

TITLE: Surface Decontamination of Crude Oil by DeconGel[™] 1101 and 1102

ABSTRACT

Surface decontamination efficacy determination of DeconGelTM 1101 and 1102 on aluminum, stainless steel, and concrete surfaces contaminated with Crude Oil (polyaromatic hydrocarbons fraction) was performed with LC/MS (Liquid Chromatography/Mass Spectrometry) according to Environmental Protection Agency (EPA) SW-846 Methods: 3500C (sampling), 8321B (analysis), and 1654A (Analysis of PAH Content of Oil).

HAZARDOUS MATERIALS RELEVANCE

Crude oil is used throughout the world as an energy source, and is used to provide a broad range of valuable and useful materials. Crude oil and its decomposition products are environmentally persistent toxins, and when released into the environment require extensive remediation typically resulting in the generation of large quantities of toxic contamination. Crude oil is comprised of 0.2-7% polyaromatic hydrocarbons (PAHs), widespread organic pollutants known for their carcinogenic, mutagenic, and tetratogenic properties.

SUMMARY RESULTS

- As seen in Table 1, excellent surface decontamination was achieved by applying DeconGel 1101 and 1102 both via brushing or pouring (non-brushed) onto contaminated surfaces, resulting in encapsulation of Crude oil contaminant by DeconGel's active components. Decontamination efficacies of poured DeconGel 1101 ranged from 85.9% (on concrete) to 93.9% (on aluminum) to 93.9% (on stainless steel), brushed DeconGel 1101 ranged from 98.4% (on concrete) to 99.0% (on stainless steel) to 99.1% (on aluminum), as determined by residual swipe analysis. Decontamination efficacies of poured DeconGel 1102 ranged from 86.6% (on concrete) to 91.3% (on stainless steel) to 94.1% (on aluminum), brushed DeconGel 1102 ranged from 98.9% (on concrete) to 99.5% (on stainless steel) to 99.6% (on aluminum), as determined by residual swipe analysis.
- Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guidelines for determination of organics in polar solvent solvated samples. When necessary, the digestion methods were customized to result in the complete dissolution of the organic contaminants and to ensure accurate decontamination efficacy determination of DeconGel.

RESULTS

Table 1 shows the decontamination efficacies of DeconGel 1101 on stainless steel, aluminum, and concrete surfaces contaminated with Crude oil as determined by residual swipe testing.

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Table 1. Decontamination efficacies of DeconGel 1101 and 1102 on Crude Oil on stainless steel, aluminum, and concrete surfaces as determined by residual swipe testing.

Swipe Testing (ppm)		Formulation	Formulation
		DeconGel 1101	DeconGel 1102
Stainless Steel	Control	213.63 <u>+</u> 1.60	213.63 <u>+</u> 1.60
	Residual (non-brushed)	12.88 <u>+</u> 0.29	18.63 <u>+</u> 0.33
	Residual (brushed)	2.04 <u>+</u> 0.02	0.97 <u>+</u> 0.03
	Decon. Efficacy (non-brushed) (%)	93.9 <u>+</u> 0.10	91.3 <u>+</u> 0.16
	Decon. Efficacy (brushed) (%)	99.0 <u>+</u> 0.10	99.5 <u>+</u> 0.10
Aluminum	Control	215.82 <u>+</u> 1.07	215.82 <u>+</u> 1.07
	Residual (non-brushed)	13.16 <u>+</u> 0.66	19.10 <u>+</u> 0.41
	Residual (brushed)	2.02 <u>+</u> 0.01	0.93 <u>+</u> 0.02
	Decon. Efficacy (non-brushed) (%)	93.9 <u>+</u> 0.10	94.1 <u>+</u> 0.34
	Decon. Efficacy (brushed) (%)	99.1 <u>+</u> 0.10	99.6 <u>+</u> 0.10
Concrete	Control	156.03 <u>+</u> 1.93	156.03 <u>+</u> 1.93
	Residual (non-brushed)	22.02 <u>+</u> 0.26	20.88 <u>+</u> 0.12
	Residual (brushed)	2.44 <u>+</u> 0.10	1.79 <u>+</u> 0.02
	Decon. Efficacy (non-brushed) (%)	85.9 <u>+</u> 0.18	86.6 <u>+</u> 0.36
	Decon. Efficacy (brushed) (%)	98.4 <u>+</u> 0.10	98.9 <u>+</u> 0.10

