Laboratory evaluation of airborne particulate control treatments for simulated aircraft crash recovery operations involving carbon fiber composite materials

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Abstract

Objective: *This study compared four treatment protocols to reduce airborne composite fiber particulates during simulated aircraft crash recovery operations.*

Design: *Four different treatments were applied to determine effectiveness in reducing airborne composite fiber particulates as compared to a "no treatment" protocol. Both "gold standard" gravimetric methods and real-time instruments were used to describe mass per volume concentration, particle size distribution, and surface area. The treatment protocols were applying water, wetted water, wax, or aqueous film-forming foam (AFFF) to both burnt and intact tickets of aircraft composite skin panels. The tickets were then cut using a small high-speed rotary tool to simulate crash recovery operations.*

Setting: *Aerosol test chamber.*

Subjects, participants: *None.*

Interventions: *Airborne particulate control treatments.*

Main outcome measures: *Measures included concentration units of milligrams per cubic meter of air, particle size distribution as described by both count median diameter and mass median diameter and geometric standard deviation of particles in micrometers, and surface area concentration in units of square micrometers per cubic centimeter. Finally, a Monte Carlo simulation was run on the particle size distribution results. Comparison was made via one-way analysis of variance.*

Results: *A significant difference ($p < 0.0001$) in idealized particle size distribution was found between the water and wetted water treatments as compared to the other treatments for burnt tickets.*

Conclusions: *Emergency crash recovery operations should include a treatment of the debris with water or wetted water. The resulting increase in particle size will make respiratory protection more effective in protecting the response crews.*

Key words: composite materials, aircraft crash recovery, dust control

Introduction

Advanced composite materials (ACMs) are present in many of the new aircraft used in the military, including the B-2 Spirit, F-22 Raptor, and F-35 Lightning II along with modern commercial aircraft like the Boeing 787.[1,2] ACM uses layers of fiber sheets bound with resin.

Carbon fibers when burnt reduce in size[3] and cause pulmonary lesions and inflammation.[4] It has been shown that the majority of fibers released from burning composites meet the nanoparticle definition.[5] As technology develops, carbon nanotubes (CNTs) are being introduced to composite fiber applications. CNTs have been shown to cause a number of lung health issues to include epithelioid granulomas,[6,7] fibrosis,[8] lung lesions,[9] and asbestos like effects.[10]

During aircraft crashes this is of concern due to the potential release of composite and nanofibers. It has been shown that sanding of ACM can release free CNTs as well as particles that comprise an epoxy matrix with CNT protrusions.[11] A literature review of CNT exposure by Schlagenhauf et al.[12] shows free CNT release and CNT protrusion on released particles during mechanical impact. CNTs were also discovered in the char after burning of CNT materials and these

DOI:10.5055/ajdm.2015.0214

could be released during the shaking of char materials. Interaction with fire damaged composite panels and the mechanical cutting of these panels is expected during the aftermath of an aircraft crash.

Such an incident occurred on February 28, 2008, when a B-2 bomber crashed on takeoff at Andersen Air Force Base (AFB), Guam.[13] Once the fire had been extinguished, there was a need to remove the airframe from the runway so that the runway could be returned to service. Within the US Air Force this is termed “crash recovery operations.” To remove the airframe, crash recovery personnel cut the airframe into sections that would fit onto a standard flat-bed trailer (~15 m). The workers commonly use gasoline-powered concrete saws. The US Air Force has conducted studies to evaluate tools and methods for these operations.[14]

This study was designed to look at several common treatment protocols to determine their effectiveness at controlling particle emission during the cutting of composite fibers panels. Current Air Force guidance recommends the use of an acrylic floor wax solution in an attempt to prevent aerosolization of the ACM fibers.[15] Water is also recommended for reduction of dry aerosols.[16,17] For asbestos fibers, wetted water is recommended.[18] Also, aqueous film-forming foam (AFFF) is used to extinguish aircraft fires at crash sites and may be present during crash recovery.[1] This laboratory-based study examined the use of four treatments (water, wetted water, wax, and AFFF) as compared to a control set of no treatment, on burnt and intact ACM tickets to determine their ability to reduce airborne exposure to ACM particulate during simulated crash recovery operations. Three samples were taken of each combination, resulting in 30 trials overall. The effectiveness was measured by gravimetric, optical, and surface area methods. Given that laboratory grade meters are not typically available to emergency and hazardous material responders, a field instrument technique was used as described by Heitbrink et al.[19] This protocol gives a field industrial hygienist or hazardous materials technician the ability to estimate the exposure at the scene and determine protective measures.

