INL/CRADA-07-18

CRADA FINAL REPORT

Evaluation of Trace Particle Removal Techniques for Enhanced Detection of Explosives in Air Cargo

Idaho National Laboratory

and

Battelle Memorial Institute

Completed: May 12, 2010

Prepared by
Idaho National Laboratory
Idaho Falls, Idaho 83415
http://www.inl.gov
Under DOE Idaho Operations Office
Contract No. DE-AC07-05ID14517

Defer Release Until May 12, 2015

The INL is a U.S. Department of Energy National Laboratory
operated by Battelle Energy Alliance

INL/EXT-10-18072

Evaluation of Trace Particle Removal Techniques for Enhanced Detection of Explosives in Air Cargo

Ernesto Cespedes, Carla J. Miller, Kendell Horman, and Alyssa Melander

March 2010

NL/EXT-10-18072

Evaluation of Trace Particle Removal Techniques for Enhanced Detection of Explosives in Air Cargo

March 2010

Idaho National Laboratory
Security Systems Technology Department
Idaho Falls, Idaho 83415

Evaluation of Trace Particle Removal Techniques for Enhanced Detection of Explosives in Air Cargo

Performed by
Idaho National Laboratory
Security Systems Technology Department
Idaho Falls, ID 83415

External Report: INL/EXT-10-18072

March 2010

Authors:

Dr. Ernesto Cespedes
Explosives Program Lead, Defense Systems and Technology
Battelle Energy Alliance, LLC

Date

Carla Miller
Chemist
Battelle Energy Alliance, LLC

Date

ABSTRACT

This report describes the procedures used and the test results obtained at the Idaho National Laboratory (INL), to determine the ability of thermal and mechanical (vibration and airflow) methods to remove/harvest trace explosives from various material substrate surfaces relevant to air cargo. As part of the Battelle Memorial Institute's (BMI) Air Cargo Screening (ACS) Initiative, INL conducted mechanical and thermal/mechanical removal tests using three different military explosives: C-4(RDX), cast TNT, and SEMTEX (PETN, 76% and RDX, 4.6%) on two substrate materials relevant to air cargo: metal and cardboard. This work leveraged similar experiments using homemade explosives (HME) which were funded by the Department of Homeland Security's Science and Technology Directorate (DHS S&T). The DHS S&T tests involved evaluating the removal efficiencies of thermal and mechanical methods for four different HME: Ammonium Nitrate prills (AN, explosive grade), Guanidine Nitrate (GN, 98% pure in Methanol), Triacetone Triperoxide (TATP, 99.5% pure in Acetonitrile), and Hexamethylene Triperoxide Diamine (HMTD, 98.4% pure in Acetonitrile). Substrate materials used for the HME removal tests included metal, cotton, canvas, leather, shrink wrap, cardboard, and vinyl. This report documents the procedures that were used during the testing as well as the test results and recommendations for future Air Cargo Screening applications.

EXECUTIVE SUMMARY

This report describes the procedures used and the test results obtained at the Idaho National Laboratory (INL), to determine the ability of thermal and mechanical (vibration and airflow) methods to remove/harvest trace explosives from various material substrate surfaces relevant to air cargo. As part of the Battelle Memorial Institute's (BMI) Air Cargo Screening (ACS) Initiative, INL conducted mechanical and thermal/mechanical removal tests using three different military explosives: the plastic explosives C-4 (RDX, 91%) and SEMTEX (PETN, 76% and RDX, 4.6%) and cast TNT deposited on two substrate materials relevant to air cargo: metal and cardboard. This work leveraged similar experiments using homemade explosives (HME) which were funded by the Department of Homeland Security's Science and Technology Directorate (DHS S&T). The DHS S&T tests involved evaluating the removal efficiencies of thermal and mechanical methods for four different HME: Ammonium Nitrate prills (AN, explosive grade), Guanidine Nitrate (GN, 98% pure in Methanol), Triacetone Triperoxide (TATP, 99.5% pure in Acetonitrile), and Hexamethylene Triperoxide Diamine (HMTD, 98.4% pure in Acetonitrile). Substrate materials used for the HME removal tests included metal, cotton, canvas, leather, shrink wrap, cardboard, and vinyl.

The Military explosives test samples consisted of first-generation fingerprints produced using standard TSA transfer techniques. The HME test samples consisted of explosive crystals that were dry transferred to the substrate of interest using techniques similar to the Transportation Security Laboratory's (TSL) patented dry transfer technique (United States Patent 6470730). GN, TATP, and HMTD were purchased from Accustandard and allowed to dry. The AN prills were crushed and fingerprinted onto the substrate surfaces. Five hundred nanograms of guanidine nitrate, one milligram of HMTD and one milligram of TATP were re-crystallized for each substrate. However, during the transferring technique, the entire amount did not usually adhere to the surface of the material, so the actual amount tested was less than that crystallized. The amounts of AN and Military explosives used for each sample were not quantified, but the white powder was clearly visible on the substrate surfaces without the use of a microscope.

The vibration tests were conducted using the three military explosives on two substrates, and four HME types on seven substrates. Vibration testing involved using three frequencies (35 Hz, 70 Hz, and 100 Hz) and vibrating the sample at a constant acceleration at 5g root mean square (RMS) for one minute. A Vibration Test Systems Model number VG 100-8 desktop vibrator was used in these experiments. This vibrator was powered by a Techron Model 5515 power amplifier. The signal was generated from a Wavetek, 2 MHz sweep/function generator, Model 19. The accelerometer used was a PCB Piezotronics Vibration Division, Model number 393B04 with a sensitivity of 1000mV/g. The samples were photographed using a Zeiss Discover V12 stereoscope with Axiocam ICc1 3 megapixel digital camera prior to vibrating and then after the one minute time period. The changes in the pre-vibration and post-vibration samples were optically compared by layering the photographs and flickering between the co-registered images to detect changes. The tests were repeated in triplicate.

The explosive fingerprint/sample was placed on the substrate of interest and loaded onto the sample holder on the vibration drum. The sample was rotated upside down to remove any unattached particles that were sitting on the surface. The sample holder was then rotated back to the starting position under the microscope lens and the original picture was taken. The sample was then rotated upside down again and shaken at the designated frequency (35Hz, 70 Hz, or 100 Hz) and acceleration (5 g RMS).

In all of the tests performed using vibration for removal of HME particles, 48% had visible particle removal after shaking. This overall 48% removal rate consisted of, 74% of the HMTD samples, 19% of the TATP samples, 71% of the AN samples, and 27% of the guanidine nitrate samples. The vibration

removal rate for Military explosives was much poorer, with only two of the TNT on metal samples showing any particle removal. Overall vibration removal rates for Military explosives were 11% for TNT, 0% for SEMTEX, and 0% of C-4.

At 35 Hz: 86% HMTD, 5% TATP, 81% ammonium nitrate, and 33% guanidine nitrate samples had visible particle removal after shaking. At 70 Hz: 71% HMTD and ammonium nitrate, 33% TATP, and 24% guanidine nitrate samples had visible particle removal after shaking. At 100 Hz: 67% HMTD, 19% TATP, 62% ammonium nitrate, and 25% guanidine nitrate samples had visible particle removal after shaking. At all frequencies, 0% of C-4 and SEMTEX had any visible changes, and only TNT showed any particle removal (11% at 35Hz).

In addition, different surfaces had significantly different particle removal efficiencies that varied with different explosive types. Overall 27% of the HME samples visibly showed particles were removed from metal, 38% from vinyl and canvas, 41% from cardboard and shrink wrap, 59% from leather, and 76% from cotton when all of the explosives were considered. Further analysis shows that 50% of the HMTD on metal samples had visible particle removal, 71% of the vinyl and canvas samples, 29% of the cardboard samples, 100% of the shrink wrap and leather samples, and 86% of the cotton samples. It was assumed that HMTD and TATP would have similar results since they are both peroxide based explosives; however, 11% of the metal samples showed visible particle removal, 33% from vinyl, 0% from canvas and leather, 44% from cardboard, and 22% from shrink wrap and cotton when TATP was the homemade explosive used. Similarly, one would expect ammonium nitrate and guanidine nitrate to behave similarly since they are both nitrated crystals; however, this did not hold true. For ammonium nitrate, 33% of the metal samples showed that particles were removed from the surface after shaking, 67% from vinyl and canvas, 89% from cardboard, 56% from shrink wrap and 100% from leather and cotton. Twenty-five percent of the metal samples showed particle removal, 0% of vinyl, cardboard, and shrink wrap samples, 22% of canvas samples, 44% of leather samples, and 100% of the cotton samples when guanidine nitrate was used.

Based on the vibration testing results; the optimal HME particle removal was obtained at 70 Hz at 5g RMS. There was a loss in the sample removal efficiency above 70 Hz at 5g RMS which made the results at 100 Hz at 5g RMS similar to those at 35Hz at 5g RMS. Metal is the most difficult substrate from which to remove the HME by means of mechanical vibration. This is probably due to the flattening of the explosive crystals onto the substrate during the transfer technique, thereby providing more surface area against the metal surface and a flatter crystal to be removed. Testing showed that it is very difficult to remove military explosives by means of mechanical vibrations. None of the Military explosives could be removed from cardboard substrates, and only a few TNT particles could be removed from two of the metal samples.

Airflow tests were conducted using the three military explosive types on two substrates and four HME types on seven substrates. The airflow tests involved photographing a sample prior to passing air at one of three angles and three flows over the surface. The angles used were 0° (sample parallel to air flow), 45°, and 90° (sample normal to airflow) and the air flows were 25 liters per minute, 50 liters per minute, and 80 liters per minute. The sample was rotated upside down to remove any free standing particles. The sample was then rotated face up and photographed, then placed in the desired positioning for the testing. The tests were performed at each angle and each airflow combination in triplicate for each substrate. The samples were then photographed and layered to determine if any particles were removed.

Test results indicate that a flow of 50 LPM is the most effective at removing both HME and Military explosives particles. Holding the sample normal to the airflow was found to be the optimal condition for all substrates and explosive types. The worst HME results were seen at 25 LPM when the sample was

held parallel to the airflow. Only 7% of the Military explosives were removed by the 25 LPM air flow (0% of the SEMTEX, 16% of the TNT, and 6% of the C-4). At 80 LPM, 22% of the samples showed particle removal (11% of the SEMTEX, 39% of the TNT, and 17% of the C-4 samples). Best results for Military explosives were obtained at 50 LPM, where 60% of the samples showed particle removal (78 % of the SEMTEX samples, 78 % of the TNT samples, and 33% of the C-4 samples). Overall, TNT was the easiest Military explosive to remove by means of airflow on both substrates.

Thermal (heat cycling) tests were conducted on the HME samples. The heat tests involved using an environmental chamber to control the humidity at 10-20% RH while holding the temperature at 40°C, 60°C, and 75°C. The humidity for the environmental chambers was turned off and not controlled; however, the humidity was measured and ranged from 10%-20% at any given time during the testing. This is due to the portholes in the chamber that are used to assure that over pressurization of the chamber does not occur. The portholes allow the chambers to come into equilibrium with the humidity of the external surroundings. The fingerprint was placed on the substrate of interest and photographed using a Zeiss Discover V.12 stereoscope with Axiocam ICc1 3 megapixel digital camera. Once photographed, the sample was placed in an environmental chamber at the designated temperature for 3 minutes. Prior testing has shown that it takes ~2 minutes for the sample to reach the desired temperature, and then the sample was left for one additional minute at the desired temperature. The samples were removed and photographed again. The amount of sample that remained on the material was compared to the pre-heating sample to determine the probability of removing the homemade explosive from the surface by heating. The tests were repeated in triplicate.

During this test series, no testing was performed with guanidine nitrate due to a lack of standard solution to perform the tests. Also no testing was performed on cotton material. For all other samples, the overall percentage of samples that showed visible particle loss was 82%. At 40°C, 56% of the samples had visible particle removal when HMTD was used, 100% when TATP was used and 72% when ammonium nitrate was used. At 60°C, 56% of the samples had visible changes in the amount of particles on the surface when HMTD was used, 100% of the TATP, and 100% of the ammonium nitrate. At 75°C, 56% of the samples had visible changes in the amount of particles on the surface when HMTD was used, and 100% of the TATP and ammonium nitrate samples.

For all temperatures and HME studied, 85% of the metal samples had visible changes, 74% of the vinyl and leather, 81% of the canvas and shrink wrap, and 96% of the cardboard. All of the TATP samples had visible changes, regardless of, temperature, or substrate. When HMTD was used 67% of the metal and canvas samples, 33% of the vinyl and leather, 44% of the shrink wrap and 89% of the cardboard samples showed visible changes When ammonium nitrate was used: 89% of metal, vinyl and leather, 78% of canvas and 100% of cardboard and shrink wrap had visible changes.

Based on the results of the HME heat test studies, the optimal temperature for removing the peroxide explosives from the substrate surfaces is 40°C or less. The sample tends to disappear at higher temperatures, which is expected due to the low vaporization potential. For explosives such as ammonium nitrate, the higher temperatures result in melting of the sample and re-crystallization. Unexpectedly, the shrink wrap did not appear to have any visible melting at the higher temperature; however, none of the samples were monitored for any type of off gas that may affect the vapor sampling.

Tests combining heat and mechanical techniques were conducted on the Military explosive fingerprints on metal and cardboard substrates. For the heat/vibration tests, flexible heating pads and a temperature controller were used to heat the samples and vibrator sample holder to 150° F. Unlike the previous tests where tests were repeated three times, this series of tests were repeated only twice. Photos were taken prior to heating, after vibrating for 1 minute without heat, and again after the sample was heated and

shaken for 1 minute. These tests were run at 35 Hz and 70 Hz, and only one set of samples (C-4 on metal at 70 Hz and 150° F indicated any particle removal. Similar tests were conducted using heating to 150° F and air flow of 80 LPM, and this time only one of the TNT on cardboard samples showed any particle removal.

ACRONYMS

AN	Ammonium Nitrate
ACS	Air Cargo Screening
BDL	Below Detection Limits
BMI	Battelle Memorial Institute
C-4	Composition 4 plastic explosive containing RDX (91%) with binders and plasticizers
DHS	Department of Homeland Security
DOE	Department of Energy
GN	Guanidine Nitrate
HME	Homemade Explosive
HMTD	Hexamethylene Triperoxide Diamine
IED	Improvised Explosive Device
INL	Idaho National Laboratory
PETN	Pentaerythritol Tetranitrate
RDX	Cyclotrimethylene Trinitramine
SEMTEX	Plastic explosive containing PETN (76%) and RDX (4.6%)
TATP	Triacetone Triperoxide
TNT	Trinitro Toluene
TSL	Transportation Security Laboratory
S&T	Science and Technology Directorate

1. INTRODUCTION

This report describes the procedures used and the test results obtained at the Idaho National Laboratory (INL), to determine the ability of thermal and mechanical (vibration and airflow) methods to remove/harvest trace explosives from various material substrate surfaces relevant to air cargo. As part of the Battelle Memorial Institute's (BMI) Air Cargo Screening (ACS) Initiative, INL conducted mechanical and thermal/mechanical removal tests using three different Military explosives: the plastic explosives C-4 (RDX, 91%) and SEMTEX (PETN, 76% and RDX, 4.6%) and cast TNT deposited on two substrate materials relevant to air cargo: metal and cardboard. This work leveraged similar experiments using homemade explosives (HME) which were funded by the Department of Homeland Security's Science and Technology Directorate (DHS S&T). The DHS S&T tests involved evaluating the removal efficiencies of thermal and mechanical methods for four different HME: Ammonium Nitrate prills (AN, explosive grade), Guanidine Nitrate (GN, 98% pure in Methanol), Triacetone Triperoxide (TATP, 99.5% pure in Acetonitrile), and Hexamethylene Triperoxide Diamine (HMTD, 98.4% pure in Acetonitrile). Substrate materials used for the HME removal tests included metal, cotton, canvas, leather, shrink wrap, cardboard, and vinyl.

