfin-Mh95	0.5	0.22	0.35
Peak rate, lb-in/min	0.7	1.6	2.9
Original Properties			
Tensile strength, psi	1196.9	1213.6	1450.7
100% modulus, psi	169.8	156.5	199.6
200% modulus, psi	201.8	212.4	335.6
Elongation, %	670.5	676.6	791.0
Hardness, shore A, points	45	45	46
Bashore rebound, %	24	42	28
Die C tear, lb/in	109	144	230
Specific gravity	1.6357	1.3326	1.3391

Note: SBR = styrene butadiene rubber, ACN = acrylonitrile content, and DBP = dibutyl phthalate.

Figure 1. Basic assembly for strip adhesion.

The panels with the bonded strips were conditioned and tested, as shown in table 3. The following letter designations were used:

- L – Dead weight load of 2.5 lb/ft^2 per square inch of rubber gasket areas applied as a loading pressure on the strips bonded to the steel panel, condition at 23 ± 1.1 °C (73.5 ± 2 °F).
- R – Rest time under no load at 23 ± 1.1 °C (73.5 ± 2 °F).
- I – Specimens immersed in salt water (5% sodium chloride), under no load at 23 ± 1.1 °C (73.5 ± 2 °F).
- T – Tests conducted at 23 ± 1.1 °C (73.5 ± 2 °F) within 1 hr after end of conditioning period, except where otherwise indicated.
- T1 – Tests conducted at 60 ± 1.1 °C (140 ± 2 °F).

Table 3. Conditioning and testing schedule.

	Elapsed Time After Assembly (hr)				
Strip Adhesion Test	**1.0 ± 0.1**	**0 to 48**	**48 to 120**	**120 to 144**	**144**
Wet adhesion	T	—	—	—	—
Stability (wet adhesion)	T	—	—	—	—
Initial	—	L	R	R	T
After immersion	—	L	I	R	T
At 60 °C	—	L	R	R	T1

2.4.1 Procedure for Determining Strip Adhesion Wet, Initially, and After Immersion

The tests for wet adhesion before and after aging of the adhesive, initial adhesion, and adhesion after immersion were conducted on the assemblies prepared, as specified in table 3. The steel panel was supported on the sides in a horizontal position. One end of the bonded rubber strip was separated from the metal panel for a distance of about 2 in. The weight specified in table 4 was suspended from the free end of the rubber strip (figures 2 and 3). The weight was allowed to act on the strip for 3 min, and the average distance of stripping of the specimen from the panel under the influence of weight was recorded.

Table 4. Weights for strip adhesion.

Strip Adhesion Test	**Weight Suspended From Rubber Strip (lb)**
Wet adhesion, initial	1.5
Wet adhesion, after aging (stability test)	1.5
Initial	—
Using classes 1 and 2 gasket stock	5
Using class 3 gasket stock	4
After immersion	4
At 60 °C	1

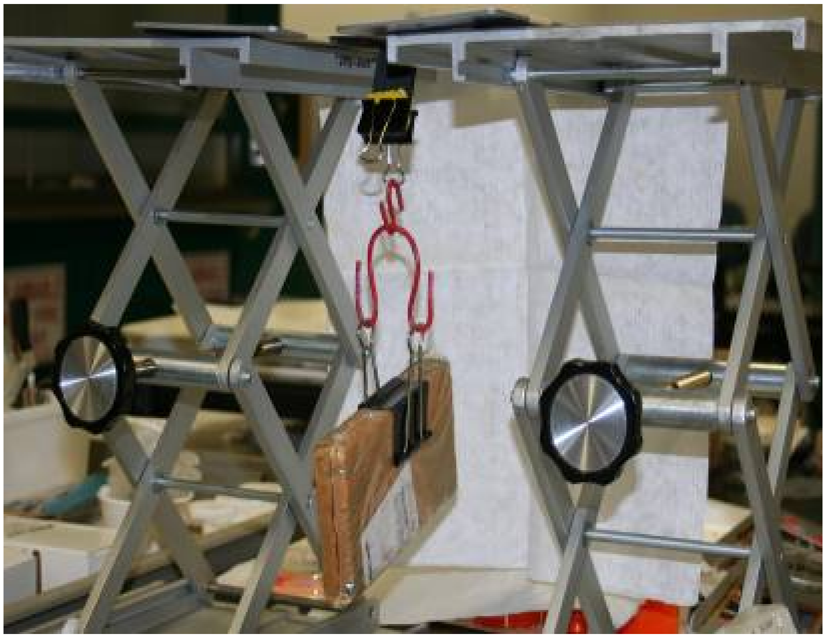

Figure 2. Basic strip adhesion setup for MMM-A-121.

