Fixatives Used for Decommissioning and Maintenance of Radiological Facilities – 17537

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ABSTRACT

The Idaho National Laboratory (INL) and the United Kingdom (UK) National Nuclear Laboratory (NNL) have been collaborating for several years on materials and methods for the fogged/misted introduction of fixatives into radiologically contaminated facilities. The objective of the project is to deliver a process for reducing airborne radiological and/or mercury contamination and affixing loose contamination in place, thereby reducing contamination risk to employees and decreasing D&D cost and schedule. The developed process provides a reliable, unmanned method of introducing a coating that captures and fixes contamination in place within facilities.

The INL coating, termed FX2, has undergone extensive non-radiological testing, including determination that it is non-flammable, affixes contamination and flows well through unusual geometries (testing at Florida International University). A series of non-active fogging trials for activity knock/tie-down application have been completed at NNL Workington on behalf of Idaho National Laboratory (INL). These trials performed by the NNL employed commercially available agricultural fogging equipment and the INL's knock/tie-down latex formulation (FX-2). This testing successfully demonstrated the ability of the fogging devices to successfully spray the FX-2 formulation within various scenarios, and prepared the project for a radioactive trial. The INL has also developed a mercury vapor reducing form of the coating termed FX-Hg, which has shown great promise in laboratory studies.

INTRODUCTION

One important consideration during the decommissioning of highly contaminated nuclear facilities is to prevent the spread of airborne contamination. This is a serious issue for facilities where large accumulations of radioactive dust and lint are present, such as disposal site exhumation, laundry facilities, exhaust ventilation ducting and exhaust stacks. During a 2004 demolition of large ventilation ducting (about 10' cross section and 100' long) at Brookhaven National Laboratory a spray coating was applied to the duct from inside using a painter dressed in anti-contamination clothing and an airline respirator system.[1] In another case at the Idaho Site there were significant accumulations of contaminated lint throughout the ductwork of the radioactive laundry facility. Traditional methods of capturing the lint prior to decommissioning, like simple glycerin fogging, were not successful because it didn't penetrate and bind the lint.[2] The estimated loss of productivity typically exceeds fifty percent on most projects requiring respiratory protection. These conditions exist in Department of Energy (DOE) Facilities where airborne contamination mandates the use of costly contamination controls and significantly reduces worker productivity.

These hazards may be mitigated with the use of a strippable or permanent coating (termed a fixative). A strippable coating is removable, and relatively temporary (typically no more than a few weeks of duration) while the fixative is used for longer durations and is not removable; either may be used for short term fixation (lock-down, tie-down) of contamination. One particularly hazardous task is the decommissioning of very large contaminated facilities that must undergo extensive “open air” demolition. A successful case is the on-going decommissioning of the Plutonium Finishing Plant/Plutonium Reclamation Facility(PFP/PRF) at the DOE Hanford Site. This complex of buildings is considered the most highly contaminated at the Hanford Site.[3] The Bartlette PBS fixative is being used extensively to prevent airborne contamination while these buildings are being torn down (Fig. 1).[4] The roof of the PRF is currently open with no contamination transfer detected.