The reason several concentration measures were used was to determine any relation between them and relevance for exposure estimations. Gravimetric methods are still the accepted standard for aerosol exposure measurements. Occupational exposure limits are defined based on total and respirable fractions of gravimetric concentrations.[20] Optical methods are useful to estimate gravimetric concentrations in real-time, given several assumptions on the instrument response to the measured aerosol. They can also give information on particle size distribution.

Surface area has also been shown to be a possible dose metric for nanoparticles and is one of the three properties that Maynard[21] has recommended being measured when performing evaluations of nanoparticle operations. Particle surface area can be estimated using measurements of particle size and distribution utilizing the concept of mobility diameters. This is the technique used for Scanning Mobility Particle Sizers. This technique is very complex to execute and therefore expensive as well. An easier method for the measurement of surface area utilizing an Electrical Aerosol Detector (EAD) is put forth by Wilson et al.[22] The EAD is based on the concept of diffusion charging where a charge is attached to the surface of an aerosol, measured, and then used to determine surface properties of the aerosol.[22]

The EAD samples the aerosol and splits the flow into two parts. The first part is sent into a mixing chamber without any change. The second part is sent through activated carbon and high-efficiency particulate air (HEPA) filters which are used to clean the air. The clean air is then charged using a corona needle and sent to the mixing chamber. Within the mixing chamber, the charged ions attach to the particles that have been sampled. The mixture is then sent through an aerosol electrometer where the current generated is measured. This current is then related to the amount of surface area of the particles which could be deposited in the lungs.[22]

In an effort to use relevant measures to estimate treatment effectiveness, traditional gravimetric, optical, and surface area methods were used for this study.

Methodology

Overall

For this study, a control of no treatment and four treatments were used on the ACM tickets: water, wetted water, acrylic wax solution, and AFFF. Ticket type was selected from available representative ACM "builds" of aircraft skin currently in Air Force inventory. These tickets were 16-ply bismaleimide (BMI) graphite composite, representative of the BMI composite material that is used within the F-22 and F-35.[23] All tickets were of identical ACM composition. Ticket dimensions were 2.5 cm × 5.1 cm, and 0.16 cm thick (1 in × 2 in, 1/16 in thick). Half the tickets were heat treated (burned) as described below. Then one of the four treatments was applied, or no treatment for the control, and the ticket cut, simulating crash recovery operations. For the five different treatment options (four treatments and one control), there were three trials each for both burned and intact ACM tickets. This resulted in (5 × 3 × 2) 30 total trials.

Ticket heat treatment

To simulate the burning of ACM panels during an aircraft crash, ACM tickets were placed in an aluminum container where 100 mL of JP-8 jet fuel was then added. JP-8 is the jet fuel used in US Air Force aircraft and would be the most likely fuel for any fire that would occur during an aircraft crash. The JP-8 was then ignited and allowed to burn to extinction.

Cut experiment

To perform the cuts, a glove bag was set up to prevent exposure to any aerosol generated when the cuts were performed. The ACM tickets were secured on a ring stand within the glove bag. All measurement devices were located next to the glove bag with hoses connected to the samplers, through the glove bag port and attached to the ring stand inside the glove bag.

