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### Environmentally Friendly Adhesives And Sealants For Army Applications

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<td>j. Information on military applications in space, nuclear chemical or biological efforts: high energy laser information, particle beam technology; etc?</td>
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<td>k. Contain information with foreign policy or foreign relations implications?</td>
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Excellent demonstration of material substitution to impact the Army. Public domain.

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32. Public/Limited release information

- a. Material has been reviewed for OPSEC policy.
- b. The information contained in this material is approved for public release that received appropriate tech/editorial review.

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**Date**
The Environmental Protection Agency (EPA) is in the process of mandating the Defense Land Systems and Miscellaneous Equipment National Emission Standard for Hazardous Air Pollutants (HAP) that will affect Army surface coating operations. The Army uses numerous adhesives and sealants among other coating materials that contain significant amounts of HAPs. This work examines a laboratory and weapons platform demonstration of two of the most highly used adhesives/sealants throughout the Army. The adhesives and sealants thrust area of the SPOTA program has identified HAP-free alternatives to Torque Seal, an anti-tamper sealant. Laboratory testing has shown that HAP-free Torque Seal using ethanol as the lone solvent had similar solids content, viscosity, adhesion, fluid resistance, and weathering relative to the baseline product. Dry time is slightly longer, but not enough to raise any concerns with weapons platforms maintainers. Furthermore, a demonstration/validation study at Fort Rucker on a UH-1 rotor shows that this HAP-free sealant has performed well as a substitute material. The SPOTA program has also identified 3M Scotch-Weld™ 847 as an alternative to current adhesives conforming to specification MMM-A-121, rubber to metal bonding adhesives. Other potentials materials such as 3M FastBond™ 30NF 3M-4491 Scotch-Weld™ did not have the required adhesive properties. A demonstration/validation study must be performed to ensure that 3M-847 can be used to effectively replace other MMM-A-121 adhesives on weapons platforms. Overall, these two substitute materials should reduce the Army HAP emissions by ~1300 lbs/yr and VOC emissions by ~1200 lbs/yr.

1. INTRODUCTION

The Environmental Protection Agency (EPA) is in the process of mandating the Defense Land Systems and Miscellaneous Equipment (DLSME) National Emission Standard for Hazardous Air Pollutants (NESHAP) that will affect Army surface coating operations (Concurrent Technologies Corporation, 2004a). The materials used for coatings operations at many Army installations was surveyed, and it was found that the Army uses numerous adhesives and sealants among other coating materials that contain significant amounts of hazardous air pollutants (HAP) (Concurrent Technologies Corporation, 2004a). 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 (Vallone, J, 2004). Therefore, the goal of the Sustainable Painting Operations for the Total Army (SPOTA) program is to severely reduce the amount of HAP emissions produced in coatings operations, including adhesives and sealant application and removal.

Figure 1: Breakdown of HAP content for adhesives and sealants.

Army adhesives and sealants account for 10% of the Army’s surface coating materials and 5% of the total HAP emissions. The top 4 adhesives/sealants make up 80% of the adhesives/sealants HAP emissions, and the top 10 make up 93% (Figure 1). Hundreds of materials make up the last 7 percent with none amounting to more that 0.5% of the overall HAP emissions generated by the Army. Therefore, the near-term goal of the SPOTA sealants and adhesives thrust area is to replace the top 4
of priority materials: Torque Seal anti-sabotage sealants, MMM-A-121 rubber-to-steel adhesives, A-A-1936 neoprene adhesives, and tent/canvas sealants. Longer-term efforts will focus on replacing the remaining top priority materials. This work in particular focuses on two of the top priority adhesives and sealants: Torque Seal and MMM-A-121.