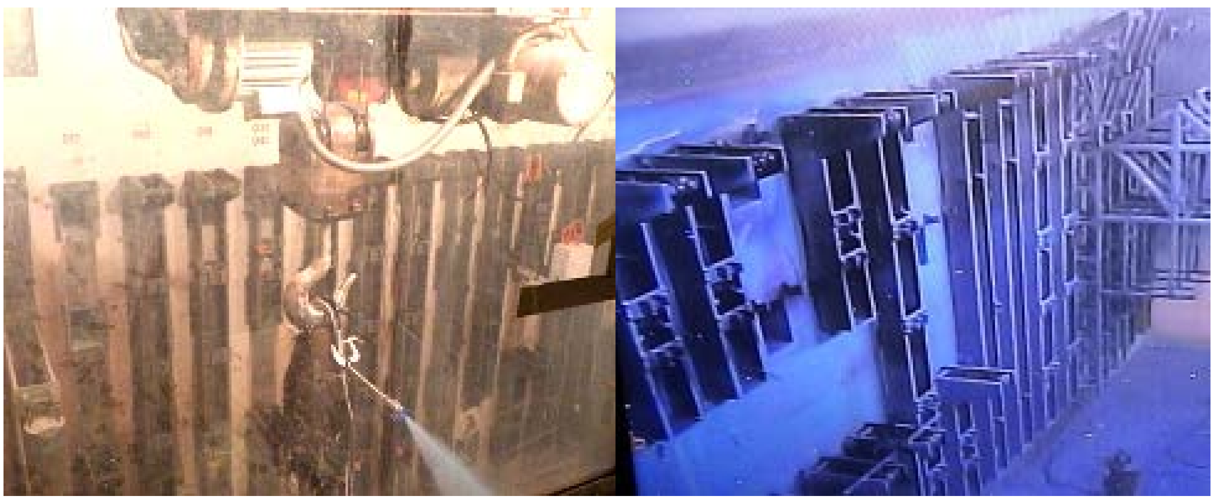


Fig. 1, Spraying PBS at the PFP/PRF and “Fixed” Contamination in Blue.

Historical Background

The beginning of fixative use for the nuclear industry is shrouded in time. Likely in the early days of the nuclear industry (certainly by 1950) it was recognized that a “coat of paint” was the final arbitrator for some types of facility contamination. Part of any old nuclear facilities’ legacy is the “layer of red paint”, that, if exposed, needed immediate attention. During the In the 1980s a new emphasis was placed on better fixative agents by the asbestos industry when asbestos bridging encapsulants, penetrating encapsulants and lockdown encapsulants were marketed. These technologies allowed use of facilities without removal of the hazardous asbestos while managing the hazard in place.[5]

While the need for contamination control is acute and obvious during facility decommissioning tasks, a more recent and increasing need is the availability of a fixative for use in urban environments in the event of a terrorist action or nuclear accident. Fixative use was reviewed by Parra, et al, for application in the field of urban remediation and specific examples of different coatings that can be used.[6] These coatings included: Aerosol Fogging, Clays, Zeolites, Molecular Sieves, Inorganic Salts, Biopolymers, Acrylics, Foams, Gels, Epoxy, Organic Binders and Lignosulfonates. These materials could be used in a wide variety of ways and concepts for the widespread application of fixatives are discussed in this article. Sutton found that fire-fighting foams, especially those incorporating clays were advantageous for urban fixation of radioactive contamination; though use of fire-fighting equipment with Cs-137 contamination may tend to spread this soluble contamination quickly.[7]

By the 1990s the use of fixatives in radiologically contaminated, DOE facilities was gaining strength. Ebadian reviewed strippable and fixative coatings as protective agents applied to an uncontaminated surface in an area where contamination is present so that on its removal the surface remains uncontaminated, as a decontamination agent when applied to a contaminated surface so that on its removal a significant decontamination of loose particulate activity is achieved, and as a fixative or tie-down coating when applied to a contaminated surface so that any loose contamination is tied down, thus preventing the spread of contamination.[8] Archibald, et al, performed successful testing at the INL on radiological contamination removal with five different strippable coatings: ALARA 1146, SensorCoat, Bartlett Stripcoat TLC, WES Strip and PENTEK 604. He found the self-stripping PENTEK 604 to be very effective at contaminant removal.[9]

Unfortunately there have been problems associated with the use of fixative materials in DOE facility decommissioning. One case the loss of control of contamination from the Separations Process Research Unit (SPRU) facility in Niskayuna, N.Y. on September 29, 2010, where contaminated facilities and equipment were being size reduced in the open air. The contamination resulted (partially) from the ineffective use of fixative materials, where fixatives were applied to accessible areas, but not thoroughly and not on process equipment internals. An area about the size of two football fields, including other facilities nearby, was contaminated from the releases. Cleanup from this event was estimated to cost more than $1M.[10] A different kind of event occurred in the same PFP/PRF facility discussed before, where PBS is being used successfully. In this case, in September of 2015, a glycerin based fogging fixative was found to have reacted with nitrate process residues in the "Pan J" area of the facility. No loss of contamination control occurred there, but chemical reactions were noted within that specific facility area and work was terminated until the extent of the circumstances were known.[11]