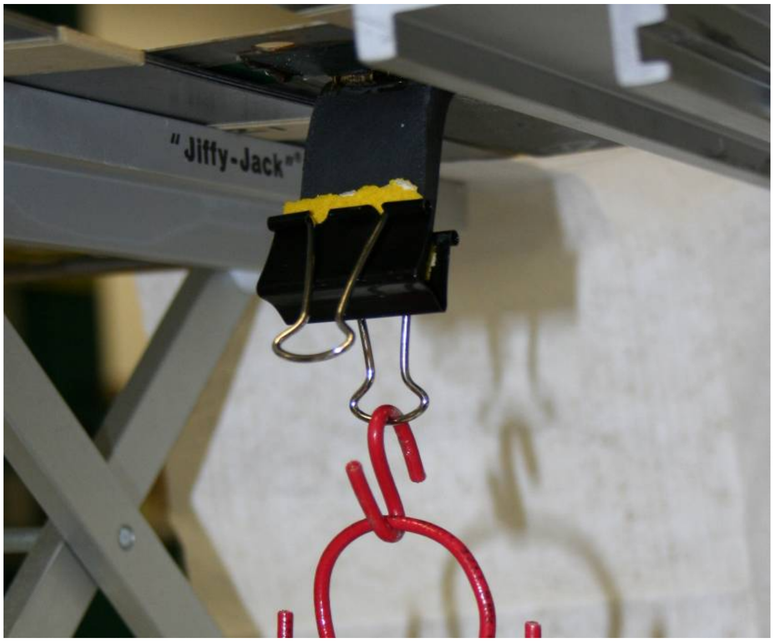

Figure 3. Basic strip adhesion close-up for MMM-A-121.

2.4.2 Procedure for Determining Strip Adhesion After Salt Water Immersion

Samples were immersed in salt water during their conditioning period, as specified by table 3. A 5 weight-percent NaCl solution was prepared. The samples were immersed where rubber and bond line were completely submerged throughout the process for 72 hr (figure 4). The strip adhesion samples were then tested as just described.

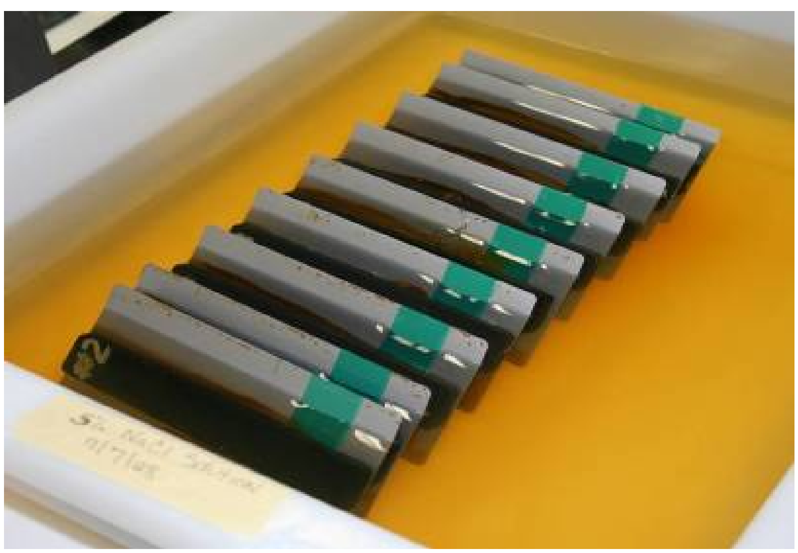

Figure 4. NaCl solution—5% for immersion with MMM-A-121.