13133x dilution factor for samples and controls

NOTES

- Application of a homogenous, thin layer of Crude oil contaminant on the respective substrate facilitated an optimized interaction between contaminant and DeconGel, and an accurate measure of DeconGel's decontamination efficacy in a scaled-down yet real-world setting. No less than 6.0 g of DeconGel was used for each experiment to allow an optimized interaction between contaminant and DeconGel.
- ASTM method E1728-03, a standardized swipe testing method used for sampling of contaminants, was the integral method used to accurately evaluate DeconGel's decontamination efficacy. Air-dried GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes wetted with methanol/DMSO (1:1) solvent were utilized in this swipe testing method.
- Standardized EPA SW-846 Sampling Method 3500C "Organic Extraction and Sample Preparation" and EPA Method 1654A "PAH Content of Oil by HPLC/UV) were followed as a guideline to prepare all samples and controls. All samples, controls, and standards were prepared using the same solvent and appropriate experimental conditions to ensure accurate and LC/MS instrumental analysis.
- LC/MS instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of organic compounds. Standardized EPA SW-846 Analytical Method 8321B "Solvent-Extractable Nonvolatile Compounds by High-

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- Performance Liquid Chromatography/Thermospray/Mass Spectrometry (HPLC/TS/MS) or Ultraviolet (UV) Detection" was followed as a guideline to prepare all samples and controls.
- To ensure accurate determination of DeconGel decontamination efficacy, a standard curve
 of the analyte of interest was prepared using sufficiently pure analyte; the respective
 standards were diluted to a known concentration (ppm) using the same solvent as used for
 samples and controls.

CALCULATIONS

Decontamination Efficacy (Swipe Testing) =

[(Contaminant (ppm) of Swipe Control) – (Contaminant (ppm) of Residual Swipe)/Contaminant (ppm) of Swipe Control] x 100%

MATERIALS AND METHODS

Sample Method

In a typical procedure, 0.10 g (1.0 mL of 5 g/50 mL hexane) Crude oil contaminant was evenly applied via brushing on 1) aluminum (surface area: 56.3 cm²), 2) stainless steel (surface area: 56.3 cm²), or 3) concrete (industrial grade, surface area: 56.3 cm²) coupons, and then the hexane carrier solvent allowed to evaporate for 20 min. Approximately 6.0 g of DeconGel 1101 or 1102 was either poured or brushed (brushed first in a top-bottom, then in a left-right fashion) onto the contaminated surface and let to dry for 24 hours. Dried DeconGel samples were peeled off the contaminated surface, and the surface was swipe tested (ASTM method E1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC) wetted with methanol:DMSO (1:1) solvent (2 mL). Swipe and dried film samples were suspended in 50 mL methanol:DMSO (1:1) for 24 hours. Samples were then analyzed via LC/MS (see below).

Control Methods

For swipe control samples, a respective amount of contaminant was evenly applied via brushing on 1) aluminum (surface area: 56.3 cm²), 2) stainless steel (surface area: 56.3 cm²), or 3) concrete (industrial grade, surface area: 56.3 cm²) coupons, and the surface was swipe tested (ASTM method 1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC) wetted with methanol:DMSO (1:1) solvent (2 mL). Swipe and dried film samples were suspended in 50 mL methanol:DMSO (1:1) for 24 hours. Samples were then analyzed via LC/MS (see below).

Reagents and Standards

Crude oil (light, sweet Chevron crude oil sourced Oahu, Hawaii) was suspended in methanol/DMSO (1:1) solvent mixture, sonicated for 10 min, let to stand for 24 hours, and then the yellow liquid decanted from undissolved solids used to generate standard curves.