To simulate the gas-powered concrete saw commonly used in the field, a high-speed rotary tool (Bosch; Farmington Hills, MI) with a cutoff head was used to cut the tickets. To reduce bias, a tool extension was used so that only the cutting head of the rotary tool was inside the glove bag. A speed of 10,000 rotations per minute (RPM) was used. Typical speeds of the gas-powered saws in the field are in the range of 2,500-5,000 RPM. However, the 5,000 RPM speed for the rotary tool was not sufficient to perform the cuts on the tickets, so the next setting of 10,000 RPM was used in the experiment. One study suggested that an increased speed would tend to shift the particle size distribution to larger particles.[24]

The four treatments used on the ACM tickets were water, wetted water, acrylic wax solution, and AFFF. Deionized water was used for the water treatment. Wetted water, also known as amended water within the asbestos community, is simply water with a surfactant added. For this study, Cascade Crystal Clear (Proctor & Gamble; Cincinnati, OH) was diluted to one part surfactant and 150 parts water. This dilution reduced the surface tension of the water so that it would spread on the ticket but would not excessively foam. For the acrylic wax treatment, P&G pro line super durable finish (Proctor & Gamble) was mixed 2:1 with water per Air Force Technical Order 00-105E-00.[15] The AFFF treatment was 3M AFFF (3M; St Paul, MN) and was not diluted. A volume of 8 mL of treatment solution was applied by a spray bottle to the ACM ticket before cutting. An 8-mL treatment was sufficient to saturate the surface of the tickets and the spray bottle was used for even application across the surface.

Each ticket had its treatment applied and was then inserted into the glove bag. Once the glove bag was sealed, a 7-mm cut was made into the ACM ticket using the rotary tool. The order of the tickets was randomized to reduce bias due to increased proficiency gained during the execution of each trial. The rotary tool cutting heads were also replaced after each trial.

Instrument setup

Gravimetric concentration. Two air sample trains were used to measure gravimetric concentration per the National Institute for Occupational Safety and Health (NIOSH) standard methods 0500 (total dust) and 0600 (respirable dust). Both trains used a personal air sample pump calibrated to 2.5 lpm before and after each trial with an electronic soap bubble flow meter (Sensidyne Gilian Gilibrator-2; Clearwater, FL). The respirable sampler (NIOSH 0600) used was an aluminum cyclone size selector (SKC; Eighty-four, PA). The filters used were 37 mm matched-weight 0.8 μm pore size mixed cellulose ester (SKC).

Particle size measurement. To sample the particle size distribution produced during the cutting of the ACM tickets, a TSI PTrak (TSI; Shoreview, MN) condensation particle counter (CPC) and a TSI 8220 (TSI) optical particle counter (OPC) were used. Both instruments were plumbed into the glove bag. The OPC had a particle size range of 0.3-20 μm. The coincidence loss for the TSI 8220 OPC was 5 percent for particle concentrations less than 70 particles/cc. Coincidence loss is when several particles in the optical sensing volume are counted as a single particle due to the concentration being higher than the designed range.[25] The OPC accuracy was 50 ± 10 percent at 0.3 μm. Accuracy achieved 100 percent above 0.45 μm. The OPC was set with the default bin size ranges of 0.3-0.5 μm, 0.5-0.7 μm, 0.7-1 μm, 1-5 μm, 5-10 μm, and >10 μm. The OPC outputted samples as a raw count in each bin. The OPC was set for a 1 minute sample time with the standard instrument flow rate of 2.8 lpm.[26] The CPC had a particle size range of 0.02-1.0 μm and a linear concentration range of 0-500,000 particles/cc. The CPC standard flow rate was 100 cc/min (0.1 lpm).[27]

The OPC was started first. The CPC was started immediately after the OPC and then stopped manually immediately after the OPC's sample for a run time of approximately 1 minute for each instrument.

