TESTING OF POLYMERIC HYDROGELS FOR RADIATION DECONTAMINATION FOR THE DEPARTMENT OF ENERGY: FINAL REPORT — PROJECT NARRATIVE

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This collaborative effort between the Department of Energy, Office of Environmental Management (DOE EM) and CBI Polymers, Inc. focused on testing/demonstrating a unique polymer hydrogel and modifying the product's performance to meet specific DOE goals to drive down overall decontamination and waste remediation costs.

Saving money through more efficient use of technology

DISCLAIMER

Approximate Conversions from Metric to U.S. Customary Measures

Length

Symbol	When You Know	Multiply By	To Find	Symbol
mm	millimeters	0.04	inches	in
cm	centimeters	0.4	inches	in
m	meters	3.3	feet	ft
m	meters	1.1	yards	yd
km	kilometers	0.6	miles	mi

Area

Symbol	When You Know	Multiply By	To Find	Symbol
cm^2	square centimeters	0.16	square inches	in^2
m^2	square meters	1.2	square yards	yd^2
km^2	square kilometers	0.4	square miles	mi^2
ha	Hectares (10,000 m^2)	2.5	acres	acs

Mass (weight)

Symbol	When You Know	Multiply By	To Find	Symbol
g	grams	0.035	ounces	oz
kg	kilograms	2.2	pounds	lb
t	metric ton (1,000 kg)	1.1	short tons	

VOLUME

Symbol	When You Know	Multiply By	To Find	Symbol
mL	milliliters	0.03	fluid ounces	fl oz
mL	milliliters	0.06	cubic inches	in^3
L	liters	2.1	pints	pt
L	liters	1.06	quarts	qt
L	liters	0.26	gallons	gal
m^3	cubic meters	35	cubic feet	ft^3
m^3	cubic meters	1.3	cubic yards	yd^3

TEMPERATURE (EXACT)

Symbol	When You Know	Multiply By	To Find	Symbol
°C	degrees Celsius	9/5, add 32	degrees Fahrenheit	°F
K	Kelvin	subtract 273.15	degrees Celsius	°C

ABSTRACT

This collaborative effort between the United States Department of Energy, Office of Environmental Management (DOE EM) and CBI Polymers, Inc. (CBIP) ascertained whether a unique, commercial hydrogel product, (DeconGel™) could be modified and/or improved to meet specific DOE goals. The product was modified and tested under various scenarios, to include field testing, to determine if the formulation changes improved efficacy and allowed wider use of the product in deactivation and decommissioning (D&D), and specifically decontamination activities. A major cost driver for the government is the decontamination of infrastructure and equipment, and the subsequent disposal of hazardous waste. DOE objectives achieved under this contract were to improve the product's efficacy on radioisotopes, improve its safety profile (i.e., flammability and toxicity), develop and validate commercial scale manufacturing processes, and to validate the product's efficacy and utility in field test evaluations. The end result was the validation of DeconGel technology as a viable and efficacious decontamination tool available to decontamination solution providers worldwide. The newly improved products were tested at DOE facilities, the Environmental Protection Agency (EPA), and the US military with great success in meeting DOE's D&D goals. Subsequent use in the immediate aftermath of the Fukushima Daiichi nuclear incident in Japan further validated the ability and efficacy of the DeconGel formulations. Further information can be found at the vendor's website at http://www.decongel.com.

ACKNOWLEDGEMENT

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ABBREVIATIONS

AAS	Atomic Absorption Spectroscopy
AATD	Accelerated Age Time Duration
AAR	Accelerated Aging Rate
ACGIH	American Conference of Governmental Industrial Hygienists
AVG	Average
BWR	Boiling Water Reactor
CAS	Chemical Abstracts Service
CBD	Chronic Beryllium Disease
CPM	Centipoise Per Minute
D&D	Deactivation and Decommissioning
DD&R	Decommissioning, Decontamination & Reutilization
DE	Decontamination Efficacy (% removal of original activity of the surface)
DF	Decontamination Factor (DF = original activity / final activity)
DI	Deionized Water
DMSO	Dimethyl Sulfoxide
DOE EM	United States Department of Energy, Office of Environmental Management
EtOH	Ethyl Alcohol
FLAA	Flame Atomic Absorption Analysis
FTS	Foam Testing System
GE	General Electric
GC-MS	Gas Chromatography-Mass Spectroscopy
HSRC	Hazardous Substance Research Center
ICP-OES	Inductively Coupled Plasma - Optical Emission Spectrometry
kWh	Kilowatt Hour
LC-MS	Liquid Chromatography-Mass Spectroscopy
LOD	Limit of Detection
μCi	MicroCurie
mCi	MilliCurie
μg	Micrograms
MPa	Mega Pascals
nCi	NanoCurie
N	Newtons
N/D	None Detected
NFPA	National Fire Protection Association
NIOSH	National Institute for Occupational Safety and Health
NMP	N-Methylpyrrolidone
OSHA	Occupational Safety Health Administration
PHNSY&IMF	Pearl Harbor Naval Shipyard and Intermediate Maintenance Facility

PCBs	Polychlorinated Biphenyls
PLM	Polarized Light Microscopy
PPH	Parts Per Hundred
PPMV	Parts Per Million by Volume
PPM	Parts Per Million
PPT	Parts Per Trillion
%R	% Removal (% removal of original radioactivity on a surface)
RT	Room Temperature
SD	Standard Deviation
TEM	Transmission Electron Microscopy
TICs	Toxic Industrial Chemicals
TIMs	Toxic Industrial Materials
TRU	Transuranic
TS	Thermospray
TTEP	Technology Testing and Evaluation Program
TWA	Time Weighted Average
VOCs	Volatile Organic Compounds
WAC	Waste Acceptance Criteria
WIPP	Waste Isolation Pilot Plant

EXECUTIVE SUMMARY

This collaborative effort between the United States Department of Energy, Office of Environmental Management (DOE EM) and CBI Polymers, Inc. (CBIP), resulted in the development, improvement and commercialization of a multi-purpose (radiological, chemical and certain biological materials [mold]) peelable decontamination hydrogel (DeconGel™). In government field testing and actual commercial use it effectively decreased the time and costs associated with decontamination and waste disposal. When used in certain scenarios, the improved hydrogel can substantially reduce total costs for government and commercial sector customers involved in deactivation and decommissioning (D&D) and specifically decontamination activities.

CBIP improved the efficacy (% Decontamination and % removal of original radioactivity on a surface) of DeconGel on radioisotopes and hydrophobic and hydrophilic contaminants. They also improved the product's safety profile, resulting in a non-flammable and non-hazardous product as defined by the Occupational Safety Health Administration (OSHA) and the National Fire Protection Association Flammable and Combustible Liquids Code 30 (NFPA 30). Additionally, a commercial scale manufacturing process was developed and validated at the 2000 gallon scale for the new DeconGel product formulas. The newly improved products were tested at DOE facilities, the Environmental Protection Agency (EPA), and with the US military. The successful tests resulted in acceptance of DeconGel products as a viable, and on a case-by-case basis, a preferred decontamination agent for use at DOE and other facilities. The technology was tested in the laboratory against non-radioactive materials including beryllium surrogates, mercury, lead, cadmium, chromium, asbestos, and mold. Laboratory analysis was followed by field testing at 100KE Basin, Richland, Washington; Y-12 National Security Complex; EPA testing on cesium and americium; polychlorinated biphenyl (PCB) decontamination aboard the USS Missouri (BB-63) and finally, lead dust decontamination at a Pearl Harbor Naval Shipyard and Intermediate Maintenance Facility (PHNSY&IMF) foundry.

Near the end of the project in a twist of fate, the earthquake/tsunami of March 2011 in Japan resulted in the meltdown of the Fukushima-Daiichi Nuclear Power Plant. Subsequent urban contamination (extending several hundred miles from the stricken plant) led the Japanese government to conduct an exhaustive review of decontamination technologies. The result of that review and testing was selection of DeconGel 1108 and 1128 as approved decontamination products for use in Japan. DeconGel was expedited to Japan where it is currently being used by civilian first responders, Tokyo Electric Power Company, and the Japanese Self Defense Forces.

Achievements of this collaborative effort include:

- Improved DeconGel efficacy over the previous formulation of DeconGel by 49% in the nondestructive decontamination of acidic solutions of Cesium-137 from bare concrete (% Decontamination of 67% vs. 45% for previous formulations) in testing performed by the EPA.
- DeconGel out-performed Bartlett's Stripcoat TLC Free™ by 83% in the nondestructive decontamination of acidic solutions of Americium-243 from bare concrete (%Decontamination of 84% vs. 46% for Stripcoat TLC Free) in testing performed by the EPA. Bartlett Stripcoat TLC Free is a popular product in D&D operations throughout the world.
- Demonstrated DeconGel application and efficacy at DOE facilities, the EPA, and the US military.
- The safety profile (i.e. flammability and toxicity) of the improved products as defined by OSHA and NFPA resulting in non-flammable and non-hazardous products with an improved odor (mild citrus).
- The development and validation of a commercial scale manufacturing process at the 2000 gallon scale.
- Demonstrated to effectively decontaminate a variety of porous and nonporous surfaces contaminated with toxic industrial chemicals and materials, as well as radioisotopes.

Based on the performance of the improved products, the authors recommend that contractors provide independent test results from head-to-head product comparisons to prove that the most cost effective decontamination method is being used to meet DOE D&D goals. This testing would be done as part of the standard contractual process before a decontamination effort was approved or funded. The reasoning is that the government must be assured that the most cost effective solution (from a total cost perspective including product, labor, waste disposal, and regulatory reporting requirements) is being presented.

1 INTRODUCTION

The mission of the DOE Office of Environmental Management (DOE EM) is the safe cleanup of the environment after five decades of nuclear weapons development and government-sponsored nuclear energy research. The EM program has made significant progress in shifting away from risk management to embracing a mission completion philosophy. This includes deactivation and decommissioning (D&D) facilities that no longer have utility, thus reducing long-term liabilities and maximizing resources for cleanup. To accomplish these objectives, EM identifies innovative technologies and ideas, and measures how well they contribute to the EM mission.

