ARMY RESEARCH LABORATORY

Federal Specification MMM-A-1617B for Adhesive, Rubber-Base, General-Purpose HAP-Free Replacement

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14. ABSTRACT

The goal of the Sustainable Painting Operations for the Total Army (SPOTA) program is to significantly reduce the amount of hazardous air pollutant (HAP) emissions produced in coatings operations, including application and removal of adhesives and sealants. This report focuses on potential HAP-free replacements for Federal Specification MMM-A-1617B: Adhesive, Rubber Base, General Purpose. A 180° peel adhesion test was conducted on a series of nine rubber-based contact adhesives using a variety of substrate combinations and conditioning parameters. The conditioning parameters consisted of various lengths of time, temperature, and immersion in liquids such as deionized (DI) water, IRM-901 reference oil, and JP-8 fuel. Three Clifton Adhesive, Inc., MMM-A-1617B qualified baseline contact adhesives containing HAPs were included in the test series. Clifton also provided three experimental contact adhesives that were either HAP free or low HAP and claimed to meet the MMM-A-1617B requirements. Three possible HAP-free alternative contact adhesives made by the 3M Company were also included in the test series. The alternative, HAP-free 3M-847 adhesive consistently exceeded the minimum requirements for MMM-A-1617B specification, while the Clifton products failed many of the minimum requirements.

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adhesive, HAP free, nitrile, rubber, SBR, neoprene, duck, SPOTA, MMM-A-1617, EPA

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1. Introduction

The U.S. Environmental Protection Agency (EPA) is planning to propose the Defense Land Systems and Miscellaneous Equipment (DLSME) National Emission Standard for Hazardous Air Pollutants (NESHAP) that will affect U.S. Army surface coating operations (*1*). Hazardous air pollutants (HAPs) are pollutants that are known or suspected to cause cancer or other serious health effects or adverse environmental effects. A survey of many Army installations found numerous adhesives, sealants, and other coating materials that contain significant amounts of HAPs (*2*). The Army has determined that it is more cost effective to reduce or eliminate HAP emissions from surface coating operations rather than using emissions control devices to capture and treat them (*2*). The goal of the Sustainable Painting Operations for the Total Army (SPOTA) program is to significantly reduce the amount of HAP emissions produced in coatings operations, including application and removal of adhesives and sealants. This report focuses on Federal Specification MMM-A-1617B adhesive, rubber-base, general-purpose adhesives currently used by the Army and potential HAP-free replacements.

1.1 Materials

1.1.1 Baseline Adhesives

The scope of MMM-A-1617B covers natural and synthetic rubber-base adhesives. The Clifton adhesives in table 1 appear on the qualified product list (QPL) for MMM-A-1617B. These elastomeric adhesives are classified into three types (*3*):

- Type I: Non-oil-resistant natural rubber base, synthetic natural (polyisoprene), styrene butadiene (SBR), reclaim, or combinations.
- Type II: Oil-resistant polychloroprene rubber base.
- Type III: Fuel-resistant butadiene acrylonitrile (nitrile) rubber base.

Table 1. Physical properties of MMM-A-1617B baseline adhesives (*4–9*).

Adhesive	Type	Solvents	Solids (% by wt.)	HAP (% by wt.)	VOC (g/L)	Density (lb/gal)
Clifton FA-1053 (baseline)	I	Hexane	40 ± 2	58–62	580–620	6.40–6.70
Clifton FA-1051 (baseline)	II	Toluene Hexane MEK	25 ± 1	15–30	716–752	6.75–7.05
Clifton FA-1076 (baseline)	III	MIBK MEK THF	21 ± 1	1–5	630–670	7.15–7.45

Note: MEK = methyl ethyl ketone, MIBK = methyl isobutyl ketone, and THF = tetrahydrofuran.

Clifton FA-1053 adhesive (baseline) is currently registered on the QPL for a MMM-A-1617B Type-I adhesive (table 1) (*5*). This adhesive is a natural rubber and SBR combination recommended for bonding cotton duck, leather, felt, and cork to themselves, aluminum, steel, or natural rubber (*5*). Cotton duck is a heavy, plain-woven fabric also referred to as canvas used to make tents and tarps (*3*). This adhesive contains 60% hexane (*4*), which is a known HAP and volatile organic compound (VOC) (*10, 11*).

Clifton FA-1051 adhesive (baseline) is currently registered on the QPL for a MMM-A-1617B Type-II adhesive (table 1) (*6*). This adhesive is oil resistant with a polychloroprene rubber base and is recommended for bonding natural and neoprene rubber. This product contains hexane and toluene, which are both HAPs and VOCs (*7*). This adhesive also contains methyl ethyl ketone (MEK) (*7*), which is non-HAP but is a VOC (*10, 11*).

Clifton FA-1076 adhesive (baseline) is registered on the QPL for a MMM-A-1617B Type-III adhesive (table 1) (*8*). This adhesive has a nitrile rubber base that will bond to Buna-N rubber and is resistant to fuels. Recommended uses are for the bonding of foams, vinyl, nitrile-coated fabric, and cotton fabric to steel. This adhesive has methyl isobutyl ketone (MIBK) (*9*), which is listed as a HAP and a VOC (*10, 11*). The two other solvents present in this adhesive are MEK and tetrahydrofuran (THF) (*9*), which are non-HAP but are considered VOCs.

1.1.2 Experimental Adhesive

Clifton Adhesive, Inc., provided experimental versions for each type of the MMM-A-1617B adhesive classification. Table 2 lists the physical properties of the experimental Clifton adhesives that contain either low or no HAPs.

Table 2. Physical properties of Clifton experimental MMM-A-1617B adhesives (*12–14*).

Adhesive	Type	Solvents	Solids (% by wt.)	HAP (% by wt.)	VOC (g/L)	Density (lb/gal)
Clifton FA-1053 non-HAP (experimental)	I	Heptane	40 ± 2	0	460–496	6.50–6.80
Clifton FA-1051 low-HAP (experimental)	II	Toluene Acetone MEK Heptane	25 ± 1	10–20	734–770	8.45–8.75
Clifton FA-1076 non-HAP (experimental)	III	Acetone	21 ± 1	0	9–17	8.00–8.30

Clifton FA-1053 non-HAP adhesive (experimental) is recommended for Type-I applications (*5*) (table 2) and contains ~50%–60% heptane, which is non-HAP but is a VOC (*10*, *11*). This adhesive is a natural rubber and SBR combination recommended for bonding cotton duck, leather, felt, and cork to themselves, aluminum, steel, or natural rubber (*5*).

Clifton FA-1051 low-HAP (experimental) adhesive is recommended for Type-II applications (table 2) (*6*). It contains a low percentage of HAPs but has a high VOC level consisting of 10%–20% toluene, 10%–20% MEK, and 20%–30% heptane (*10, 11*). This formulation also contains 10%–20% acetone, which is non-HAP and VOC exempt (*10, 11*). This adhesive is an oil-resistant, polychloroprene rubber base recommended for bonding natural and neoprene rubber (*6*).

Clifton FA-1076 non-HAP adhesive (experimental) is recommended for Type-III applications (table 2) (*8*). The adhesive is HAP-free and low-VOC because it contains 70%–80% acetone (*10*, *11*). This adhesive is a fuel-resistant, nitrile rubber base and is recommended for bonding nitrile rubbers or vinyl (*8*).

1.1.3 Alternative HAP-Free Adhesives

3M* adhesives, listed in table 3, were selected as possible HAP-free alternatives for the current HAP-containing baseline adhesives. The technical literature for these 3M products does not claim to meet the MMM-A-1617B specification. However, the reported physical properties and the technical data sheets (TDS) recommendations of these 3M adhesives were sufficient to include in this study.

* 3M is a trademark of 3M Company.