2. MATERIALS

Torque Seal is used to detect tampering or loosening of mechanical fasteners on aircraft. Applied after bolts or fittings are in proper torque or position, this product gives inspectors visual evidence of any movement or tampering. Torque Seal dries to form a very brittle film which will crack, flake or crumble when minimal force is applied. Other key product attributes are excellent adhesion to most surfaces and fast drying. The manufacturer refers to this product as an inspection seal lacquer or anti-sabotage lacquer. The fast drying characteristic of Torque Seal is achieved by using low boiling point solvents as carriers, specifically ethanol and methanol. While both solvents are volatile organic compounds (VOC), methanol has been classified by the EPA as a hazardous air pollutant. Torque Seal contains approximately 20 wt% methanol HAP and 30 wt% ethanol (Organic Products Company, 2006). Organic Products Co. (Irving, TX) is the only known manufacturer of this product, and no HAP-free versions of this material are commercially produced (Concurrent Technologies Corporation, 2006). Torque Seal comes in a variety of colors, including, red, orange, green, white, blue, yellow, and black. According to the manufacturer and our measurements, there are no significant differences in the properties of different colored versions of this product.

To eliminate HAP content in Torque Seal, ARL commissioned Organics Products Co. to manufacture a HAP-free batch of Torque Seal where the entire methanol content is completely replaced with ethanol. Organic Products Co. manufactured this HAP-free Torque Seal only in the color red, the Army’s highest used color (Concurrent Technologies Corporation, 2008). Red and green baseline Torque Seal was used for comparison to the HAP-free product.

The scope of federal specification MMM-A-121 involves adhesives used in bonding vulcanized synthetic rubber to steel (MMM-A-121, 1966). The two most commonly used baseline products under the MMM-A-121 specification are 3M-1357 Scotch-Weld™ Neoprene High Performance Contact Adhesive (3M-1357) containing petroleum distillate, acetone, MEK, toluene and n-hexane (3M, 2007a).

A possible HAP free alternative product for MMM-A-121 was identified as 3M 847 Scotch-Weld™ Nitrile High Performance Rubber & Gasket Adhesive (3M-847) (Concurrent Technologies Corporation, 2006) containing acetone (an exempt solvent) (3M, 2007b). However, testing is needed to ensure performance, compatibility and compliance to MMM-A-121. Two additional products were also identified as potential low HAP alternatives (Concurrent Technologies Corporation, 2006). These products are 3M-4491 Scotch-Weld™ Nitrile Industrial Adhesive (3M-4491) containing acetone and cyclohexanone (3M, 2002), and 3M-30NF Fastbond™ Contact Adhesive (3M-30NF) containing primarily water (3M, 2008). Only the two baseline adhesives claim compliance with the requirements of MMM-A-121.

3. TORQUE SEAL LABORATORY VALIDATION

Various testing was done in the laboratory to determine the relative similarity of baseline Torque Seal to the HAP-free product. This testing included solids content, adhesion, dry-time, rheology, fluid resistance, weathering, among others.

3.1 Solids Content

Solids content of the sealants was measured by weighing out a few drops of the sealant into a glass petri dish. The bulk of the solvent was allowed to evaporate for 1 day at room temperature. The sample was reweighed and then heated to 50°C for 1 week to increase the evaporation rate of the residual solvent. To ensure complete removal of the solvent, the sample was then pulverized followed by heating at 50°C for an additional week. The solids content for the HAP-free Torque Seal was 54 ± 2 wt% and was within experimental error of the baseline Torque Seal with 51 ± 3 wt% solids content.

3.2 Rheology

The rheology of Torque Seal before evaporation of the solvent was measured using a TA Instruments AR2000 Rheometer (New Castle, DE) 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 is to keep the carrier solvents from volatilizing during the experiment and causing 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 two seconds. The shear rate and viscosity were recorded when the viscosity stabilized to within 5% tolerance for three consecutive intervals.

The viscosity was Newtonian from $10^{-2}$ 1/s to $10^{-4}$ 1/s, but then began to shear thin. The Newtonian viscosities were within experimental error: $2*10^4 \pm 1*10^4$ Pa·s. Furthermore, the magnitude and rate of shear thinning for the baseline and HAP-free Torque Seal were similar.