DOE EM tasked CBI Polymers (CBIP) to conduct research and development on its commercial hydrogel with the aim of improving its decontamination capabilities against radioactive isotopes, transuranic (TRU) isotopes, heavy metals (e.g., cesium, cobalt, etc.), and radioactive and toxic industrial chemicals (TICs) and materials (TIMs) of concern in DOE facilities.

While many decontamination technologies have been initiated by DOE research, relatively few have advanced into the commercial marketplace. Without commercial marketability, the ability of the government to leverage market forces to drive down costs or to keep a successful product viable and available can be severely curtailed. Therefore, as part of this effort, DOE required development of product marketing materials to include education of end-users, development of end-user awareness, and training materials (written or electronic). CBIP also attended industry meetings, provided support for conferences and symposia (e.g., research papers and talks as directed by the government), and engaged in market research to determine if an improved product would have market sustainability.

1.1 Project Background

In 2006 CBIP discovered a remarkable characteristic of polymer hydrogels. When a hydrogel is combined with a specialized blend of chelators, thixotropes, surfactants, biocides, de-foaming agents, and other proprietary ingredients, it has the ability to bind heavy metal ions and other materials in a semi-crystalline polymer matrix. When dry, the polymer matrix with its encapsulated contaminants could be peeled away from hard substrates (concrete, wood, metal, plastic, rubber, etc.) leaving a surface effectively free of loose contamination. The technology was named “DeconGel” and was patented, manufactured and commercialized, and is currently sold world-wide.

DeconGel came to the attention of DOE EM via congressional interest in discovering more efficient ways of managing the nation’s cleanup of the environment due to nuclear weapons development and government-sponsored nuclear energy research. DOE EM’s mission philosophy focuses on cleanup completion and securing effective results while mitigating risk. With safety being a central concern, the department expressed a keen interest in technologies that can deliver superior decontamination results via a safe-to-use application and deployment process.

The current state of technology for decontamination products include soap and water, foaming technologies, latex and polymer based strippable coatings, and even corrosive acids that are designed to dissolve substrate surfaces. The latter can damage and/or destroy infrastructure or equipment being decontaminated. Foaming or soap and water technologies show dubious efficacy, are labor intensive, produce massive amounts of secondary waste, and can spread contaminants over a larger area. Fixative products (durable paint-like coatings that cover the contamination thus preventing its removal) can prevent or reduce the spread of contamination during D&D operations; however, some fixatives also prevent or increase the difficulty and expense of subsequent decontamination and add to the total weight of the contaminated waste, resulting in higher disposal costs. These aforementioned products, though effective in select situations, are often difficult to use, may contain highly corrosive ingredients, are toxic, require mixing, or require specialized equipment. Some produce significant amounts of secondary waste thus increasing remediation, storage and disposal costs. This in turn, increases labor costs and extends the logistics tail for decontamination and mitigation operations.

The original DeconGel family of products (DeconGel 1101, 1120, 1121) originally demonstrated many characteristics that DOE EM desired:

- Safe-to-use and handle
- Non-corrosive and non-destructive to surfaces
- Single component (no mixing)
- Rehydratable (allowing forensic analysis)
- Could immediately fix[1] contaminants in-place preventing spread
- A five-year shelf life with freeze/thaw stability
- Effective in the decontamination of radioactive isotopes, TICs and TIMs

Because the product was designed specifically for surface decontamination, DOE EM's goal was to improve upon the product's decontamination effectiveness on radioisotopes, heavy metals, hydrophobic contaminants; improve the technology's safety profile, and demonstrate effectiveness of the products via a variety of field tests.

1.2 Project Concept & Scope

In collaboration with DOE EM, the project was structured around 11 tasks designed with commercialization strategies in mind (Table 1). This ensured the products would have broad-use capabilities applicable to both government and civilian needs. The authors felt the

[1] It is important to note that while DeconGel can effectively "fix" contaminants, it was neither designed nor tested as a fixative.

marketing discussion detracted from the overall technical data overview and thus has been omitted from this report. Specific technical data pertaining to laboratory work and testing is available in the Appendix.

Table 1 – Project Tasks and Status.

Milestone / Deliverable	Completion Date	Status
Task 1. In a collaborative effort with DoE, perform field testing/product demonstrations.	May 2010	✓ Completed
Task 2. Evaluate, test, and document the suitability of DeconGel 1101, 1120, 1121 and second generation products for multiple disposal options	September 2010	✓ Completed
Task 3. Complete the development of improved second generation products	March 2010	✓ Completed
Task 4a. Perform in-house laboratory evaluations - Decontamination of toxic non-radioactive chemicals/compounds (and surrogates)	May 2010	✓ Completed
Task 4b. Perform in-house laboratory evaluations - Simultaneous decontamination of radioactive contaminants and grease/oil	July 2010	✓ Completed
Task 4c. Perform in-house laboratory evaluations - Simultaneous decontamination of radioactive contaminants and mold	September 2010	✓ Completed
Task 5. Develop dual-source manufacturing capability for DeconGel	March 2010	✓ Completed
Task 6. Develop and approve the commercial production process for second generation DeconGel products	June 2010	✓ Completed

Task 7. Perform analyses and obtain certifications important to DOE end-users	September 2010	✓	Completed
Task 8. Evaluate efficacy of DeconGel™ in the decontamination of Cesium-137 through the EPA's TTEP	March 2010	✓	Completed
Task 9. Support the evaluation, demonstration, and dissemination of training materials resulting in the inclusion of DeconGel in the D&D Toolbox	September 2010	✓	Completed
Task 10. Perform market research to learn end-user needs	September 2010	✓	Completed
Task 11. Educational, support and awareness activities for DOE and DOE approved sites	September 2010	✓	Completed

The scope of the project included research and development, scale-up, demonstrations, and deployment of an improved, effective, safe product aimed at the decontamination of radioactive (including TRU) isotopes, heavy metal isotopes, heavy metals, and TICs & TIMs of concern in DOE facilities. Additionally, the team examined end-user requirements, educated end-users via trade shows and conferences, and produced end-user training materials.

1.3 Benefits of the Project

This project resulted in several noteworthy accomplishments that are described in this document. Most notably, the DeconGel family of products was improved and demonstrated increased decontamination effectiveness against radioisotopes, TICs, and TIMs, including hydrophobic contaminants. These results were achieved while improving the environmental aspects of the technology by making the product even safer (OSHA non-hazardous and non-flammable) to use and handle. Additionally, market research demonstrated the viability of the technology in the marketplace and how it could reduce total costs. The developed products are environmentally friendly and reduce secondary waste stream generation, and the amount of labor required for decontamination efforts. The overall result was improvement of a technology that has successfully transitioned to the marketplace with dual-use capabilities for the D&D and commercial industrial landscapes.

The importance of the benefits derived could not be illustrated any better than the actions taken in response to the hysteria that occurred in Japan in the aftermath of the March 2011 disaster. The Japanese government was compelled to find readily available decontamination technologies with broad spectrum decontamination capabilities, and safe enough for potential public use. Because of this DOE EM/CBIP effort, the newly improved DeconGel products were available to Japan for testing and subsequently approved for use by the government.

2 BASELINE TECHNOLOGY DESCRIPTION

DeconGel was originally designed to meet the requirements for aircraft decontamination from radioisotopes collected during airborne sampling missions. Three formulas were developed: DeconGel 1101, a brush-on application for all hard surfaces; DeconGel 1120, a spray version designed for hard, smooth, non-porous surfaces; and DeconGel 1121, a spray version of 1101 designed for all hard vertical and horizontal surfaces.

DeconGel was designed to be a water-soluble and environmentally friendly product for decontamination of radioactive contaminants, TICs and TIMs from hard surfaces. This unique polymer hydrogel attracts and mechanically locks radionuclides into a semi-crystalline polymer matrix and safely removes them from substrates. The hydrogel coating can be applied to horizontal, vertical, inverted and complex surfaces, and can be applied to most hard substrates; for example, bare, coated and painted concrete, aluminum, steel, lead, rubber, Plexiglas™, herculite, wood, porcelain, tile grout, and vinyl, ceramic, and linoleum floor tiles. When dry, the film containing the encapsulated contamination can be peeled and disposed of according to appropriate local, state, and federal regulations.

DeconGel main characteristics include:

- Easy, rapid application and removal (apply, dry, and peel)
- Short term fixation[2] of surface contaminants
- Reduction of surface contamination
- No ablation of underlying surfaces, non-corrosive
- No water required in application
- Minimal secondary waste generated, with no runoff
- OSHA defined non-hazardous, safe-to-use, and OSHA/FRPA defined non-flammable
- Five-year shelf life under normal storage conditions (sub-100^{o}F)

DeconGel was a novel technology and generally superior compared to other strippable technologies because of its water-soluble polymer-based foundation and proprietary blend of ingredients. These characteristics enable it to penetrate deep into substrates for greater efficacy, and allow it to be easily peeled from a surface without tearing. DOE EM believed that there were improvements that could be made to the technology to facilitate greater acceptance within the D&D community, especially since there had been relatively little recent advancement in other decontamination technologies.

3 Summary of Technology Improvements

Successive improvements developed during the DOE EM/CBIP collaboration include:

- Two improved second generation DeconGel formulations (1108 and 1128) that have been scaled to commercial production. These products were shipped to Japan for use in urban decontamination efforts.

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[2] Although short term fixation has been observed, DeconGel has not been officially evaluated by DOE EM as a long-term fixative.

- A formulation (DeconGel 1102) that has increased affinity for oil, grease, and hydrophobic toxic chemicals such as PCBs. This product has been embraced by the US Navy for use in surface preparation (cleaning) of tanks and voids prior to surface blasting with grit.

- A prototype formulation with low impurities for Boiling Water Reactors (BWR).

- Modifications of DeconGel 1108 and 1128 into fast-dry formulations (DeconGel 1108FD and 1128FD)[3] with substantially reduced drying time. This characteristic was specifically requested by the Japanese and the product is currently under evaluation in Japan for use in cold and humid environments.