Table 3. Physical properties of potential alternative HAP-free adhesives (*15*–*20*).

Adhesive	**Type**	**Solvents**	**Solids (% by wt.)**	**HAP (% by wt.)**	**VOC (g/L)**	**Density (lb/gal)**
3M-847 (alternative)	NA	Acetone	33–39	0	0	7.40–7.80
3M-4491 (alternative)	NA	Acetone Cyclohexanone	22–26	0	42–52	7.00–7.40
3M-1099 (alternative)	NA	Acetone	31–37	0	0	7.30–7.50

Note: NA = not applicable.

3M Scotch-Weld (alternative) Nitrile High Performance Rubber and Gasket Adhesive 847 (3M-847) is a medium-viscosity grade for brush or flow application while providing strong, flexible bonds (*16*). 3M-847 is quick drying, with excellent resistance to many fuels and oils. This adhesive bonds leather, nitrile rubber, most plastics, and gasketing materials to a variety of substrates. The carrier solvent is acetone (table 3) (*15*). This product meets the requirements for MIL-C-4003 Federal Specification: Cement General Purpose and Synthetic Based (*16*). MIL-C-4003 was cancelled and superseded by MIL-A-5092 Federal Specification: Adhesive, Rubber Base, General Purpose. MIL-A-5092 was cancelled and superseded by MMM-A-1617A and superseded by the updated specification MMM-A-1617B (*3*). 3M-847 should meet the requirements of MMM-A-1617B. However, this product is not currently included on the QPL (*3*, *16*).

3M Scotch-Weld (alternative) Nitrile Industrial Adhesive 4491 (3M-4491) dries fast while providing a strong and flexible bond (*17*). 3M-4491 is resistant to weathering, water, fuels, oil, and plasticizer. This product bonds fabrics, leather, foams, plastics, vinyl extrusions, and sheeting. This formulation contains acetone and cyclohexanone (table 3) (*18*), which are both non-HAP solvents; however, cyclohexanone is a VOC (*10, 11*).

3M Scotch-Weld (alternative) Nitrile High Performance Plastic Adhesive 1099 (3M-1099) is a medium-viscosity grade for brush and flow applications, is fast drying, and forms strong and flexible bonds (*19*). 3M-1099 is resistant to weathering, water, fuels, oil, and plasticizers. This adhesive bonds fabrics, leather, foams, plastics, vinyl extrusions, and sheeting. The carrier solvent is acetone (table 3) (*20*), which is non-HAP and VOC exempt (*10, 11*).

2. Experimental Method

2.1 Substrate Preparation

Aluminum 7075-T6 (Al-7075), shown in figure 1, was cut to the dimensions of 6 × 1 × 0.25 in. The Al substrate was cleaned with MEK, followed by isopropyl alcohol (IPA). No resurfacing of the Al substrate was required. Unbleached cotton duck, plied yarn, and hard texture no. 10 (figure 1) were cut to the dimensions of 12.5 × 1.25 in (*21*). The duck substrates were cleaned with a brush to remove any loose dirt and debris. Neoprene rubber with a Duro-hardness rating of 60 (figure 2) was cut to the dimensions of 12.5 × 1 × 0.062 in. The rubber was hand sanded with a coarse abrasive paper and cleaned with acetone to remove any excess dirt and debris. The vinyl plastic substrate was transparent and flexible (figure 3), cut to the dimensions of 12.5 × 1 × 0.03 in, and cleaned with IPA. Release tape was placed ~4 in from the top of each substrate to create a clean and consistent edge for adhesive application. This also provided an unbounded section of material to insert into the upper grip of the Instron machine.

Figure 1. Al-7075 and cotton duck substrates.

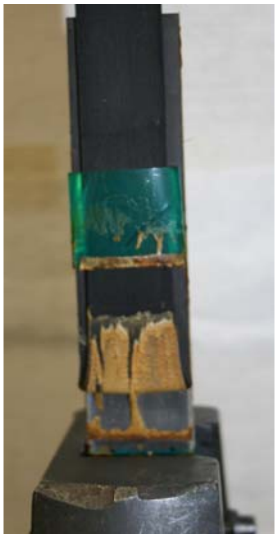

Figure 2. Al-7075 and neoprene rubber substrate.

Figure 3. Al-7075 and vinyl plastic substrates coated with adhesive.

2.2 Sample Assembly

Each adhesive was thoroughly mixed prior to application, and brushes were rinsed with IPA prior to use to remove any dirt or debris. The lab temperature was 69–72 °F, and relative humidity was 48%–53% at the time of adhesive application. A thin uniform coat of adhesive was brushed onto both substrates per assembly (figure 3). A second coat of adhesive was applied to both substrates, allowing drying between coats. A third coat of adhesive was required on the cotton duck substrate due to the porosity of the material. Before bonding the substrates together, the adhesives were allowed to dry until tacky. A hand roller 2 inches in diameter was used to remove trapped air in the adhesive layer of the assembly and to aid in the bonding process.

2.3 Strip Adhesion Testing

The American Society of Testing and Materials Method D 413-98 (ASTM D 413-98) Standard Test Methods for Rubber Property—Adhesion to Flexible Substrate was used as a guide to determine the adhesion strength of the 180° peel assemblies (*22*). Prior to the peel adhesion test, digital calipers were used to measure the thickness of the peel assemblies. The substrate thicknesses were subtracted from the measurement to determine the value for the dry adhesive film. Measurements were taken in triplicate, and the average thickness values were used to determine consistency in the dry adhesive film. Prior to testing, the samples were cut at the substrate/adhesive interface to prevent edge effects in the sample bond area, which could result in an incorrect tension measurement (*23*). The Al-7075 substrate was placed into the stationary lower wedge grip of the Instron machine (figure 4). The flexible substrate (duck, rubber, or vinyl plastic) was placed in the upper pneumatic air grip of the Instron machine (figure 4) and pulled away at a rate of 2 in/min from the adhesive layer at an angle of 180° to the Al substrate. The force needed to peel the substrates apart was measured in pounds of force (lbf) per square inch (in^2) (*22*).

Figure 4. Instron model no. 1123 (left) and stationary lower wedge grip (right).

After the 180° peel test was completed, the sample was examined for the type of failure that occurred. Adhesive failure (figure 5, left) is a rupture in the adhesive in which separation appears to be at the adhesive/substrate interface. Cohesive failure (figure 5, right) is the rupture of a bonded assembly in which the separation appears to be in the adhesive itself (*23*).

Figure 5. Adhesive failure to Al-7075 substrate (left) and cohesive failure (right).

2.4 Test Series

Table 4 summarizes the substrate combinations, conditioning, and results required for strip adhesion testing. All variations in table 4 required standard conditioning of 7 days at room temperature prior to any additional conditioning parameters. The following sections describe the parameters of each test.

Table 4. Strip adhesion strength specifications for MMM-A-1617B.

Substrate Combinations and Conditioning	Requirement		
	Type I (lbf)	**Type II (lbf)**	**Type III (lbf)**
After standard conditioning:			
Al/duck 7 days at RT (2.4.1)	12	15	10
Al/rubber 7 days at RT (2.4.2)	—	15	—
Al/vinyl 7 days at RT (2.4.3)	—	—	8
After immersion for 22 h:			
Al/duck DI H_20 at RT (2.4.4)	6	12	5
Al/duck IRM-901oil at 70 °C (2.4.5)	—	12	8
Al/duck JP-8 fuel at RT (2.4.6)	—	—	8
After accelerated heat aging:			
Al/duck 7 days at 70 °C (2.4.7)	12	15	10

Note: DI = deionized and RT = room temperature.