3.3 Dry Time Evaluation

Dry time studies based on ASTM D1640 (ASTM, 2003) and qualitative methods commonly used in industry were conducted for the HAP-free Torque Seal formulation and the current commercially available Torque Seal. Beads of the sealants were applied to a steel panel and evaluated for skin time. Skin time (also know as open time) is the formation of a cohesive film that can withstand a light touch with a wooden dowel. Next, samples were applied as a uniform film with a thickness of 6.5 mils. The tack free time of the samples was then measured periodically with a common qualitative industry “touch-test” until the samples were no longer tacky and resisted transfer. Lastly, tack free time of the samples was evaluated using cotton fibers. Tack free with cotton fibers is a more sensitive touch-test that determines the ability of the sample to resist adhesion to the fibers. All of the results (Table 1) are recorded as a range of time, as these tests are qualitative in nature (ASTM, 2003). Dry time studies were performed at -11ºF, 75ºF, and 140ºF. At all times, the dry time is slightly longer for the HAP-free sealant relative to the baseline. This was expected because ethanol has a lower volatility than methanol.

Table 1: Torque Seal dry time results.

<table>
<thead>
<tr>
<th>Test Temperature (°F)</th>
<th>Skin on bead (minutes)</th>
<th>Tack free by touch (minutes)</th>
<th>Tack free by cotton (minutes)</th>
</tr>
</thead>
<tbody>
<tr>
<td>CONTROL</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>-11</td>
<td>27-29</td>
<td>37-39</td>
<td>38-40</td>
</tr>
<tr>
<td>75</td>
<td>10-12</td>
<td>19-21</td>
<td>20-22</td>
</tr>
<tr>
<td>140</td>
<td>5-7</td>
<td>6-8</td>
<td>8-10</td>
</tr>
<tr>
<td>HAP FREE Replacement</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>-11</td>
<td>33-35</td>
<td>70-72</td>
<td>71-73</td>
</tr>
<tr>
<td>75</td>
<td>13-15</td>
<td>23-25</td>
<td>24-26</td>
</tr>
<tr>
<td>140</td>
<td>7-9</td>
<td>9-11</td>
<td>10-12</td>
</tr>
</tbody>
</table>

3.4 Adhesion Testing

The baseline and HAP-free sealants were applied to steel substrates with a uniform thickness of 6.5 mils. The sealants were allowed to dry for 1 day and then were tested for adhesion using ASTM D3359 Test Method B-Cross-Cut tape test. Tape was applied to the cross-hatched area and then removed. The amount of sealant remaining in the cross-hatched area was rated according to ASTM D 3359 to assess the adhesion. Adhesion testing was conducted at -5ºF, 75ºF, and 140ºF. Adhesion results for both the HAP-free and baseline sealants were excellent with ratings of 5B at 75ºF, and performed only slightly worse at -5ºF (Fig. 2) and 140ºF with 4B ratings. Thus, the HAP-free product performed similarly to the baseline product in all three temperature settings.

3.5 Flexibility Testing

Flexibility testing was performed using the mandrel bend test. The sealants with uniform thickness of 6.5 mils were applied to tin coated steel Q-panels substrates. The samples were allowed to dry for 1 day and then were bent on a 1/8” mandrel. The samples were conditioned at -5ºF, 75ºF, and 140ºF for 2 hrs prior to testing. Interestingly, because this is an anti-tamper sealant, some degree of cracking and breaking is desired. In fact, both the baseline and HAP-free Torque Seal showed similar white stress fractures at the site of the bend across the sample (Fig. 3).

3.6 Resistance to Fluid Immersion

The baseline and HAP-free sealants were applied to steel substrates with a uniform thickness of 6.5 mils and allowed to dry 24 hours at room temperature. These specimens were then partially immersed by standing the panels vertically in either MIL-L-23699 lubricating oil at...
121°C for 24 hours, MIL-PRF-83282 hydraulic fluid at 66°C for 24 hours or JP-8 fuel at room temperature for 7 days according to MIL-PRF-8528D section 4.6.8. The films were examined one hour after removal from the fluid for blistering, delamination, and discoloration.