2. BACKGROUND

Characterization of the adhesive properties of explosive particles, and determination of the efficacy of various methods to remove/harvest explosive particles from substrate surfaces, are important for developing effective explosive trace detection components of Air Cargo Screening systems. There are a number of commercially available explosive trace detection (ETD) systems that are capable of detecting and identifying nanogram levels of explosives once the particles are collected and transported to the instrument. Examples include, but are not limited to, ion mobility spectrometers, mass spectrometers, gas chromatographs, surface acoustic wave sensors, cantilever beam sensors, fluorescence quenching sensors, cavity-ringdown spectroscopy sensors, colorimetric sensors, and electron capture detectors. Similarly, there are several ways that may be utilized to collect and transport the explosive particles to the sensor, some of which are incorporated within the instrument and some which may require an operator to perform the transfer. Examples include, but are not limited to, manual swiping, air puffers, vacuum collection of vapor or particles, and vortex vacuum sampling. There are many applications of interest to DHS in which it is impractical to manually wipe a surface. These include, for example, sampling without an operator, large area sampling, high throughput personnel screening, consolidated cargo screening, remote sampling, robotic sampling, and situations in which the frequent replacement of wiping materials is not acceptable. As a result, there is considerable interest in developing efficient non-contact methods for removing and harvesting explosive particles. The research described in this report was focused on evaluating removal techniques applicable to both Military explosives and HME threats in consolidated Air Cargo

3. PROCEDURES

The INL conducted a series of experiments to characterize the adhesive properties of trace levels of HMEs and Military explosives deposited on various types of materials relevant to Air Cargo by monitoring the effectiveness of three removal techniques: forced air, vibration, heat, and combinations of heat and vibration as well as heat and airflow. The materials consisted of 1" by

1” samples of material substrates: metal, canvas, vinyl, suede leather, cotton, cardboard, and shrink wrap. These substrates provided a broad range of materials that were likely to be sampled as air cargo, airline baggage, or person-borne materials. The HMEs that were identified for use were urea nitrate, ammonium nitrate, and TATP. However, during the process of obtaining HMEs, it became apparent that nitrated fertilizers such as urea nitrate and ammonium nitrate have been replaced by sulfated fertilizers. Therefore, only ammonium nitrate was used. In addition, guanidine nitrate standards were used to provide a secondary nitrated sample. HMTD was also added to the test matrix to provide two peroxide based explosives. The Military explosives used in these tests included two plastic explosives, C-4 (RDX, 91%) and SEMTEX (PETN, 76% and RDX, 4.6%), as well as cast TNT. The Military explosives were deposited (as first-generation fingerprints) on two substrate materials relevant to air cargo: metal and cardboard.

The HME was placed on the surface of the material substrates by dabbing explosive crystals from a Teflon coated surface. To create the explosive crystals, Accustandard was deposited onto the Teflon coated surface and allowed to dry. The amount of Accustandard used was 1mL at 1mg/mL for TATP and HMTD and 500 µL at 1 mg/mL of guanidine nitrate. The ammonium nitrate (explosive grade prills) were crushed and fingerprinted onto the surface of the substrate. The Military explosives were deposited as first-generation fingerprints on two substrate materials relevant to Air Cargo: metal and cardboard.

Testing was divided into four series. The first series was designed to determine the amount of explosives (both HME and Military) that was removed from the surface of material substrates by using vibration at frequencies of 35 Hz, 70 Hz, and 100 Hz at a constant acceleration of 5g RMS. The second series was designed to determine the amount of HME and Military explosives that was removed from the surface of the material substrates using airflows of 25 L/min, 50 L/min, and 80 L/min at angles of 0°, 45°, and 90°. The third series was designed to determine the amount of HME that was removed from the surface by heating the sample in a controlled environment to 40°C, 60°C, and 75°C. The fourth series of tests was designed to determine the amount of Military explosives that was removed by combinations of heat and vibration as well as heat and airflow. The amount of explosive that was removed during each test was determined optically by comparing pre and post test micro-photographs of the substrate.

3.1 Removal of Explosive Particles Using Vibration

This portion of the test involved determining the efficacy of removing HME and Military explosives from the material substrate surfaces using vibrations at frequencies of 35Hz, 70Hz, and 100Hz at a constant acceleration of 5g RMS. A mechanical vibrator, Vibration Test Systems Inc. Model VG-100-8,with a Piezo-Electronics, Inc. Model PCB 393B04 accelerometer were used for these measurements. The homemade explosive was placed on the surface of the material substrates by dabbing explosive crystals from a Teflon coated surface. To create the HME crystals, Accustandard was deposited onto the Teflon coated surface and allowed to dry. The amount of Accustandard used was 1mL at 1mg/mL for TATP and HMTD and 500 µL at 1 mg/mL of guanidine nitrate. The ammonium nitrate that was used was fertilizer pellets. These pellets were crushed and fingerprinted onto the surface of the substrate. The Military explosives were deposited on metal and cardboard substrates using first-generation fingerprints. Three samples of each matrix were contaminated with each type of explosive for testing at each frequency/angle combination. A photograph of the substrate was taken using a using a Zeiss Discover V12 stereoscope with Axiocam ICc1 3 megapixel digital camera (Figure 2) to determine the difference in the crystalline structure and surface contamination prior to and after

vibrating the sample for one minute. The experimental design for this phase appears in Figure 1a. Figure 1b and 1c are close up views of the vibration drum and the sample holder. The sample holder was designed so that it could be rotated directly under the microscope to ensure that the position of the substrate was not displaced so that the photograph (before and after) could be co-registered.

Figure 1a. Experimental design for vibration testing

Figure 1b. View of the vibration drum

Figure 1c. View of the sample holder on the vibration drum

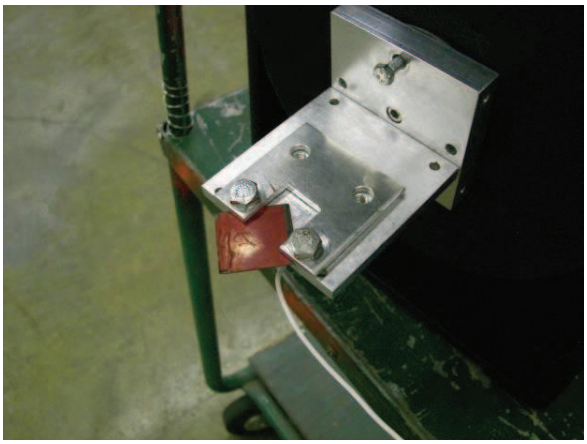
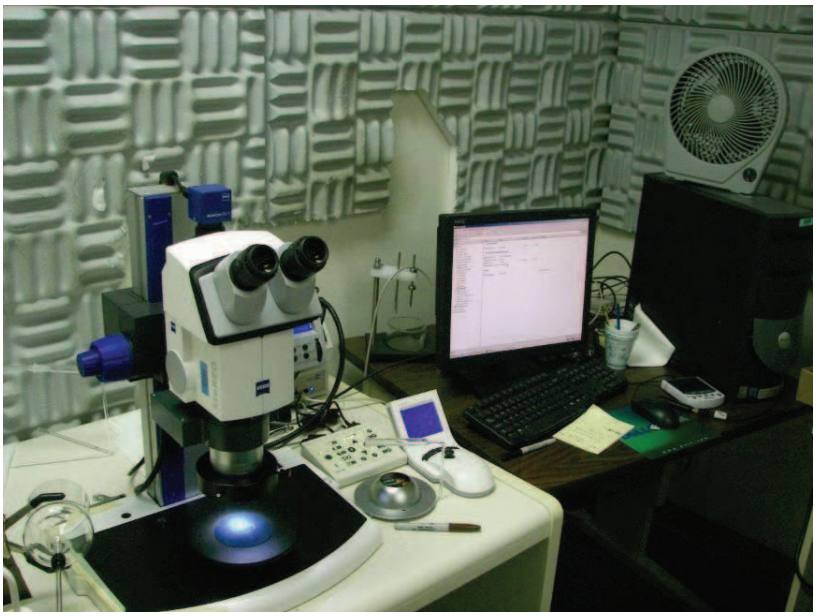


Figure 2. Zeiss Discover V12 stereoscope with Axiocam ICc1 3 megapixel digital camera setup with computer system



3.2 Removal of Explosive Particles Using Airflow

This portion of the test involved determining the efficacy of removing Military and HME from the material substrate surfaces using air to dislodge the explosive. Airflows of 25 L/min, 50 L/min, and 80 L/min were used. The substrate surfaces were held at three different angles relative to the airflow for each air flow rate: 0°(sample parallel to the airflow), 45°, and 90°(sample normal to the airflow). The air tube was directed toward the sample and held approximately four inches from the surface of the substrate for these tests as shown in Figure 3a. Additional tests were performed with the same three angles and an air flow of 80 L/min with the air occupying a larger volume and not directed onto the substrate as shown in Figure 3c. The explosive samples were placed on the surface of the material substrates using the procedures described in section 3.2 above.

The instrument setup for the airflow tests are shown in Figure 3a-3c. Figure 3a is the overall setup for the airflow testing. Figure 3b is the airflow controllers for the testing, and Figure 3c is the flow tube and the sample holder for the airflow tests. The sample holder could be rotated and positioned at different angles so that the air flow was horizontal across the surface of the sample, vertical along the surface of the sample, or hitting the sample at an angle of ~45º. The sample holder could also be positioned at different depths in the tube; however, a standard depth of 1" into the tube was used which resulted in the air entering the widest part of the tube approximately 3" from the sample. In addition, there was an approximately 1/2" gap prior to the wide part of the tube. The flow throughout the tube was assumed to be laminar for these tests. The Reynolds number was calculated for the three air flows and show that the air flow is laminar through the tube. In an attempt to create turbulent air, the sample was place such that the air had to flow

around the flat surface of the sample holder. The results from the trial tests did not show improved sample removal; therefore this technique was not used throughout the tests.

Figure 3a:. Instrument setup for airflow testing

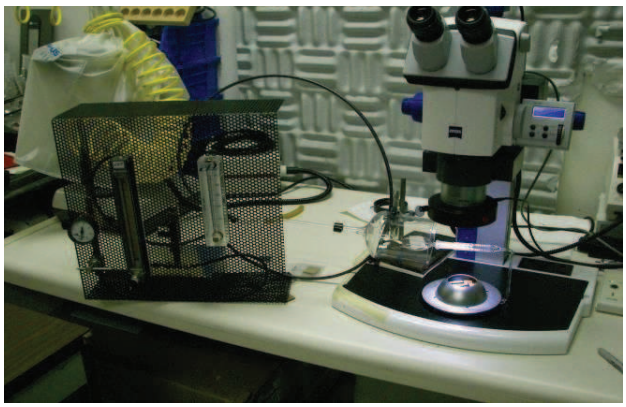


Figure 3b. Airflow control gauges used during airflow testing

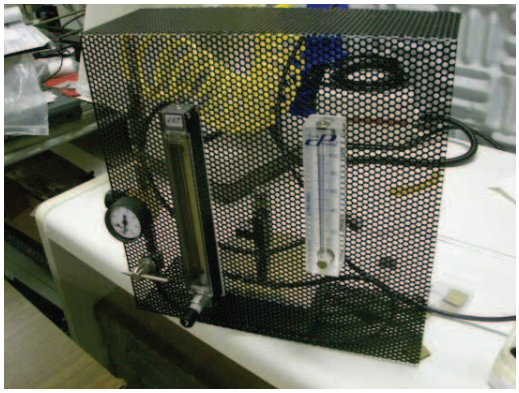
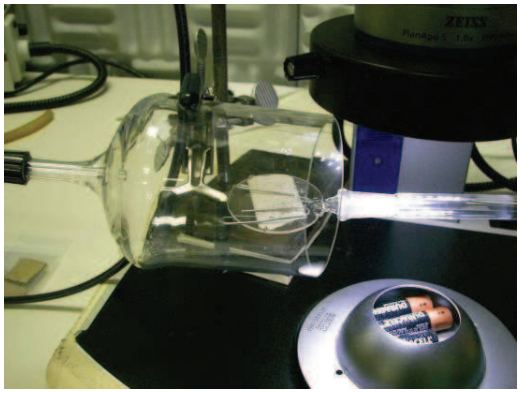


Figure 3c. Tube to control direction of airflow and sample holder for airflow testing



3.3 Removal of HME Using Heat

This portion of the testing involved determining the efficacy of removing HME from the material substrate surfaces using heat at 15% relative humidity and temperatures of 40°C, 60°C, and 75°C. The HME was placed on the surface of the material substrates by dabbing explosive crystals from a Teflon coated surface. To create the HME crystals, Accustandard was deposited onto the Teflon coated surface and allowed to dry. The amount of Accustandard used was 1mL at 1mg/mL for TATP and HMTD. The ammonium nitrate that was used was fertilizer pellets. These pellets were crushed and fingerprinted onto the surface of the substrate. Three samples of

each matrix were contaminated with homemade explosive for each heat setting used, for a total of 42 samples. Guanidine nitrate was not used for these tests. A photograph of the substrate was taken using a using a Zeiss Discover V12 stereoscope with Axiocam ICc1 3 megapixel digital camera (Figure 2) to determine the difference in the crystalline structure and surface contamination prior to and after heating the sample for one minute.

3.4 Removal of Military Explosives Using Combined Heat and Mechanical Methods

This portion of the test involved determining the efficacy of removing Military explosives from metal and cardboard surfaces using a combination of heat/vibration and heat/air flow. Due to the very low vapor pressures associated with military explosives and their high thermal stability (compared to HME), we chose relatively high temperatures (150° F minimum) for these tests. For the heat/vibration tests, flexible heating pads and a temperature controller were used to heat the samples and vibrator sample holder to 150° F. Photos were taken prior to heating, after vibrating for 1 minute without heat, and again after the sample was heated and shaken for 1 minute. Similar tests were conducted using heating the sample and holder to temperatures of 150° to 175° F and subjecting the samples to air flows of 80 LPM.

4. RESULTS AND DISCUSSION

An attempt was made to enhance the optical microscopy by using fluorescent dyes and taking the photographs using ultraviolet lighting. This technique was adequate for taking photographs without the microscope, but the black lighting could not be used with the microscope unless it was underneath the sample and shining up through the sample due to the lighting necessary to obtain microscopic results. Unfortunately, most of the substrates did not allow for the black light to penetrate through the sample. In addition, when the explosive were mixed with the fluorescent dyes and then dried to obtain the explosive crystals, the dyes tended to cause the matrix to dry in a clump and the entire clump never completely dried. Therefore, when the explosive was transferred, it smeared into the material rather than remaining on the surface and could not be removed by any of the three techniques attempted. An attempt was also made using high speed cameras to photograph the particles as they were being removed. However, this technique was also not sufficient due to the small size of the particles that were removed as well as reflections from the glass of the air flow tube. Therefore, a determination was made to look only at the optical changes before and after the airflow tests.

4.1 Removal of Explosive Particles Using Vibration - Test Results

The vibration tests involved using three frequencies (35 Hz, 70 Hz, and 100 Hz) and vibrating the sample using mechanical vibration for one minute at 5g RMS. The sample was photographed using a Zeiss Discover V12 stereoscope with Axiocam ICc1 3 megapixel digital camera prior to vibrating and then after the one minute time period. The changes in the pre-vibration and post-vibration samples were optically compared by layering the photographs and flickering back and forth to determine if any changes had occurred. The tests were repeated in triplicate.

The fingerprint was placed on the substrate of interest and loaded onto the sample holder on the vibration drum. The sample was rotated upside down to remove any unattached particles on the surface. The sample holder was then rotated back to the starting position under the microscope

lens and the original picture was taken. The sample was then rotated upside down again and shaken at the designated frequency of 35Hz, 70 Hz, or 100 Hz at an acceleration of 5g RMS for 1 minute based on preliminary test results.

Preliminary tests were performed to determine the range of frequencies and accelerations that provided optimal particle loss. The preliminary tests were performed at a range of 10Hz at 1.6g to as high as 3kHz at 7 g. These preliminary tests indicated that the bulk of the testing should be focused on the 35-100 Hz range at 5g.

There were a total of 70 samples at 35 Hz of which 44% had visibly lost particles from the surface. Fifty percent of the 84 samples at 70 Hz and 43% of the 83 samples at 100 Hz had visible changes in the amount of particles on the surface after vibration. In all of the tests performed using vibration for removal, ~46% had visible particle removal after shaking. This overall 48% removal rate consisted of ,74% of the HMTD samples, 19% of the TATP samples, 71% of the ammonium nitrate samples, and 27% of the guanidine nitrate samples.
At 35 Hz: 86% HMTD, 5% TATP, 81% ammonium nitrate, and 33% guanidine nitrate samples had visible particle removal after shaking. At 70 Hz: 71% HMTD and ammonium nitrate, 33% TATP, and 24% guanidine nitrate samples had visible particle removal after shaking. At 100 Hz: 67% HMTD, 19% TATP, 62% ammonium nitrate, and 25% guanidine nitrate samples had visible particle removal after shaking.