Components incorporated in these second generation products (DeconGel 1108 and 1128) were selected for improved efficacy against radioisotopes commonly found in DOE D&D operations. In addition to improved efficacy, improvements in the toxicological and flammability profiles resulted in OSHA non-hazardous and non-flammable designations. NFPA 30 uses the OSHA definitions for flammable and non-flammable, therefore DeconGel meets NFPA requirements as a non-flammable liquid. The application characteristics of the final formulations were improved demonstrating decreased foam generation during spray application and improved sag resistance. These improved second generation products have a pleasant mild citrus odor, are non-flammable, freeze-thaw stable,

[3] The fast-dry formulations were experimental batches unique to the emergency requirements of the Fukushima Daiichi Nuclear Power Plant, whose flooded interior resulted in exceptionally high (100%) humidity levels and could not facilitate the normal drying needs for DeconGel. Decontamination efforts also had to take place in below freezing conditions. Although the flammability and toxicological profiles have been altered (due to the high concentration of ethanol), both of these formulations show 30-50% faster drying times relative to the original (DeconGel 1101; 1121) and the second generation (DeconGel 1108; 1128) products. The fast-dry formulations are not commercially available.

and are OSHA non-hazardous.[4] Detailed descriptions of the development, components incorporated, and evaluations performed that led to the development of these improved DeconGel formulations are provided in the Appendix I. under Technical Details.

The DeconGel intellectual property improvements can be summarized as follows:

- Decontamination Efficacy Improvements
    - Two new products (DeconGel 1108 and 1128) were developed.
    - An improved sequestering agent has been developed (among other improvements) to improve efficacy on radioisotopes and heavy metals.

- Reduced Foam Generation
    - Second generation products show reduced foam generation and fast foam decay, improving the peel performance of the dry film.

- Improved Safety and Flammability Profiles
    - Lower flammability and higher flashpoints were achieved through the partial replacement of highly volatile and flammable solvents with less flammable solvents resulting in a DOT rating for unrestricted shipping and OSHA/NFPA designation as non-hazardous products (DeconGel 1108 and 1128).

- Reduced Toxicity (DeconGel 1108 and 1128)
    - Concentrations of hazardous components have been reduced to less than 1wt%. These improvements resulted in the development of low toxicity, OSHA non-hazardous formulations (DeconGel 1108 and 1128).

4 A flash point >200°F (93°C) is defined as non-flammable.

- Similar Mechanical Properties

    - The mechanical properties (most importantly tear resistance) of the dried gels for DeconGel 1108 and 1128 were retained.

- Rheological Properties

    - The viscosity, thixotropic behavior and sag resistance of these improved formulations were statistically equal to or better than DeconGel 1101 and 1121.

- Shelf-Life

    - These formulations are freeze-thaw stable and have a five year shelf life based upon accelerated aging tests.

- Improved Odor

    - These improved second generation formulations have a light, pleasant citrus smell.

- Improved Decontamination Efficacy for Hydrophobic Toxic Chemicals

    - DeconGel 1102, a new product formulated specifically for the improved affinity towards hydrophobic organic toxic chemicals, met Environmental Protection Agency PCB limit standards for unrestricted public access aboard the USS Missouri (BB-63).[5]

[5] Regulations 761.79, 761.123 of ≤ 10 μg/100 cm^2

- Reduced Dry Time (pre-commercialization)

    - ➢ Modified products demonstrating significantly faster drying times have been developed for use in cold and/or humid outdoor areas (DeconGel 1108FD; 1128FD).

Scaling to commercial production for 1102, 1108, and 1128, along with the approval of a second toll manufacturer, Capital Resins, Inc. (Columbus, OH), was successfully completed. Approval of a second toll manufacturer ensures an uninterrupted supply of DeconGel products to DOE, Japan and other industrial marketplace users. At the onset of the effort, three 800-gallon batches each of DeconGel 1101 and 1121 were successfully produced, within specification, at this new toll manufacturer. Subsequently, a single 800-gallon batch of 1102 was produced within specification at this same toll manufacture. Finally, two 2000-gallon batches each of DeconGel 1108 and 1128 were produced within specification to prove that the product and technology could be successfully scaled to commercial manufacturing volumes without loss of quality or product effectiveness. DOE EM and CBIP were aware that without scale-up testing data there was no valid method to ascertain whether or not laboratory produced products could be scaled to commercial marketability.

Testing of the product resulted in its approval for disposal by Energy Solutions as low level waste in the Clive, Utah, and Barnwell, South Carolina facilities, and for incineration at the Bear Creek facility in Tennessee. In a report given by Savannah River National Laboratory (SRNL) on August 31, 2010 titled, "Technical Basis for Approval of DeconGel as an Acceptable Component of WIPP Waste Streams," field demonstrations indicate DeconGel was efficient at removing loose and fixed TRU contamination, but some proprietary ingredients produce VOCs that are considered flammable gases. The Waste Isolation Pilot Plant (WIPP) TRAMPAC (Transuranic Waste Authorized Methods for Payload Control) requirements for flammable VOCs in waste container headspace dictate that VOC's must be

< 500 ppmv. Through laboratory testing, SRNL determined that cured DeconGel will not emit flammable VOCs at levels that exceed the headspace criterion of < 500 ppmv. VOC headspace concentrations were noted to remain at near steady-state levels after a container hold time of 42 days. The sum of the maximum value for each flammable VOC detected in DeconGel was 108 ppmv which is 22% of the 500 ppmv limit. Therefore, DeconGel was determined to be an acceptable component of the WIPP waste stream when the encapsulated contaminants are below the WIPP WAC. This information was presented at the ANS DD&R 2010 Conference in Idaho Falls, ID and the finding allows DeconGel to be stored in large containers for transportation to WIPP, and storage/disposal of generated waste at WIPP.

The remaining sections briefly describe evaluations performed on the new formulations and the test methods used to arrive at the efficacies presented. Additional information is presented in the Appendix.

4 LABORATORY EVALUATIONS OF DECONGEL TECHNOLOGIES ON TOXIC CHEMICALS AND MOLD

Formulation efficacy on a variety of non-radioactive toxic and hazardous contaminants was performed for this project. DeconGel products (1101 and 1102) were evaluated (as noted in the following chart) for their efficacy to remove toxic and hazardous non-radioactive chemicals and materials from substrates of importance to the DOE. Evaluations were performed on a variety of substrates including industrial grade concrete, aluminum, stainless steel, carbon steel, linoleum tile, glass, and painted surfaces. Decontamination efficacy values were determined primarily via residue swipe analysis (comparison of residue swipes before and after decontamination). Swipe sampling methods are used in most industrial standard practices for the evaluation of the contamination (powder, dust or liquid) levels on a substrate. Except for asbestos analysis, which was performed at an independent lab, all other analysis was performed at CBIP.

Table 2 – Decontamination Efficacy of DeconGel products on non-radioactive TICs (or surrogates) of importance to DOE (see notes at bottom of chart for specific products).

Contaminant	Substrate Evaluated	% Decontamination via Swipe[1]/Extraction[2]/ Vapor Analysis[3] Method
PCBs	Stainless Steel[1]	92.4 ± 0.10[4]/ 94.1 ± 0.10[5]
		100*[6]/100*[7]
	Aluminum[1]	92.4 ± 0.10[4]/
		94.1 ± 0.10[5]
		100*[6]/100*[7]
	Concrete[1]	83.9 ± 0.18[4,8]/
		87.2 ± 0.11[5,8]
		100*[6,8]/100*[7,8]
		98.6 ± 0.10[4,9]/
		98.8 ± 0.15[5,9]
		100*[6,9]/100*[7,9]
		98.8 ± 0.10[4,10]/
		99.0 ± 0.12[5,10]
		100*[6,10]/100*[7,10]
Mercury	**Elemental Mercury**	
	Stainless Steel[3]	98.4 ± 0.7[11]
	Concrete[3]	97.8 ± 0.7[11]
	Porcelain Tile[3],**	99.2 ± 0.4[11]
		85.9 ± 11.7[12]
	Composite Tile[3],**	91.7 ± 2.1[11]

		66.0 ± 11.7[12]
	Linoleum Tile[3],**	95.4 ± 0.9[11]
		90.8 ± 5.8[12]
		99.0 ± 0.2[13]
Lead Compounds	**Lead (II) Oxide**	
	Stainless Steel[1],**	91.5 ± 5.0
	Aluminum[1],**	81.2 ± 7.4
	Concrete[1],**	91.2 ± 8.7
	Lead (II) Sulfate	
	Stainless Steel[1]	99.4 ± 3.5
	Aluminum[1]	99.8 ± 8.9
	Concrete[1]	99.8 ± 7.6
Asbestos	**Chrysotile Fibers**	
	Linoleum Tile	100*,[14]
		99.9715
	Painted Drywall	100*,[14]
	Concrete	100*,[15]
Chromium Compounds	**Potassium Chromate**	
	Stainless Steel[1]	99.6 ± 5.9
	Carbon Steel[1]	99.2 ± 9.6
	Concrete[1],**	91.9 ± 3.5
Beryllium Compounds	**Aluminum powder[7]**	
	Stainless Steel	97.9 ± 2.0**
	Carbon Steel	99.5 ± 4.6**
	Concrete	94.0 ± 2.8
	Aluminum Oxide[7]	
	Stainless Steel	99.7 ± 7.7
	Carbon Steel	99.9 ± 14.4
	Concrete	99.7 ± 5.9

Cadmium Compounds	**Cadmium Chloride**	
	Stainless Steel	99.5 ± 1.7
	Aluminum	N/A[8]
	Carbon Steel	98.5 ± 4.2
	Concrete	97.6 ± 2.2
	Cadmium Oxide	
	Stainless Steel	100.0 ± 0.2
	Carbon Steel	100.0 ± 0.4
	Concrete	98.4 ± 0.2

Note: Except asbestos analysis, which was conducted by an independent lab –EMSL Analytical- all analysis was conducted by CBIP research scientists.

* Contaminant residue amount below limit of detection (LOD).