Both the baseline sealant (green) and the HAP-free sealant (red) showed a darkening of color after immersion for 24 hours in lubricating oil at 121°C, no blistering or delamination occurred. Fig 4 shows the baseline sealant (green) and the HAP-free sealant (red) panels after immersion for 24 hours in lubricating oil at 121°C. Below the fluid line, the samples darkened considerably, but did not blister or delaminate. Both the baseline sealant and the HAP-free sealant showed no change after immersion for 24 hours in hydraulic fluid at 66°C. Similarly, both the baseline sealant and the HAP-free sealant showed no change after immersion in JP-8 fuel at room temperature for 7 days.

Approximately two weeks following the completion of the fluid immersion evaluation, ASTM D3359 Test Method B-Cross-Cut tape test at 75ºF was conducted on the portion of sealant which had been submerged in fluid. The HAP-free sealant performed similarly to the baseline sealant with a 5B rating and had no reduction in adhesion performance relative to the non-immersed samples.

For humidity testing, the specimens were then subjected to constant conditions of 40°C and 100% relative humidity. Samples were evaluated after one-day, three-days, one-week, two-weeks and four-weeks of exposure. No delamination or blistering occurred on any of the panels. However, fading in color to a similar extent for both the baseline and HAP free sealants was visible. The samples exposed for one-day were as faded in color as the samples exposed at four-weeks. Any uneven fading can be attributed to the position of the spray nozzle. This phenomenon was verified by repositioning the various sample panels to duplicate the effect.

Torque seal samples were exposed to QUV testing, an accelerated form of weathering, which simulates high intensity light exposure, heat, and humidity. In particular, the samples were exposed to a 340A light source for 8 hrs at 60°C followed by 4 hrs darkness with condensation at 50°C. After 100 hrs, both the baseline and HAP-free samples faded considerably and gloss was reduced from ~40 units at 60º to approximately 2 units. After 500 hrs, the gloss and color remained the same, but the samples cracked and delaminated from the substrate (Fig 5).

4. TORQUE SEAL DEMONSTRATION/VALIDATION ON UH-1 HELICOPTER

4.1 Torque Seal Demonstration at Fort Rucker

As a result of the success of the HAP-free ethanol-based Torque Seal in the laboratory testing, this product was demonstrated/validated at Fort Rucker, AL on a UH-1 rotor assembly. On May 6, 2008, the UH-1 maintainers applied the baseline and HAP-free Torque Seal to every nut and bolt on the assembly (Fig. 6). The maintainers were asked to rate the ease of application and time required for drying. Overall, they were unable to determine any difference between the baseline and HAP-free material. From May through November 5, 2008, the UH-1 was fielded and the Torque Seal was inspected by the maintainers regularly for damage and to
compare the baseline to the HAP-free material. As time progressed, some beads of Torque Seal cracked, fractured, or rotated out of alignment (and thus the bolt/nut required tightening). As of August 6, 2008, the HAP-free material performed very similarly to the baseline material. It is expected that the HAP-free material will pass all requirements and will be validated for Army use in the near future.

Figure 6: Photograph showing red HAP-free beads and green baseline Torque Seal beads on the UH-1 rotor assembly.

4.2 HAP-Free Torque Seal Conclusions

The HAP-free ethanol-based Torque Seal produced by Organic Products Co. performs in every way very similarly relative to the HAP-containing Torque Seal. Solids content, viscosity, adhesion, fluid resistance, and weathering are all comparable for the HAP-free product. Dry time is slightly longer, but not enough to raise any concerns with weapons platforms maintainers. Furthermore, the demonstration on a real weapons platform appears that it will be successful, although it is still on-going. As a result, it is expected that HAP-free Torque Seal will soon replace the methanol-containing Torque Seal throughout the Army, reducing over 100 lbs/yr HAP emissions (Concurrent Technologies Corporation, 2008).

5. MMM-A-121 ADHESIVE LABORATORY VALIDATION

Various testing was done in the laboratory to determine the relative similarity of baseline 3M-1357 and 3M-1300 to the potential alternative materials. This testing included solids content, rheology, dry time, and adhesion strength.

5.1 Non Volatile Content (Solids)

A suitable container was weighed, and approximately 10 grams 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 room temperature before weighing. Each sample was run in duplicate (ASTM, 2005).