2.4.1 Aluminum Bonded to Cotton Duck Test Series

The Al bonded to cotton duck test series included all types (I, II, and III) of adhesives, and three assemblies per adhesive were made. Samples were assembled by bonding the cotton duck substrate to an Al-7075 substrate with a particular adhesive conditioned for 7 days at room temperature (RT) and peel tested at an angle of 180° (*3*). Products in this test series were Clifton FA-1053 (baseline), Clifton FA-1051 (baseline), Clifton FA-1076 (baseline), 3M-847 (alternative), 3M-4491 (alternative), 3M-1099 (alternative), Clifton FA-1053 (experimental) Clifton FA-1051 (experimental), and Clifton FA-1076 (experimental).

2.4.2 Aluminum Bonded to Neoprene Rubber Test Series

The Al bonded to neoprene rubber test series included Type-II and Type-III adhesives (oil and fuel resistant), and three assemblies per adhesive were made. Samples were assembled by bonding the neoprene rubber substrate to an Al-7075 substrate with a particular adhesive conditioned for 7 days at RT and peel tested at an angle of 180° (*3*). Products in this test series were Clifton FA-1051 (baseline), Clifton FA-1076 (baseline), 3M-847(alternative), 3M-4491 (alternative), 3M-1099 (alternative), Clifton FA-1051 (experimental), and Clifton FA-1076 (experimental).

2.4.3 Aluminum Bonded to Vinyl Plastic Test Series

The Al bonded to vinyl plastic test series included Type-III adhesives (fuel resistant), and three assemblies per adhesive were made. Samples were assembled by bonding the vinyl plastic substrate to an Al-7075 substrate with a particular adhesive conditioned for 7 days at RT and peel tested at an angle of 180° (*3*). Products in this test series were Clifton FA-1076 (baseline), 3M-847 (alternative), 3M-4491 (alternative), 3M-1099 (alternative), and Clifton FA-1076 (experimental).

2.4.4 Deionized Water Immersion Test Series

The DI water immersion test series included all types (I, II, and III) of adhesives, and three assemblies per adhesive were made. Samples were assembled by bonding the cotton duck substrate to an Al-7075 substrate with a particular adhesive conditioned for 7 days at RT, immersed in DI water for 22 h at RT, allowed to dry at RT after removal from DI water, and peel tested at an angle of 180° within 24 h (*3*). Products in this test series were Clifton FA-1053 (baseline), Clifton FA-1051 (baseline), Clifton FA-1076 (baseline), 3M-847 (alternative), 3M-4491 (alternative), 3M-1099 (alternative), Clifton FA-1053 (experimental), Clifton FA-1051(experimental), and Clifton FA-1076 (experimental).

2.4.5 IRM-901 Oil Immersion Test Series

The oil immersion test series included Type-II and Type-III adhesives (oil and fuel resistant), and three assemblies per adhesive were made. Samples were assembled by bonding the cotton duck substrate to an Al-7075 substrate with a particular adhesive conditioned for 7 days at RT, immersed in IRM-901 oil for 22 h at 70 °C, and peel tested at an angle of 180° (*3*). IRM-901 oil is "severely solvent refined heavy paraffinic petroleum oil" (*24*) that is referenced in ASTM D 471-98 (*25*). Products used in this test series were Clifton FA-1051 (baseline), Clifton FA-1076 (baseline), 3M-847 (alternative), 3M-4491 (alternative), Clifton FA-1051(experimental), and Clifton FA-1076 (experimental).

2.4.6 JP-8 Fuel Immersion Test Series

Fuel immersion test series included Type-III adhesives (fuel resistant), and three assemblies per adhesive were made. Samples were assembled by bonding the cotton duck substrate to an Al-7075 substrate with a particular adhesive conditioned for 7 days at RT, immersed in JP-8 fuel for 22 h at RT, and peel tested at an angle of 180° (*3*). MMM-A-1617 suggests ASTM D 471-98, fuel B as the reference liquid for fuel immersion, which has a composition of isooctane (70%) and toluene (30%) (*25*). A fuel commonly used by the Army (JP-8) was selected as a replacement for fuel B in this research. JP-8 has a composition of ~99% kerosene and ~1% diethylene glycol monomethyl ether (*26*). Products in this test series were Clifton FA-1076 (baseline), 3M-847 (alternative), 3M-4491 (alternative), and Clifton FA-1076 (experimental).

2.4.7 Accelerated Heat Aging Test Series

The accelerated heat aging test series included all types (I, II, and III) of adhesives, and three assemblies per adhesive were made. Samples were assembled by bonding the cotton duck substrate to an Al-7075 substrate with a particular adhesive conditioned for 7 days at RT, heat aged for 7 days at 70 °C, and peel tested at an angle of 180° (*3*). Products in this test series were Clifton FA-1053 (baseline), Clifton FA-1051 (baseline), Clifton FA-1076 (baseline), 3M-847 (alternative), 3M-4491 (alternative), 3M-1099 (alternative), Clifton FA-1053 (experimental), Clifton FA-1051 (experimental), and Clifton FA-1076 (experimental).

3. Results

3.1 Aluminum Bonded to Cotton Duck Test Series

The required adhesion strength (table 4) for a bond between a cotton duck substrate to an Al-7075 substrate in this test series (section 2.4.1) was 12 lbf for Type I, 15 lbf for Type II, and 10 lbf for Type III (table 4). Figure 6 illustrates the bond strength of all nine adhesives in this test series. Clifton FA-1051 (baseline), 3M-847 (alternative), 3M-4491 (alternative), 3M-1099 (alternative), and Clifton FA-1053 (experimental) exceeded the minimum requirements for this test series. Clifton FA-1051 and 3M-847 had a bond strength exceeding 23 lbf, which is approximately double the specification for Type-I and Type-III adhesives. 3M-1099 (alternative) and Clifton FA-1053 (experimental) both had a bond strength of ~18 lbf, and 3M-4491 had a bond strength of ~16 lbf, which exceeded the minimum specification for all three types of adhesive. Clifton FA-1053 (baseline) and Clifton FA-1076 (baseline) did not meet the minimum requirements for bond strength in this test series despite being registered on the QPL. Clifton FA-1051 (experimental) and Clifton FA-1076 (experimental) did not meet the requirements for bond strength in this test series and are therefore not acceptable replacements.

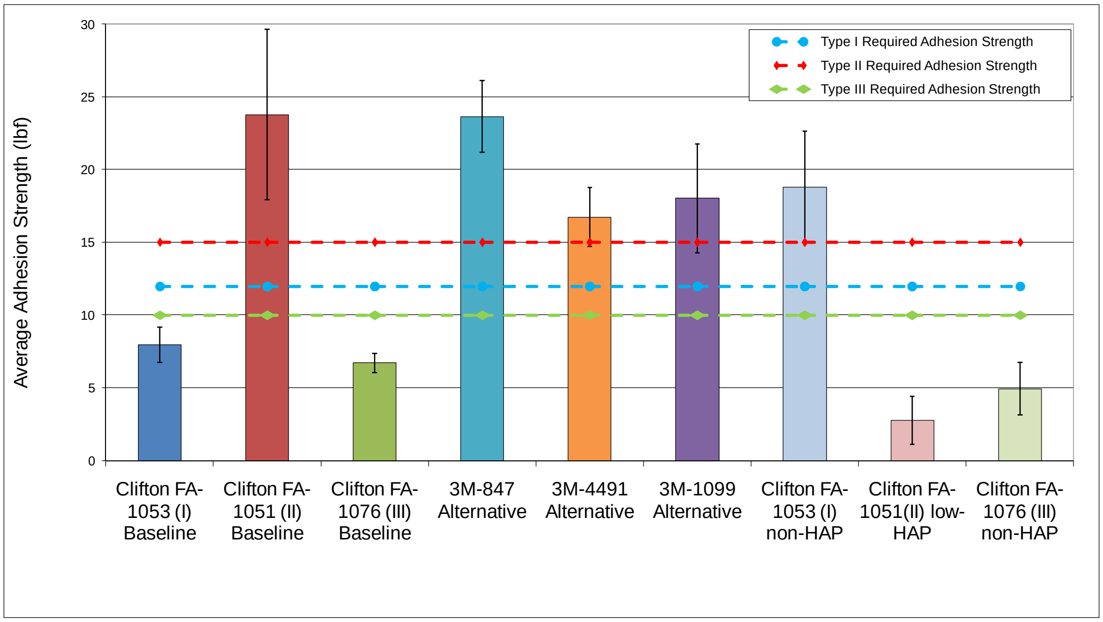

Figure 6. Adhesion results for Al-7075 bonded to duck test series.