** Contaminant reacted with substrate and/or with trace amounts of water, or absorbed into substrate, creating a fixed residue on/within the contaminated surface that could not be completely removed by DeconGel; as evidenced by a lower than expected Decontamination Efficacy (if applicable).

[1] Swipe method of analysis: use of swipe testing.

[2] Extraction method of analysis: use of solvent extraction.

[3] Vapor analysis method of analysis: use of mercury vapor analyzer.

[4] DeconGel 1101 non-brushed (poured) onto contaminated surface.

[5] DeconGel 1102 non-brushed (poured) onto contaminated surface.

[6] DeconGel 1101 brushed onto contaminated surface.

[7] DeconGel 1102 brushed onto contaminated surface.

[8] Standard 24 h decontamination protocol utilized.

[9] Second DeconGel application (3 days/72 h after 1st application).

[10] Second DeconGel application (7 days/168 h after 1st application).

[11] Contaminant administration controlled using pipet-aided removal of bulk mercury liquid to yield contaminant as very small liquid beads (<1 mm diameter); DeconGel was poured onto contaminated surface.

[12] Contaminant administration controlled using zinc dust-assisted removal of bulk mercury liquid to yield contaminant as very small liquid beads (<1 mm diameter); DeconGel was poured onto contaminated surface.

[13] Contaminant administration controlled using zinc dust-assisted removal of bulk mercury liquid to yield contaminant as very small liquid beads (<1 mm diameter); DeconGel was brushed onto contaminated surface.

[14] Decontamination Efficacy determined by tape lift adhesion sampling, polarized light microscopy analysis method.

[15] Decontamination Efficacy determined by swipe sampling, transmission electron microscopy analysis method.

4.1 Analytical Methods

The analytical methods used to determine the decontamination efficacy of DeconGel products in the preceding table included the following:

- Gas Chromatography-Mass Spectrometry (GC-MS)[6]
- Liquid Chromatography-Mass Spectrometry (LC-MS)[7]
- Inductively Coupled Plasma-Optical Emission Spectroscopy (ICP-OES)[8]
- Mercury Vapor Analysis[9]
- Transmission Electron Microscopy[10]

To ensure accurate determination of a DeconGel product's decontamination efficacy against compounds analyzed via ICP-OES, calibration standards of the analyte of interest were prepared using either a sufficiently pure analyte or an appropriate ICP-MS Standard (Ricca

[6] Following standardized Environmental Protection Agency (EPA) SW-846 Analytical Method 8270C "Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry" (GC/MS) for organic TICs (or their simulants)/chemicals of concern.

[7] Following standardized EPA SW-846 Analytical Method 8321B "Solvent-Extractable Nonvolatile Compounds by High-Performance Liquid Chromatography/Thermospray/Mass Spectrometry (HPLC/TS/MS) or Ultraviolet (UV) Detection" for organic TICs (or their simulants)/chemicals of concern, and for inorganic TICs (or their simulants)/chemicals of concern.

[8] Following standardized EPA SW-846 Analytical Method 6010C "Inductively Coupled Plasma-Atomic Emission Spectrometry."

[9] Following Environmental Protection Agency (EPA) SW-846 Method 7471B: "Mercury in Solid or Semisolid Waste (Manual Cold-Vapor Technique)."

[10] Following ASTM standard test method D6480-05 "Standard Test Method for Wipe Sampling of Surfaces, Indirect Preparation, and Analysis for Asbestos Structure Number Concentration by Transmission Electron Microscopy," and Polarized Light Microscopy following EPA/600/R-93/116 "Interim Method for the Determination of Asbestos in Bulk Insulation Sample."

Chemical Company; Arlington, TX); the respective standards were diluted to a known concentration (ppm) using the same digestion method as the one used for samples and controls.

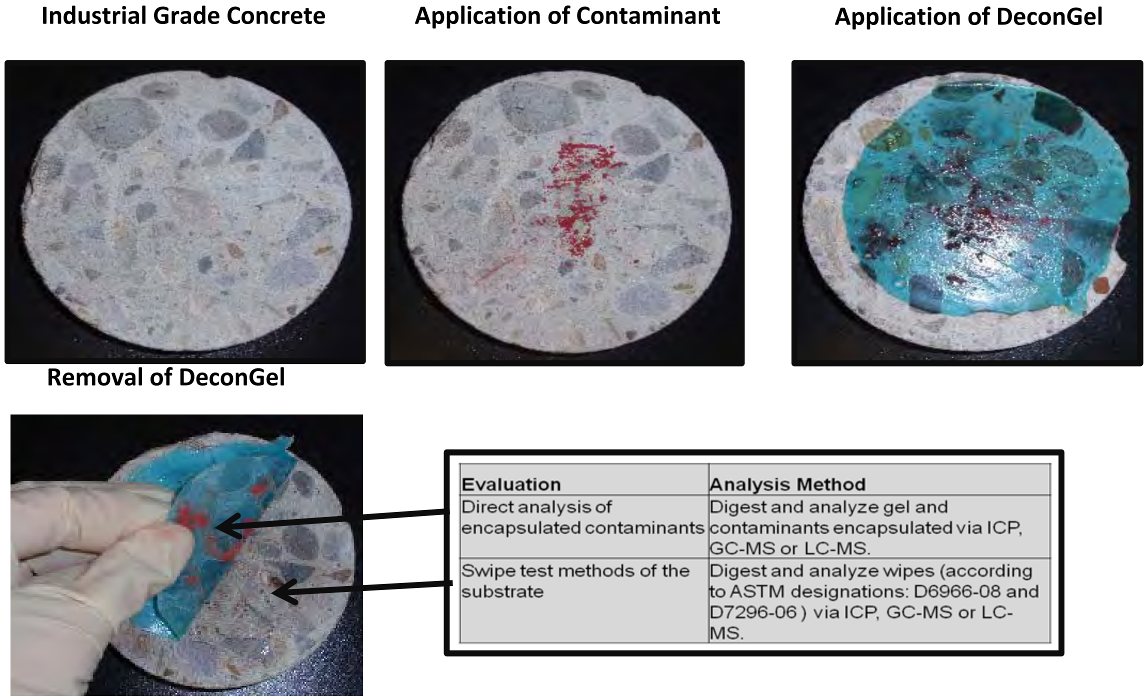
4.2 Experimental Description

A known amount of contaminant was applied to a substrate. For evaluation of DeconGel 1102 against polychlorinated biphenyls (PCBs), contaminants were applied on top of the respective test surface with a small brush. In some cases (depending on the contaminant) drops of methanol (or other solvents) were applied on top of the contamination and allowed to dry. This would represent a contaminant deposit more likely to be found in a "real-world" contamination scenario where personnel may have attempted to remove PCBs with solvents and were unsuccessful. Samples containing compounds that could not be dissolved in a solvent (e.g., elemental metals or other metallic compounds) were digested according to EPA method-3010A or a variation thereof.[11]

Application of a uniform thin layer of contaminant optimized the interaction of the contaminant and DeconGel for an accurate measure of decontamination efficacy. A specific amount of DeconGel formulation (1101 or 1102) was then placed on top of the contaminant. Unless otherwise specified, the hydrogel formulations were left to dry for 24 hours.[12] The figure below depicts the general steps followed for these evaluations.

[11] EPA method-3010A is the acid digestion of aqueous samples, and extracts for total metals for analysis by FLAA or ICP-OES spectroscopy. Variations may include a change in acid type and/or concentration as well as digestion temperature adjustments.

[12] Some contaminants might prolong the drying time of the gel.



Evaluation	Analysis Method
Direct analysis of encapsulated contaminants	Digest and analyze gel and contaminants encapsulated via ICP, GC-MS or LC-MS.
Swipe test methods of the substrate	Digest and analyze wipes (according to ASTM designations: D6966-08 and D7296-06) via ICP, GC-MS or LC-MS.

Figure 1 – Application of contamination on the substrate of interest; application of DeconGel; removal of the dried gel followed by the method of analysis.

4.3 Substrates Evaluated

Hydrogel formulations were evaluated on the following substrates:

- commercial grade stainless steel (surface area: 56.3 cm^2)
- commercial grade carbon steel (surface areas: 17.85 cm^2, 100 cm^2)
- commercial grade aluminum (surface area: 56.3 cm^2)
- commercial grade floor tile (surface area: 100 cm^2)
- industrial grade concrete (surface area: 56.3 cm^2)
- commercial grade composite tile (surface area: 48.8 cm^2)
- commercial grade linoleum tile (surface areas: 10cm^2, 48.8 cm^2)
- commercial grade porcelain tile (surface area: 48.8 cm^2)
- painted drywall (surface area: 10cm^2)

Unless specified, all substrates were used as received with no further pretreatment before application of the contaminants.

4.4 Method of Sampling and Analysis

Swipe sampling methods are used in most industrial standard practices for the evaluation of the contamination (powder, dust, or liquid) level on a substrate. The method consists of applying a specific amount of a contaminant on the substrate of interest, applying the gel on top, removing the gel, and swipe sampling the remaining contaminant on the surface via standard swipe methods for metal sample collection. A respective standardized swipe testing method (see footnote 13) was used for qualitative and quantitative analysis.

All samples and controls were run in triplicate. In a typical experimental procedure, contaminant was evenly applied using careful spreading with a spatula (solids) or using a small paint brush (liquids) on substrate surfaces. Approximately 6.0 g of DeconGel was applied onto the contaminated surface and allowed to dry for 24 hours. Dried gel samples were peeled off the contaminated surface, and the surface was swipe tested. Swipe samples were suspended in the appropriate solvent found to dissolve contaminants. Samples were then analyzed using the appropriate analytical method (as listed in section 4.1). For organic and inorganic contaminants, standardized methods were used to prepare samples and controls.[13]

[13] Organic contaminants: standardized EPA SW-846 Sampling Method 3500C "Organic Extraction and Sample Preparation" was used to prepare samples and controls; inorganic contaminants: standardized EPA SW-846 Sampling Method 3005A "Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA or ICP-OES Spectroscopy" was used to prepare samples and controls.