A summary of the HME test results is located in Table 1. Figure 4, 5, 6, and 7 represent the four HME types at 35 Hz, 70 Hz, and 100 Hz at an acceleration of 5g RMS. The left picture is the pre-vibration picture and the picture on the right represents the material after the substrate was shaken for one minute. Figure 4 is ammonium nitrate samples at 35 Hz (a), 70 Hz (b), and 100 Hz (c). Figure 5 is guanidine nitrate samples at 35 Hz (a), 70 Hz (b), and 100 Hz (c). Figure 6 is HMTD samples at 35 Hz (a), 70 Hz (b), and 100 Hz (c). Figure 7 is TATP samples at 35 Hz (a), 70 Hz (b), and 100 Hz (c). As can be seen in the figures, in many cases, there is no visible removal of any explosive and in some cases only the larger crystals are removed. Because the changes are difficult to see in the photographs, the pictures are overlaid and flickered between the co-registered images to detect changes. These overlaid picture results are the ones listed in Table 1.

In addition, different surfaces had significantly different particle removal efficiencies that varied with different explosive types. Overall 27% of the samples visibly showed particles were removed from metal, 38% from vinyl and canvas, 41% from cardboard and shrink wrap, 59% from leather, and 76% from cotton when all of the explosives were considered. Further analysis shows that 50% of the samples showed visible particle removed from metal, 71% from vinyl and canvas, 29% from cardboard, 100% from shrink wrap and leather and 86% from cotton when the explosive was HMTD. It was assumed that HMTD and TATP would have similar results since they are both peroxide based explosives; however, 11% of the metal samples showed visible particle removal, 33% from vinyl, 0% from canvas and leather, 44% from cardboard, and 22% from shrink wrap and cotton when TATP was the homemade explosive used. Similarly, one would expect ammonium nitrate and guanidine nitrate to behave similarly since they are both nitrated crystals; however, this did not hold true. For ammonium nitrate, 33% of the metal samples showed that particles were removed from the surface after shaking, 67% from vinyl and canvas, 89% from cardboard, 56% from shrink wrap and 100% from leather and cotton. Twenty-five percent of the metal samples showed particle removal, 0% of vinyl, cardboard, and shrink wrap samples, 22% of canvas samples, 44% of leather samples, and 100% of the cotton samples when guanidine nitrate was used.

Based on the vibration testing results; the optimal condition was 70 Hz. There was an insignificant gain in the sample loss above 70 Hz. TATP appears to be difficult to remove using vibration techniques. In addition, it appeared that it was difficult to remove the explosive particles from a metal surface, regardless of the explosive type or the frequency of the vibration. This is probably due to the amount of surface area that holds the crystal to the metallic surface. When a sample is fingerprinted onto a hard surface such as metal, the crystals tend to become flattened.

Table 1. A summary of the HME vibration test results. The results are in number with visible removal of explosives over number of samples analyzed for each material, frequency, and explosive.

	HMTD			***TATP***			***AN***			***GN***			***TOTALS***
Substrate	*35 Hz*	*70Hz*	*100 Hz*	*35 Hz*	*70 Hz*	*100 Hz*	*35 Hz*	*70 Hz*	*100 Hz*	*35 Hz*	*70 Hz*	*100 Hz*	
Metal	*0/1*	*2/3*	*1/3*	*0/3*	*1/3*	*0/3*	*0/3*	*1/3*	*2/3*	*1/3*	*1/3*	*0/2*	***9/33***
Vinyl	*1/1*	*2/3*	*2/3*	*1/3*	*2/3*	*0/3*	*3/3*	*2/3*	*1/3*	*0/3*	*0/3*	*0/3*	***13/34***
Canvas	*1/1*	*2/3*	*2/3*	*0/3*	*0/3*	*0/3*	*3/3*	*2/3*	*1/3*	*2/3*	*0/3*	*0/3*	***13/34***
Cardboard	*1/1*	*0/3*	*1/3*	*0/3*	*1/3*	*3/3*	*3/3*	*2/3*	*3/3*	*0/3*	*0/3*	*0/3*	***14/34***
Shrink wrap	*1/1*	*3/3*	*3/3*	*0/3*	*1/3*	*1/3*	*2/3*	*2/3*	*1/3*	*0/3*	*0/3*	*0/3*	***14/34***
Leather	*1/1*	*3/3*	*3/3*	*0/3*	*0/3*	*0/3*	*3/3*	*3/3*	*3/3*	*1/3*	*1/3*	*2/3*	***20/34***
Cotton	*1/1*	*3/3*	*2/3*	*0/3*	*2/3*	*0/3*	*3/3*	*3/3*	*3/3*	*3/3*	*3/3*	*3/3*	***26/34***
TOTALS	***6/7***	***15/21***	***14/21***	***1/21***	***7/21***	***4/21***	***17/21***	***15/21***	***13/21***	***7/21***	***5/21***	***5/20***	***109/237***

Figure 4. Ammonium Nitrate Explosive on a) canvas at 35 Hz; b) cotton at 70 Hz; and c) leather at 100 Hz

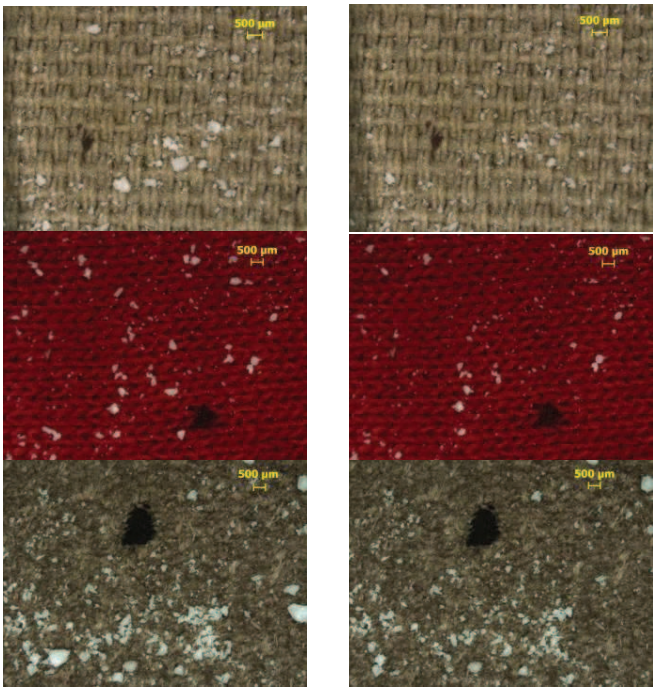


Figure 5. Guanidine Nitrate Explosive on a) cotton at 35 Hz; b) shrink wrap over metal at 70 Hz; and c) cotton at 100 Hz

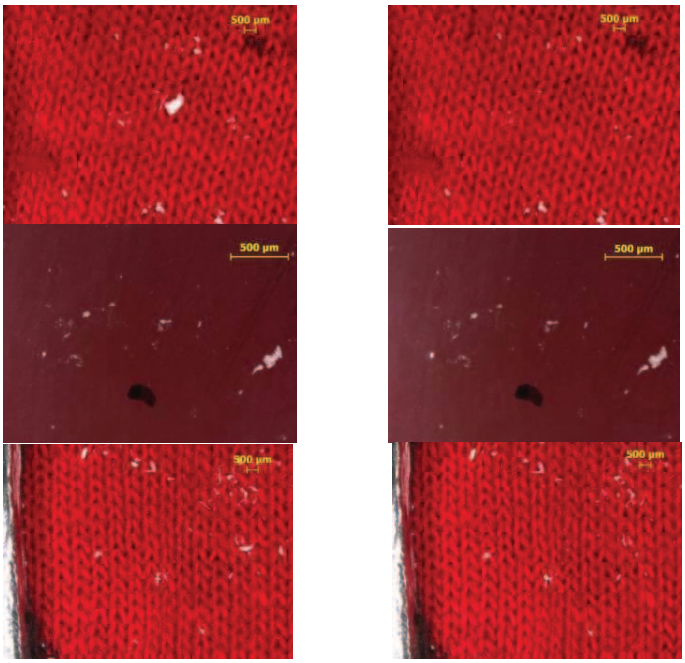


Figure 6. HMTD Explosive on a) canvas at 35 Hz; b) cardboard at 70 Hz; and c) leather at 100 Hz

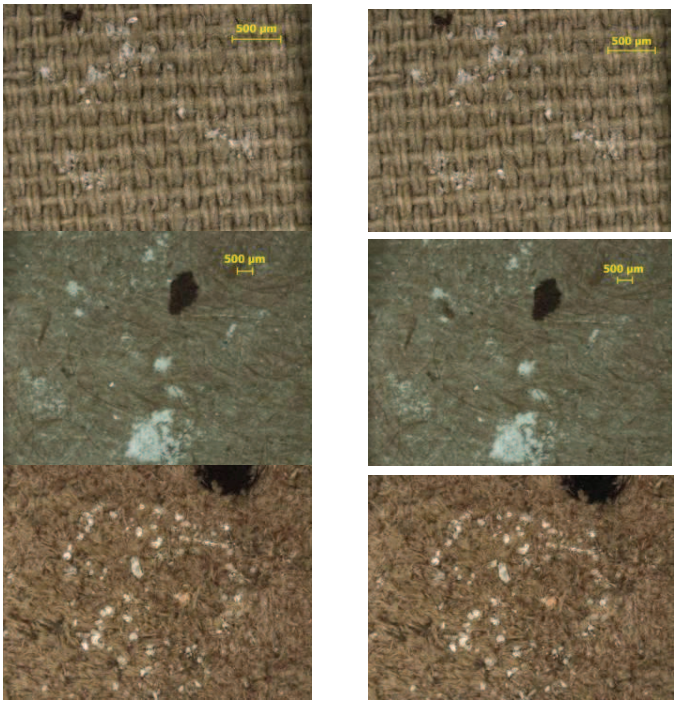
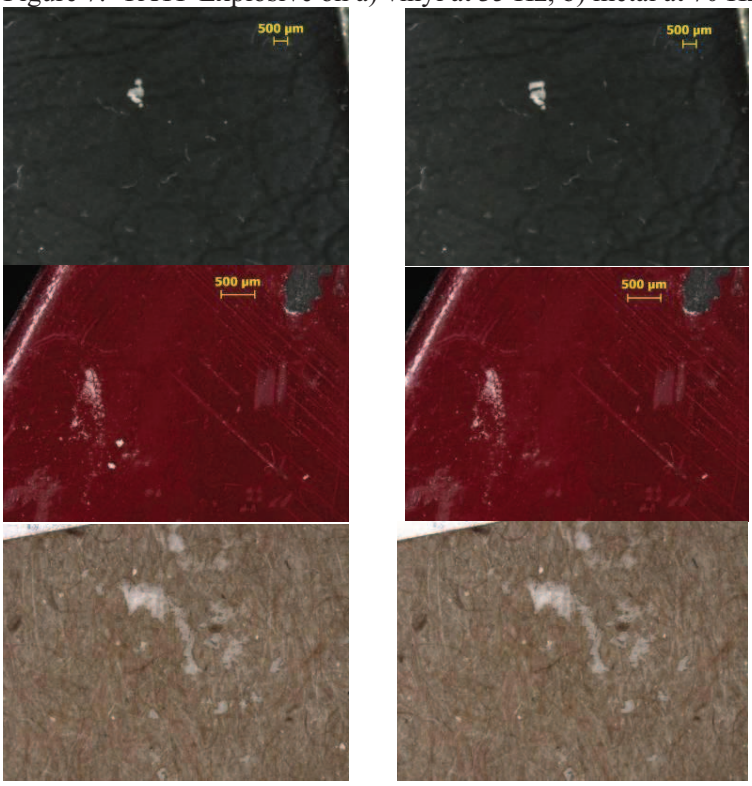


Figure 7. TATP Explosive on a) vinyl at 35 Hz; b) metal at 70 Hz; and c) cardboard at 100 Hz



The results of the vibration tests using Military explosives are summarized on Table 2 and illustrated in Figures 8 through 10. The vibration removal rates for Military explosives were much poorer than those for HME. Only two of the TNT on metal samples showed any particle removal. Overall vibration removal rates for Military explosives were 11% for TNT, 0% for SEMTEX, and 0% of C-4. These tests confirm the fact that it is very difficult to remove military explosives by means of mechanical vibrations. None of the Military explosives could be removed from cardboard substrates, and only a few TNT particles could be removed from two of the metal samples.

Table 2. A summary of the Military explosives vibration test results. The results are in number with visible removal of explosives over number of samples analyzed for each material, frequency, and explosive type.

	SEMTEX			***C-4***			***TNT***			***TOTALS***
Substrate	*35 Hz*	*70Hz*	*100 Hz*	*35 Hz*	*70 Hz*	*100 Hz*	*35 Hz*	*70 Hz*	*100 Hz*	
Metal	*0/3*	*0/3*	*0/3*	*0/3*	*0/3*	*0/3*	*2/3*	*0/3*	*0/3*	***2/27***
Cardboard	*0/1*	*0/2*	*0/2*	*0/1*	*0/1*	*0/1*	*0/3*	*0/3*	*0/3*	***0/17***
TOTALS	***0/4***	***0/5***	***0/5***	***0/4***	***0/4***	***0/4***	***2/6***	***0/6***	***0/6***	*2/44*

Figure 8. SEMTEX Vibration Testing Results: a) SEMTEX on cardboard, 70Hz (no particles removed); b) SEMTEX on cardboard, 100Hz (no particles removed); c) SEMTEX on metal, 35Hz (no particles removed); d) SEMTEX on metal, 100 Hz (no particles removed).