3.2 Aluminum Bonded to Neoprene Rubber Test Series

The required adhesion strength of the bond between a neoprene rubber substrate to an Al-7075 substrate was 15 lbf for Type-II adhesives (table 4). The graph in figure 7 shows that 3M-847 (alternative) had a bond strength of 15.1 lbf, which meets the minimum required adhesion strength. The remaining adhesives in this test series did not meet that specification, including Clifton FA-1051 (baseline).

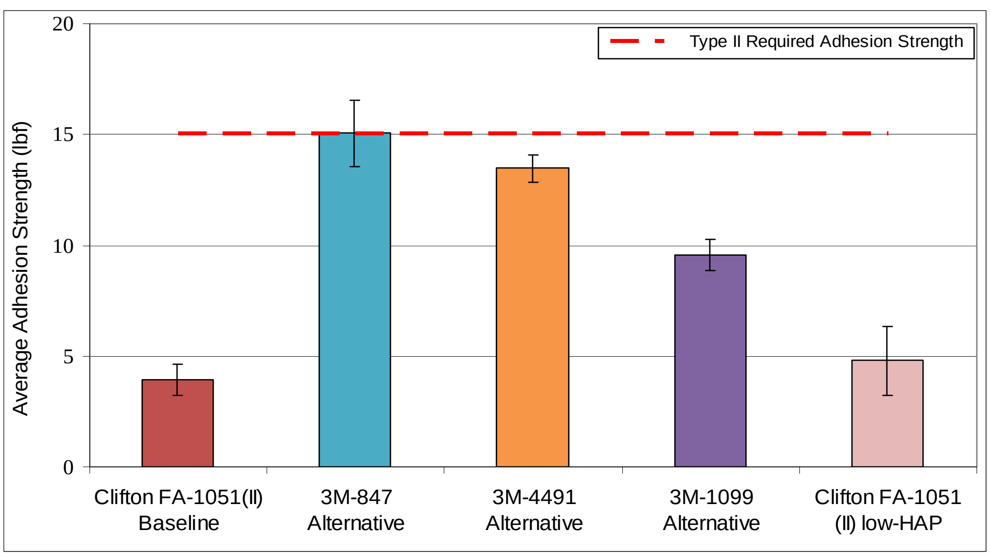

Figure 7. Adhesion results for Al-7075 bonded to neoprene rubber test series.

3.3 Aluminum Bonded to Vinyl Plastic Test Series

The required adhesion strength of the bond between a vinyl plastic substrate to an Al-7075 substrate was 8 lbf for Type-III adhesives (table 4). The 3M-847 (alternative) and Clifton FA-1076 (experimental) both exceeded the minimum requirement with adhesion strength values of 9.7 and 9.9 lbf, respectively (figure 8). The remaining adhesives in this test series did not meet the minimum specification, including Clifton FA-1076 (baseline).

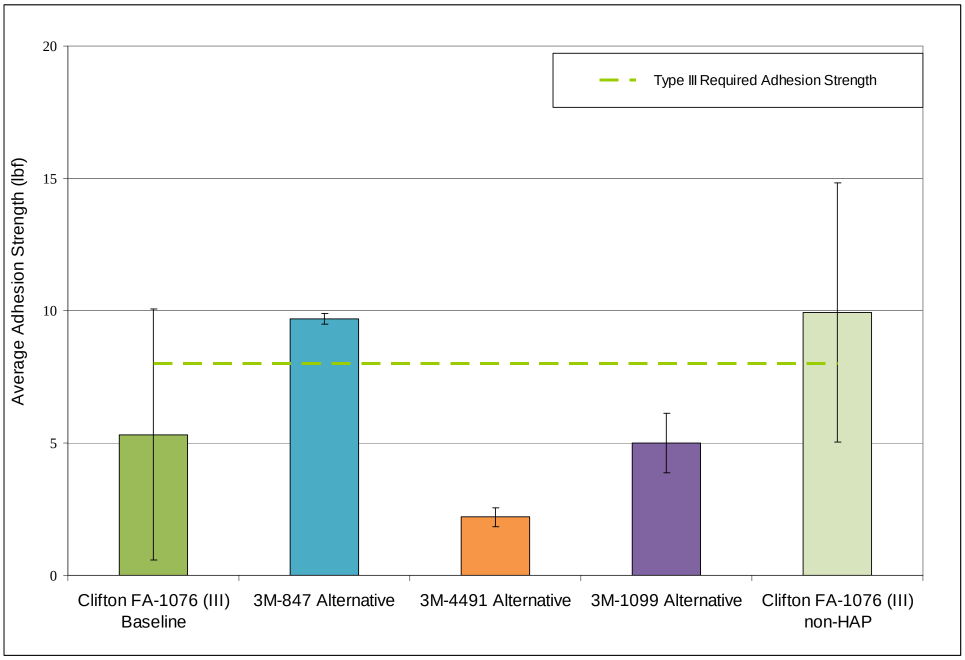

Figure 8. Adhesion results for Al-7075 bonded to vinyl plastic test series.

3.4 DI Water Immersion Test Series

The required adhesion strength of the bond between a cotton duck substrate to an Al-7075 substrate, conditioned at RT for 7 days and immersed in deionized (DI) water for 22 h at RT, was 6 lbf for Type-I, 12 lbf for Type-II, and 5 lbf for Type-III adhesives (table 4). Clifton FA-1053 (baseline), Clifton FA-1051 (baseline), 3M-847 (alternative), 3M-4491 (alternative), and 3M-1099 (alternative) exceeded the adhesion strength minimum requirements with values of 6.7, 19.2, 30.5, 20.7, and 20.1 lbf, respectively, as shown in figure 9. The remaining adhesives in this test series did not meet the required specification, including the baseline adhesive Clifton FA-1076.

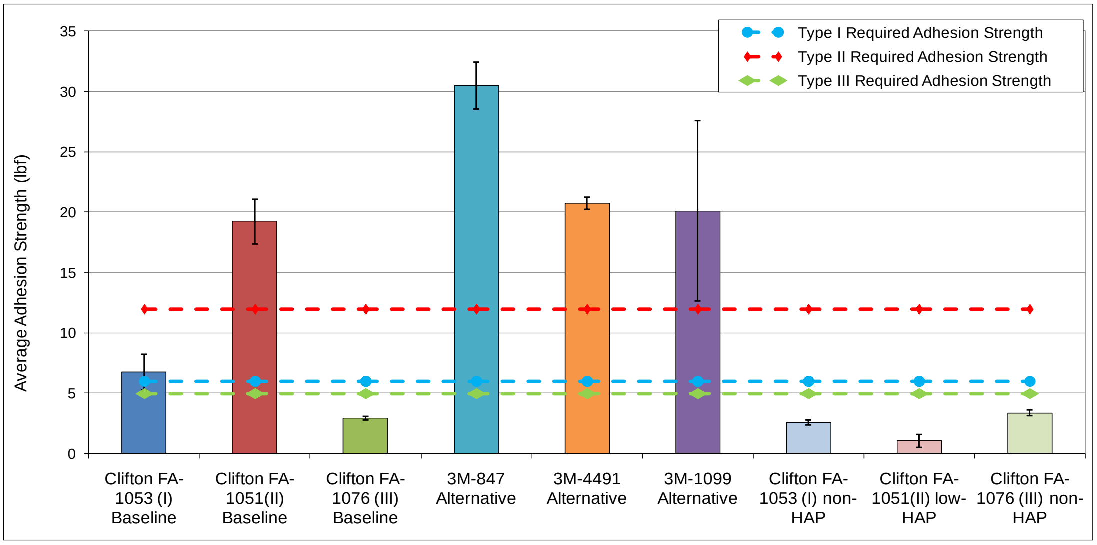

Figure 9. Adhesion results for Al-7075 bonded to duck, DI water immersion test series.