2.4.3 Procedure for Determining Strip Adhesion at 60 °C

The specimen panels, conditioned as specified in table 3, were supported on the sides in a horizontal position in an oven at a temperature of 60 ± 1.1 °C (140 ± 2 °F). The panels were conditioned for 20 min, with the rubber strips facing down. While still in the oven and after the conditioning period, one end of each rubber strip was separated from the metal panel for a distance of about 2 in and a 1-lb weight was suspended from each strip (figure 5). After 3 min, the distance of stripping of each specimen from the panel was noted and the results averaged.

Figure 5. Strip adhesion test in 60 °C oven.

2.4.4 Procedure for Stability Test

A closed 1-pt container of the adhesive material was placed in an oven for 2 weeks at 49 ± 1.1 °C (120 ± 2 °F). The sample was removed from the oven and allowed to cool for 3 hr at 23 ± 1.1 °C (73.5 ± 2 °F). The wet adhesion test specified in table 3 was conducted.

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3. Results and Discussion

3.1 Rheology

All of the adhesives tested were non-Newtonian (*10*) shear thinning fluids; thus, the viscosity changed with shear rate. There was typically a Newtonian plateau at very low shear prior to shear thinning behavior where viscosity was independent of shear rate. The power law region of the shear thinning curve had the following viscosity:

$$\eta = \kappa\gamma^{n-1}, \quad (1)$$

where η is the viscosity, κ is the flow consistency index, γ is the shear rate, and n is the flow behavior index. The values of K and n were calculated and used to characterize each product formulation. Rheology testing was conducted at 20 °C using a 40-mm cross-hatched plate with a solvent trap containing ethanol.

Figure 6 shows rheological behavior of the various adhesives. Figure 7 focuses on the shear thinning range for each adhesive. The Newtonian plateau of 3M-1357 was 3× higher than that of 3M-1300L (table 5), showing that a range of viscosities was acceptable for MMM-A-121. The Newtonian viscosity of 3M-847 matched that of the baseline 3M-1357, although 3M-1357 shear thinned to a higher degree (figure 7) noted by the lower value of *n* (table 5). While 3M-4491 and 3M-30NF matched the Newtonian viscosity of 3M-1300L (table 5), the onset for shear-thinning occurred at much lower shear rates for the two potential replacements (figure 6).