Development of INL/NNL FX Fixative Process

The initial objective of the Vista Technologies/INL DOE Sponsored small business innovative research grant (SBIR) project in 2006 was to develop a fogged capture coating that would allow for easy application, unmanned operation and permeate throughout facilities and equipment. The solution needed to penetrate through the surface layer of dust and "freeze" the dusty contaminants, fixing them to the substrate. Laboratory tests were performed to develop new formulations using different surfactants, binders and other components. These were applied in simple scoping studies to samples of lint and dust to simulate contamination. The simple tests consisted of spraying these solutions onto pads of lint mixed with talcum powder. These tests indicated whether solutions were able to capture the dusty lint type of contaminants prepared in the beakers. Twenty different simple tests were completed using latex paint, glue, surfactants, glycerin, waxes and commercial fixatives common to the dust and asbestos industries. These solutions were evaluated on the ability of the solution to penetrate the top layer, contain dust and bind the material. Based on the results of these simple spray tests, solutions containing latex paint and surfactants were taken to fogging tests (Fig. 2) and a successful solution, FX1, was identified.

Fig. 2, Initial testing apparatus for INL/Vista SBIR.

In 2011 it was recognized that two separate technologies, the United Kingdom's (UK) National Nuclear Laboratory's (NNL), Pursuit Dynamics (PDX) large volume atomization technology (Fig. 3) and the Idaho National Laboratory's FX1 fogging solution, could be integrated into a powerful contamination capture system. The NNL's PDX was developed through the National Nuclear Laboratories work within the Chemical, Biological, Radiological and Nuclear (CBRN) response arena, where the laboratory has been using its expertise in waste characterization, waste management, and decontamination to allow post terrorist-incident recovery.

Fig. 3, PDX Atomizer and laser particle analyzer testing

The integration tests of the fogging solution and the misting technology showed that they are compatible and have significant potential for use as a contamination control method. The two technologies were developed without consideration of using them together, thus it is little surprise that some further development would be required to achieve a proper balance of characteristics. The FX1 fogging solution had been developed to a “bench-top” level of maturity, using entirely different equipment and conditions than the pneumatic PDX atomizer. Given that, the first formulation of FX1 and PDX showed many good qualities:

- A dense fog was produced that lasted over 10 minutes.
- Coalesence that binds dry contamination to the substrate.
- Line of sight and non-line of sight coverage.

These are highly desirable traits and balanced the lack of thickness and dryness of the product that were observed. However, the results of those first tests were inadequate to recommend immediate transfer into a full demonstration.

In 2012 the NNL and the INL demonstrated that a large scale application of the fog was possible with pneumatic, "aerosonic" dispersions. Unfortunately, the UK PDx Company's Aerosonix fogging equipment was discontinued and a suitable substitute was not easily discovered during 2013. In 2014 the INL fabricated and began to run tests in their new fogging enclosure[3]. The fogging experiments were conducted inside a 2.5 m square enclosure with about 2 m height at the roof peak. It was equipped with remote access provided by sample ports and gloves. The purpose of this chamber was to contain the low-pressure (< 1 atm.) fogging solution, while workers are located outside the chamber. The enclosure was constructed using commercially available flame retardant polyethylene sheeting (6 mil thick Americover) with a plastic support frame (in accordance with NFPA 701-04). A commercially available intrinsically safe (explosion proof) exhaust fan (Mod. EPF-

10P.3, MagnaLight.com) with ductwork and air filters were used to provide a slight negative pressure in the enclosure and to prevent environmental release of fog during enclosure evacuation. The fogging enclosure is shown in Fig. 4.