For swipe control samples, the contaminant was evenly applied via spreading or brushing and the surface was swipe tested according to ASTM methods using GhostWipe™ swipes.[14] For solid contaminants such as lead, solvent-wetted GhostWipe swipes were used, whereas for liquid contaminants such as PCBs, air-dried GhostWipe swipes were used. Swipe controls were suspended in the appropriate solvent found to dissolve contaminants for 24 hours. Controls were then analyzed using the appropriate analytical method.

Decontamination Efficacy via swipe testing was calculated using the following equation:

Decontamination Efficacy (Swipe Testing) =

$$\frac{[\text{Contaminant (ppm) of Swipe Control} - \text{Contaminant (ppm) of Residual Swipe}]}{\text{Contaminant (ppm) of Swipe Control}} \times 100\%$$

[14] Depending on the type of contamination, one of the following ASTM standards were used: ASTM E1728-03: "Standard Practice for Collection of Settled Dust Samples Using Wipe Sampling Methods for Subsequent Lead Determination;" ASTM D6966-08: "Standard Practice for Collection of Settled Dust Samples Using Wipe Sampling Methods for Subsequent Determination of Metals;" ASTM 7296-06: "Standard Practice for Collection of Settled Dust Samples Using Dry Wipe Sampling Methods for Subsequent Determination of Beryllium and Compounds;" ASTM D6661-06: "Standard Practice for Field Collection of Organic Compounds from Surfaces Using Wipe Sampling." GhostWipe™ swipes are available from Environmental Express (www.environmentalexpress.com).

4.5 Decontamination Efficacy on Selected Toxic Industrial Chemicals and Materials

4.5.1 Decontamination Efficacy Evaluations on Beryllium Compounds

Beryllium and beryllium compounds are used in defense, telecommunications, aerospace industries, semiconductors, and nuclear facilities. They are known to cause Chronic Beryllium Disease (CBD), an irreversible, chronic, and incurable disease which may result in death.[15] The maximum limits of exposure as defined by OSHA are 0.5µg/m^3 as a Time Weighted Average concentration (TWA) for an 8 hour work day. This small amount can easily be left in cracks/pores on the spill substrate leading to the closure and demolition of full facilities.

In several case studies, DeconGel has been proven successful in removing beryllium compounds from a variety of substrates. The DOE National Energy Technology Laboratory (NETL) and their cleaning contractor, Safety and Ecology Corporation (SEC), won the Environmental, Security, Safety and Health (ESS&H) Achievement Award in recognition of their innovative use of DeconGel as a beryllium decontamination technique. Use of DeconGel in beryllium decontamination met the decontamination objectives set by DOE NETL while reducing overall costs by a reported 70% and resulted in significant reductions in the volume of waste generated. Most of the cost savings were obtained by reductions in labor required and waste generation while meeting decontamination objectives.

CBIP evaluations included testing of the decontamination efficacy of DeconGel 1101 on stainless steel, aluminum, carbon steel, and concrete surfaces contaminated with beryllium

[15] The Beryllium Disease Website, http://www.berylliumdisease.net, 2010.

surrogate compounds (aluminum powder (Al) and aluminum oxide (Al_2O_3)).[16] Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guidelines for determination of inorganic compounds in aqueous samples. The results are summarized as follows:

- Decontamination efficacies for DeconGel 1101 ranged from 99.7% (on concrete) to 99.9% (on carbon steel) to 99.7% (on stainless steel) for aluminum oxide (surrogate for beryllium oxide); and from 97.9% (on concrete) to 99.5% (on carbon steel) to 99.6% (on stainless steel) for aluminum dust (surrogate for beryllium metal dust) as determined by residual swipe analysis.

4.5.2 Decontamination Efficacy Evaluations on Mercury (Elemental)

Mercury is a compound encountered anywhere from coal-fueled power plants to dental offices. The maximum exposure limits defined by OSHA are even less than beryllium at $0.1\mu g/m^3$ as a TWA concentration for an 8-hour work day. Current cleanup procedures require special mercury vacuums and/or vapor depressants prior to disposal. DeconGel has been proven effective in removing elemental mercury and other mercury compounds from a variety of substrates. CBIP evaluations included the decontamination efficacy of DeconGel products on stainless steel, porcelain tile, composite tile (unwaxed), linoleum tile, and concrete surfaces contaminated with mercury (elemental).[17] Optimized experimental and analytical methods were successfully developed following a standardized EPA analysis method as a guideline for determination of elemental mercury vapor on solid surfaces.

[16] Evaluations performed using ICP-OES (Inductively Coupled Plasma-Optical Emission Spectroscopy) following Environmental Protection Agency (EPA) SW-846 Methods 3005A (sampling) and 6010C (analysis).

[17] Evaluations performed using Mercury Vapor Analysis Sensor (Mercury Tracker 3000) EPA SW-846 Method 7471B: “Mercury in Solid or Semisolid Waste (Manual Cold-Vapor Technique).”

When necessary, experimental methods were customized to ensure accurate decontamination efficacy determination. The results are summarized as follows:

- When DeconGel 1101 was poured on top of the mercury contaminated surfaces (using Zn-controlled contaminant loading) decontamination efficacies were 66.0% (composite tile), 85.9% (porcelain tile), and 90.8% (linoleum tile). When DeconGel 1101 was brushed on the surfaces contaminated with mercury (using Zn-controlled contaminant loading) the decontamination efficacy was 99.0% on linoleum tile.

- Mercury (elemental) was shown to interact with most plastic surfaces such as un-waxed composite and linoleum tiles, adhering to the plastic surfaces and forming a fixed residue that could not be completely removed by DeconGel. Nevertheless, DeconGel was effective in the decontamination of loose mercury (small beads <1 mm diameter) from such surfaces.

4.5.3 Decontamination Efficacy Evaluations on Lead Compounds

Lead and lead compounds can be found in facilities where lead based batteries, lead based pigments, and lead based paints are made. Spills and contamination are often cleaned using moist paper towels and vacuums. The maximum exposure limits as defined by OSHA are 50μg/m^3 as a TWA concentration for an 8-hour work day. DeconGel has been evaluated by CBIP for its efficacy to remove a variety of lead based compounds from various substrates including stainless steel, aluminum, and concrete surfaces contaminated with Lead (PbO, $PbSO_4$).[18] The lead compounds PbO and $PbSO_4$ were chosen as representative lead compounds for evaluating DeconGel efficacy. DeconGel is expected to have similar

[18] Evaluations performed using ICP-OES (Inductively Coupled Plasma-Optical Emission Spectroscopy) following Environmental Protection Agency (EPA) SW-846 Methods 3005A (sampling) and 6010C (analysis).

efficacy towards the wide range of lead compounds. Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guidelines for determination of inorganic compounds in aqueous samples. When necessary, experimental methods were customized to afford complete dissolution of inorganic contaminants, and to ensure accurate decontamination efficacy determination of DeconGel. The results are summarized as follows:

- Decontamination efficacies of DeconGel 1101 on lead (II) oxide ranged from 81.2% (on aluminum) to 91.2% (concrete) to 91.5% (stainless steel) as determined by residual swipe analysis. Decontamination efficacies of DeconGel 1101 on lead (II) sulfate ranged from 99.4% (stainless steel) to 99.8% (concrete) to 99.8% (aluminum) as determined by residual swipe analysis.

- Lead (II) oxide is very hygroscopic and reacts with trace amounts of water (on or within porous and non-porous test surfaces), forming a fixed residue that was not able to be completely removed by DeconGel. Nevertheless, DeconGel was effective in the decontamination of loose lead (II) oxide contamination from such surfaces.

4.5.4 Decontamination Efficacy Evaluations on Cadmium Compounds

Cadmium metal and cadmium compounds are highly toxic. Cadmium chloride is often used in the process of making cadmium sulfide, a common yellow pigment while cadmium oxide is a conductive material used in the making of photovoltaic cells and photodiodes. Cadmium compounds are suspected carcinogens (classified suspected carcinogen A2 by the American Conference of Governmental Industrial Hygienists, ACGIH) and fatal inhalation hazards. It is important to contain and remove cadmium particles which can become airborne. Cadmium chloride and cadmium oxide were chosen as representative cadmium compounds for evaluating DeconGel efficacy; DeconGel is expected to have similar efficacy towards a wide range of cadmium compounds.

CBIP evaluations included the determination of the surface decontamination efficacy of DeconGel 1101 on stainless steel, aluminum, carbon steel, and concrete surfaces contaminated with cadmium compounds (cadmium chloride ($CdCl_2$) and cadmium oxide (CdO)).[19] The results are summarized as follows:

- Decontamination efficacies for DeconGel 1101 ranged from 99.5% (stainless steel), to 98.5% (carbon steel), to 97.6% (concrete) for cadmium chloride and from 100% (stainless steel), to 100% (carbon steel), to 98.4% (concrete) for cadmium oxide as determined by residue swipe analysis.

4.5.5 Decontamination Efficacy Evaluations on Chromium Compounds

Hexavalent chromium is often found in manufacturing plants of stainless steel, anticorrosion coatings, and textile dyes. Current clean-up procedures include sand, vacuums, and paper towels. Recently, hexavalent chromium was recognized as a human carcinogen via inhalation and has been banned from many applications. The maximum exposure limits are $0.1 \mu g/m^3$ as a TWA concentration for an 8-hour work day as defined by National Institute for Occupational Safety and Health (NIOSH). Potassium chromate was chosen as a representative chromium surrogate for evaluating DeconGel efficacy. DeconGel is expected to have similar efficacy towards the wide range of chromium compounds. The results are summarized as follows:

- Decontamination efficacies of DeconGel 1101 ranged from 91.9% (on concrete) to 99.2% (on carbon steel) to 99.6% (on stainless steel) against potassium chromate as determined by residual swipe analysis.

[19] Evaluations performed using ICP-OES (Inductively Coupled Plasma-Optical Emission Spectroscopy) following Environmental Protection Agency (EPA) SW-846 Methods 3005A (sampling) and 6010C (analysis).

- Potassium chromate was evidenced to react with concrete surfaces, forming a fixed residue that was not able to be completely removed from concrete by DeconGel. Nevertheless, DeconGel was effective in the decontamination of loose potassium chromate as noted in the previous paragraph.