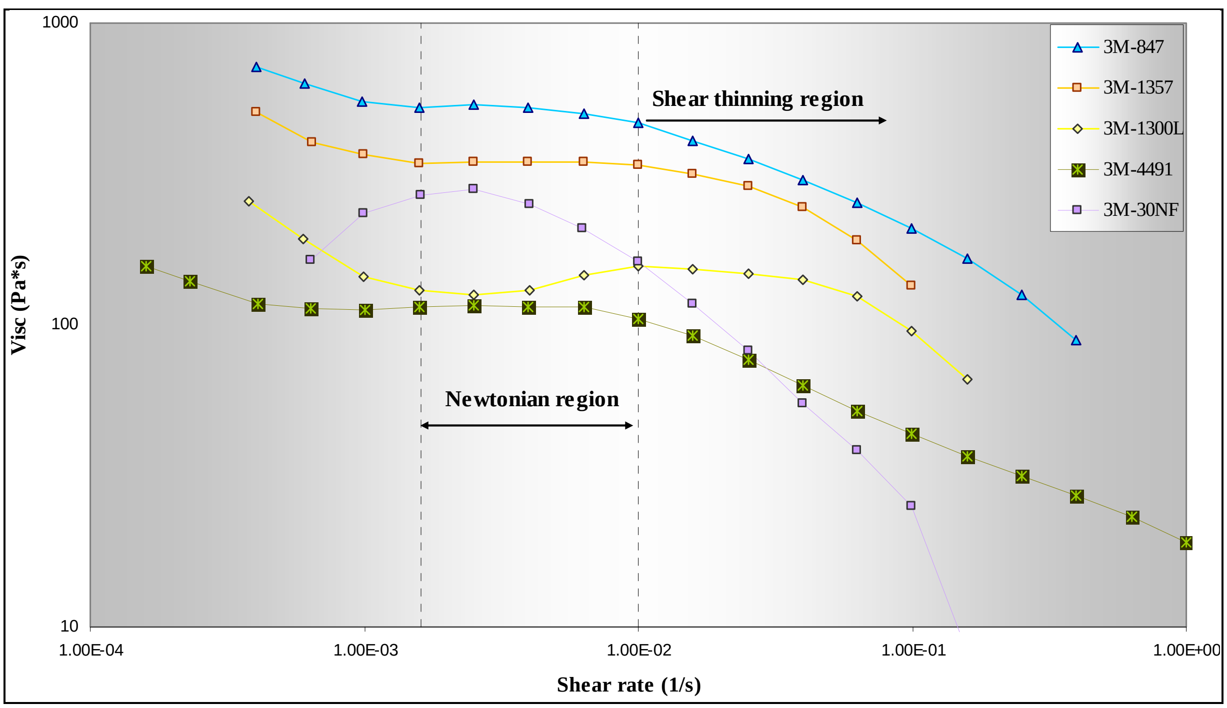

Figure 6. Rheological characteristics of adhesives.

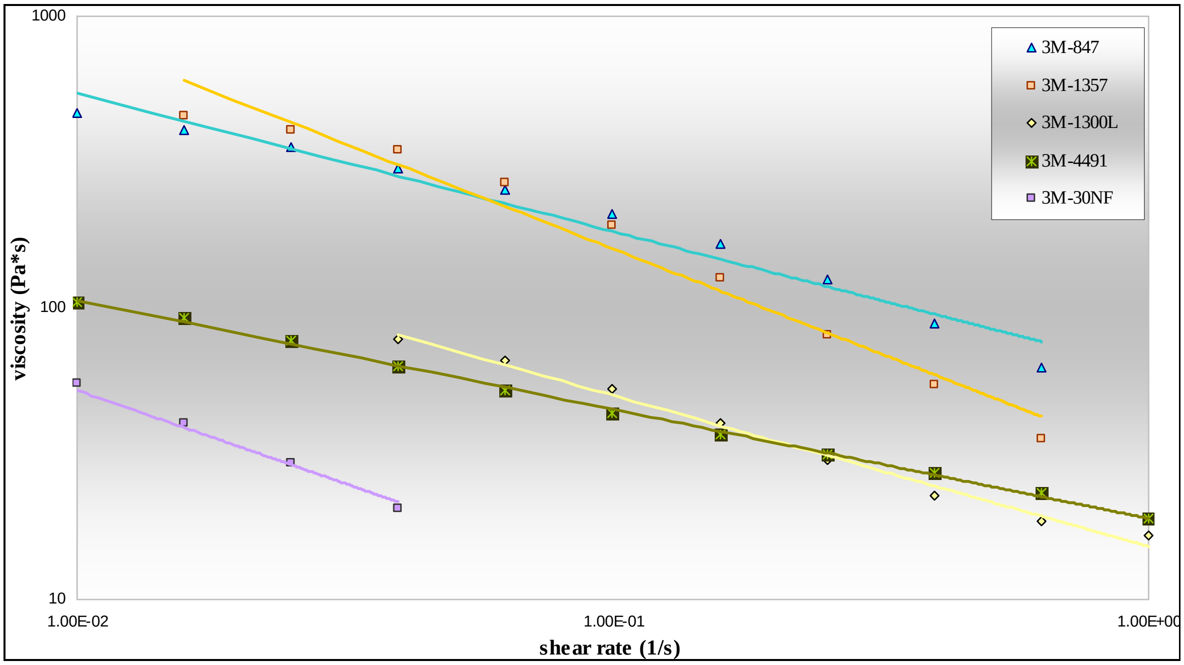

Figure 7. Least-squares curve fitting of the shear thinning viscosity.

Table 5. Rheology data for adhesives.