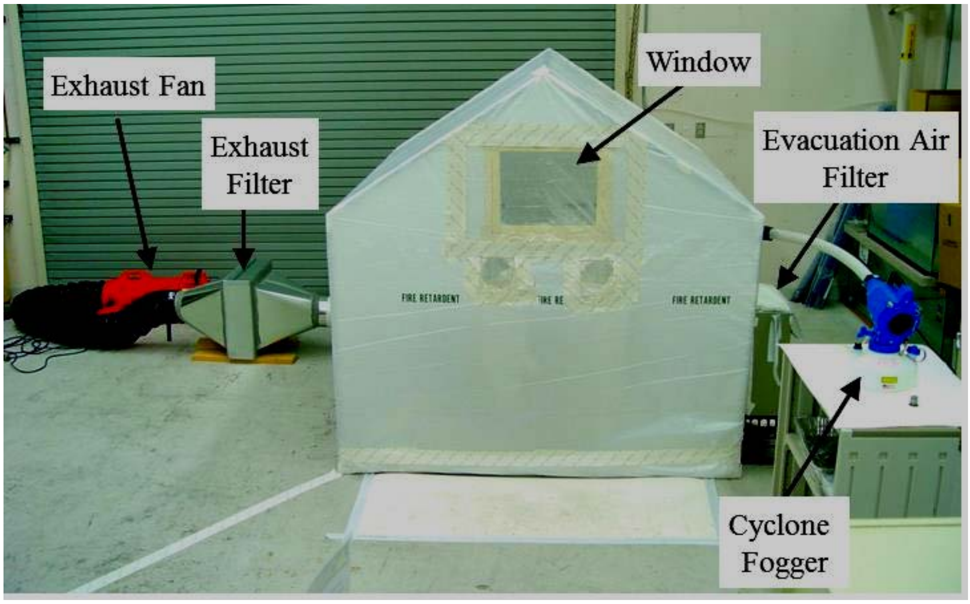


Fig. 4, INL Pilot scale fogging enclosure.

Various mixtures of water, latex paint (LTX), glycerin (GLY) and sodium lauryl sulfate (SLS) were tested in the enclosure. The Fractional Factorial method of optimizing the solution concentration based on the desired characteristics was employed. In this method a conceptual “box” is constructed using the high and low concentrations of the different constituents. The results are tabulated and compared in a relative manner and an arithmetic model is constructed to determine the optimum concentration of the overall solution. About 2.5 liters of each solution was prepared using different formulations (the high and low concentrations of each constituent). A view inside the tent before and during fogging is shown in Fig. 5.

Fig. 5, Before (left) and during (right) fogging in the enclosure.

The test materials used to quantify these tests were talcum powder placed in a small plastic dish and a thin and thick polypropylene cloth/batting coupon cut into 10 cm squares. The talcum powder was used to determine the "dustiness" (the relative movement of the dry powder) after fogging and the batting coupons (coupons) were used to evaluate surface coverage and fog penetration into the coupons. Although the coupon's loosely woven fibers provided ample "open space" for fog penetration, the fibers of thin coupons were more closely packed in comparison to the relatively thick coupons. As such, penetration into thin coupons was likely constrained by the relatively close fiber packing.

The results were judged based on the reduction of dustiness, coverage over the surface and penetration of the fog into the surface of the coupon. Dustiness was qualitatively evaluated by the ability of a dried pan (a 100 cm^2 weighing dish) containing talcum powder to be "fixed" (encapsulated) after fogging and remain immobile when being rubbed and inverted. Penetration of the fog was judged by the relative depth of fog penetration into the central portion of the coupon. Surface coverage was subjectively determined by the relative amount of fog solution deposited on exposed coupon faces (the side facing up). Penetration and coverage evaluations incorporated digital microscope images that were acquired using a Leica digital camera (DFC450) and microscope (Z16 APOA) (Fig. 6). The "stickiness" (good) and "oiliness" (bad) of the fog solution played a role in helping to determine the quality of fog produced. Relative to oily solutions, a sticky solution produced fog that better encapsulated, penetrated, and covered test materials.