4.5.6 Decontamination Efficacy Evaluations on Asbestos

Asbestos is a set of six naturally occurring silicate minerals exploited commercially for their desirable physical properties. The inhalation of asbestos fibers can cause serious illnesses, including malignant lung cancer**,** mesothelioma and asbestosis. Most asbestos fibers that can enter the respiratory system are invisible to the unaided human eye because their size is about 3.0–20.0 µm long and can be as thin as 0.01 µm.

DeconGel has been shown effective in the decontamination of asbestos from a variety of surfaces including linoleum tile, painted drywall, and concrete surfaces contaminated with asbestos fibers (chrysotile fibers).[20] Application of asbestos contamination on the respective substrate and sampling tests were performed in CBIP's labs; analysis of the samples was performed at independent labs (EMSL Analytical, Centennial, CO; and Bureau Veritas North America, Inc., Kennesaw, GA).

The results are summarized as follows:

- Decontamination efficacies (wt% based on the residual asbestos fibers on the surface of interest) were 99.9+% from painted drywall, linoleum tile, and concrete surfaces.

[20] Evaluations performed using Transmission Electron Microscopy (TEM; ASTM standard test method D6480-05) and Polarized Light Microscopy (PLM; EPA approved method for analysis of asbestos from bulk samples.

- Both semi-qualitative (tape lift adhesion sampling method followed by EPA approved PLM analysis) and semi-quantitative (ASTM standard test swipe sampling method followed by TEM analysis) methods have been utilized in these evaluations.

4.5.7 Decontamination Efficacy Evaluations on Mold

DeconGel has been shown effective in removing outdoor mold previous field tests on outdoor mold on various surfaces (including concrete and asphalt). The figures below show mold contamination on outdoor concrete sidewalks before and after application of 1101.

Figure 2 – Before and after pictures of DeconGel 1101 on mold contamination.

The advantage of using DeconGel (a waterless method) to remove mold is that this method eliminates the generation of wastewater and decreases the likelihood of contaminating surrounding areas with mold spores. Empirical evidence also suggests that it takes longer for the mold to return to areas treated with DeconGel. The objective was to physically remove and/or chemically inactivate the mold fungi.[21] Various compounds known for their anti-fungal performance were evaluated for incorporation into DeconGel formulations.

Efficacy evaluations of modified and unmodified DeconGel 1101 and second generation DeconGel 1108 products were performed by an independent lab.[22] Four separate formulations were evaluated in these tests. Formulations evaluated include 1101, modified 1101, 1108, and modified 1108.[23] The tests were performed on very rough concrete that was contaminated with high loads of mold spores and biomass, thus representing a very challenging contaminant/substrate combination. The results from the independent lab showed that no viable spores were left on the surface after decontamination with the modified DeconGel 1101; any remaining spores/colonies appeared as remnants and were non-viable. Additionally, modified second generation DeconGel 1108 removed/killed all pathogenic molds types associated with allergenic fungi illness or related to toxicity. *Ulocladiu*, which is non-pathogenic, was the only type of mold left on the surface after decontamination with this formulation. Unmodified DeconGel 1101 and 1108 performed well but did not completely remove all pathogenic mold spores and colonies. The results validate that the incorporation of an antifungal agent results in formulations that are

21 The ultimate objective was to develop a formulation effective in the simultaneous decontamination of radioactive compounds and mold.

22 Pacific Enterprises Hawaii, Inc. was the independent lab performing the evaluations.

23 Modified DeconGel 1101 and modified DeconGel 1108 both contained 0.5ppH of acetic acid.

effective against mold spores via the combined mechanisms of physical removal and chemical inactivation.

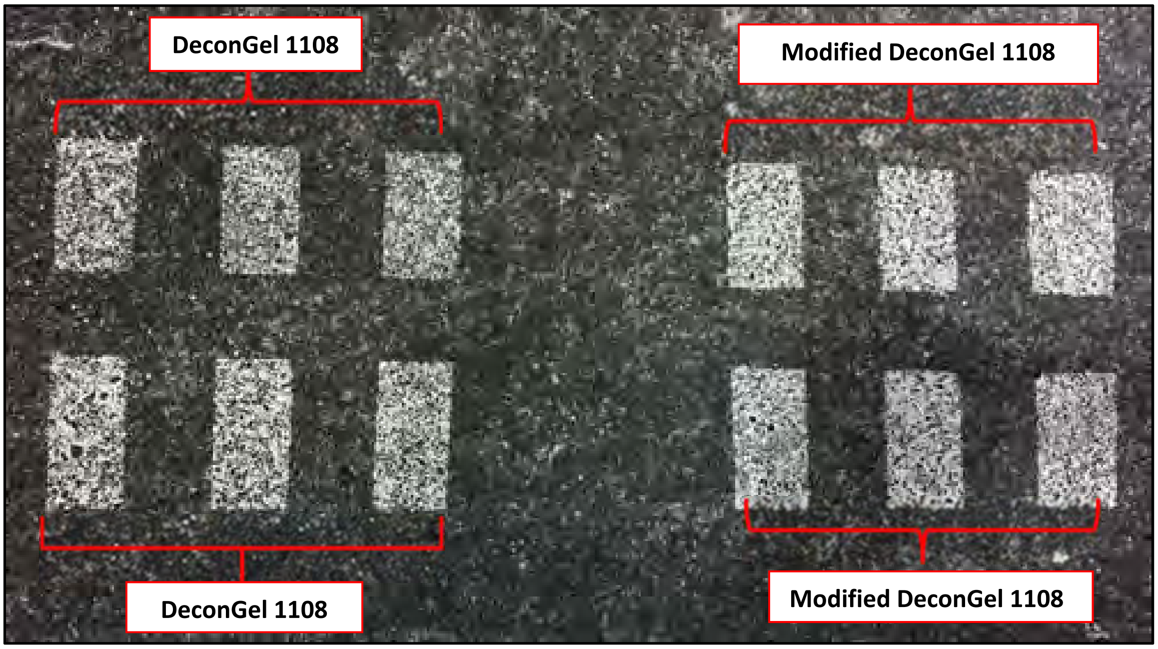


Figure 3 – Decontaminated sections after peeling off the dried gels**.**

The figure above shows the decontaminated sections after removal of the tested formulations. A grid was used to evaluate the efficacy of the four different formulations and to avoid cross contamination. Three replicates of each formulation were performed to verify reproducibility of results; each section of the grid was labeled to identify respective formulations. The test grid was then covered to prevent any rainwater from impacting the test site. Swipe samples from all sections before and after decontamination were analyzed for the presence of spores (number of spores per unit area).

5 FIELD TEST EVALUATIONS

DeconGel products (1101 and 1121) and next generation products developed under this effort (1102, 1108, & 1128) have been field tested under a diverse set of test scenarios. The field tests evaluations included decontamination efficacy, operational factors, safety, waste minimization, and cost. Evaluations performed included the following:

- Successful decontamination of Komatsu 800 Excavator Shears used in the demolition of facilities at 100KE Basin (Richland, Washington).
- Tc-99m decontamination of bare and painted concrete at the Critical Experiments Facility at the Y-12 National Security Complex (Oak Ridge, Tennessee).
- Cs-137 and Am-243 decontamination of bare concrete under the EPA's Technology Testing and Evaluation Program (TTEP) program.
- PCB decontamination aboard the USS Missouri maritime museum vessel (Pearl Harbor, Hawaii).
- Lead dust decontamination at a foundry built in 1915 at Pearl Harbor Naval Shipyard and Intermediate Maintenance Facility (PHNSY&IMF).

Successful evaluations under this effort were instrumental in DeconGel's inclusion in DOE EM's suite of innovative deactivation and decontamination (D&D) technologies known as the D&D Toolbox Project. As part of the Toolbox initiative, DOE EM chose DeconGel for demonstrations on contaminated Kamatsu 800 Excavator Shears that were used for the 100KE Basin demolition in Richland, Washington.

5.1 Demonstration of DeconGel at 100KE Basin, Richland, Washington

The KE Reactor, its associated basin for underwater fuel storage, and the superstructure over the basin operated between 1955 and 1971. The KE Basin was determined to have leaked into the surrounding soil during its operational life. Cleaning up the KE facility is a high priority for the DOE and regulators due to its proximity to the Columbia River half a kilometer away. DeconGel (1101 & 1121) removed alpha and beta contamination from Kamatsu 800 Excavator Shears used in the demolition of facilities at 100KE Basin.

Earlier decontamination efforts with hot, high pressure water were unsuccessful and the shears were coated with a water insoluble fixative used to suppress (fix) dust and loose contamination.[24] Approximately two gallons of DeconGel 1121 were applied with an airless sprayer, and 0.5 gallons of DeconGel 1101 were applied with a paint brush. It was estimated that DeconGel was able to remove about 25% of the fixative coating with the first application.

Figure 4 – Kamatsu 800 Excavator Shears used in demolition of 100KE Basin.

[24] Soil Sement® fixative, Midwest Industrial Supply, Inc., Canton, Ohio.

Application methods were then developed that provided excellent decontamination results. Application of DeconGel, with a wire brush or emery cloth (followed by brush or spray), was used to provide an effective means of removing the fixative while decontaminating the surface. As emery cloth is a wet sanding tool, all scrubbing/sanding was done with liberal amounts of DeconGel, which contained and encapsulated the particulates produced, as well as, incorporating the old fixative into the DeconGel film for removal. This technique provided good decontamination results (<1000 dpm/100cm^{2} removable Beta).

Figure 5 – Application of DeconGel 1101 & 1121 on contaminated excavator sheers.

Figure 6 – Peeling of dried DeconGel from the now decontaminated sheers.

Background radiation near the shear was too high to allow direct reads of the surfaces on the shear. Additionally, swipe evaluation of the contaminated surfaces before decontamination with DeconGel was unavailable because the surfaces were coated with the Soil-Sement fixative. Therefore, a GM probe was used to survey both sides of the peeled films as well as swipes to test for loose contamination of the decontaminated surfaces. The data generated show that substantial radioactive contamination was removed from surfaces that were previously cleaned with the hot high-pressure water. Additionally, loose contamination on the decontaminated surfaces was non-detectable as all the readings were equal to background readings.