Product Name	Newtonian Viscosity (Pa*s)	Standard Deviation	"κ" Flow Consistency Index	"n" Flow Behavior Index	R^2
3M-1357	428.93	67.53	45.92	0.4111	0.9665
3M-1300L	142.57	1.42	19.03	0.3804	0.9655
3M-847	454.72	74.88	46.96	0.5152	0.9667
3M-4491	119.28	29.42	17.50	0.6014	0.9981
3M-30NF	125.68	10.73	2.76	0.3616	0.9935

3.2 Nonvolatile Content (Solids)

Samples dried over several days in an oven at 70 °C until no further weight change was measured. The percentage of total solids was calculated as follows:

$$\text{Total solids, percent} = (\text{weight of residue/weight of sample}) \times 100. \quad (2)$$

All products were compared to their respective technical data sheets for the specified percent solids, and the results were reported in table 6. The solids content matched the technical data sheet, except for 3M-1357 and 3M-4491, both of which had slightly higher solids content than expected. The 3M-30-NF had the highest solids content, while the 3M-1357 had the lowest. Except for the 3M-30-NF (colored in red in table 6), all of the adhesives had solids content within the acceptable limits (green in table 6) of the MMM-A-121 specification.

Table 6. Solids content of adhesives.

Product Name	Tech Data Sheet Solids (Weight-Percent)	Calculated Solids (Weight-Percent)
3M-1357	23–27	28
3M-1300L	26–33	32
3M-847	33–39	36
3M-4491	22–26	29
3M-30NF	50–51	50

3.3 Dry Time

The time required for the sample to become tack-free was recorded as a range rather than a single data point. The HAP-free adhesive (3M-847) contained acetone as the primary solvent, which had a very fast evaporation rate, whereas the baseline adhesives (3M-1300L and 3M-1357) contained toluene, petroleum distillate, and MEK, which had a relatively slower evaporation rate. This and the slightly higher solids content for 3M-847 were the reasons for the slightly shorter dry time relative to the baseline products. The 3M-4491 adhesive contained a blend of acetone and an extremely low evaporating solvent cyclohexanone, aiding in the longer dry time of this product. The 3M-30NF was ~40%–50% water based, which explained the significantly longer dry time than that of the other products in this series. Results are listed in table 7. Although the dry times for the two baseline adhesives was slightly longer than the HAP-free 3M-847, the difference was not observed by the user during application.

Table 7. Dry time of adhesives.

Product Name	Time Range (min)
3M-1300L	12–14
3M-1357	11–13
3M-847	10–12
3M-4491	36–38
3M-30NF	67–69

3.4 Strip Adhesion - MMM-A-121

The maximum adhesive/cohesive loss allowed for any of the five strip adhesion tests was 3 in. All passing results in table 3 exhibited an adhesive/cohesive loss ranging from 0 to less than 1 in. The two baseline products (3M-1300L and 3M-1357) passed all versions of the strip adhesion tests specified by MMM-A-121 (table 8, figure 8), with all three classes of rubber. The HAP-free adhesive (3M-847) also passed all versions of the strip adhesion tests (figure 8). Overall, the strip adhesion performance of the 3M-847 very closely matched that of the control adhesives

Table 8. Strip adhesion results.

Test	3M-1300L	3M-1357	3M-847	3M-4491	3M-30NF
Wet adhesion	Pass	Pass	Pass	Fail	Fail
Stability adhesion	Pass	Pass	Pass	Fail	Fail
Initial adhesion	Pass	Pass	Pass	Pass	Fail
After immersion	Pass	Pass	Pass	Pass	Fail
At 60 °C	Pass	Pass	Pass	Fail	Fail

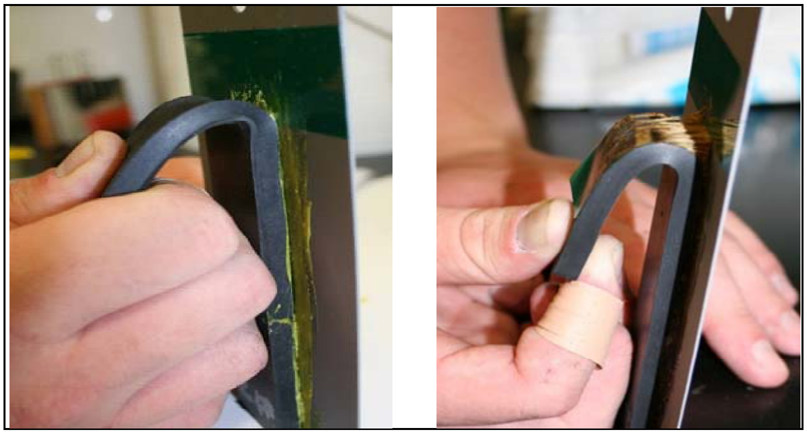

Figure 8. The 3M-1357 (left) and 3M-847 (right).