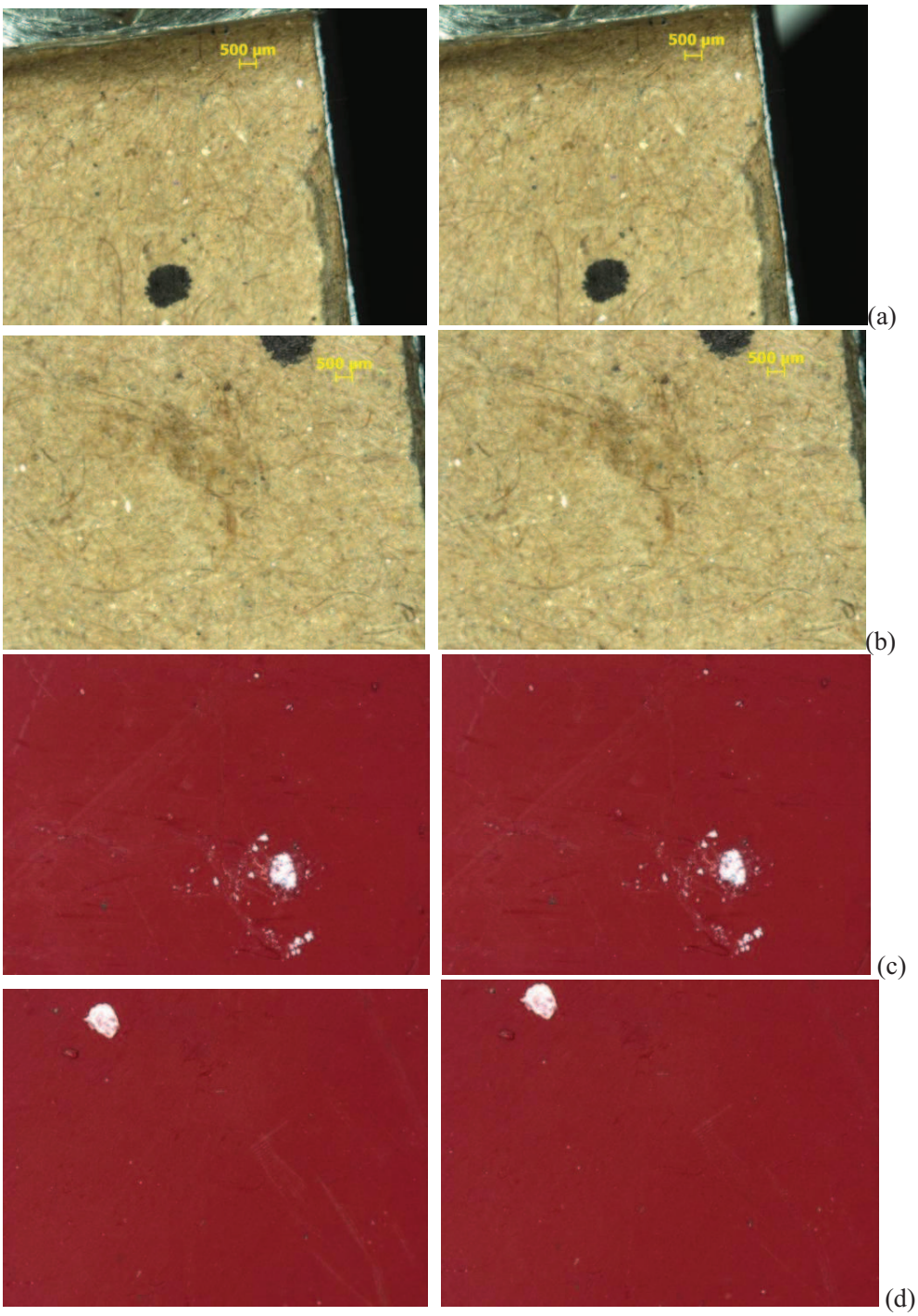
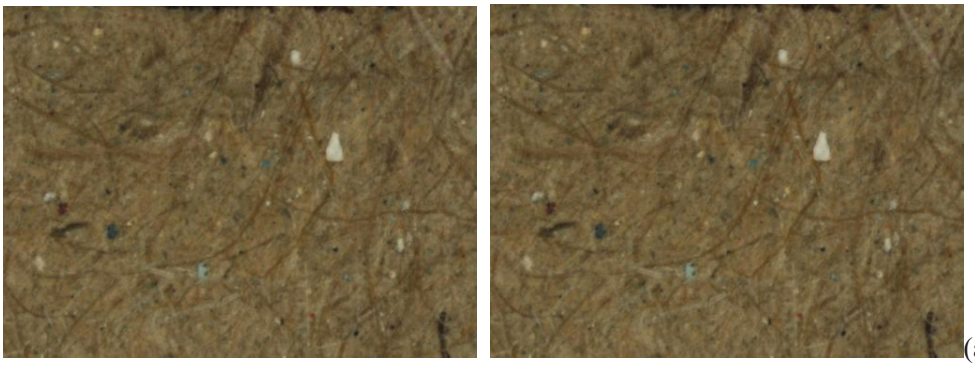
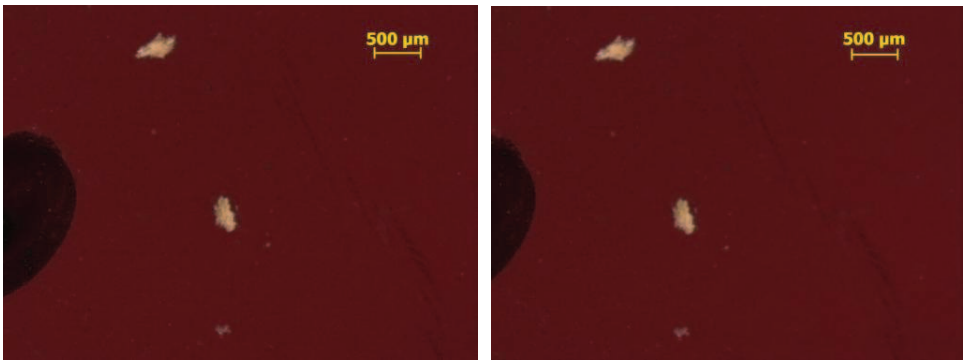


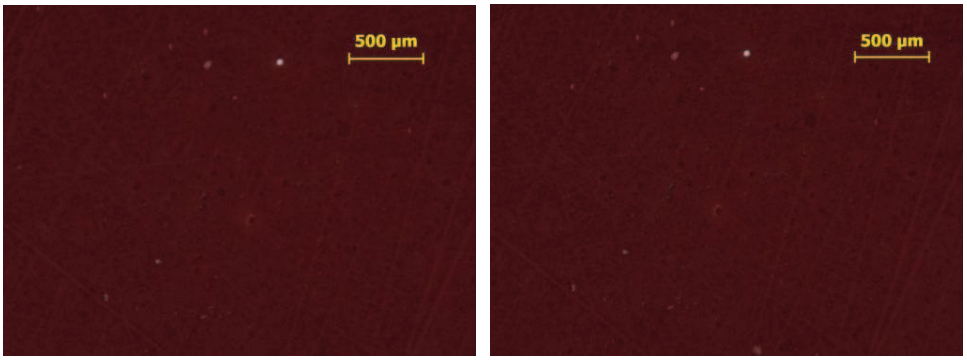
Figure 9. C-4 Vibration Testing Results: a) C-4 on cardboard, 70Hz (no particles removed); b) C-4 on metal, 35Hz (no particles removed); c) C-4 on metal, 100 Hz (no particles removed).



(a)

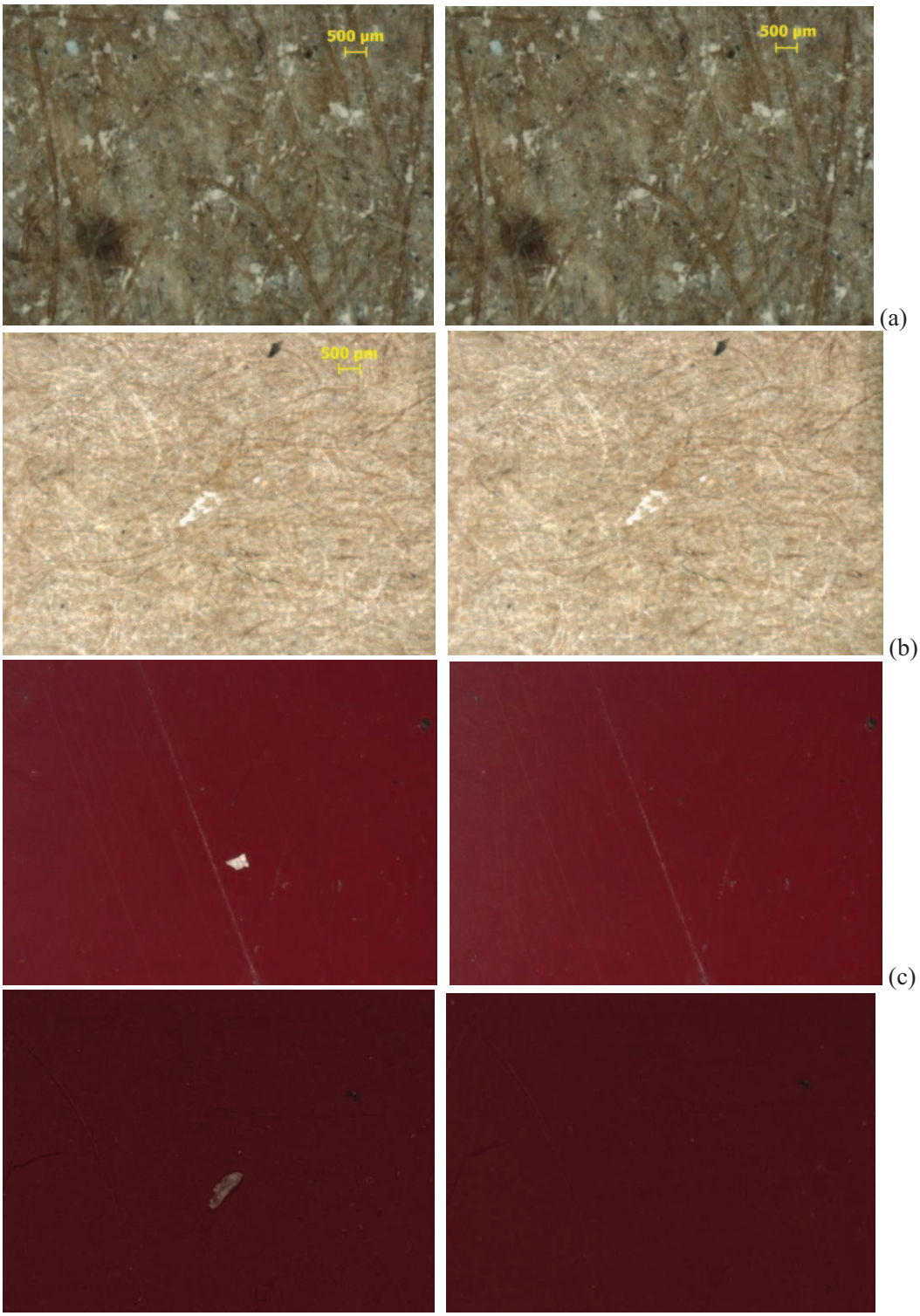


(b)



(c)

Figure 10. TNT Vibration Testing Results: a) TNT on cardboard, 35Hz (no particles removed); b) TNT on cardboard, 100 Hz (no particles removed); c) TNT on metal, 35 Hz (large particle removed); d) TNT on metal, 35 Hz (large particle removed).



4.2 Removal of Explosive Particles Using Air Flow - Test Results

The airflow tests involved photographing a sample prior to passing air at one of three angles and three flows over the surface. The angles used were 0°(parallel to the airflow), 45°, and 90°(normal to the airflow) and the air flow rates were 25 liters per minute, 50 liters per minute, and 80 liters per minute. The tests were performed at each angle and each airflow combination in triplicate for each substrate. The air was flowed across the surface for 30 seconds. The samples were then photographed and layered to optically identify any changes that occurred. The fingerprint was placed on the substrate of interest, then placed on the sample holder of the air flow system. The sample was rotated upside down to remove and free standing particles. The sample was then rotated face up and photographed, then placed in the desired positioning for the testing. Eighty-eight percent of the total 756 samples had visible changes to the amount of explosive on the surface: 82% at 25 LPM, 97% at 50 LPM, and 86% at 80 LPM. Of the 82% at 25 LPM, 80% of the visible changes occurred when the sample was held horizontally to the airflow, 85% was when the sample was held vertically to the airflow, and 82% was when the sample was held at a 45° angle. Similar results were seen with the other two air flows. When the flow was 50 LPM, 96% of the samples that were horizontal to the airflow, 100% of the samples vertical to the airflow and 94% of the samples at a 45° angle to the airflow had visible changes on the surface. At 80 LPM the values observed were 81% when the sample was held horizontal, 92% when the sample was held vertically and 85% when the sample was at a 45° angle.

A summary of the HME test results appear in Table 3. Results of the HME tests are also shown in Figures 11-14. Figure 11 (a-i) are examples of ammonium nitrate on cotton at 25 LPM (11a-c), 50 LPM (11d-f), and 80 LPM (11g-i). As can be seen in this series of photos, the explosive is removed from the surface of the material using all three airflows and at each of the three angles. Figure 12 (a-i) are examples of guanidine nitrate on cotton at 25LPM (12a-c), 50 LPM (12d-f), and 80 LPM (12g-i). As can be seen in this series of photos, the explosive is removed from the surface of the material using all three airflows and at each of the three angles. Figure 13 (a-i) are examples of HMTD on metal at 25 LPM (13a-c), 50 LPM (13d-f), and 80 LPM (13g-i). As can be seen in this series of photos, the explosive is not visibly removed from the surface of the material using any of the three airflows and at any of the three angles. Figure 14 (a-i) are examples of TATP on vinyl at 25 LPM (14a-c), TATP on cardboard at 50 LPM (14d-f), and TATP on canvas at 80 LPM (14g-i). As can be seen in this series of photos, some of the explosive is visibly removed from the surface of the material using all three airflows and at each of the three angles.

When ammonium nitrate was used, regardless of substrate, airflow, or angles, 86% of the samples had visible changes in the amount of particles on the surface after testing. When guanidine nitrate was used 79% of the samples had changes, 88% of the TATP samples, and 100% of the HMTD samples had visible changes after airflow tests were performed.

When ammonium nitrate was used, the substrate that had the fewest number of samples with visible changes was vinyl at 41% and the most was leather and canvas at 100%. Metal had 89%, shrink wrap had 81%, cardboard had 93% and cotton had 96%. When guanidine nitrate was used, the substrate that had the fewest number of samples with visible changes was metal at 41%. Shrink wrap had 63%, cotton and canvas had 93%, vinyl had 89%, leather had 81% and cardboard had 96%. When TATP was used, leather and metal had the least number of samples with visible changes at 81%. Shrink wrap had 85%, cotton and canvas had 89%, vinyl had 93%

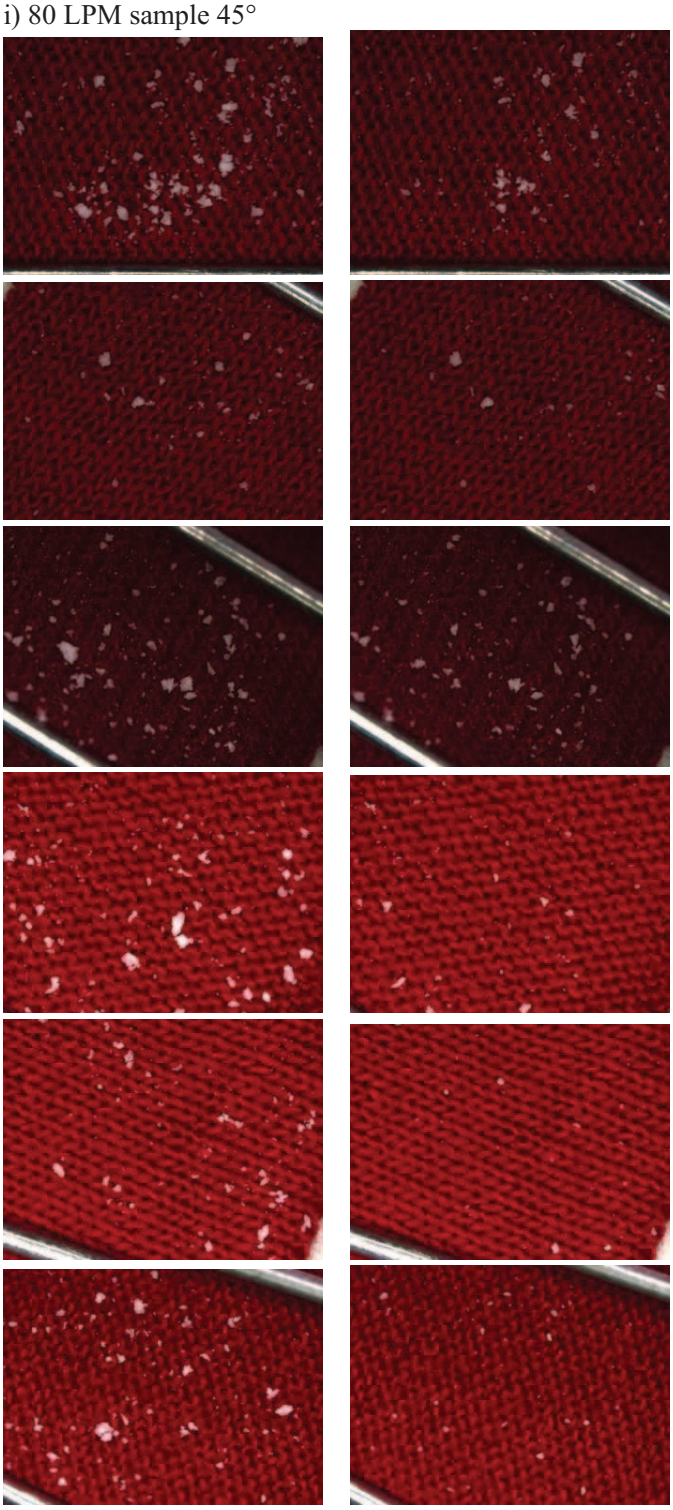
and cardboard had 100%. For HMTD all of the substrates had 100% of the samples with visible changes.

Based on the results of the airflow tests, there is no pattern as to which substrates or explosives will provide the best results. It would appear that a flow of 50 LPM is the most effective at removing sample particles. Best results were obtained when the sample is held in the vertical position with the airflow at 50 LPM for all substrates and explosive types. The worst results were seen at 25 LPM when the sample was held horizontal to the airflow.

Table 3. A summary of the air flow test results for all HME at all air flows, and all sample positions tested. The results are listed as number of samples with visible changes over the total number of samples tested.