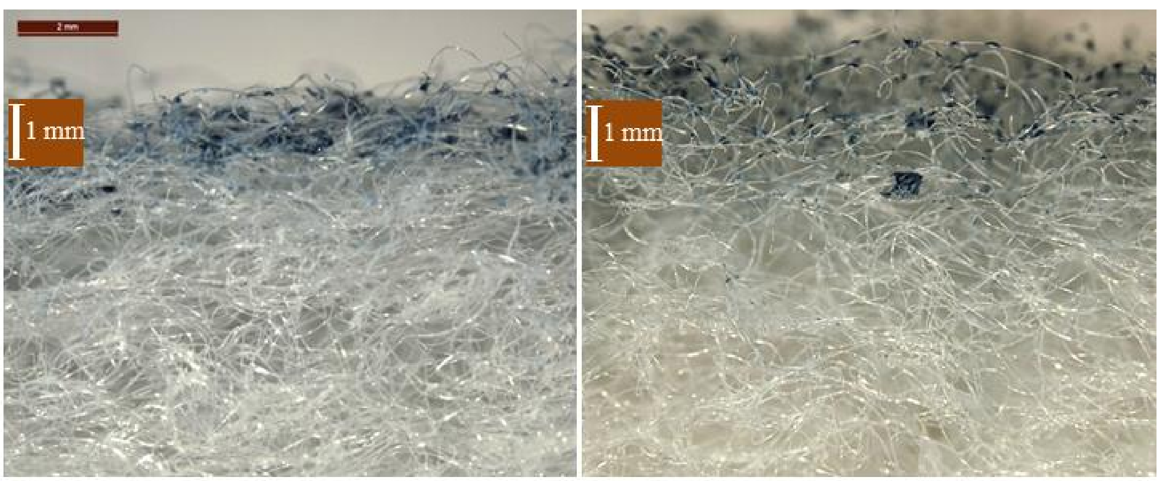


Fig. 6, Microscopic comparison of FX1 versus FX2 penetration of polyester coupon.

The tests showed that the coverage is driven to a great extent by the concentration of latex, especially if higher concentrations of glycerin or sodium lauryl sulfate are used. Glycerin greatly affected the dusty character of the encapsulated material; the greater the glycerin concentration the greater the dust encapsulation and cohesion. While glycerin was the highest factor for this criterion, latex also had a significant contribution. Finally, penetration below the superficial level was dramatically improved by the increased levels of surfactant (and hampered by higher latex paint concentrations). Results of these experiments produced an optimized fogging solution and a practical, inexpensive approach to demonstrating this technology in the field.

Florida International University Collaborative Testing

Building on the INL's practical and inexpensive approach, FIU's Applied Research Center (ARC), in collaboration with INL and Savannah River National Laboratory (SRNL), developed a fogging agent test plan designed to evaluate the operational performance of the agent against critical performance criteria and metrics. The test plan was reviewed and formally approved by all stakeholders, including FIU ARC, INL, SRNL, and DOE's Office of Environmental Management. Testing occurred in late March and early April 2015. INL provided the fogging apparatus (e.g., foggers, exhaust fan, and filter housing) and the FX2 fogging solution. FIU staff and undergraduate students in the DOE-FIU Science and Technology Workforce Development Program (a.k.a. DOE Fellows) prepared the mockup facility, executed the tests, and conducted subsequent data collection and analysis. INL personnel participated in testing and provided guidance and support in conduct of the fogging process.

The testing protocol set out to evaluate several aspects of the FX2 fogging process. The dispersion characteristics of FX2 dispensed via a commercial fogger (the Curtis Dyna-Fog Cyclone Ultra-Flex fogger) were examined. Specifically, the ability to fix loose contamination to different types of surfaces (glass, concrete, steel, plastic, and wood) and to cover locations outside the direct line-of-sight of the fogger were tested. An attempt was made to quantify the FX2 fog's ability to knock down airborne particulate; however, this aspect of testing was inconclusive.[12]

These tests were conducted by fogging a portion of the ARC hot cell. A moveable temporary wall was constructed in the hot cell to allow testing of two room volumes – 28 m^3 and 42 m^3. Two fogging units were run concurrently, injecting fog through ports in the temporary wall at approximately 3.3 m in height. Total runtime was 30 minutes for each room configuration. The exhaust fan was activated periodically for short durations to provide air movement; however, the fog generally was allowed to billow and fill the volume. Clear plastic petri dishes were used as sample coupons. The coupons were placed on shelves throughout the space. Some were empty and used to evaluate the degree of coating applied; others contained dryer lint or talcum powder to evaluate FX2's fixative ability. Coverage of horizontal and inclined surfaces in the test space was thorough. Vertical surfaces were covered to a lesser degree. Even sample coupons not in the line-of-sight of the fogging units were thoroughly coated (Fig. 7).

FIU also conducted quantitative analyses of the coating laid down by the fogging process, measuring the following attributes:

- Flammability (American Society for Testing and Materials [ASTM] D3065)
- Reactivity to flame and heat sources (during application and after dried/cured)
- Ability to shield against alpha radiation
- Adhesiveness to surfaces

- Coverage of surface area.