The decontamination of radioactive surfaces on the shears was highly effective when DeconGel was applied with scrubbing (wire brush/emery cloth). Since the surfaces were previously decontaminated by the current "best" method (high pressure water/steam followed by the fixative coating), it was shown that DeconGel provides a method of effectively decontaminating surfaces that could not be fully decontaminated with high pressure water/steam cleaning. Higher efficacy is expected if the water insoluble fixative is not applied and DeconGel is applied in its place, or the equipment is first decontaminated with DeconGel. Of note is that use of DeconGel may allow subsequent release of the equipment from the 100KE Basin.

5.2 Operational Evaluation of DeconGel 1128 at the Y-12 National Security Complex, (Critical Experiments Facility, Building 9213)

As part of the U.S. Environmental Protection Agency's mission to protect human health and the environment from acts of terror, certain performance tests on homeland security technologies are conducted to determine their effectiveness. The EPA and Department of Homeland Security (DHS) conducted an Emergency Preparedness technology evaluation of several products, including DeconGel 1128 (spray version of DeconGel 1108),[25] on its ability to remove radioactive technetium-99 metastable (Tc-99m) from concrete wall and floor surfaces similar to those found in homes and buildings. The purpose of the evaluation was to demonstrate the effectiveness of the decontamination technology for remediation and decontamination of concrete surfaces, following a nuclear or radiological emergency (e.g., use of a radiological dispersal device). Specific objectives of the evaluation included: (1) determining the operational requirements for conducting radiological decontamination field trials; (2) evaluating the Y-12 National Security Complex, Nuclear and Radiological Field Training Center for use in future investigations of technologies; (3) assessing the operational factors associated with use of 1128;[26] and (4) evaluating the efficacy of the product as a decontamination technology.

DeconGel 1128 was applied to four test areas, consisting of 20 square feet of wall surface and 33 square feet of floor surface contaminated with approximately 2 milliCuries (mCi) of acidic solutions of Tc-99m. No positive or negative control areas were designated for this trial. The amount of contamination deposited on each grid square was measured using beta, gamma G-M, and sodium iodide (NaI) detectors connected to a Ludlum radiation ratemeter/scaler. Two coats of DeconGel 1128 were sprayed onto the vertical wall surface, and one coat was sprayed onto the floor surface areas.[27] Approximately 20 hours after the second coat was applied, the dried DeconGel 1128 was peeled from the surfaces and the grids were resurveyed for remaining contamination.

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[25] "Operational Evaluation of DeconGel 1128 Spray Product for Radiological Decontamination on Concrete Surfaces."

[26] Some operational factors are skilled labor or special training required, method and rate of product application, applicability to different surfaces, portability, and volume of waste generated.

[27] The second coat of DeconGel 1128 was applied to the wall surface approximately two hours after the initial coat in order to allow the first coat to partially dry.

Decontamination of the wall surface resulted in an average % Decontamination (% removal of original radioactivity on a surface) of approximately 71%. Decontamination of the floor surface resulted in an average % Decontamination of approximately 80%. In general, less porous materials (sealant-coated, concrete flooring) were decontaminated more effectively than the more porous wall and the rough raised concrete ledge at the base of the wall.

Regarding operational factors, other than the skill to operate a commercial airless sprayer, no specialized skills or training were required of personnel; the only required tool was the airless sprayer. Secondary waste consisted entirely of disposable personal protective gear. The radiation control technician, using a NaI gamma detector, conducted a full-body examination of EPA personnel performing the decontamination each time the team exited the contaminated test area. In all instances, no measurable activity was detected on the personnel. Retail material cost of a 5.28-gallon (20-liter) container of DeconGel 1128 is $800. Utilizing the retail price, the material cost for this evaluation was determined to be approximately $2.26 /ft^2.

5.3 EPA Evaluation of DeconGel 1101 & 1108 on Cesium-137

In 2011 the U.S. Environmental Protection Agency's (EPA's) National Homeland Security Research Center (NHSRC) Technology Testing and Evaluation Program (TTEP) evaluated the performance of DeconGel (1101 & 1108) in removing radioactive cesium (Cs-137) from the surface of unpainted concrete. See report at:

http://cfpub.epa.gov/si/si_public_record_report.cfm?dirEntryId=236312

DeconGel 1101 and 1108 were applied as paint-like coatings and then cured in order to bind the Cs-137; the cured coating containing Cs-137 could then be removed from the surface causing little or no surface damage. Prior to the evaluation, eight 15 × 15 centimeter unpainted concrete coupons were contaminated with Cs-137 at a level of approximately 1 microcurie (μCi, measured by gamma spectroscopy). The eight contaminated coupons were placed in a test stand (along with one uncontaminated blank coupon) in a vertical orientation to simulate the wall of a building. Both the 1101 and 1108 were painted onto separate concrete coupons with a 4 inch paint brush. The time required to apply each coating to a coupon was an average of 30 seconds for each coat that was applied. Two coats were applied to each coupon and then the coupons were allowed to dry overnight and the coatings were removed. Then the cycle was repeated. Thus, the coatings were applied and removed twice for each coupon (four coupons each for 1101 and 1108) before the residual activity of the contaminated coupons was measured.

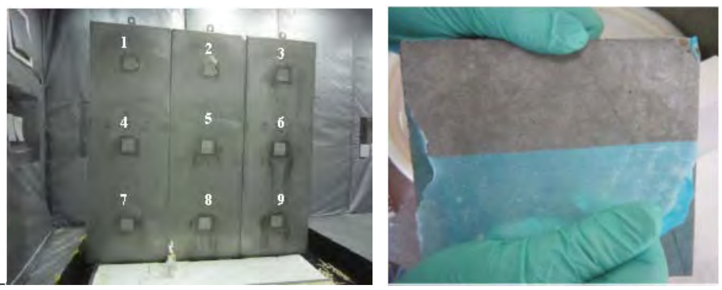

Figure 7 – Test stand containing contaminated numbered coupons (left); and DeconGel peel (right).

The decontamination efficacy (% Decontamination = %R = % removal of original activity on a surface) of the four contaminated coupons for DeconGel 1101 demonstrated an average % Decontamination for 45 ± 7%.

The average % Decontamination for DeconGel 1108 (second generation product) was 67 ± 9% demonstrating a significant improvement in decontamination efficacy compared to the original DeconGel products (1101) in addition to it being non-flammable and OSHA non-hazardous.

5.4 Evaluation of DeconGel 1108 on Americium-243

As a result of previous testing by the EPA NHSRC on cesium-137 (described above), the EPA subsequently evaluated the performance of DeconGel 1108. The objective of the evaluation was to test the ability of 1108 to remove radioactive americium (Am-243) from the surface of unpainted concrete. The decontamination efficacy of 1108 was evaluated following the contamination of porous concrete coupons with acidic solutions of Am-243. This contamination model is considered to be one the most difficult challenges for radiological decontamination due to the absorption and migration of the heavy metal isotopes into the porous concrete. See report at:

http://cfpub.epa.gov/si/si_public_record_report.cfm?dirEntryId=248392&fed_org_id=1253&address=nhsrc/&view=desc&sortBy=pubDateYear&showCriteria=1&count=25&searchall=TTEP

Prior to the evaluation of DeconGel 1108, 15 x 15 centimeter unpainted concrete coupons were contaminated with an activity level of approximately 50 nanoCuries (nCi) of an acidic solution of Am-243 as measured by gamma spectroscopy. Several of these contaminated coupons were then placed on a test stand to create a vertical concrete surface to which 1108 was applied and removed.

Two coats of DeconGel 1108 were applied to all of the coupons in the test stand; the application of 1108 was performed using a standard 10 cm paint brush. The coupons were then allowed to dry overnight and the procedure was repeated. The temperature and relative humidity were recorded during the application and removal of 1108. These conditions did not vary significantly in the laboratory where the coupons were stored and evaluated. Over the duration of testing, the temperature was always within the range of 22–23^{o}C and relative humidity was 16%.

DeconGel 1108 was tested side by side (using identical application and testing methods) against Bartlett's Stripcoat TLC Free, an industrial strippable decontamination coating. Bartlett's Stripcoat TLC Free demonstrated a % Decontamination (%R) of 46%. DeconGel 1108 demonstrated a % Decontamination (%R) of 84%.

5.5 Decontamination of PCB Oil Onboard the USS Missouri (BB-63)

CBIP performed a four-part field test aboard USS Missouri using DeconGel 1102. The 1102 formulation was designed to decontaminate hydrophobic chemicals including Toxic Industrial Chemicals (TICs).

Polychlorinated Biphenyls (PCBs) were widely used as industrial dielectric additives in transformer and capacitor coolants, and insulating fluids. Due to carcinogenicity, PCB production has been banned since the 1970s. PCBs are considered chemically inert and resist environmental degradation, and as such remain environmentally persistent pollutants.

The USS Missouri maritime museum vessel contains several areas with limited access to the public due to PCB oil contamination. These areas had previously received extensive decontamination by repeatedly scrubbing with hexanes, a highly flammable solvent, and wiping. Even after several attempts, PCB levels did not meet EPA regulatory limits for unrestricted public access.

Figure 9 – USS Missouri maritime museum vessel.

DeconGel 1102 achieved these limits after only one application on all four types of decades-old contaminated surfaces.[28] Cost estimates prepared by a Pearl Harbor Naval Shipyard and Intermediate Maintenance Facility (PHNSY&IMF) subcontractor put the cost of the standard decontamination method (cleaning and scrubbing with a solvent) to be $20.83/sqft. When 1102 is used, the cost estimate dropped to $7.88/sqft., demonstrating a reduction in cost of more than 62%.