(i.e., 3M-1357 and 3M-1300L). The 3M-4491 product only passed the "initial" and the "after immersion" strip adhesion. Figure 9 (left) shows that the adhesive failure of 3M-30NF starts immediately after a weight was suspended from the rubber test assembly. At 1 min 30 s, figure 9 (right) illustrates the rapid progression of adhesive failure. Complete failure occurred at 1 min 45 s. The 3M-30NF product experienced total adhesive failure (*10*) to the steel substrate (figure 10) across the board. This adhesive failure to the steel substrate was not unexpected since the technical data sheet specifically stated that the product was not for use on metal (*12*). However, the 3M-30NF product was still included in this test series because of the low HAP and volatile organic compound (VOC) properties in the formulation.

Figure 9. The 3M-30NF at 10 s (left) and at 1 min 30 s (right).

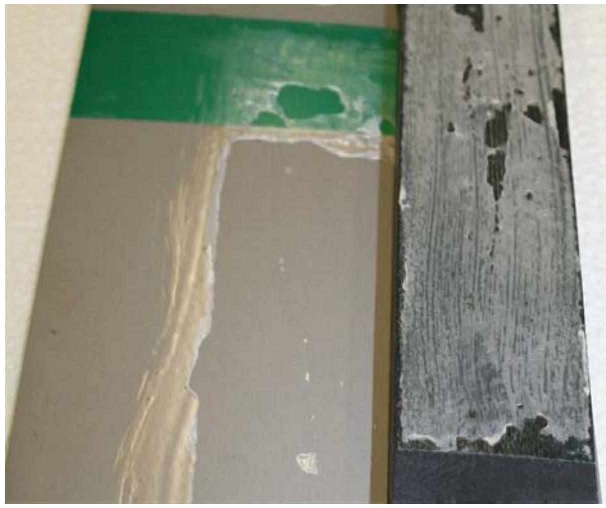

Figure 10. Total adhesive failure of 3M-30NF to steel.

4. Conclusions

Two commercial products, 3M-1357 and 3M-1300L, commonly used for applications covered by federal specification MMM-A-121 contained unacceptably high levels of HAPs and VOCs. Three possible alternative commercial off-the-shelf products were tested vs. the baseline materials for performance in order to identify suitable replacements, resulting in lower HAP and VOC emissions. Strip adhesion results clearly distinguished only one alternative, 3M-847, as acceptable. Furthermore, 3M-847 passed all other performance metrics according to MMM-A-121 including rheology/viscosity, dry time, and solids content. This study also determined that 3M-4491 and 3M-30NF were not suitable materials for vulcanized rubber to steel bonding, as prescribed by MMM-A-121. Switching from current baseline materials to the 3M-847 replacement would mean a reduction of ~1200 lb/year of HAP and VOC emissions (*13*). However, to approve 3M-847 for military use, a demonstration/validation study at an actual Army facility is necessary.

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List of Symbols, Abbreviations, and Acronyms

ACN	acrylonitrile content
ASTM	American Society for Testing and Materials
DBP	dibutyl phthalate
Eta (η)	viscosity
Gamma (γ)	shear rate
HAP	hazardous air pollutant
Kappa (κ)	flow consistency index
MEK	methyl ethyl ketone
MSDS	material safety data sheet
n	flow behavior index
NDCEE	National Defense Center for Energy and Environment
NESHAP	National Emission Standard for Hazardous Air Pollutants
Pa*s	Pascal second
R^2	coefficient of determination
SPOTA	Sustainable Painting Operations for the Total Army
VOC	volatile organic compound

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