		25 LPM			**50 LPM**			**80 LPM**			**TOTALS**
Explosive	**Substrate**	**Horiz.**	**Vert.**	**45º**	**Horiz.**	**Vert.**	**45º**	**Horiz.**	**Vert.**	**45º**	
Ammonium Nitrate	Cotton	3	3	3	3	3	3	1	3	3	**26/27**
	Vinyl	2	0	0	2	3	2	0	2	0	**11/27**
	Metal	3	3	2	3	3	3	3	2	2	**24/27**
	Canvas	3	3	3	3	3	3	3	3	3	**27/27**
	Shrink Wrap	2	3	2	3	3	3	0	3	3	**22/27**
	Leather	3	3	3	3	3	3	3	3	3	**27/27**
	Cardboard	3	3	3	3	3	3	3	2	2	**25/27**
Guanidine Nitrate	Cotton	3	2	2	3	3	3	3	3	3	**25/27**
	Vinyl	2	2	3	3	3	3	2	3	3	**24/27**
	Metal	1	2	1	1	3	1	0	2	0	**11/27**
	Canvas	3	3	2	3	3	3	2	3	3	**25/27**
	Shrink Wrap	0	2	3	3	3	3	1	1	1	**17/27**
	Leather	2	3	3	3	3	3	2	2	1	**22/27**
	Cardboard	3	3	3	3	3	3	3	3	2	**26/27**
TATP	Cotton	2	2	2	3	3	3	3	3	3	**24/27**
	Vinyl	2	3	2	3	3	3	3	3	3	**25/27**
	Metal	1	2	2	3	3	2	3	3	3	**22/27**
	Canvas	1	3	2	3	3	3	3	3	3	**24/27**
	Shrink Wrap	1	1	3	3	3	3	3	3	3	**23/27**
	Leather	3	1	1	3	3	2	3	3	3	**22/27**
	Cardboard	3	3	3	3	3	3	3	3	3	**27/27**
HMTD	Cotton	3	3	3	3	3	3	3	3	3	**27/27**
	Vinyl	3	3	3	3	3	3	3	3	3	**27/27**
	Metal	3	3	3	3	3	3	3	3	3	**27/27**
	Canvas	3	3	3	3	3	3	3	3	3	**27/27**
	Shrink Wrap	3	3	3	3	3	3	3	3	3	**27/27**
	Leather	3	3	3	3	3	3	3	3	3	**27/27**
	Cardboard	3	3	3	3	3	3	3	3	3	**27/27**
TOTALS		**67/84**	**71/84**	**69/84**	**81/84**	**84/84**	**79/84**	**68/84**	**77/84**	**71/84**	**667/756**

Figure 11. Ammonium Nitrate explosive on cotton material at: a) 25LPM sample horizontal; b) 25 LPM sample vertical, c) 25 LPM sample 45°, d) 50 LPM sample horizontal; e) 50 LPM sample vertical, f) 50 LPM sample 45°, g) 80LPM sample horizontal; h) 80 LPM sample vertical, i) 80 LPM sample 45°



(a)

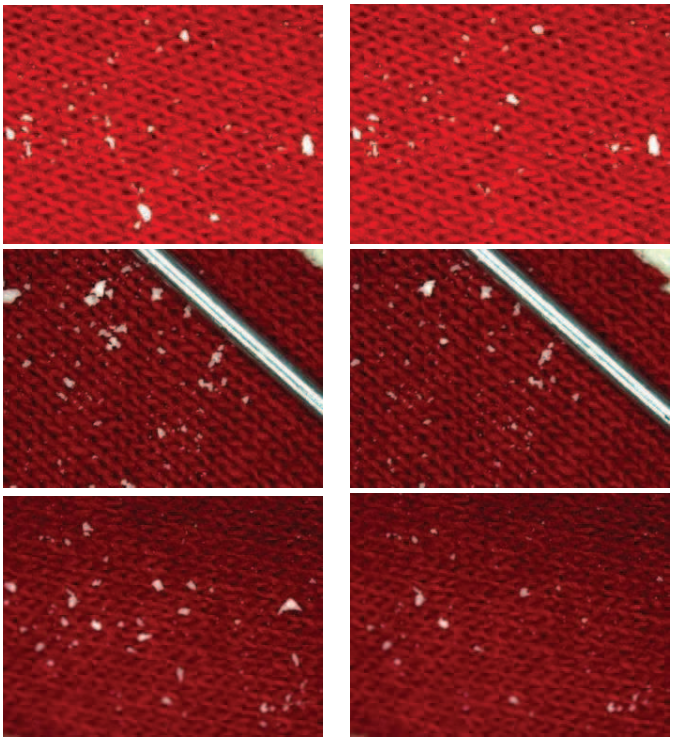
(b)

(c)

(d)

(e)

(f)



(g)

(h)

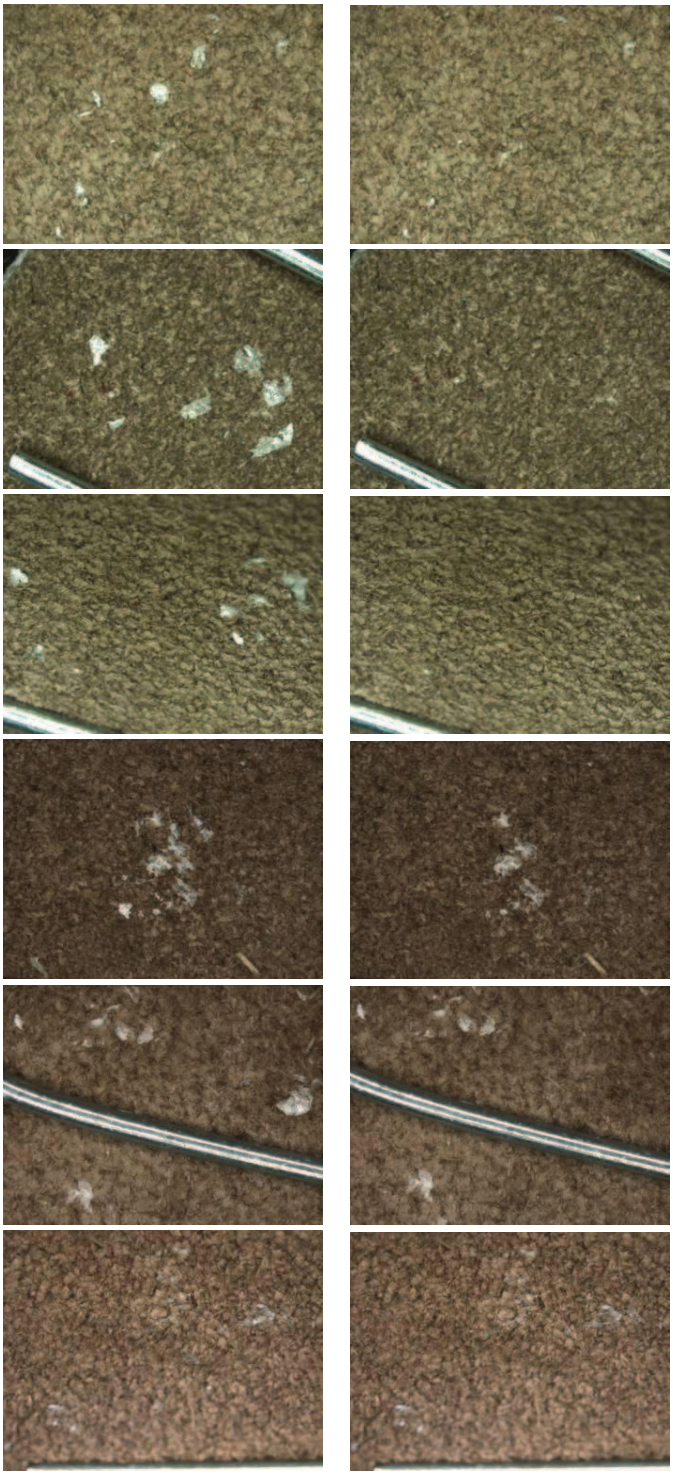
(i)

Figure 12. Guanidine Nitrate explosive on leather material at: a) 25LPM sample horizontal; b) 25 LPM sample vertical, c) 25 LPM sample 45°, d) 50 LPM sample horizontal; e) 50 LPM sample vertical, f) 50 LPM sample 45°, g) 80LPM sample horizontal; h) 80 LPM sample vertical, i) 80 LPM sample 45°

(a)

(b)

(c)



(d)

(e)

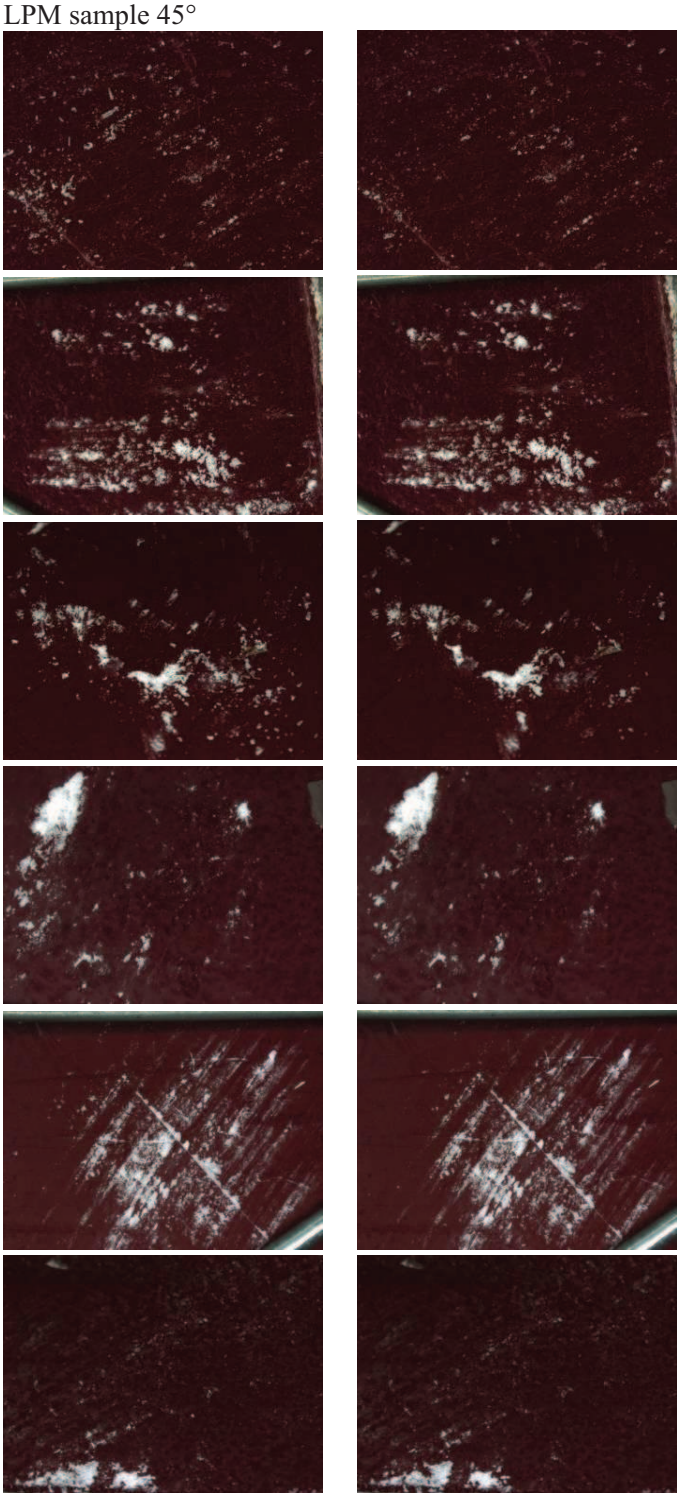
(f)

(g)

(h)

(i)

Figure 13. HMTD explosive on metal material at: a) 25LPM sample horizontal; b) 25 LPM sample vertical, c) 25 LPM sample 45°, d) 50 LPM sample horizontal; e) 50 LPM sample vertical, f) 50 LPM sample 45°, g) 80LPM sample horizontal; h) 80 LPM sample vertical, i) 80 LPM sample 45°



(a)

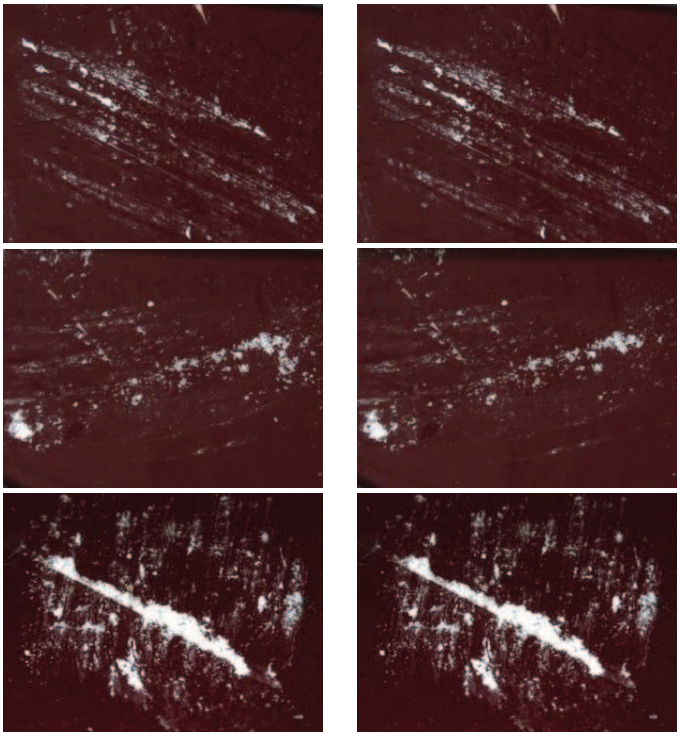
(b)

(c)

(d)

(e)

(f)

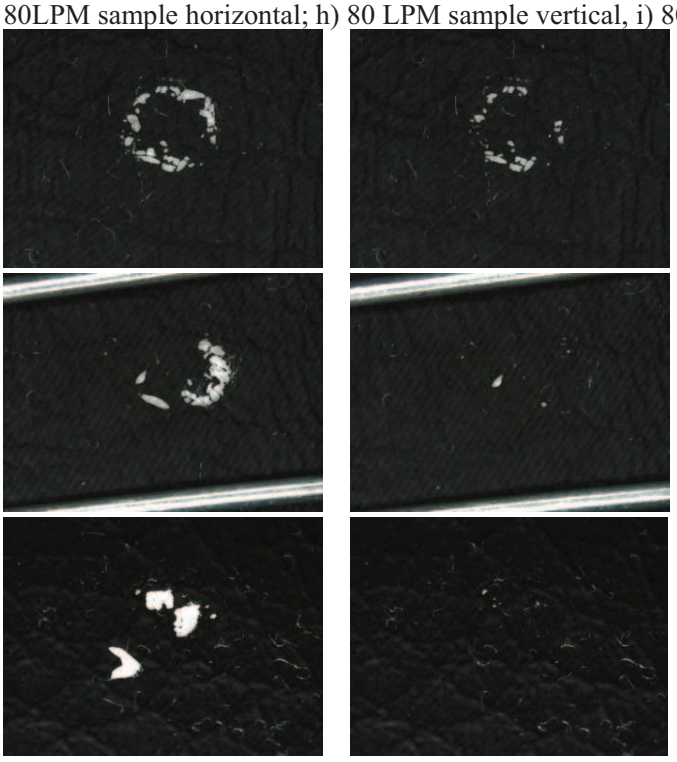


(g)

(h)

(i)

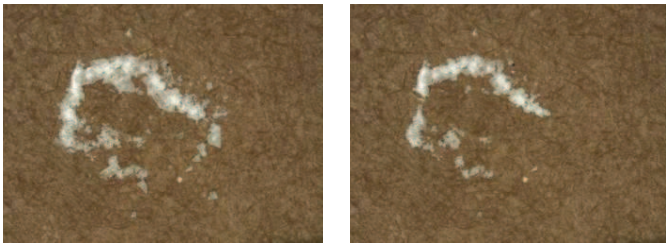
Figure 14. TATP explosive on vinyl material at: a) 25LPM sample horizontal; b) 25 LPM sample vertical, c) 25 LPM sample 45°. TATP on cardboard material at d) 50 LPM sample horizontal; e) 50 LPM sample vertical, f) 50 LPM sample 45°. TATP on canvas material g) 80LPM sample horizontal; h) 80 LPM sample vertical, i) 80 LPM sample 45°