5.6 Lead Dust Decontamination at a PHNSY&IMF Foundry

Building 6 Foundry Complex at PHNSY&IMF is a 52,000-square-foot structure built in 1915 as a facility for casting metal ship replacement parts. Foundry operations were discontinued in 1997. Currently, the primary objective is remedial action to reduce or eliminate risk to human health under continued industrial or future commercial land

[28] For all contaminated surfaces tested (oily stainless steel, cement levelling compound, Cosmoline coated brass and painted steel deck plate) EPA PCB limit standards (Regulations 761.79, 761.123) of ≤ 10 μg/100 cm^2 were achieved upon decontamination with a single application DeconGel 1102.

scenarios. DeconGel was evaluated as a decontamination method on a variety of complex equipment and substrates, e.g., a smelter, a metal circuit board, bare and painted concrete, bare and painted metal, and glass window surfaces.

Among other contaminants, Building 6 contains significant amounts of dust contamination comprising heavy metals (mostly lead). Lead compounds are used in batteries, bullets, as part of solders and alloys, vulcanized rubber, and as radiation shields. However, lead is a toxic heavy metal. Upon exposure and ingestion/inhalation, lead and its compounds accumulate in soft tissue and bone, and act as potent neurotoxins.

While there are standards set by OSHA for the amount of airborne lead dust workers can be exposed to, there are no legal standards for the amount of lead on surfaces in the workplace. To protect workers, OSHA requires that wipe samples collected on surfaces in the eating areas in workplaces not exceed 200 micrograms/sqft ($\mu g/ft^2$). While PHNSY&IMF believes that this limit is more stringent than that required for remediation of Building 6, it also believes this limit could be used as a conservative estimated limit to determine the suitability of using DeconGel for the decontamination of lead dust.

DeconGel formulations (both DeconGel 1101 and DeconGel 1121 were evaluated) achieved excellent surface decontamination efficacy against lead dust. The % Decontamination was ≥85% for most of the areas tested. With the exception of the smelting equipment that is planned to be removed from the building, all other areas decontaminated had average post-decontamination lead levels below the stringent 200 $\mu g/ft^2$ specification. Lead contamination on bare metal horizontal surfaces within the foundry was 1600 $\mu g/ft^2$. Lead remaining after the first application and removal of DeconGel 1101 was 405 ± 15 $\mu g/ft^2$ and lead remaining after the 2nd application and removal of DeconGel 1101 was 65 ± 10 $\mu g/ft^2$. The % Decontamination after the first application was 74.69 ± 0.94, and after the second application was 95.94 ± 0.63.

For bare concrete floor areas the lead contamination before DeconGel 1101 was 857 ± 471.78 μg/ft^2. Lead contamination remaining after the first application and removal of DeconGel 1101 was 234.4 ± 98.81 μg/ft^2 and lead remaining after the second application and removal of DeconGel 1101 was 84.82 ± 42.54 μg/ft^2. This resulted in a % Decontamination of 69.8 ± 11.06 (on average) after one application and a % Decontamination of 87.84 ± 8.02 after two applications.

During this field test, DeconGel was also evaluated as a "fixative" to keep particulates in place on equipment that is to be removed before the decontamination project starts. The photos below were taken by PHNSY&IMF photographers during field testing at Building 6. Note in Figure 10 the use of DeconGel in preventing lead dust from aerosolizing off of the encapsulated lead furnace.

Figure 10 – Application of DeconGel 1121 by spraying vertical and three-dimensional surfaces.

Figure 11 – **Dried DeconGel can be easily peeled off from a variety of surfaces**.

For costing, a job consisting of 50% porous bare concrete, 25% glass/painted metal/bare metal, and 25% painted walls was estimated to cost $13.24/ft^2. No direct cost comparison could be performed as PHNSY&IMF previously considered the cost to decontaminate the facility to be infeasible. Use of the next generation products, DeconGel 1108 and DeconGel 1128, are predicted to provide results superior to the successful results in this study.

Because the type of surface determines the amount of gel required for a project, only generalized cost figures can be given. DeconGel (all versions) retails for $800 per 20 liter (5.28) gallon pail, and each gallon covers anywhere from 35 ft^2 (rough uncoated concrete) to 150 ft^2 (nonporous surfaces) per gallon, giving a cost of approximately $4.57 to $1.06 per square foot. Government agencies receive a discount from the stated retail pricing when purchased directly from CBIP.

6 REGULATORY AND LEGAL CONSIDERATIONS

Because this collaborative effort resulted in changes to the baseline DeconGel formulas, an overview of the regulatory and legal considerations relevant to the new formulations was required. CBIP has achieved approval for use at key U.S. DOE disposal sites. Chemical testing has been completed for major commercial nuclear facilities, and approved for use at General Electric's (GE) U.S. service locations and on GE Nuclear reactor systems.

6.1 Regulatory Approval and Certification

CBIP has assessed the relevant regulatory approvals and certifications for use required for exporting to major commercial markets. The following provides an overview of the current of DeconGel approvals in the U.S., Europe and Japan. Evaluation of the requirements for markets in Singapore and Korea is currently underway.

U.S. regulatory approval for products such as DeconGel entails technical specifications rather than specific product approvals. DeconGel has obtained the following approvals, each of which required an evaluation of DeconGel by each site and the associated facility's regulatory license authority.

DeconGel has obtained the following waste disposal approvals, subject to the restrictions that apply to nature of the contaminant encapsulated:

- Waste Isolation Pilot Plant (WIPP) Transuranic Waste Disposal facility
- Disposal at low-level radioactive waste sites
- Incineration at U.S. commercial low level waste facilities

DeconGel has also completed chemical testing and has been approved for use at commercial nuclear facilities, including:

- All GE Nuclear facilities
- Perry Nuclear Power Plant in Perry, OH
- Kewaunee Power Station in Kewaunee, WI
- San Onofre Nuclear Generating Station in San Onofre, CA
- Bruce Power and Ontario Power Generation in Ontario, CN

CBIP is in the process of obtaining product certification for the commercial use of DeconGel within the European Union (EU). One important note is that the US trademark name “DeconGel” cannot be used in the EU since the word, “Decon” has been trademarked by DECON Labs, UK. CBIP is currently using the name “CBI Polymers Decontamination Gel 11XX” where “XX” will be the numerical designation of the gel (1101, 1102, 1120, 1121, 1108, 1128, etc.) for all products sold in the EU.

In respect to the immediate needs of the Japanese market, DeconGel is already being used on a selected basis by the Japanese Self-Defense Force and is being tested by Tokyo Electric Power Company and other organizations. The key driver for use of DeconGel within Japan may lie with large commercial remediation companies who have been contracted by the government for clean-up.

6.2 Intellectual Property

To protect the Company’s proprietary intellectual property, U.S. and Patent Cooperation Treaty (PCT) applications entitled “POLYMER COMPOSITION AND METHOD FOR REMOVING CONTAMINATES FROM A SUBSTRATE” were filed on February 28,

2007. This case is currently pending in the U.S, Canada, Japan, China, Korea, and Australia and patents have been granted in China, Singapore and Europe. The European patent was validated in France, Germany and the U.K.

A related application entitled “POLYMER COMPOSITION FOR PROTECTING SUBSTRATES AND REMOVING CONTAMINANTS FROM SUBSTRATES” has issued in Europe and is currently pending in the US, Canada, China, Japan, and Korea. The European patent was validated in France, Germany and the UK.

A provisional application incorporating intellectual property generated under this contract was filed 8/11/11. A full application was filed on 8/10/12.

Additional patent applications will be completed for new products/versions/technological breakthroughs/uses.

7 SUMMARY

This effort made significant improvements to, and improved the efficacy of, DeconGel on radioisotopes and hydrophobic contaminants and improved the products' safety profile, meeting all DOE EM goals set out in the effort.

The technology was field tested at 100KE Basin, Richland, Washington; Y-12 National Security Complex; and the National Energy Technology Laboratory–Albany. EPA testing on cesium and americium; PCB decontamination aboard the USS Missouri (BB-63) and finally, lead dust decontamination at a Pearl Harbor Naval Shipyard and Intermediate Maintenance Facility (PHNSY&IMF) foundry.

Rarely does an improved product development effort meet with an immediate need for its use. However the impact of the earthquake/tsunami of March 2011 in Japan to the Fukushima-Daiichi Nuclear Power Plant resulted in widespread contamination extending several hundred miles from the affected plant. This led the Japanese government to conduct an exhaustive review of decontamination technologies. The result of that review and testing was the listing of DeconGel as an approved decontamination product for use in Japan. DeconGel 1108 and 1128 were expedited to Japan where they are currently being used by civilian first responders, Tokyo Electric Power Company, and the Japanese Self-Defense Forces. Aspects of the product's acceptance by the Japanese included the efficacy of the product, its ease of use especially by potential civilian users within the general population, its safety and flammability profile, and most importantly reduction in total operational costs through improved efficacy, efficiency and reduction in waste as compared to traditional methods of decontamination. This, in itself, justified the research and development efforts that have led to the development of these substantially proven products.

8 RECOMMENDATIONS

The authors recommend that:

1. Contractors supply at least three side-by-side product comparison testing results to the government when proposing to clean assets with decontamination products. Additionally, the total costs to complete the effort using each comparison product should be provided. This would facilitate the selection and use of products, labor, and waste disposal resulting in optimum cost-effective solutions from a total lifecycle perspective.
2. DOE establish a list of decontamination processes with pros and cons, similar to the list of fixatives developed by the former Hanford ALARA Center.
3. DOE EM evaluate DeconGel as a removable fixative. A lesson learned in Japan was the utility of removable fixatives and the value of stockpiling such products for immediate use. Errors made by decontamination experts included the use of non-removable fixatives and the delay in obtaining and deploying (since they were not readily available) such products. Non- removable fixatives eventually result in the requirement to demolish assets coated with these fixatives, and removal and storage of the waste generated. DeconGel may be suited for use as a removable fixative for those assets which require non-destructive decontamination. However, DeconGel has not been DOE tested as a permanent or removable fixative.
4. Finally, the authors recommend that DOE EM initiate and fund an outreach effort to brief the results of this program, e.g., product efficacy and cost-saving applications. Each DOE contractor and all DOE divisions involved with decontamination or clean-up of power generation systems should be apprised.