Fig. 7. Sample dish inside a trash can facing away from the fogging units.

The test results proved that the FX2 solution was not flammable, and was capable of permeating and coating non-line-of-sight areas within a moderate sized facility. Not only was the FX2 solution non-flammable, but it tended to suppress a flame. While it was not flammable, it also did not retard flame on a combustible surface. Some DOE customers have expressed a desire for a "fire retardant" coating, in those instances FX2 does not meet the requirements.

Development of a Mercury Abatement Fog

An outgrowth of the versatility of the robust FX2 system is the ability to add ingredients to target specific contaminants. One focus of the FX development processes has been to create a fogging solution that will reduce mercury vapor within contaminated facilities. The presence of mercury within such facilities causes significant hazards to workers and increased costs during decommissioning. Mercury is extremely toxic, causing neurological damage and other significant health hazards. Mercury contamination has been found to be a challenge in decommissioning facilities as diverse as research reactors and spent fuel reprocessing facilities, where certain coolant types contained mercury (1) to mercury in switchgear and piping (2), and in such diverse areas as facilities from Oak Ridge to Brookhaven. Traditional treatment of mercury contaminated facilities involves the total removal of the mercury. For D&D facilities, the issue is not as clear; there is little benefit in doing costly mercury remediation on facilities that are destined for disposal.

Tests on the INL mercury abatement coating, using the basic fog formula, began in 2015. Micro-sized absorbent materials were incorporated into the fog and sprayed on mercury contaminated samples. Gas samples were taken before and after the treatment. Each test ran for some 150 hours. In all cases, the reduction in mercury vapor continued to improve throughout the test, showing a good potential for long term applications. Fig. 8 shows a reduction in maximum vapor concentrations as high as 40x was achieved for three FX2 formulations (~0.5 mg/m^3), compared to the base case (~21 mg/m^3). The formulation containing Additive 1 was among the three coatings achieving a 40x maximum concentration reduction. The inset in Fig. 8 shows "FX2 Only" and "FX2+Additive 2" performed relatively well in reducing Hg evaporation, as indicated by a maximum vapor concentration of ~1.5 mg/m^3. However, greater reductions of maximum concentration were achieved when with Additive 3, which is simply a combination of Additives 1 and 2.. Additives 4 and 5 are chemically similar to Additive 2, but considerable more expensive, engineered materials. While they outperform Additive 2, their performance was not significantly different than Additive 1, which is comparable in cost to Additive 2. Therefore, this study down selected Additive 1 as the winner.