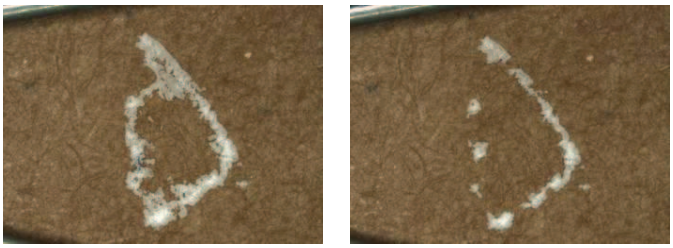
(a)

(b)

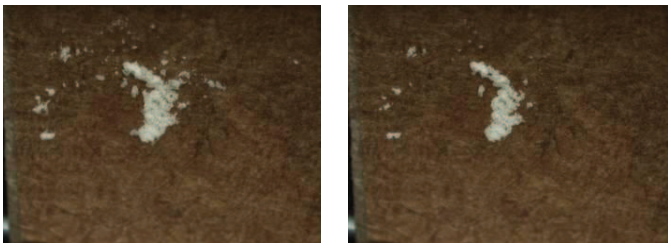
(c)



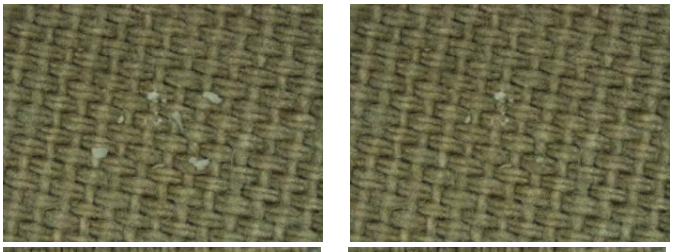
(d)



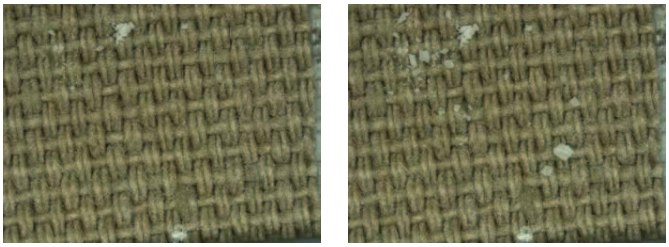
(e)



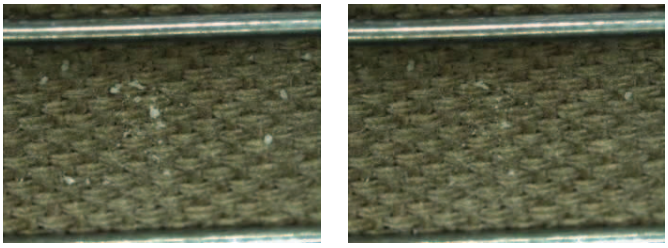
(f)



(g)



(h)



(i)

The results of the airflow tests using Military explosives are summarized on Table 4 and illustrated in Figures 15 through 20. The airflow removal rates for Military explosives were much better than those obtained via mechanical vibrations. Test results indicate that a flow of 50 LPM is the most effective at removing Military explosives particles (same as for HME). Only 7% of the Military explosives were removed by the 25 LPM air flow (0% of the SEMTEX, 16% of the TNT, and 6% of the C-4). At 80 LPM, 22% of the samples showed particle removal (11% of the SEMTEX, 39% of the TNT, and 17% of the C-4 samples). Best results for Military explosives were obtained at 50 LPM, where 60% of the samples showed particle removal (78 % of the SEMTEX samples, 78 % of the TNT samples, and 33% of the C-4 samples). Overall, TNT was the easiest Military explosive to remove by means of airflow on both substrates.

Table 4. A summary of the air flow test results for the three Military explosives at all air flows and all sample positions tested. The results are listed as number of samples with visible changes over the total number of samples tested.

		25 LPM			**50 LPM**			**80 LPM**			**TOTALS**
Explosive	**Substrate**	**Horiz.**	**Vert.**	**45°**	**Horiz.**	**Vert.**	**45°**	**Horiz.**	**Vert.**	**45°**	
SEMTEX	Cardboard	0/3	0/3	0/3	0/0	0/0	0/0	0/3	0/3	0/3	**0/18**
	Metal	0/3	0/3	0/3	3/3	2/3	2/3	0/3	1/3	1/3	**9/27**
C-4	Cardboard	0/3	0/3	0/3	0/3	0/3	0/3	0/3	0/3	0/3	**0/27**
	Metal	0/3	0/3	1/3	3/3	1/3	2/3	1/3	1/3	1/3	**10/27**
TNT	Cardboard	1/3	0/3	2/3	2/3	2/3	1/3	2/3	2/3	1/3	**13/27**
	Metal	0/3	0/3	0/3	3/3	3/3	3/3	0/3	1/3	1/3	**11/27**
TOTALS		**1/18**	**0/18**	**3/18**	**11/15**	**8/15**	**8/15**	**3/18**	**5/18**	**4/18**	**43/153**

Figure 15. SEMTEX on Cardboard at: a) 25 LPM sample horizontal (no particles removed); b) 25 LPM sample at 45° (no particles removed); c) 80 LPM sample horizontal (no particles removed); d) 80 LPM sample horizontal (no particles removed).

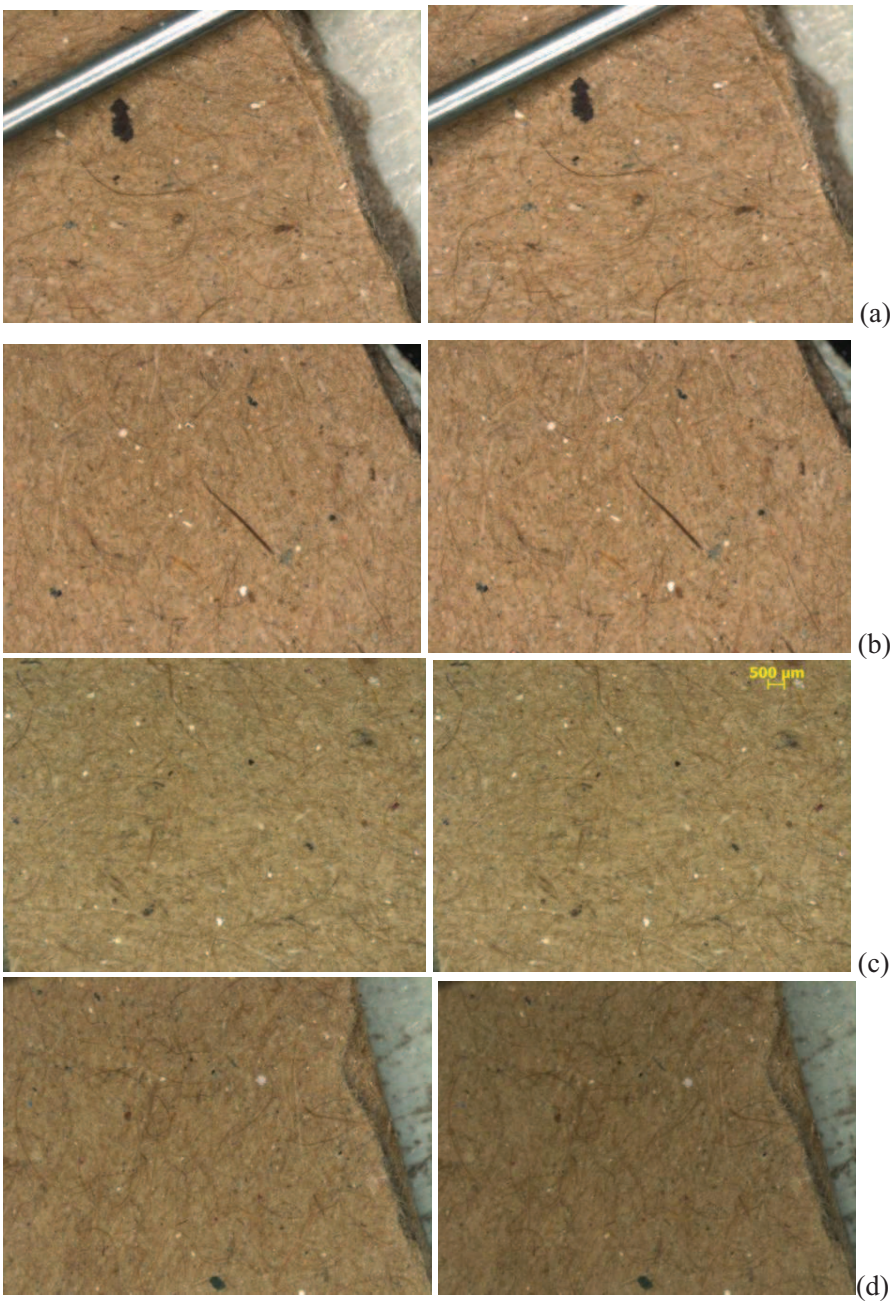
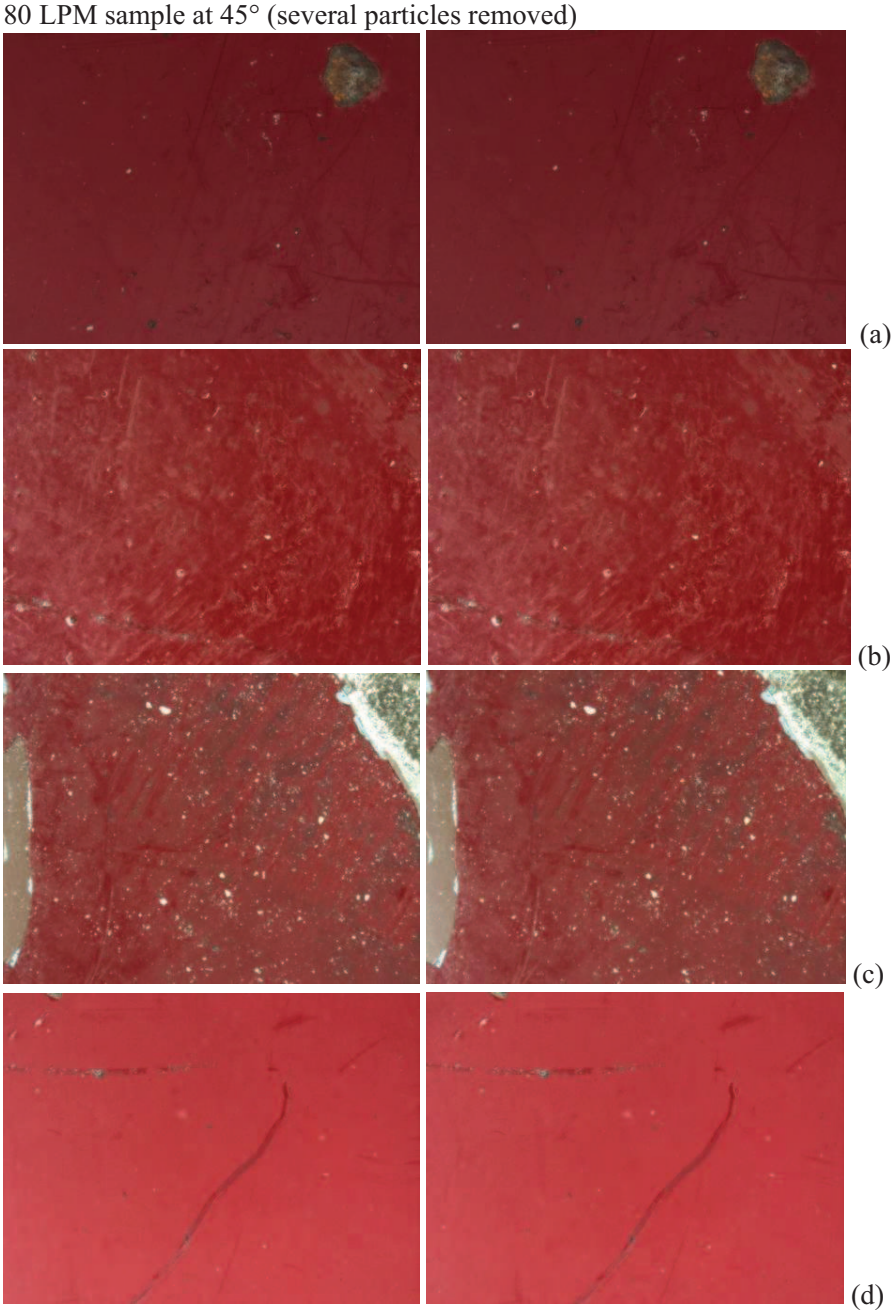


Figure 16. SEMTEX on Metal at: a) 25 LPM sample horizontal (no particles removed); b) 50 LPM sample horizontal (one small particle removed); c) 50 LPM, sample vertical (many small/medium particles removed); d) 80 LPM sample vertical (two small particles removed); e) 80 LPM sample at 45° (several particles removed)



(a)

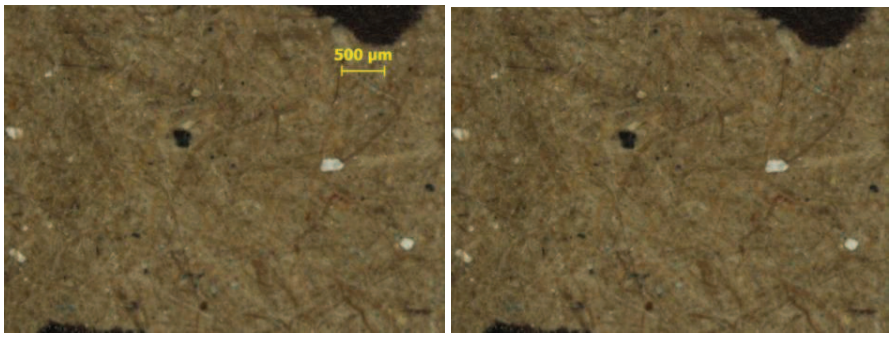
(b)

(c)

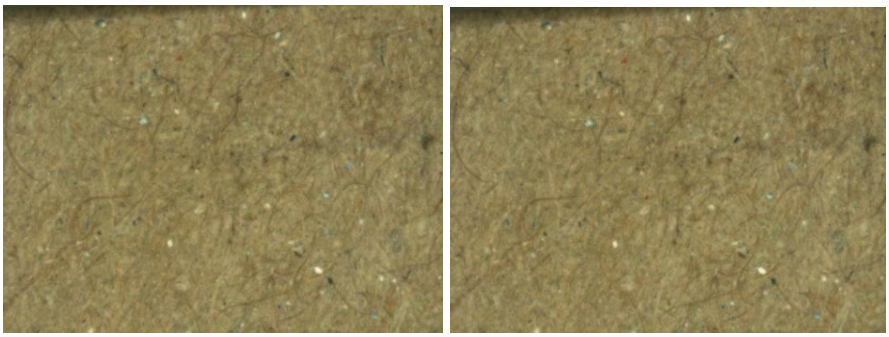
(d)

(e)

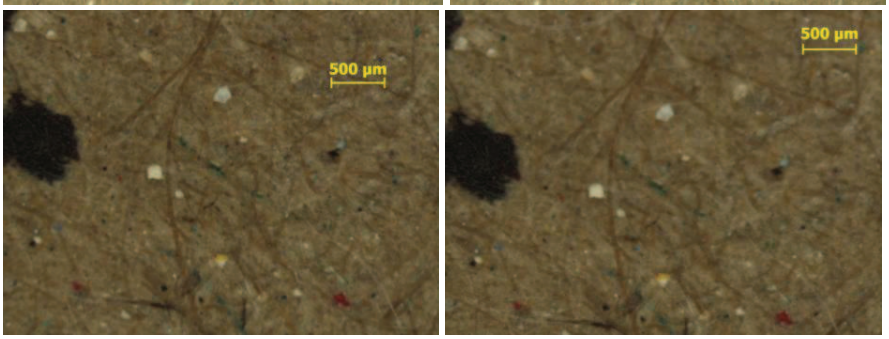
Figure 17. C-4 on Cardboard at: a) 25 LPM sample horizontal (no particles removed); b) 50 LPM sample horizontal (no particles removed); c) 50 LPM, sample vertical (no particles removed); d) 80 LPM sample horizontal (no particles removed).