Analytical Instrumentation

A Thermo LCQ LC/MS with autosampler in positive mode was used to determine PAHs (polyaromatic hydrocarbons) fraction concentration (ppm, wt/wt) of all samples and controls, using a Grace Davison (Deerfield, IL) C18-Select analytical column (150 x 4.6 mm, 5 um).

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A 7-point standard curve derived from three independently prepared stock solutions (crude oil dissolved in methanol:DMSO (1:1) was prepared. The calibration curve exhibits a curve fitting as approximated by the coefficient of determination of linear regression R^2 , where $R^2 = 0.99$ (see Figure 1).

LC method using A= water (0.1% formic acid), B=acetonitrile (0.1% formic acid); start at 90%A to 2 min at 70%A, to 3 min at 50%A, to 5 min at 30%A, to 6 min at 100%B, hold until 16 min at 90% A hold until 17 min.

Crude oil (PAHs fraction) LC/MS data: 9.2 min; lambda max = 275 nm; no data acquired for MS.

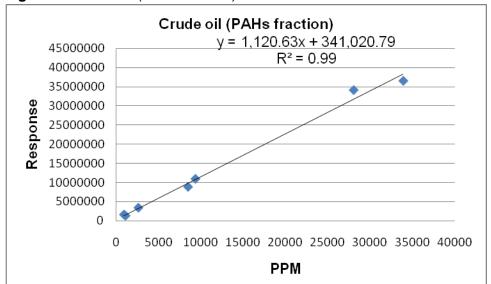


Figure 1. Crude oil (PAHs fraction) standard calibration curve

APPLICATION INSTRUCTIONS FOR END-USERS

Use product directly as is from container. DO NOT DILUTE. Masking or painters tape can be applied along one edge of the area that is to be decontaminated to aid creating a peeled edge to grip for peeling the dried film. Apply DeconGel using a paint brush, a trowel, a handheld sprayer, or an industrial grade sprayer (use DeconGel 1120 or 1121 for spray application).



The thickness of the gel and the number of coats is dictated by the surface to be decontaminated. Coating thickness required for good peel characteristics varies with substrate

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and generally increases with substrate porosity. It is recommended that first time customers test DeconGel on a small sample area to confirm the required film thickness and dry time for their specific application. If the film is difficult to peel, please apply an additional coat. A razor blade is useful to start the peel. Lay the blade nearly flat and fillet the edge of the film to create a tab that can be pulled. For surfaces that the gel adheres to well, such as concrete, 12" - 24" strips can be cut in the film resulting in less force being required to peel the film.

➤ Let DeconGel dry for 24 hours

Dry time will vary depending on humidity, temperature, air flow and thickness of the DeconGel. This can take from as little time as an hour for thin coats in a dry environment with plenty of airflow, to overnight or longer if thicker coats are applied in humid environments. Dry times exceeding 24 hours may sometimes be required for good peel performance on bare concrete, wood and other highly porous substrates and substrates with deep cracks or grooves. However, 18-24 hours is often sufficient dry time on good quality concrete. It is recommended that users test a small area to determine drying time prior to applying DeconGel for an entire job. Supplemental heat or air circulation will accelerate DeconGel's drying time for any job.

> Peel DeconGel off the surface by starting from one of the edges



When dry, the product locks the contaminants into a polymer matrix. The film containing the encapsulated contamination can then be peeled. DeconGel peels from most non-porous and porous hard surfaces if the dried film is thick enough. If the film is difficult to peel, add another coat, let dry, and peel. In most cases the DeconGel will come off in a single sheet but for odd shaped surfaces you may be required to score DeconGel in order to peel it off.

Dispose of the dried DeconGel in accordance with the local, state and Federal disposal regulations of the contaminant/substance you are removing. DeconGel itself has no special disposal restrictions.



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For questions about DeconGel or to place an order, visit our website at $\underline{\text{www.decongel.com}}$ or contact us at:

CBI Polymers Inc. 1946 Young Street, Suite 480 Honolulu, Hawaii 96826 (808) 949-2215 ext. 146 orders@decongel.com

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