Initial experiments determined that particle concentrations would exceed the OPC coincidence range and the CPC linear concentration range. OPC data were adjusted with a calculated efficiency and the CPC sample train was adjusted using in-line sample dilution. The OPC adjustment was accomplished using coincidence data generated during TSI's development of the OPC used in this study. Technical experts from TSI shared data from their coincidence trials with the authors.[28] These data compared OPC bin count data to CPC counts across different concentration ranges. These data were used to construct a trend line with Microsoft Excel to determine the amount of coincidence at the concentration encountered by the OPC. The trend line produced equation (1) which gives the counting efficiency in percent at the CPC concentration.

$$\text{Counting eEfficiency} = -5.8(*\ln(\text{CPC count}) + 110.67 \quad (1)$$

The OPC count for each bin was then divided by the counting efficiency at the average CPC count for that trial to provide an estimation of the actual particle count in each bin. A further limitation in optical data was the efficiency at different particle sizes. O'Shaughnessy and Slagley[29] gave efficiency curves for two optical particle meters by particle size. A response factor was taken from the handheld aerosol monitor of that study for the midpoint of each OPC bin size. The final OPC bin count was then the raw OPC count divided by the counting efficiency from equation (1), divided by the response factor from ref. [29].

To bring the levels down to the linear region for the CPC, a solution of dilution was selected. The sample air was diluted with "clean" air from a tedlar bag. An electronic soap bubble flow meter (Sensidyne Gilian Gilibrator-2) was used to determine the dilution factor that would keep the particle concentration entering the CPC sensing volume in the midrange of its linear region. A dilution factor of 5.82 (5.82:1 clean air to sample air ratio) was calculated for the setup that was used for the cut experiments. This resulted in a 0.085 lpm flow of clean air into the CPC sample train to achieve the necessary dilution. The count concentration (count/cc) data were converted to count data by multiplying the count/cc value by the volume sampled by the CPC.

Surface area. An AeroTrak 9000 Nanoparticle Aerosol Monitor EAD (TSI) was used for measuring surface area in this study. This AeroTrak 9000 has a particle size detection range of 10.01-1.00 μm with its 1 μm cyclone in place. It is designed with its 1 μm cyclone to measure smaller particles of alveolar and tracheobronchial deposition importance and features a setting to estimate alveolar or tracheobronchial concentrations. The alveolar concentration setting was used for this experiment. For the alveolar deposition region, the AeroTrak 9000 has a surface area detection range of 1-10,000 μm²/cc. It has an accuracy of ±20 percent for particles in the 0.02-0.20 μm size range.[30]

Analysis

Once the data were collected, a series of averages and standard deviations were computed for each treatment using Microsoft Excel (Microsoft; Redmond, WA). The OPC and CPC data were then used to calculate the mass median diameter (MMD) and count median diameter (CMD) in accordance with Hinds.[31]

The OPC was unable to detect particles smaller than 0.30 μm. To calculate a CMD and MMD utilizing particle sizes smaller than 0.3 μm, a 0.02-0.30 μm bin was calculated utilizing the technique published by Heitbrink et al.[19] Their equation had to be modified as a different OPC with different bin sizes and widths was used during this study. The CPC has a detection particle size range of 0.02-1.00 μm. The technique uses the number of particles from the CPC, subtracting off the OPC size bins of 0.30-1.00 μm, as the smallest size bin. Thus, the two instruments give an estimation of particle size distribution from 0.02 to 20 μm. Equation (2) shows the modified equation used to combine the OPC and CPC count data giving the particle count for the 0.02-0.30 μm bin.

$$C_{0.02\text{-}0.30\mu m} = N_{cpc} - \sum_{i=1}^{3} C_{n,i} \qquad (2)$$

CMD and MMD values were then calculated using the OPC count data and the calculated 0.02-0.30 μm bin. A density assumption of 2.17 g/cc for sodium chloride (NaCl) was used for the calculation of MMD values. NaCl is a common reference aerosol and its density is in line with that of graphite which varies from 2.00 to 2.25 g/cc.[32] Carbon fibers are commonly referred to as graphite fiber as the fibers are of graphite.

Statistics were then performed using analytical statistics software (SAS JMP 8.0; Cary, NC). Analysis of variance (ANOVA) with Tukey's honestly significant difference (HSD) post-test was used at the $\alpha = 0.05$ level to determine if any difference between the baseline and any of the four treatments existed.

Results

Overall results of all measurements are given in Table 1. Values reported are the average of the three trials for each treatment. Standard deviations were calculated using each of the three trials for each treatment.