3.5 IRM-901 Oil Immersion Test Series

The required adhesion strength of the bond between a cotton duck substrate to an Al-7075 substrate conditioned at RT for 7 days and immersed in IRM-901 oil for 22 h at 70 °C was 12 lbf for Type-II adhesives and 8 lbf for Type-III adhesives (table 4). Clifton FA-1076 (baseline), 3M-847 (alternative), 3M-4491 (alternative), and 3M-1099 (alternative) met or exceeded the minimum requirements with adhesion values of 8.83, 17.2, 25.2, and 12.9 lbf, respectively, as shown in figure 10. The remaining adhesives in this test series did not meet the specification.

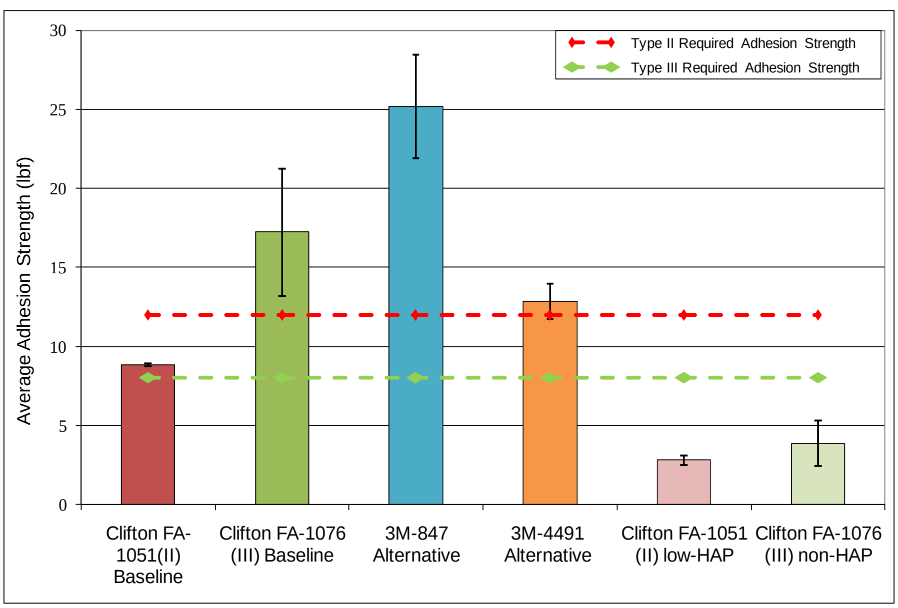

Figure 10. Adhesion results for Al-7075 bonded to duck, IRM-901 oil immersion test series.

3.6 JP-8 Fuel Immersion Test Series

The required adhesion strength of the bond between a cotton duck substrate to an Al-7075 substrate conditioned at RT for 7 days and immersed in JP-8 fuel for 22 h at RT was 8 lbf for Type-III adhesives (table 4). The 3M-847 (alternative) and 3M-4491 (alternative) exceeded the minimum requirements with adhesion values of 23.2 and 13.2 lbf, respectively (figure 11). The remaining adhesives in this test series did not meet the required specifications, including Clifton FA-1076 (baseline).

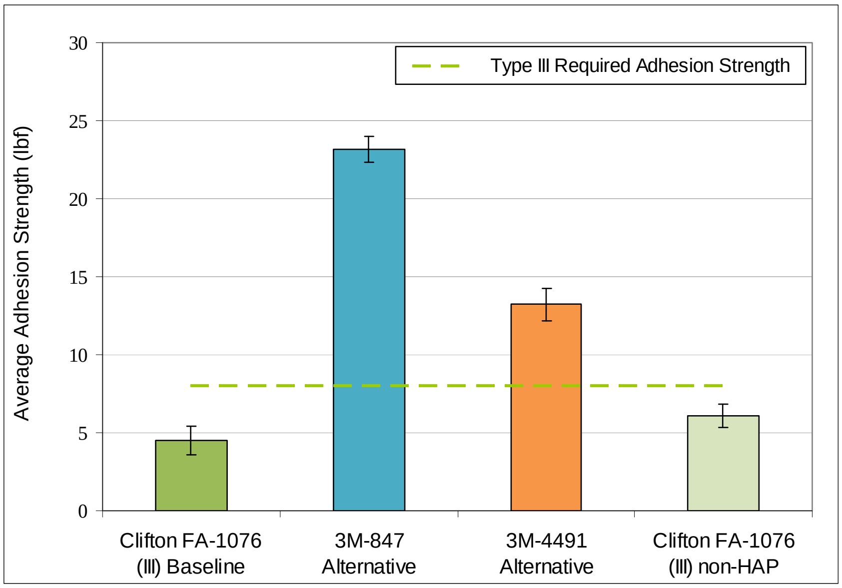

Figure 11. Adhesion results for Al-7075 bonded to duck, JP-8 fuel immersion test series.

3.7 Accelerated Heat Aging Test Series

The required adhesion strength of the bond between a cotton duck substrate to an Al-7075 substrate conditioned 7 days at RT and heat aged for 7 days at 70 °C was 12 lbf for Type-I, 15 lbf for Type-II, and 10 lbf for Type-III adhesives (table 4). In figure 12, the baseline adhesives Clifton FA-1053 (12.8 lbf), Clifton FA-1051 (27.5 lbf), and Clifton FA-1076 (10.5 lbf) met or exceeded the minimum requirements in this test series. The alternative products, 3M-847 (40.2 lbf), 3M-4491 (22.5 lbf), and 3M-1099 (22.7 lbf), exceeded the requirements for all three adhesive types. The three experimental adhesives failed to meet the minimum required specifications for adhesion in this test series.

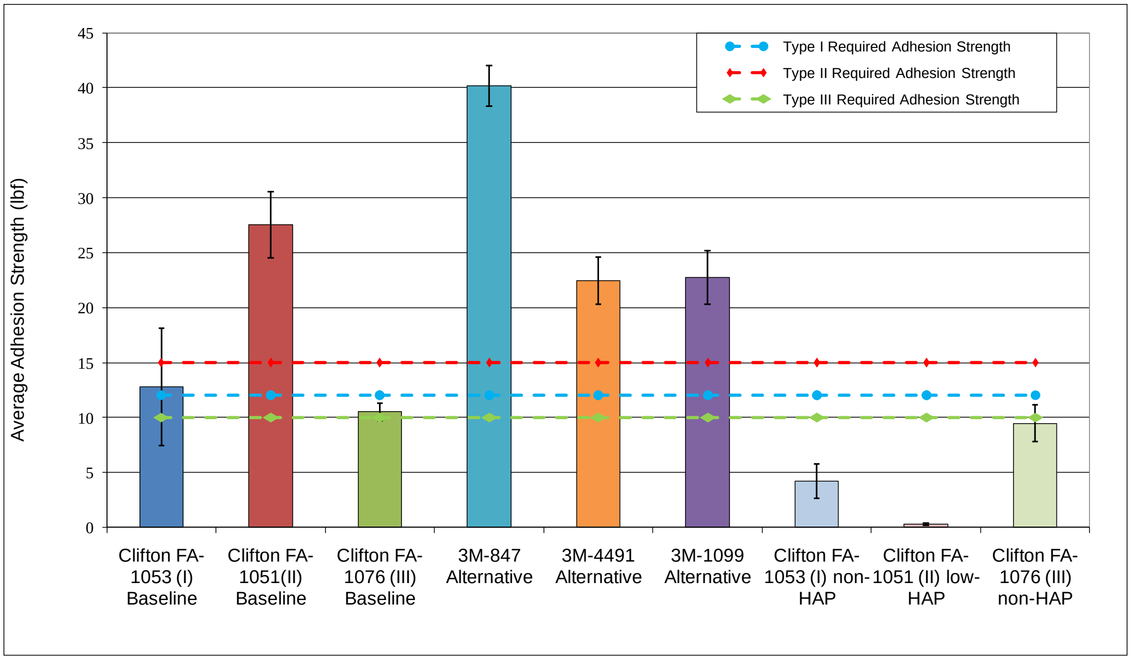

Figure 12. Adhesion results for Al-7075 bonded to duck, accelerated heat aging test series.