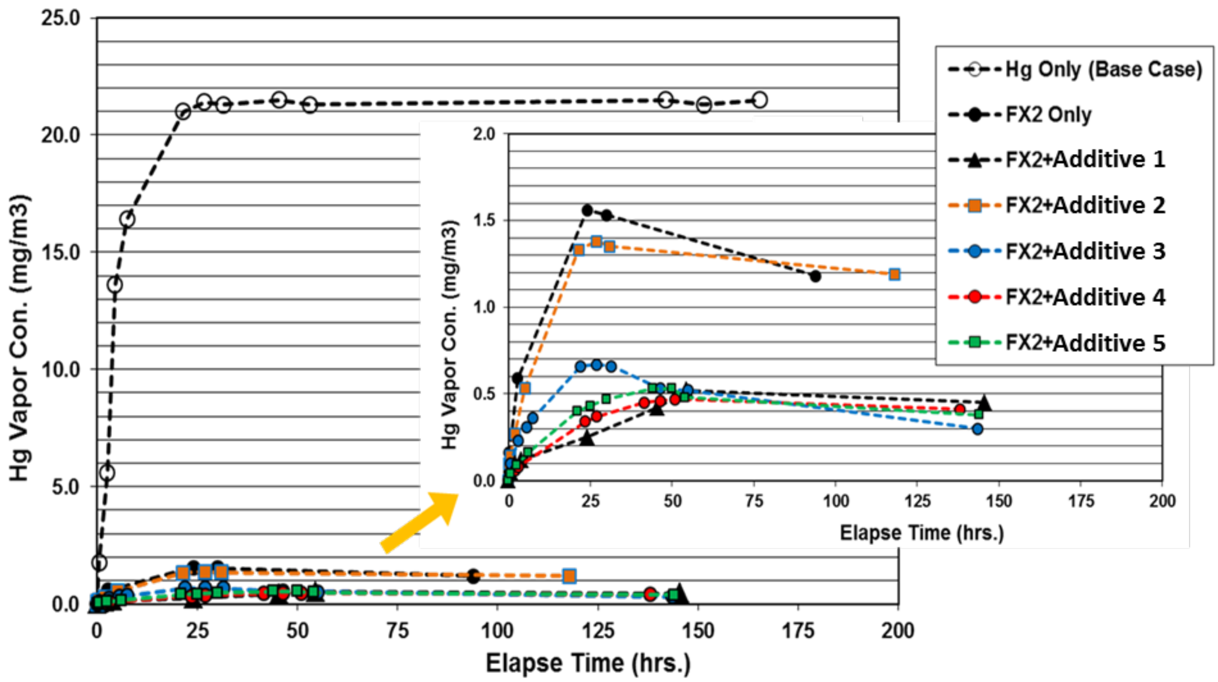
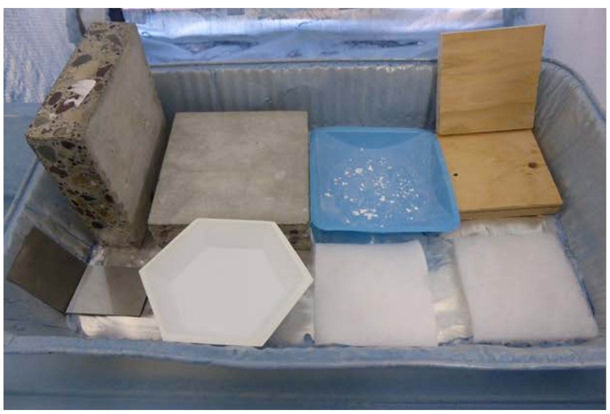


Fig. 8, Evaluation of Hg vapor reduction for various FX2 formulations, including the uncoated, Hg-only base case. The inset shows a magnification of the lower portion of the main graph, omitting the uncoated base case in order to accentuate differences between the various FX2 formulations.

Fig. 9. Red FX Hg Fog Solution in Pilot Scale Test



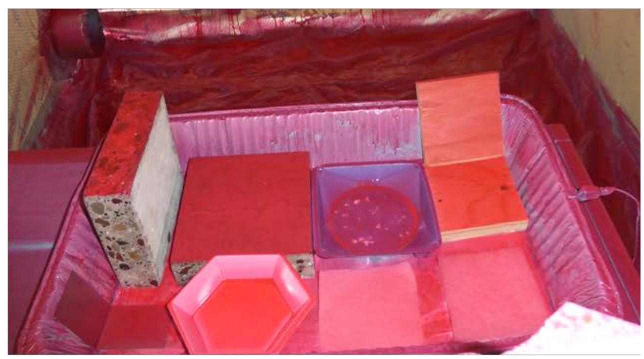


Fig. 10, Fogging Coupon Specimens Before and After Fogging

In 2016 the FX Hg solution was tested in the test tent system to determine if fogging of the solution (rather than just spraying) was appropriate. A normal fogging process was exhibited by the FX Hg during testing (Fig. 9). The only unusual observation was that there was some particulate left behind in the fogger tank afterwards. It appeared the sulfur particulate did not entirely stay in suspension in the fogger supply tank. An analysis was conducted in an effort to quantify the content of the dried coating on the test coupons (Fig. 10). However, a significant amount of active ingredient was detected in the FX Hg cured coatings than in the baseline FX2. Based on this result, combined with the success of the bench scale tests using actual mercury, FX Hg appears to be a viable approach for mercury vapor abatement – via either localized application with a spray bottle or larger scale application with a commercial fogging unit.