Univariate ANOVA was performed for each measure of particulate (respirable mass concentration, total mass concentration, surface area, CMD, and MMD) by both ticket status (burnt or intact) and treatment. The simple ANOVA model of ticket status, treatment, and the interaction of the two factors generated p-values listed in Table 2. There was generally no difference in particulate treatments for either burnt or intact tickets as compared to no treatment. The only significant factor was ticket status for the MMD measurement. From the MMD measurements in Table 1, it is clear that the particle size was larger for all treatments on burnt as compared to intact tickets, particularly water and wetted water treatments. Ticket status was also almost significant for total dust concentration and CMD measure of particle size. It should be noted that MMD measurements are estimated from CMD measures and are very sensitive to variation as a cube (power of three) is applied. Therefore, more attention was paid to the CMD measures even though no measure showed any statistically significant difference between the different treatments and the no treatment baseline.

While trying to further analyze the data, a difference in the average CMDs and CMD geometric standard deviations (GSDs) for burnt tickets was apparent to the eye. To evaluate this difference, the arithmetic average CMD and the average GSD for the particle size distributions over the three trials for each treatment control of burnt tickets were calculated. With these CMDs and GSDs, an idealized plot of the particle size distribution curve for each treatment control was plotted in Figure 1, along with the theoretical Most Penetrating Particle Size (MPPS) in classical filtration theory of 0.3 μm as shown in Hinds.[31]

While there was not a statistically significant shift between treatments in the CMD, there is a definite shift in the particle size distribution when the GSD was also taken into account. This particle size distribution shift is relevant due to the use of respirators at

Table 1. Average respirable and total mass concentration (mg/m^3), surface area ($\mu m^2/cc$), and OPC+CPC generated count median diameter and mass median diameter (μm) results with standard deviation						
Ticket status	Treatment	Respirable ± std dev (mg/m^3)	Total ± std dev (mg/m^3)	Surface area ± std dev ($\mu m^2/cc$)	CMD ± std dev (μm)	MMD ± std dev (μm)
Intact	Nothing	9.08 ± 3.10	276.89 ± 177.83	80.40 ± 39.98	0.1706 ± 0.0056	0.31 ± 0.12
Intact	Water	11.95 ± 1.80	315.26 ± 27.96	109.27 ± 28.50	0.1714 ± 0.0019	0.32 ± 0.04
Intact	Wetted water	13.75 ± 3.70	231.87 ± 62.16	142.67 ± 14.94	0.1726 ± 0.0042	0.35 ± 0.10
Intact	Wax	9.82 ± 0.91	263.22 ± 45.68	158.75 ± 14.21	0.1712 ± 0.0018	0.31 ± 0.03
Intact	AFFF	12.50 ± 0.49	405.63 ± 134.51	73.77 ± 13.62	0.1690 ± 0.0075	0.291 ± 0.13
Burnt	Nothing	13.08 ± 3.62	323.48 ± 21.21	75.97 ± 23.90	0.1767 ± 0.0027	0.42 ± 0.07
Burnt	Water	9.65 ± 1.92	197.68 ± 60.13	47.41 ± 25.91	0.3638 ± 0.2918	30.63 ± 51.41
Burnt	Wetted water	12.72 ± 2.36	241.03 ± 91.03	30.17 ± 17.77	0.7174 ± 0.5793	61.95 ± 55.22
Burnt	Wax	10.60 ± 6.01	166.91 ± 78.11	478.45 ± 731.64	0.1796 ± 0.0082	0.45 ± 0.15
Burnt	AFFF	13.41 ± 4.76	226.58 ± 100.77	90.42 ± 13.20	0.1857 ± 0.0128	0.71 ± 0.40

the crash site during aircraft disassembly. Respirator filters work better at larger particle sizes.