4. Discussion

4.1 Adhesive Performance

All adhesives used in this research were acquired shortly before testing and used before the expiration date. Table 5 summarizes the overall proficiency of the nine adhesives in this test series for Type-I applications. Clifton FA-1053 (baseline) only passed two of three required tests despite being on the QPL for Type-I applications. It failed the Al/duck performance with standard conditioning, which is among the easier performance metrics to meet for MMM-A-1617. Interestingly, Clifton FA-1051 (baseline), which is on the QPL for Type-II applications, met the necessary performance requirements for Type I. Clifton FA-1076 (baseline) failed two out of the three tests. 3M-847, 3M-4491, and 3M-1099 met the performance for all three tests. All three Clifton experimental products failed the tests for Type-I applications. In fact, only one experimental Clifton adhesive (FA-1053) passed a single test.

Table 5. Overall proficiency of adhesives for Type-I requirements.

Substrate Combination & Conditioning Parameters	Clifton FA-1053 (I) Baseline	Clifton FA-1051 (II) Baseline	Clifton FA-1076 (III) Baseline	3M- 847 Alternative	3M-4991 Alternative	3M-1099 Alternative	Clifton FA-1053 (I) Experimental	Clifton FA-1051 (II) Experimental	Clifton FA-1076 (III) Experimental
After Standard Conditioning (7 days @ RT)									
Al to Duck	fail	pass	fail	pass	pass	pass	pass	fail	fail
After Standard Conditioning & Immersion (22 hours)									
Al to Duck DI H_2O @ RT	pass	pass	fail	pass	pass	pass	fail	fail	fail
After Standard Conditioning & Accelerated Heat Aging (7 days @ 70°C)									
Al to Duck	pass	pass	pass	pass	pass	pass	fail	fail	fail

Table 6 summarizes the performance of Clifton FA-1051, baseline and experimental, and the three 3M products according to Type-II performance requirements. Only 3M-847 met all performance Type-II requirements. The baseline Clifton product failed the Al/rubber substrate combination and the IRM-901 oil immersion testing. The experimental Clifton (FA-1051) product failed all Type-II tests. The other two 3M products (3M-4491 and 3M-1099) performed relatively well in that they only failed the Al/rubber substrate combination test.

Table 6. Overall proficiency of adhesives for Type-II requirements.

Substrate Combination & Conditioning Parameters	Clifton FA-1051 (II) Baseline	3M- 847 Alternative	3M-4991 Alternative	3M-1099 Alternative	Clifton FA-1051 (II) Experimental
After Standard Conditioning (7 days @ RT)					
Al to Duck	pass	pass	pass	pass	fail
Al to Rubber	fail	pass	fail	fail	fail
After Standard Conditioning & Immersion (22 hours)					
Al to Duck DI H_2O @ RT	pass	pass	pass	pass	fail
Al to Duck IRM-901 Oil @ 70°C	fail	pass	pass	n/a	fail
After Standard Conditioning & Accelerated Heat Aging (7 days @ 70°C)					
Al to Duck	pass	pass	pass	pass	fail

Note: n/a = not applicable.

Table 7 summarizes the performance of Clifton FA-1076, baseline and experimental, and the three 3M products according to Type-III performance requirements. Clifton FA-1076 (baseline and experimental) passed only one of six required tests. On the other hand, 3M-847 and

3M-4491 passed all six tests. 3M-1099 passed four of the six tests but was not tested for oil and fuel immersion because it was originally chosen as a possible adhesive for the vinyl plastic substrate. The TDS for 3M-1099 did not list metal as a common substrate and thus was not expected to perform as well as it did.

Table 7. Overall proficiency of adhesives for Type-III requirements.

Substrate Combination & Conditioning Parameters	Clifton FA-1076 (III) Baseline	Clifton FA-1051 (II) Baseline	3M- 847 Alternative	3M-4991 Alternative	3M-1099 Alternative	Clifton FA-1076 (III) Experimental
After Standard Conditioning (7 days @ RT)						
Al to Duck	fail	pass	pass	pass	pass	fail
Al to Vinyl	fail	n/a	pass	pass	pass	pass
After Standard Conditioning & Immersion (22 hours)						
Al to Duck DI H_2O @ RT	fail	pass	pass	pass	pass	fail
Al to Duck IRM-901 Oil @ 70°C	pass	fail	pass	pass	n/a	fail
Al to Duck JP-8 Fuel @ RT	fail	n/a	pass	pass	n/a	fail
After Standard Conditioning & Accelerated Heat Aging (7 days @ 70°C)						
Al to Duck	pass	pass	pass	pass	pass	fail

Note: n/a = not applicable.

The Clifton FA-1051 baseline, although qualified as a Type-II adhesive, met the requirements for a Type-I adhesive. The 3M-4491 met Type-I and Type-III performance requirements, and 3M-1099 met the specification for Type I. Additionally, 3M-847 met all performance requirements regardless of application type. Conversely, the Clifton experimental adhesives (I, II, and II) do not meet any of the adhesives performance requirements.

Overall, the results indicate that the 3M adhesives are higher performing than the Clifton products. These adhesive formulations all contained a nitrile polymer base, also known as acrylonitrile butadiene. Nitrile has good resistance to oil, water, and heat. The adhesive properties can be increased by increasing the nitrile content in the formulation (*27*). According to the material safety data sheet (MSDS), 3M-847 has a content of 10%–30% nitrile, whereas 3M-1099 has a content of 10%–20% (*15*). Both of these 3M adhesives also contain 5%–10% phenol formaldehyde polymer (phenolic resin), which is a tackifier and adhesion promoter (*28*).

Tackifiers in adhesive formulation are materials used to enhance the “wet-grab” property that causes contacting surfaces to adhere to each other upon initial contact prior to chemical curing (*28*). According to ASTM, wet bond strength in the context of this research is used to describe the strength of a joint when the adherends are brought together with the adhesive still in the wet state (*23*). In particular, 3M-847 had a perceptible “wet grab” that was far stronger than all the other adhesives. Phenol-formaldehyde adhesion promoters are capable of enhancing adhesion durability due to improvement in substrate wetting and the formation of chemical bonds across the film/substrate interface (*28*). Adhesives with nitrile-phenolic blends have enhanced peel strength without reduction in high-temperature strength, high wet and dry strength, and are resistant to moisture (*28*).

The material "glycerol esters of rosin acids" is specified at a level of 7%–13% on the 3M-847 MSDS. This material is used as a thermoplastic resin, also known as a "modifier," unique to the 3M-847 formulation in this test series. Thermoplastic resin is defined as a material that becomes soft and pliable when heated without change in its other properties and hardens when cooled again (*29*). The addition of a thermoplastic resin to a nitrile-phenolic blend in a contact adhesive formulation assists in the flexibility of the product, thus increasing the fracture toughness and decreasing brittleness of the adhesive interface, allowing for better adhesion (*30*).