The solids content is listed below in Table 2. 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 2), all of the adhesives have solids content within the acceptable limits (green in Table 2) of the MMM-A-121 specification.

Table 2: Solids Content of Adhesives

<table>
<thead>
<tr>
<th>Product Name</th>
<th>Tech Data Sheet Solids (wt. %)</th>
<th>Calculated Solids (wt. %)</th>
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<tr>
<td>3M-1357</td>
<td>23-27</td>
<td>28</td>
</tr>
<tr>
<td>3M-1300L</td>
<td>26-33</td>
<td>32</td>
</tr>
<tr>
<td>3M-847</td>
<td>33-39</td>
<td>36</td>
</tr>
<tr>
<td>3M-4491</td>
<td>22-26</td>
<td>29</td>
</tr>
<tr>
<td>3M-30NF</td>
<td>50-51</td>
<td>50</td>
</tr>
</tbody>
</table>

5.2 Rheology

Rheology was measured as for the Torque Seal. All of the adhesives tested were non-Newtonian shear thinning fluids (ASTM, 2005), and thus the viscosity changes with shear rate. There is typically a Newtonian plateau at very low shear prior to shear thinning behavior where viscosity is independent of shear rate. The power law region of the shear thinning curve has a viscosity:

\[ \eta = \kappa \gamma^{n-1} \]  

where \( \eta \) is the viscosity, \( \kappa \) is the flow consistency index, \( \gamma \) 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.

Fig. 7 shows rheological behavior of the various adhesives. The Newtonian plateau of 3M-1357 was three times higher than that of 3M-1300L (Table 3), showing that a range of viscosities are 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 noted by the lower value of \( n \) (Table 3). While 3M-4491 and 3M-30NF matched the Newtonian viscosity of 3M-1300L (Table 3), the onset for shear-thinning occurred at much lower shear rates for the two potential replacements (Fig. 7).
Figure 7: Rheological characteristics of the baseline MMM-A-121 adhesives and potential substitutes.

<table>
<thead>
<tr>
<th>Product Name</th>
<th>Newtonian Viscosity (Pa*s)</th>
<th>Standard Deviation (Pa*s)</th>
<th>(\kappa) (Pa*s)</th>
<th>(n)</th>
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<tr>
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<td>454.72</td>
<td>74.88</td>
<td>46.96</td>
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<tr>
<td>3M-1357</td>
<td>428.93</td>
<td>67.53</td>
<td>45.92</td>
<td>0.4111</td>
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<td>3M-1300L</td>
<td>142.57</td>
<td>1.42</td>
<td>19.03</td>
<td>0.3804</td>
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<tr>
<td>3M-4491</td>
<td>119.28</td>
<td>29.42</td>
<td>17.50</td>
<td>0.6014</td>
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<tr>
<td>3M-30NF</td>
<td>125.68</td>
<td>10.73</td>
<td>2.76</td>
<td>0.3616</td>
</tr>
</tbody>
</table>

5.3 Dry Time

Dry times were measured as described previously for Torque Seal. The time required for the sample to become “tack free” was recorded as a range rather than a single data point (Table 4). The HAP free adhesive (3M-847) contains acetone as the primary solvent which has a very fast evaporation rate, whereas the baseline adhesives (3M-1300L and 3M-1357) contain toluene, petroleum distillates and methyl ethyl ketone, which have relatively slower evaporation rates. This and the slightly higher solids content for 3M-847 are the reason for its slightly shorter dry time relative to the baseline products. The 3M-4491 adhesive contains a blend of acetone and an extremely low evaporating solvent cyclohexanone, which resulted in the longer dry time of this product. The 3M-30NF is approximately 40-50% water based, which explains its significantly longer dry time relative to the other products. 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 4: Dry Time of Adhesives

<table>
<thead>
<tr>
<th>Product Name</th>
<th>Time Range (minutes)</th>
</tr>
</thead>
<tbody>
<tr>
<td>3M-1300L</td>
<td>12-14</td>
</tr>
<tr>
<td>3M-1357</td>
<td>11-13</td>
</tr>
<tr>
<td>3M-847</td>
<td>10-12</td>
</tr>
<tr>
<td>3M-4491</td>
<td>36-38</td>
</tr>
<tr>
<td>3M-30NF</td>
<td>67-69</td>
</tr>
</tbody>
</table>