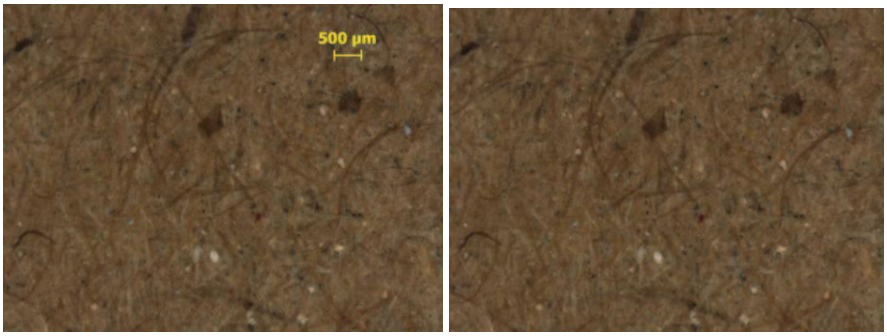
(a)



(b)

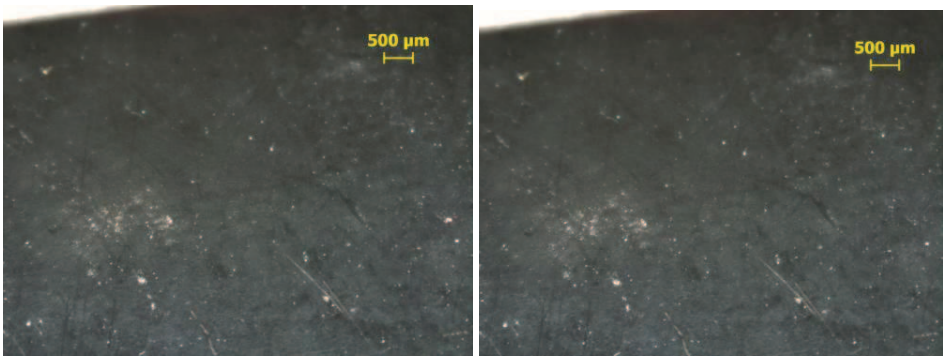


(c)

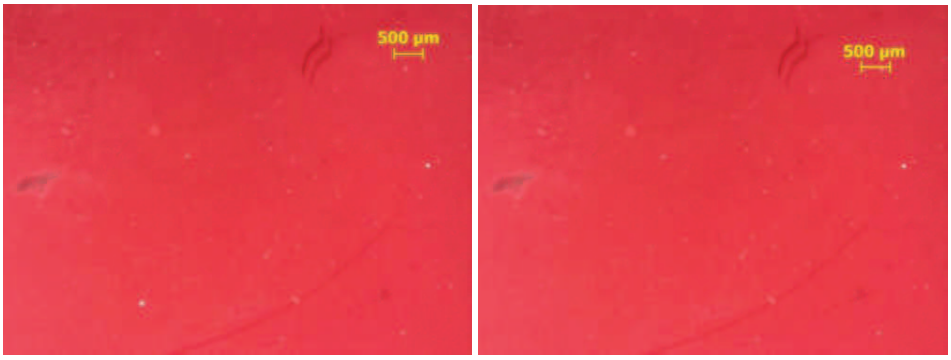


(d)

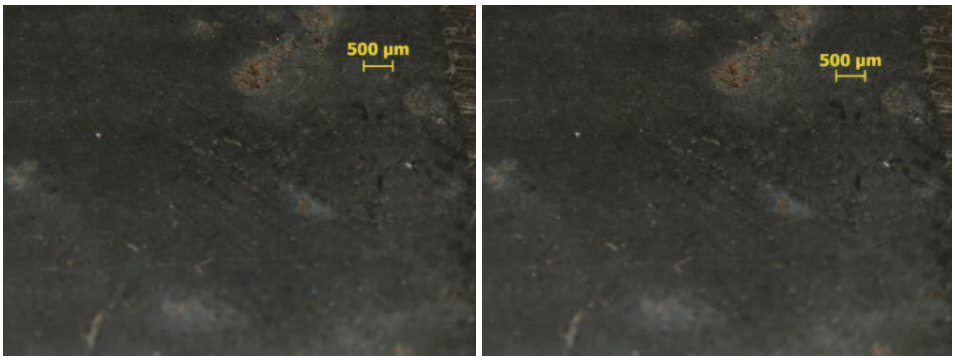
Figure 18. C-4 on metal at: a) 25 LPM, sample at 45° (one large particle on far right side removed); b) 50 LPM sample horizontal (one particle removed); c) 50 LPM, sample at 45° (several small particles removed); d) 80 LPM sample horizontal (one large particle removed).



(a)



(b)



(c)

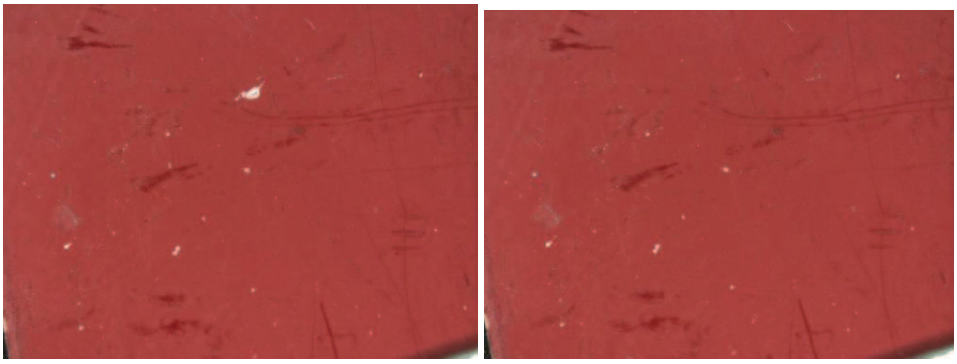
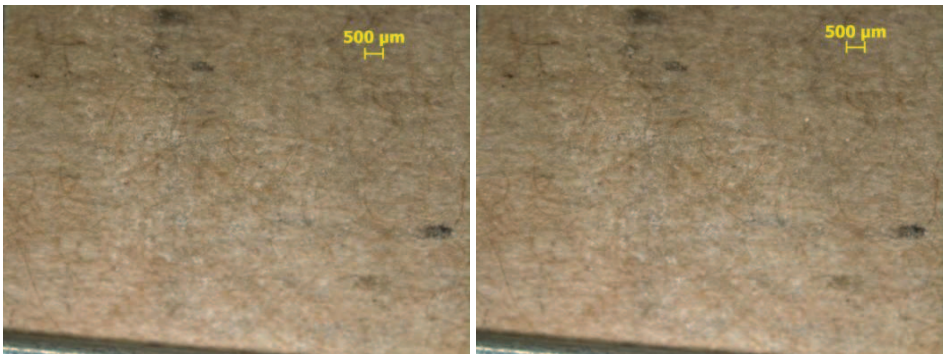
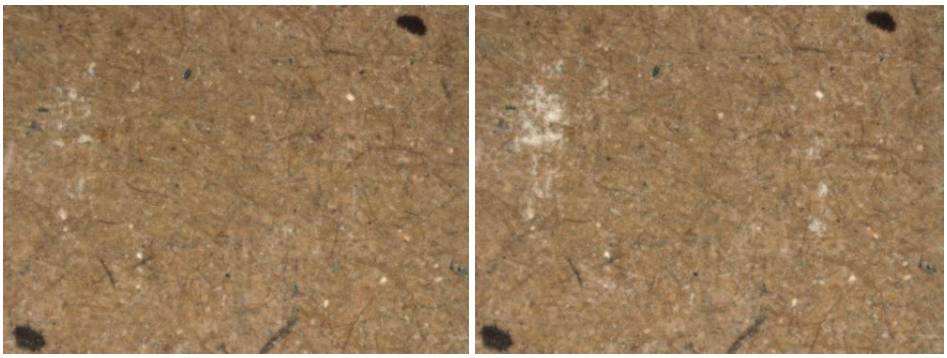
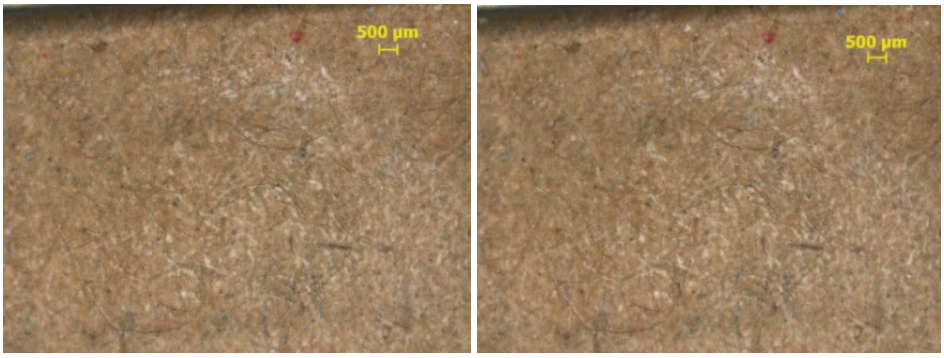

(d)

Figure 19. TNT on Cardboard at: a) 25 LPM, sample at 45° (several small particles removed); b) 50 LPM sample vertical (many particles removed); c) 80 LPM sample horizontal (several particles removed).



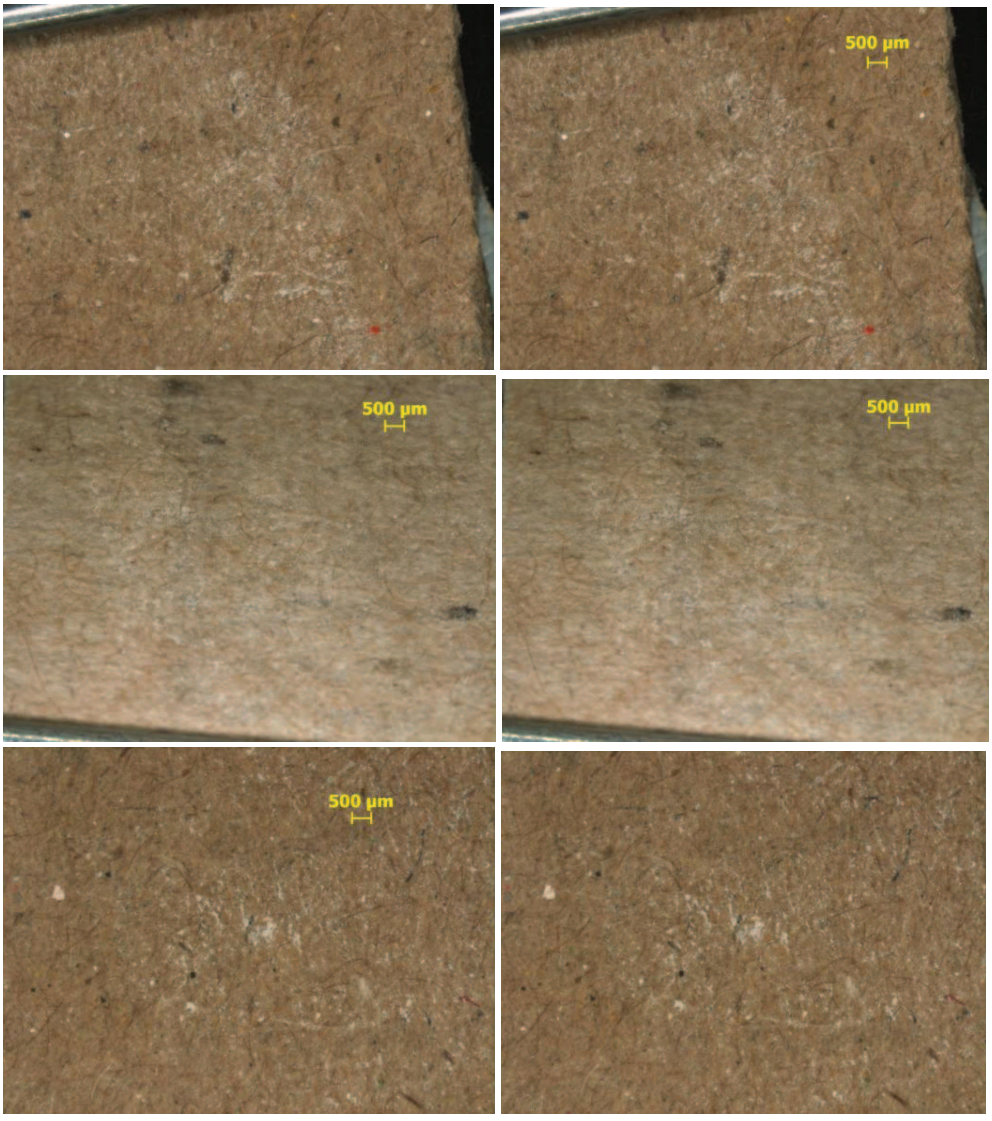
(a)

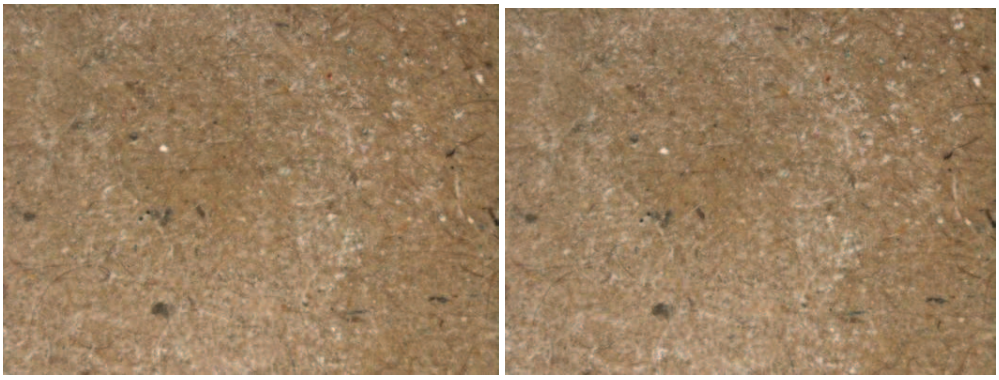

(b)



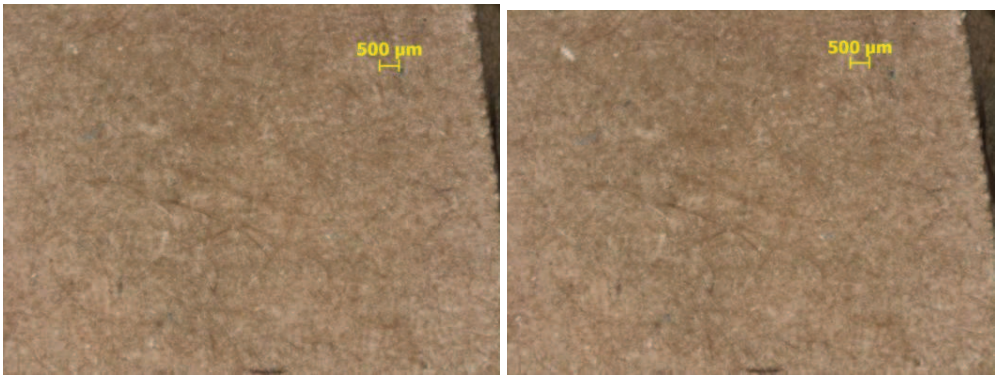
(c)

Figure 20. TNT on Metal at: a) 25 LPM, sample horizontal (no particles removed); b) 25 LPM,sample at 45° (several particles removed); c) 25 LPM, sample vertical (several particles removed); d) 50 LPM, sample horizontal (many small particles removed); e) 80 LPM, sample vertical (several large particles removed).





(d)



(e)

4.3 Removal of HME Using Heat - Test Results

The heat tests involved using an environmental chamber to control the humidity at 15% RH while holding the temperature at 40°C, 60°C, and 75°C. The fingerprint was placed on the substrate of interest and photographed using a Zeiss Discover V.12 stereoscope with Axiocam ICc1 3 megapixel digital camera. Once photographed, the sample was placed in an environmental chamber at the designated temperature for 3 minutes. Prior testing has shown that it takes ~2 minutes for the sample to reach the desired temperature, then the sample was left for one additional minute at the desired temperature. The humidity for the environmental chambers was turned off and not controlled; however, the humidity was measured and ranged from 10%-20% at any given time during the testing. This is due to the portholes in the chamber that are used to assure that over pressurization of the chamber does not occur. The portholes allow the chambers to come into equilibrium with the humidity of the external surroundings.
The samples were removed and photographed again. The amount of sample that remained on the material was compared to the pre-sample to determine the probability of removing the homemade explosive from the surface by heating. The tests were repeated in triplicate. A summary of the test results appear in Table 5. Figure 21 depicts ammonium nitrate explosive on various substrates at the various test temperatures. Figure 21a is ammonium nitrate on cardboard at 40°C. There is very little removal of the explosive at this temperature and what explosive is removed are larger pieces. It also appears that the explosive may be dissolving and re-crystallizing; however, the melting point of ammonium nitrate is169.6 °C so this does not seem probable, but from the photos of ammonium nitrate at 60°C on vinyl (Figure8c), it is apparent that some melting of the crystalline structure is occurring. Figure 22 are pictures of TATP on leather at 40°C. The left side of the picture is a before image and the right side of the picture is an after

heating image. TATP has a melting point of 91°C. In tests where temperatures of >40°C were used, there does not appear to be any residual explosive remaining on the surface.