To determine if a statistically significant difference existed between the distribution curves, a Monte Carlo simulation was run using the natural logarithm of the CMD and GSD to develop sets of data points for each idealized distribution with more statistical power. ANOVA ($\alpha = 0.05$) was then performed on each of these sets of normalized data points. A statistically significant difference was found (p-value < 0.0001). Tukey's HSD post-test was then run to determine the order and groupings of the data as shown in Figure 2. Water and wetted water treatments were statistically different, and both were different from the other treatments for burnt tickets.

Discussion

A number of metrics were compared to determine if any of the treatments offered any type of benefit when compared with the control of no treatment.

Table 2. Statistical significance of ticket status (burnt or intact), treatment, and interaction for different measures of particles			
Measure	Ticket status	Treatment	Ticket status × Treatment
Respirable dust	0.699	0.424	0.550
Total dust	0.059	0.313	0.232
Surface area	0.714	0.321	0.544
CMD	0.054	0.140	0.145
MMD	0.046*	0.136	0.137
*Significant (<0.05).			

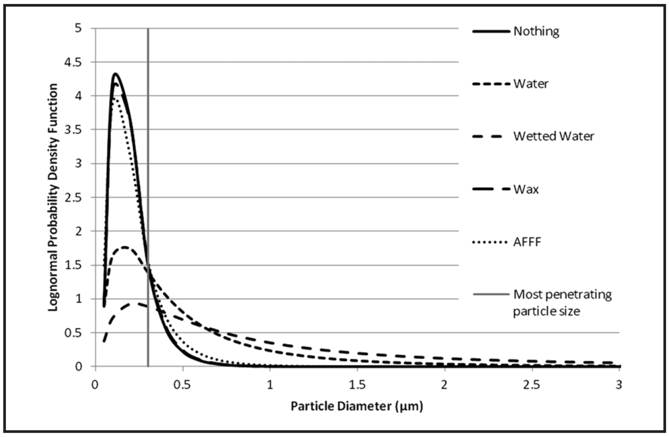

Figure 1. Plot of idealized particle size distributions for burnt tickets based on CMD and CMD GSD data.

Respirable mass concentration, total mass concentration, surface area concentration, CMD, and MMD were all compared. No measure showed any statistically significant difference between the several treatments and the no treatment baseline. However, Monte Carlo analysis showed a shift in particle size distribution, measured as CMD, by treatment for burnt tickets. There are several reasons for this.

First, the variability was high, as shown in Table 1. For intact tickets, the treatments had little effect on the different measurements. This may be because the treatment was applied to the surface. Once the ticket is cut, the treatment only affects the particles generated at or close to the surface. For burnt tickets, the surfaces begin to delaminate so that the treatment applied to the surface can get deeper into the ticket. This was more pronounced for water and wetted water than for wax or AFFF.

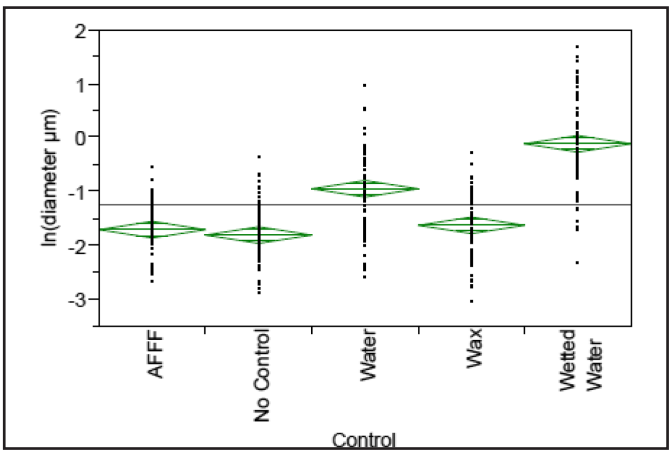

Figure 2. Natural logarithm of particle size CMD from burnt ticket Monte Carlo analysis by treatment.

However, the change in particle size for burnt tickets for water and wetted water shown in Figure 1 was interesting. There was an apparent visual shift in particle size. This would make sense as the water droplets would get deeper into the ticket material for burnt tickets where the surfaces had delaminated compared to the intact tickets. The water present would tend to agglomerate with the aerosols generated during the cut procedures as opposed to intact tickets where the water would not penetrate. The larger particle size distribution would make respiratory protection more effective at filtering particles from the air.