5.4 Strip Adhesion

Strip adhesion was tested in accordance with MMM-A-121 (MMM-A-121, 1966). Strips of rubber material measuring 1 by 6 by ¼ inch were bonded to 3 in x 6 in x 0.032 in steel panels (Fig. 8). Three different rubbers were used: neoprene, styrene-butadiene rubber (SBR), and nitrile. One brush coat of the adhesive material was applied to the prepared surfaces of the rubber strips and panels. The rubber were was rolled down with six single passes of a ten pound roller, two inches wide, to form a good bond between the adhesive and substrates. The adhesive was allowed to dry according to the MMM-A-121 requirements listed in Table 5. The steel panel was supported at the ends in a horizontal position. One end of the bonded rubber strip was separated from the metal panel for a distance of about two inches. The weight specified below was suspended from the free end of the rubber strip (Fig. 9). The weight was allowed to act on the strip for three minutes, and the average distance of stripping of the specimen from the panel under the influence of weight was recorded. Strip adhesion tests were run on newly prepared adhesive strips in triplicate for each of the following test conditions:
1. Wet adhesion before and after aging the adhesive for two weeks at 49\(^\circ\)±1.1\(^\circ\)C (120\(^\circ\)±2\(^\circ\)F).
2. Initial adhesion
3. Adhesion after immersion in salt water solution.
4. Adhesion at 60\(^\circ\)±1.1\(^\circ\)C (140\(^\circ\)±2\(^\circ\)F).
The panels with the bonded strips were conditioned and tested as shown in Table 5. The following letter designations are used:

L – Dead weight load of 2.5 pounds per square inch of rubber gasket areas applied as a loading pressure on the strips bonded to the steel panel, condition at 23°C±1.1°C (73.5°F±2°F).

R – Rest time under no load at 23°C±1.1°C (73.5°F±2°F).

I – Specimens immersed in salt water (5 percent sodium chloride), under no load at 23°C±1.1°C (73.5°F±2°F).

T – Tests conducted at 23°C±1.1°C (73.5°F±2°F) within one hour after end of conditioning period except where otherwise indicated using 1.5 lb load except where noted.

T1 – Tests conducted at 60°C±1.1°C (140°F±2°F).

The loads used for the initial adhesion testing were 5 lbs for neoprene and SBR, 4 lbs for nitrile, 4 lbs after salt water immersion, and 1 lb load for 60°C testing. For elevated temperature testing after the loading and rest stages, the samples were conditioned in an oven for 20 min at 60°C±1.1°C (140°F±2°F). The samples were then tested for adhesion in the oven as previously described.

Table 5: Conditioning and testing schedule for MMM-A-121 specification adhesion testing

<table>
<thead>
<tr>
<th>Elapsed Time after Assembly (hours)</th>
<th>1.0±0.1</th>
<th>0 to 48</th>
<th>48 to 120</th>
<th>120 to 144</th>
<th>144</th>
</tr>
</thead>
<tbody>
<tr>
<td>Wet Adhesion</td>
<td>0</td>
<td>4</td>
<td>8</td>
<td>12</td>
<td>14</td>
</tr>
<tr>
<td>Stability (wet adhesion)</td>
<td>L</td>
<td>R</td>
<td>R</td>
<td>T</td>
<td></td>
</tr>
<tr>
<td>Initial Adhesion</td>
<td>L</td>
<td>R</td>
<td>R</td>
<td>T</td>
<td></td>
</tr>
<tr>
<td>After Immersion</td>
<td>L</td>
<td>I</td>
<td>R</td>
<td>T</td>
<td></td>
</tr>
<tr>
<td>At 60°C</td>
<td>L</td>
<td>R</td>
<td>R</td>
<td>T1</td>
<td></td>
</tr>
</tbody>
</table>