Both Clifton FA-1053 (baseline) and Clifton FA-1053 (experimental) adhesives are composed of a natural rubber and SBR combination and classified as Type I (*4*, *5*). The amount of each rubber material in these formulations was not provided in the MSDS. The adhesion properties of SBR are not as good as nitrile or neoprene but have a lower cost (*27*). Both Clifton FA-1051 (baseline) and Clifton FA-1051 (experimental) adhesives are classified as Type II and are composed of a polychloroprene (neoprene) rubber base (*7*, *13*). Polychloroprene has similar properties to natural rubber but is stronger, has better aging properties, and good resistance to water (*27*). Thus, it was expected that this formulation should meet all the performance specifications for MMM-A-1617, but only the baseline Clifton FA-1051 did for Type-I applications. The amount of polychloroprene in the formulation was not provided, and thus low rubber contents could play a role in the poor adhesion (*27*). Both Clifton FA-1076 (baseline) and Clifton FA-1076 (experimental) adhesives are classified as Type III and are composed of a nitrile rubber base (*9*, *14*). Nitrile has good resistance to oil and heat. Because this is a nitrile formulation, this adhesive was expected to meet MMM-A-1617 Type-I specifications, but neither the Clifton FA-1076 baseline nor experimental product met any of the MMM-A-1617 performance requirements. Again, the amount of nitrile contained in the formulation was not provided and could play a factor in the low adhesion (*27*).

Overall, none of the Clifton baseline products met their applicable MMM-A-1617 type specifications, while in the past they must have met specifications in order to become listed on the QPL. Thus, the reduced performance relative to the specifications for the Clifton baseline adhesives are most likely due to process or formulation modifications made to the product over time that may have degraded the performance of the products. It should be noted that 1995 was the latest revision of the MMM-A-1617 specification. Most likely, these adhesives have not had to qualify according to the specification for these 15 years and probably even longer. In addition, the most recent MSDSs available for Clifton Type-I and Type-III baseline adhesives are also dated 1995 (Type II, 1998), which supports the length of time that has lapsed since the products were qualified. During that time, it is possible that raw materials were replaced because of availability or cost leading to variations in the performance of the adhesives. As a result, requalification of the baseline adhesives is recommended.

The experimental Clifton adhesives performed worse than the baseline adhesives for Type-I and Type-II MMM-A-1617 applications. According to the technical literature, the only difference between the Clifton baseline adhesives and the Clifton experimental adhesives is the solvent system used in the individual formulations (*4–9*). Thus, based on this information, similar performance would be expected. The lower performance for the experimental Clifton products is likely due to the slower evaporation of the non-HAP solvents (*31*). In fact, the Clifton FA-1053 and FA-1051 (experimental) used heptane instead of hexane that is used in the baseline products (*4*, *7*, *12*, *13*). Therefore, entrapped solvent is possible in the experimental products, not allowing the adhesive to cure to full strength. In comparison, the 3M HAP-free adhesives contain high-volatile solvents, including acetone, which are not likely to become entrapped (*15*, *18*, *19*). In addition, Clifton FA-1076 (experimental) used acetone as the non-HAP solvent and performed as well as the baseline product (*8*, *9*).

4.2 Comparison of Bond Strength With Various Substrates

Figure 13 shows a comparison of the test series adhesives with three substrate combinations (Al/duck, Al/rubber, and Al/vinyl) conditioned for 7 days at RT. In general, for the products in this test series, the adhesive strength was highest for Al/duck and lowest for Al/vinyl. The cotton duck was very porous compared to both the neoprene and the vinyl, resulting in better adhesion to the substrate. The rubber substrate surface was roughened with an abrasive paper to help the adhesion, but the vinyl substrate was simply cleaned with IPA as not to mar the surface. Only one adhesive, 3M-847, performed well across all three substrate combinations. Two adhesives, 3M-4491 and 3M-1099, performed well on duck and neoprene but poorly on vinyl. No Clifton adhesives, baseline or experimental, performed well on more than one substrate. Thus, the 3M adhesives are more versatile than the Clifton adhesives. The nitrile rubber base, tackifiers, and high volatility of acetone used in the 3M products may explain the better performance compared to both the Clifton baseline and experimental products.

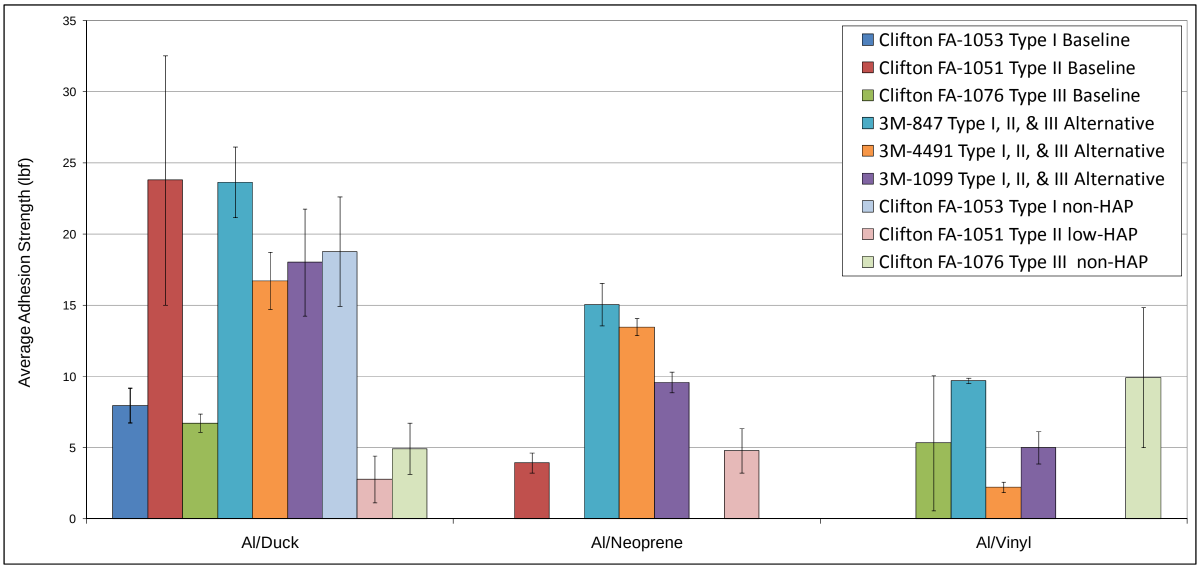

Figure 13. Comparison of adhesives with various substrates combinations.

Performance changes per substrate combination from Clifton baseline adhesives to the Clifton experimental adhesives (table 8) are noticeable. The experimental Clifton Type-I product had significantly higher performance than the baseline Clifton Type-I adhesive. On the other hand, for the Type-II and Type-III adhesives, the experimental Clifton products underperformed the Clifton baseline products for Al/duck, while it had improved performance for Al/rubber or Al/vinyl. The erratic findings may be a result of how the different solvent systems interact and aid in bonding with the particular substrates.

Table 8. Performance of Clifton experimental adhesives relative to Clifton baseline adhesives of the same type.

Substrate Combination	Type I (%)	Type II (%)	Type III (%)
Al/duck	136	–88	–27
Al/neoprene	NA	23	NA
Al/vinyl	NA	NA	87

Note: NA = not applicable.