Glycerin-free FX2 formulation

The recent concerns about the use of the glycerin (present in most fogging solutions), particularly glycerin's unwanted reaction with nitrate salts left over from processing, prompted the testing of a glycerin free FX2 product. A bench scale experiment was performed in 2016 to determine if a glycerin free formulation of FX2 could satisfy the basic criteria of a fixative application. For this experiment, standard FX2 and FX2 Glycerin-Free (FX2 GF) were sprayed onto coupons with a hand sprayer. The sprayers did not produce an ideal spray pattern. As a result the solutions were dispensed in more of a splatter than a fine mist. Coupons coated included galvanized iron, wood, concrete, smooth plastic, talcum powder, and two densities of polyester batting.

The test proved to be a success. After drying overnight, the glycerin-free coating was dryer and less slick to the touch. The FX2 GF coating was observed to coat uniformly in most cases (though spray patterns don't do it justice on many substrates), with coating characteristics and penetration depth similar to standard FX2. In some cases, as in Fig. 11, the FX2 GF looked to be a bit superior in coverage and depth of penetration.

Figure 11. FX2 GF (left) and standard FX2 (right) coating polyester coupons.

Radioactive Demonstration Tests

While significant efforts have focused on perfecting the fogging process, complimentary actions have focused on fielding a radiological demonstration. Though close on several occasions, no full U.S. facility demonstration has yet been realized. However, another collaborative test with the UK's National Nuclear Laboratory is underway. This test will be an active (i.e., radioactive) test. The test will involve fogging a section of a stainless steel filter (pictured below in Fig. 13) previously used in the MOX demonstration facility at NNL Preston Laboratory. The filter is contaminated with ~200g UO_2.

Fig. 13, Stainless steel filter representative of the type to be fogged at NNL's Preston facility.

Due to some issues with contract terms, work under this contract did not commenced until early July. Work is scheduled to be completed by the end of the calendar year. Thus far, the preliminary planning, safety case documentation, and other enabling activities have been completed, including:

- Safety assessment based on the MSDS for FX2 reagent;
- Plant Modification Proposal (Integrated Risk Assessment) (50% complete);
- The FX2 agent has been received from INL intact at NNL Workington and a viscosity measurement check showed the viscosity of a sample from one of the bottles of FX2 at 20.0°C (at a constant shear rate of 30.0 s^{-1}) was within the expected range at 10.2 cP, indicating that the solution did not freeze during shipping;
- Two Dyna-Fog Cyclone units have been procured and received at NNL Preston Lab;
- Quotes and delivery lead times for glove bag containment options have been processed.

Shakedown (non-radioactive) testing will begin in November 2016. Active trials with the UO_2 contaminated pieces are scheduled to commence in early December. Reporting of final results back to INL should be complete by the end of the calendar year.

Non-active (i.e., cold) trials will be carried out to provide information on fogging and curing times, along with coverage of the coating. The cold trials will also provide operator experience with the process and allow any necessary equipment modifications or process optimization to be accomplished in a clean environment. A fogging enclosure similar to that used in INL's 2014 tests (see in Fig. 4) will be constructed, although the footprint of the enclosure may be smaller. The enclosure will be coupled to a Curtis Dyna-Fog Cyclone Ultra Flex Fogger and an exhaust fan.

Upon completion of shakedown testing, the equipment will be disassembled and then reassembled in the NNL Preston Laboratory Total Enclosure. Following initial cleaning of the filter unit to remove gross uranium dioxide powder contamination, a ~150 mm cross-sectional test piece will be sawn from one end. The test piece will then be monitored for loose and fixed contamination, and dose rate measurements will be taken. Subsequently, the test piece will be transferred to the fogging enclosure and the active trial carried out. Photographs will also be taken to show the extent of outer and inner surface coverage by the FX2 agent. Similarly, dose rate and contamination readings will be carried out, post fogging, to compare against the earlier readings.

CONCLUSIONS

Over the past 10 years a new type of fixative coating has been developed and tested to mitigate several decommissioning concerns. The INL FX2 coating has been proven non-flammable, able to affix simulated contamination and able to flow through unusual geometries (via independent testing at Florida International University). A series of non-active fogging trials for activity knock/tie-down application at NNL Workington on behalf of Idaho National Laboratory (INL) have shown steadily improving large-scale fogging characteristics. The INL has also manipulated the basic formulation into a mercury vapor reducing form of the coating, termed FX-Hg, and another form that reduces glycerin concerns, both of which have shown great promise in laboratory studies

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