The maximum adhesive/cohesive loss allowed for any of the five strip adhesion tests is three inches. All passing results in Table 6 exhibited an adhesive/cohesive loss ranging from zero to less than one inch. The two baseline products (3M-1300L and 3M-1357) passed all versions of the strip adhesion tests specified by MMM-A-121 with all three classes of rubber (Table 6, Fig. 10). The HAP free adhesive (3M-847) also passed all versions of the strip adhesion tests. The 3M-4491 product only passed the “initial” and the “after immersion” strip adhesion, whereas the 3M-30NF failed yielding total adhesive failure (ASTM, 2005) to the steel substrate (Figure 11) for every MMM-A-121 adhesion test. This adhesive failure to the steel substrate was not unexpected, since the technical data sheet specifically states that the product is not for use on metal. Despite this, the 3M-30NF product was included in this test series because of its low HAP/VOC content.

Table 6: Strip adhesion results

<table>
<thead>
<tr>
<th>Test</th>
<th>3M-1300L</th>
<th>3M-1357</th>
<th>3M-847</th>
<th>3M-4491</th>
<th>3M-30NF</th>
</tr>
</thead>
<tbody>
<tr>
<td>Wet Adhesion</td>
<td>Pass</td>
<td>Pass</td>
<td>Pass</td>
<td>Fail</td>
<td>Fail</td>
</tr>
<tr>
<td>Stability Adhesion</td>
<td>Pass</td>
<td>Pass</td>
<td>Pass</td>
<td>Fail</td>
<td>Fail</td>
</tr>
<tr>
<td>Initial Adhesion</td>
<td>Pass</td>
<td>Pass</td>
<td>Pass</td>
<td>Pass</td>
<td>Fail</td>
</tr>
<tr>
<td>After Immersion</td>
<td>Pass</td>
<td>Pass</td>
<td>Pass</td>
<td>Pass</td>
<td>Fail</td>
</tr>
<tr>
<td>At 60°C</td>
<td>Pass</td>
<td>Pass</td>
<td>Pass</td>
<td>Fail</td>
<td>Fail</td>
</tr>
</tbody>
</table>

Figure 9: Basic strip adhesion set-up for MMM-A-121.

Figure 10: Photograph showing that no adhesive failure occurred baseline 3M-1357L.
5.5. MMM-A-121 Substitute Conclusions

Two commercial products, 3M-1357 and 3M-1300L, commonly used for applications covered by federal specification MMM-A-121 contain 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 which would result in lower HAP and VOC emissions (Concurrent Technologies Corporation, 2006). Strip adhesion results clearly distinguish only one alternative, 3M-847, as acceptable for use. This study also determined that 3M-4491 and 3M-30NF are 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 lbs/year of HAP and VOC emissions (Concurrent Technologies Corporation, 2008). However, to approve 3M-847 for military use a demonstration/validation study at an actual Army facility is necessary.

6. CONCLUSIONS

The adhesives and sealants thrust area of the SPOTA program has identified HAP-free alternatives to one of the most used sealant, Torque Seal, and one most used adhesive, 3M-1357/3M-1300L, in the Army. Laboratory testing has validated that HAP-free Torque Seal using ethanol as the lone solvent performs very similarly to the baseline HAP-containing material. Furthermore, a demonstration/validation study at Fort Rucker on a UH-1 helicopter shows that this HAP-free sealant has performed well as a substitute material. Similarly, laboratory testing shows that 3M Scotch-Weld 847 is a high-performing HAP-free and VOC-free replacement for the currently used adhesives conforming to MMM-A-121. A demonstration/validation study must be performed to ensure that 3M-847 can be used to effectively replace other MMM-A-121 adhesives on weapons platforms. Overall, these two substitute materials should reduce the Army HAP emissions by ~1300 lbs/yr and VOC emissions by ~1200 lbs/yr. Replacements for adhesives conforming to A-A-1936 and seam sealants for tents will soon be validated on the laboratory level.

ACKNOWLEDGEMENTS

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3M, 2008: Fastbond™ Contact Adhesive 30-NF MSDS, 3M Company, St. Paul MN.