During these tests, no testing was performed with guanidine nitrate or cotton material. For all other samples, the overall percentage of samples that showed visible particle loss was 82%. At 40°C, 56% of the samples had visible particle removal when HMTD was used, 100% when TATP was used and 72% when ammonium nitrate was used. At 60°C, 56% of the samples had visible changes in the amount of particles on the surface when HMTD was used, 100% of the TATP, and 100% of the ammonium nitrate. At 75°C, 56% of the samples had visible changes in the amount of particles on the surface when HMTD was used, and 100% of the TATP and ammonium nitrate samples.

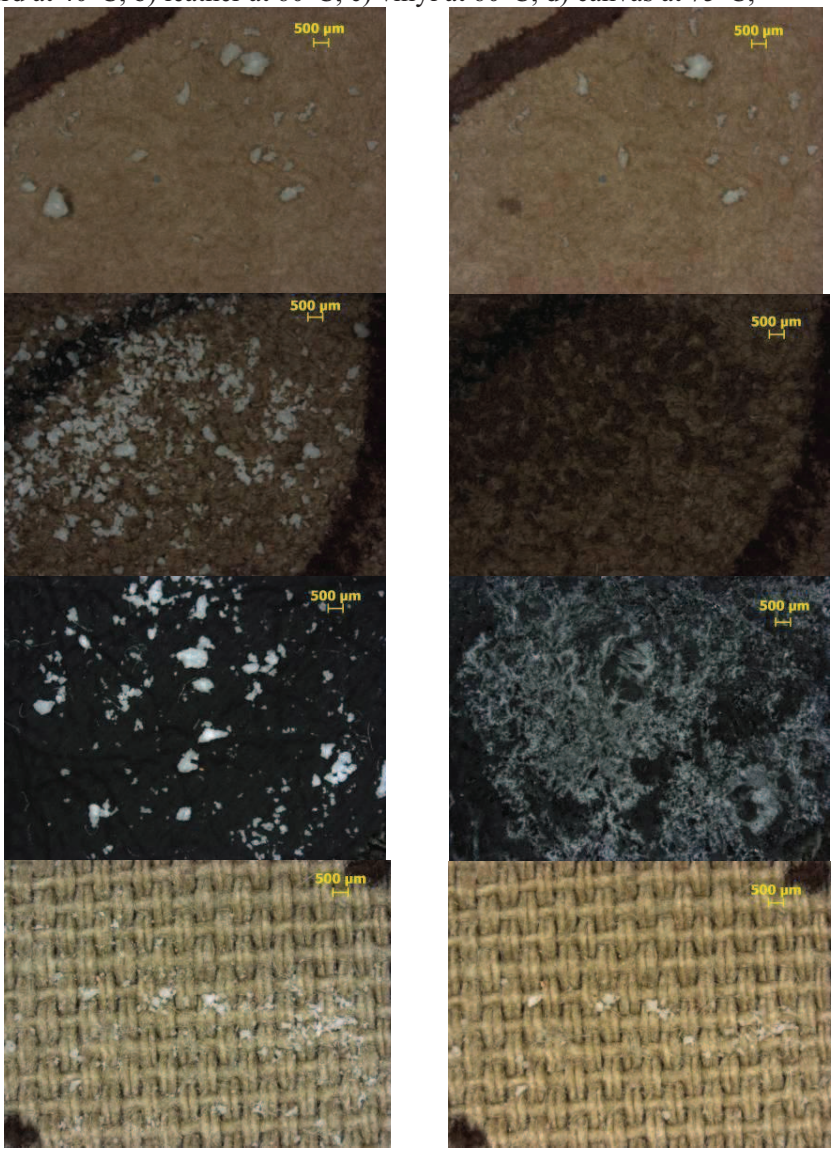
For all temperatures and explosives studied, 85% of the metal samples had visible changes, 74% of the vinyl and leather, 81% of the canvas and shrink wrap, and 96% of the cardboard. All of the TATP samples had visible changes, regardless of explosive type, temperature, or substrate. When HMTD was used 67% of the metal and canvas samples, 33% of the vinyl and leather, 44% of the shrink wrap and 89% of the cardboard samples showed visible changes When ammonium nitrate was used: 89% of metal, vinyl and leather, 78% of canvas and 100% of cardboard and shrink wrap had visible changes.

Based on the results of the heat test studies, the optimal temperature for the peroxide explosives is 40°C or less. The sample tends to volatilize or breakdown at higher temperatures, which is expected due to the low vaporization potential. For explosives such as ammonium nitrate, the higher temperatures result in melting of the sample and re-crystallization. Unexpectedly, the shrink wrap did not appear to have any visible melting at the higher temperature; however, none of the samples were monitored for any type of off gas that may affect the vapor sampling.

Table 5. Summary of heat testing results for each HME type at each of the three temperatures tested. The results are listed as number of samples with visible changes over the number of samples tested.

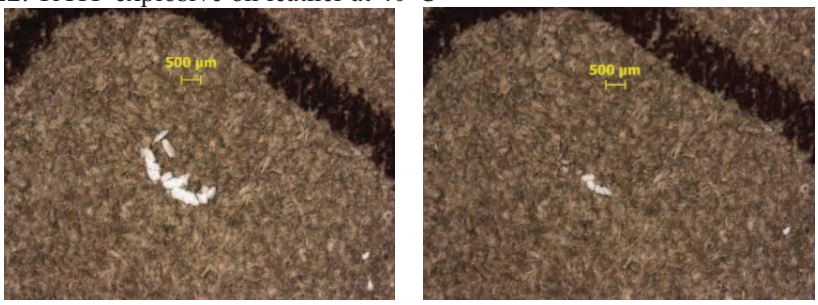
	HMTD			***TATP***			***AN***			***TOTALS***
Substrate	***40ºC***	***60ºC***	***75ºC***	***40ºC***	***60ºC***	***75ºC***	***40ºC***	***60ºC***	***75ºC***	
Metal	*3/3*	*2/3*	*1/3*	*3/3*	*3/3*	*3/3*	*2/3*	*3/3*	*3/3*	*23/27*
Vinyl	*1/3*	*1/3*	*1/3*	*3/3*	*3/3*	*3/3*	*2/3*	*3/3*	*3/3*	*20/27*
Canvas	*2/3*	*2/3*	*2/3*	*3/3*	*3/3*	*3/3*	*1/3*	*3/3*	*3/3*	*22/27*
Cardboard	*3/3*	*2/3*	*3/3*	*3/3*	*3/3*	*3/3*	*3/3*	*3/3*	*3/3*	*26/27*
Shrink wrap	*1/3*	*2/3*	*1/3*	*3/3*	*3/3*	*3/3*	*3/3*	*3/3*	*3/3*	*22/27*
Leather	*0/3*	*1/3*	*2/3*	*3/3*	*3/3*	*3/3*	*2/3*	*3/3*	*3/3*	*20/27*
Cotton	*Not done*	*Not done*	*Not done*	*Not done*	*Not done*	*Not done*	*Not done*	*Not done*	*Not done*	
TOTALS	*10/18*	*10/18*	*10/18*	*18/18*	*18/18*	*18/18*	*13/18*	*18/18*	*18/18*	*133/162*

Figure 21. Ammonium Nitrate explosive on various substrates at varying temperatures: a) cardboard at 40°C, b) leather at 60°C, c) vinyl at 60°C, d) canvas at 75°C,



There were relatively no changes in HMTD crystals on any of the substrate surfaces due to heating at any of the three temperatures. HMTD decomposes at 75°C so there should be changes in the before and after pictures. Because the data does not seem to follow decomposition behaviors for any of the explosives tested, it appears that the relative humidity in the chamber is playing a role in the changes that were seen.

Figure 22. TATP explosive on leather at 40°C



4.4 Removal of Military Explosives Using Combination of Heat and Mechanical Methods - Test Results

Tests combining heat and mechanical techniques were conducted on the Military explosive fingerprints on metal and cardboard substrates. For the heat/vibration tests, flexible heating pads and a temperature controller were used to heat the samples and vibrator sample holder to 150° F. Unlike the previous tests where tests were repeated three times, this series of tests were repeated only twice. Photos were taken prior to heating, after vibrating for 1 minute without heat, and again after the sample was heated and shaken for 1 minute. These tests were run at 35 Hz and 70 Hz, and only one set of samples (C-4 on metal at 70 Hz and 150° F shown in Figure 23) indicated any particle removal. Similar tests were conducted using heating to 150° F and air flow of 80 LPM, and this time only one of the TNT on cardboard samples showed any particle removal. These combined tests indicate that heating of the Military explosives to approximately 150° F do not increase the particle removal efficiency of either vibration or airflow methods and that significantly higher temperatures will be required to achieve significant increases in particle removal rates.

Figure 23. C-4 on metal before and after heating to 150° F and shaking at 70Hz. (several small particles removed).

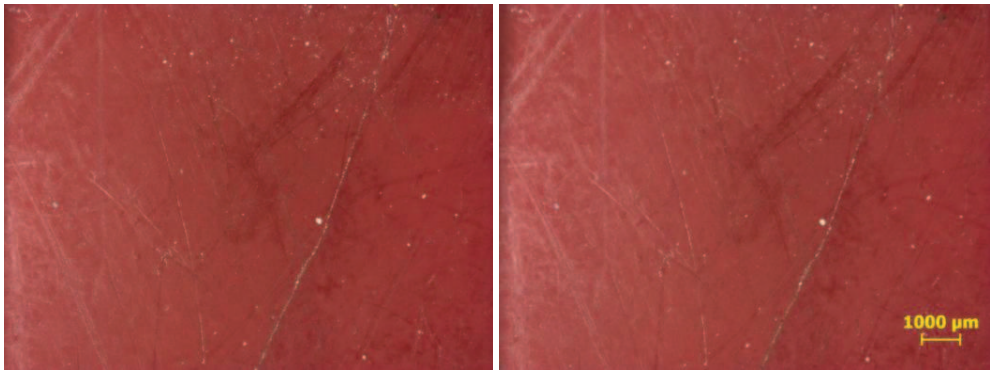
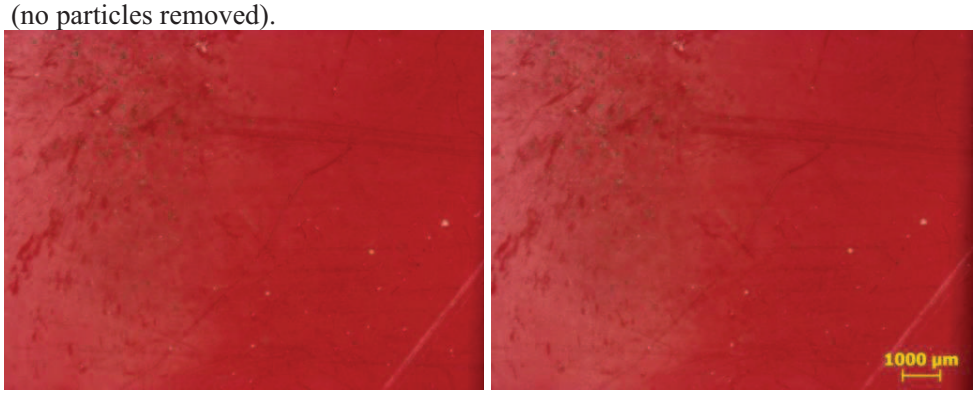


Figure 24. SEMTEX on metal before and after heating to 150° F and shaking at 70Hz (no particles removed).



5. CONCLUSIONS

Summary of vibration tests

- The overall visible removal rate for all frequencies, for all HME types, and for all substrates was 48%.
- The overall visible removal rate for all frequencies, for all Military explosive types and for all substrates was 4.6%. Only TNT on metal samples showed any particle removal (11% at 35 Hz).
- 100 Hz at 5g RMS is the least efficient frequency for HME particle removal from the substrates tested
- 35 Hz and 100 Hz have very similar results at ~43% of the HME samples tested having visible particle removal; therefore 70 Hz at 5g RMS is assumed to be the optimal frequency removal rate with 54% of the samples tested having visible particle removal.
- Least amount of HME explosive crystals are removed from metal (27%) independent of the frequency used.
- Least amount of Military explosive crystals were removed from cardboard (0%) independent of the frequency used.
- Greatest amount of HME explosives were removed from cotton (76%) independent of the frequency used.
- Greatest amount of Military explosives were removed from metal (7.4%) independent of the frequency used.
- TATP is the most difficult to remove from any substrate tested independent of the frequency used. The highest removal rate for TATP was from cardboard (44% of the samples tested).
- AN and HMTD were the easiest explosives to remove from the substrate surfaces.
- TNT was the easiest of the Military explosives to remove from all surfaces using any of the removal techniques.
- Only larger particles tend to come off independent of frequency, substrate material, or explosive type (both HME and Military). This is important for proper filter/preconcentrator design.

Summary of heat tests (HME only)

- At temperatures of 60°C and above, the explosive crystals for ammonium nitrate appear to melt and spread across the surface of the substrate in a thin film
- At temperatures of 40°C the majority of the TATP crystals vaporize from the surface of the substrate.
- Vinyl and leather were the most difficult substrates to remove the particles from using heat (~74% of the samples had visible sample losses).
- Cardboard was the easiest substrate to remove particles from using heat (96% of the samples had visible sample losses).
- HMTD was the most difficult explosive to remove, then ammonium nitrate, and TATP was the easiest. The melting points for the explosives are HMTD which decomposes at 75°C, ammonium nitrate melts at 169.6°C, and TATP melts at 91°C.
- The ammonium nitrate samples appear to melt and then re-crystallize in larger clumps or stay thinly spread across the substrate surface. The crystals change forms from β-rhombic to α-rhombic at temperatures of ~32°C which also increases the size of the crystal by ~3.6%.
- The trimeric form of TATP sublimes at room temperature and either reforms as larger crystals or completely disappears.

Summary of air flow tests

- Metal was the most difficult substrate from which to remove the GN and TATP, while vinyl was the most difficult surface from which to remove AN, independently of the flow rate or the angle of the sample.
- All explosives (HME and Military) fingerprinted onto metal surfaces tend to be flattened and are difficult to remove with techniques other than swipe sampling.
- 25 LPM, horizontal position, is the least effective configuration for HME sample removal.
- 50 LPM, vertical position, is the most effective configuration for HME sample removal.
- 50 LPM appears to be the optimal airflow for both HME and Military explosives particle removal independent of the angle the sample is being held.
- Removal rates of Military explosives by airflow was found to be fairly constant across all angles.
- HMTD was readily removed from the surface using air at any flow with the sample held at any of the three angles tested.
- TATP was the most difficult HME explosive to remove (76% of all TATP samples had visible changes).
- TNT was by far the easiest Military explosive to remove from both substrate materials tested. C-4 was only slightly easier to remove than SEMTEX.

Summary of combined heat/vibration and heat/air flow Tests

- Heating of the Military explosives did not seem to increase the particle removal efficiency of either vibration or airflow methods.

Overall Summary

- 50 LPM is the most effective airflow for removing particles from the substrate surface independent of explosive type or substrate, or vertical, horizontal, or 45º sample positioning.
- 70 Hz at 5g RMS is the most effective vibrational frequency for removing particles from the substrate surface independent of explosive type or substrate.

- TATP is the most difficult explosive to remove from the substrate surfaces using vibration.
- TNT is the easiest Military explosive type to remove using mechanical methods.
- Metal is the most difficult substrate surface to remove any of the HME from using either airflow or vibration.
- The stability of TATP is questionable. For a 1 mg sample size, the TATP sublimes and completely disappears within 24 hours if left in the open air. If the TATP has been transferred to a substrate surface, the TATP has sublimes within 2-3 hours.