Further, Eninger et al.[33] showed that the actual MPPS may be smaller than 0.3 µm when utilizing electret filters. Additionally, Eninger et al.[33] showed that the current NIOSH respirator filter testing procedure is not capable of detecting particles <0.1 µm in diameter and particles between 0.1 and 0.2 µm in diameter contribute little to the certification metric. Because of these findings, any shift in particle size distribution to larger sizes would reduce the level of aerosol in the more penetrating size range for the respirator filters, thereby increasing the respirator's effectiveness at filtering the aerosol out of the air. This would increase the protection afforded crash recovery personnel utilizing respirators during aircraft disassembly.

The limitations of the EAD include that it may not measure a geometric surface area of the particles in question, but rather the active surface area of the particles. That is, the area that is available for reaction with the environment surrounding the particle. Additionally, the relation of the output of the EAD must take into account the breathing rates of the worker population being evaluated as the output is in units of area per volume. This is relevant because workers who are performing administrative tasks will inhale a smaller volume of air than workers who are performing manual labor.[34]

Crash recovery operations using gasoline-powered concrete saws provide intense mechanical energy to both intact and burnt ACM resulting in hazardous aerosol generation in the worker's breathing zone.

While this study was only a controlled laboratory experiment, it indicated that the water and wetted water treatments may be preferred for burnt aircraft sections to increase particle size and therefore the effectiveness of respirator filter performance. There was no statistical difference between the other treatments and no treatment for any of the aerosol measures used.

The limitations of this laboratory-based study include the treatment applications, differences in cutting methods, and the glove bag setup. In the field, more than 8 mL of treatment would be applied. However, the intent would be the same—to saturate the surface. For the ACM tickets used, 8 mL was sufficient to saturate the surface. The rotary tool had a smaller cutting wheel and a higher RPM than a gas-powered saw. This would make a difference in particle size distribution as was mentioned earlier. The saw may produce a smaller particle size distribution, so the shift in particle size from water or wetted water treatment of burnt ACM may be significant but different from this study. The glove bag setup was needed to protect the researchers in the laboratory. However, it may be the largest limiting factor for application of the results. Aircraft tend to fly and crash outdoors. Outdoor air currents and conditions vary and would tend to disperse the particles and reduce the hazard compared to the interior of the glove bag. Because the glove bag was a small volume and all particles were confined, optical measures were high and adjustments had to be made to the instrument results. This may have been a significant limiting factor in being able to statistically determine an effect of the several treatment protocols.

Conclusions

Particles from ACMs present a potential airborne hazard to exposed workers. During emergency and postemergency recovery operations, intense mechanical energy supplied to the burnt and intact ACM components of crashed aircraft releases particles into the air. These airborne particles must be considered in the health risk equations of emergency managers. Application of water, wetted water, acrylic floor wax, or AFFF had no effect in reducing airborne particle hazards from intact ACM tickets when cut, as compared to the no treatment control. For burnt ACM tickets, water or wetted water treatments were shown to increase aerosol particle size measured as CMD, rendering respiratory protection more effective. Acrylic floor wax and AFFF had no effect on particle measures for burnt tickets. Recovery crews should keep ACM wet during crash recovery operation activities and continue to wear prescribed respiratory protection.

Because of the limitations in this study, further research is needed to assess the airborne particle effects of the saw interactions, glove bag versus field operations, and field application methods and volumes.

Acknowledgments

This work was completed with support from the United States Air Force School of Aerospace Medicine. ***Previous podium presentation****: This work was presented at the American Industrial Hygiene Conference and Exposition in Portland, OR, May 14-19, 2011.* ***Disclaimer:*** *The views expressed in this article are those of the authors and do not necessarily reflect the official policy or position of the United States Air Force, the US Department of Defense, or the US government.*

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Originally published in Journal of Emergency Management, 2015; 13(5): 468-476. DOI:10.5055/jem.2015.0256.