4.3 Effects of Various Conditioning Parameters on Bond Strength

Figure 14 and table 9 show the effects of various conditioning parameters on bond strength. All assemblies in this test series were made with Al-7075 bonded to cotton duck. Prior to the secondary conditioning described in the figure 14, all assemblies were conditioned for 7 days at RT (standard conditioning). The minimum bond strength requirements for the various test conditions are found in table 4. In general, accelerated heat aging improved the performance of the adhesives. The TDS for all three alternative products manufactured by 3M affirms that the adhesives may be heat cured to improve physical properties (*16*, *17*, *20*). Water immersion decreased the performance of all of the Clifton products, both baseline and experimental. Some decrease is expected because water can disrupt the bonding, thereby reducing adhesion strength. The performance of the Type-I and Type-II baseline products, FA-1053 and FA-1051, were only slightly reduced, while the Type-III FA-1076 (baseline) was more significantly reduced. The opposite trend was seen for the experimental products where the FA-1076 (experimental) retained more of its adhesive properties relative to FA-1053 (experimental) and FA-1051 (experimental). Again, this could be explained by the solvent substitutions used, since the solvents for FA-1076 are more volatile in the experimental product, while they are less volatile for experimental FA-1053 and FA-1051 (*31*). The 3M products slightly improved their adhesive performance after water immersion. The retention of performance was expected because technical literature shows that these adhesives are weather and water resistant (*16*, *18*, *20*). Oil and JP-8 immersion generally decreased the performance of the adhesives. One exception was the increased performance of Clifton FA-1076 (baseline) after oil immersion. The oil immersion was performed at elevated temperature and may allow for the adhesive to flow better, forming improved bonds with the substrates. This same effect was not seen for FA-1076 (experimental), but this product did at least retain its adhesive properties during oil and JP-8 immersion. There is a strong indication that 3M-1099 would likely meet Type-III requirements, assuming only a 20%–25% reduction in properties due to oil or JP-8 immersion as the trend exhibited for 3M-4491 (table 9). The necessary percent reduction in performance to fail immersion in oil or JP-8 would be more than 50%.

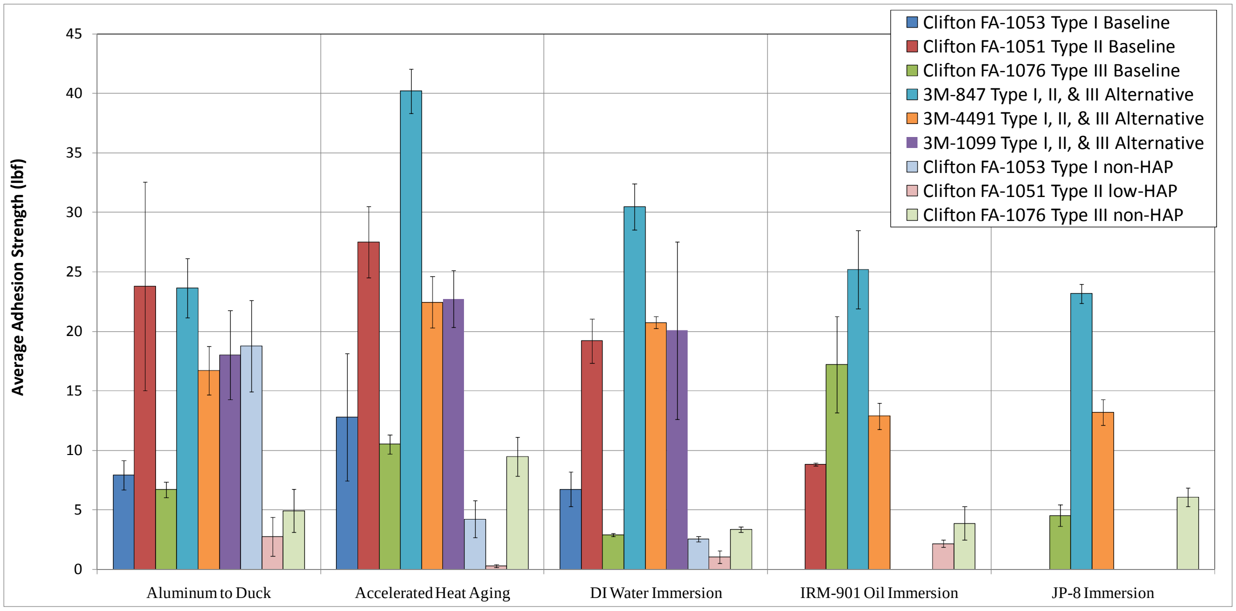

Figure 14. Comparison of various conditioning parameters on bond strength.

Table 9. Comparison of percent change under various conditioning parameters relative to standard conditioning.

Conditioning	FA-1053 baseline (%)	FA-1051 baseline (%)	FA-1076 baseline (%)	3M-847 alternative (%)	3M-4491 alternative (%)	3M-1099 alternative (%)	FA-1053 experimental (%)	FA-1051 experimental (%)	FA-1076 experimental (%)
Heat Aging	61.3	15.8	56.9	70.0	34.3	26.3	-77.5	-89.5	92.7
DI H_2O	-15.1	-19.2	-56.3	28.9	24.0	11.6	-86.4	-61.2	-31.5
IRM-Oil 901	n/a	-62.9	156.6	6.5	-23.0	n/a	n/a	-21.0	-21.1
JP-8	n/a	n/a	-32.5	-2.0	-21.0	n/a	n/a	n/a	23.4

Note: n/a = not applicable.

5. Summary and Conclusions

In accordance with the SPOTA initiative to decrease HAP-containing adhesives, sealants, and coatings, a low-HAP or HAP-free adhesive replacement is required for MMM-A-1617B Federal Specification Adhesive, Rubber Base, General Purpose. The Clifton baseline adhesives in this research were designed to meet the MMM-A-1617B (Types I, II, and III) but currently contain HAPs. Clifton Adhesives, Inc., provided low-HAP and HAP-free experimental adhesives that were also designed to meet the requirements of MMM-A-1617B. Based on testing results, the Clifton experimental and baseline adhesives did not meet all of the requirements for MMM-A-1617B, likely due to deleterious reformulation of the rubber base and solid additives in these adhesives. Because the baseline Clifton products are listed on the QPL but failed these tests, it is recommended that Clifton requalify these products through the specification-preparing activity. The experimental Clifton products performed more poorly than the baseline Clifton products, likely as a result of solvent entrapment caused by the use of less volatile solvents. The 3M alternative adhesive products performed better than the Clifton products as a result of the high content of nitrile rubber blended with phenolic tackifiers. In particular, 3M-847 had the best performance across all test parameters and substrate combinations. In fact, 3M-847 met the specifications for MMM-A-1617 for Type-I, Type-II, and Type-III applications. 3M-847 is an appropriate HAP-free/VOC-free replacement for the current MMM-A-1617B adhesives. 3M-4491 is a HAP-free and low-VOC replacement for MMM-A-1617 Type-I and Type-III specifications. 3M-1099 is a HAP/VOC-free replacement for MMM-A-1617 Type-I applications and may also meet Type-III specifications, but full testing was not completed for this adhesive. Adhesives that are used in compliance with MMM-A-1617B account for a significant percentage of the total adhesives and sealants used by the Army (*1*). Therefore, this research has shown that HAP-free 3M-847 can eliminate the associated HAP emissions from Army operations while improving performance of this adhesive classification.

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List of Symbols, Abbreviations and Acronyms

Al	aluminum
ARL	U.S. Army Research Laboratory
ASTM	American Society for Testing and Materials
DI	deionized
EPA	U.S. Environmental Protection Agency
HAP	hazardous air pollutant
IPA	isopropyl alcohol
MEK	methyl ethyl ketone
MIBK	methyl isobutyl ketone
MSDS	material safety data sheet
NDCEE	National Defense Center for Energy and Environment
NESHAP	National Emission Standard for Hazardous Air Pollutants
QPL	qualified product list
RT	room temperature
SBR	styrene butadiene rubber
SPOTA	Sustainable Painting Operations for the Total Army
TDS	technical data sheet
THF	tetrahydrofuran
VOC	volatile organic compound

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