Bureau of Safety and Environmental Enforcement Oil Spill Preparedness Division

Emerging Pollution Response Technology Evaluation: Adsorbents

Final Report

June 2023



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US Department of the Interior Bureau of Safety and Environmental Enforcement Oil Spill Preparedness Division



Final Report

OSRR # 1128

June 2023

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Prepared under Contract Number 140E0122F0101 By U.S. Coast Guard Research and Development Center and The Bureau of Safety and Environmental Enforcement

US Department of the Interior Bureau of Safety and Environmental Enforcement Oil Spill Preparedness Division



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| Bureau of Safety and | Project Number – | Testing of Oil Spill Technologies (TOST) Program |
| Environmental Enforcement | 1128 | Bureau of Safety and Environmental Enforcement |
| (BSEE) | | (bsee.gov) |
| U.S. Department of the Interio | or Emerging Pollution | https://library.doi.gov |
| Library | Response | |
| | Technology | |
| | Evaluation: | |
| | Adsorbents | |
| National Technical Reports | Emerging Pollution | https://ntrl.ntis.gov/NTRL/ |
| Library | Response | |
| - | Technology | |
| | Evaluation: | |
| | Adsorbents | |

Sources: a) BSEE (2019), b) DOI [2021], c) National Technical Information Service (2021)

CITATION

Balsley A, Wurl M, McKinney K. 2023. Emerging Pollution Response Technology: Adsorbents. U.S. Department of the Interior, Bureau of Safety and Environmental Enforcement. Report No.: 1128. Contract No: E0122F0101.

ABOUT THE COVER

The cover image, taken by Ohmsett staff, illustrates one of the adsorbents deployed in a test tray and in the process of adsorbing oil. The sorbent had partially adsorbed the available oil when the image was taken. The test oils were dyed red for visibility.



Report No. CG-D-02-23

Emerging Pollution Response Technology: Adsorbents

Distribution Statement A: Approved for public release; distribution is unlimited.

June 2023



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| | | r. | Fechnica | l Report Documenta | ation Page |
|---|----------------|--------------------------------|--------------|---------------------------|-------------|
| 1. Report No. | | 2. Government Accession Number | 3. Recipier | nt's Catalog No. | |
| CG-D-02-23 | | | | | |
| 4. Title and Subtitle | | | 5. Report I | Date | |
| Emerging Pollution Response Techno | logy Evaluatio | on: Adsorbents | June 20 | 023 | |
| | 25 | | | ing Organization Code | |
| | | | Project | No. 1011 | |
| 7. Author(s) | | | | ing Report No. | |
| Balsley, A., Wurl, M.A., McKinney, I | Κ. | | R&DC | UDI 2009 | |
| 9. Performing Organization Name and Address | | | 10. Work L | Jnit No. (TRAIS) | |
| U.S. Coast Guard | Bureau of Sa | fety and Environmental | | | |
| Research and Development Center | Enforcement | • | 11. Contra | ct or Grant No. | |
| 1 Chelsea Street | 45600 Wood | | | | |
| New London, CT 06320 | Sterling, VA | | | | |
| 12. Sponsoring Organization Name and Address | 6, | | 13. Type o | f Report & Period Covered | |
| COMMANDANT (CG-MER-3) | | | Final | | |
| US COAST GUARD STOP 7516 | | | | | |
| 2703 MARTIN LUTHER KING JR A | VE SE | | | oring Agency Code | |
| WASHINGTON, DC 20593 | | | | andant (CG-MER-3) | |
| | | | | ast Guard Stop 7516 | |
| | | | Washin | gton, DC 20593 | |
| 15. Supplementary Notes | | | | | |
| The R&D Center's technical point of | contact is Mr. | Alexander Balsley, (860) 865-0 | 474; Emai | l: Alexander.Balsley@ | uscg.mil. |
| 16. Abstract (MAXIMUM 200 WORDS) | | | | | |
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| 47. Kay Warda | | 40 Distribution Of standard | | | |
| 17. Key Words | oil amili | 18. Distribution Statement | nround f. | nublic releases distril | tionic |
| Adsorbent, sorbent, Type I, adsorption | - | Distribution Statement A: Ap | proved for | public release; distribu | tion is |
| response, reusability, buoyancy, AST | | unlimited. | | | |
| Ohmsett, water uptake, retrievability, | emerging, | | | | |
| technology, mechanical | | | | | |
| 10. Socurity Close (This Percent) | | 20 Security Class (This Page) | | 21 No of Pages | |
| 19. Security Class (This Report) | | 20. Security Class (This Page) | | 21. No of Pages | 22. Price |
| UNCLAS | | UNCLAS\\Public | | 74 | 1 |



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EXECUTIVE SUMMARY

The U.S. Coast Guard Research and Development Center (USCG RDC) partnered with the Bureau of Safety and Environmental Enforcement Oil Spill Preparedness Division (BSEE OSPD) to execute systematic and unbiased evaluation of four different Type I sorbents with a technology readiness level (TRL) of 6-8¹ that might be of interest to spill responders. According to ASTM F726-17 ("Sorbent Performance of Adsorbents for use on Crude Oil and Related Spills"), Type I sorbents are defined as roll, film, sheet, pad, blanket, and web sorbents (ASTM, 2017). Oil spill responders frequently use this category of sorbents during oil spill incidents nearshore or at smaller spill sites. After RDC executed market research, and with agreement from product suppliers that RDC would make the results public, the project team selected four sorbents for testing at the National Oil Spill Response Research and Renewable Energy Test Facility (Ohmsett).

The project team designed the test setup to provide the most relevant performance data that would predict how a sorbent might perform during typical field use. More specifically, the project team tested the sorbents' maximum oil capacity, adsorption rate, water uptake, buoyancy, retrievability, and in one case, reusability for a manufacturer that provided a recovery system for processing the sorbents for reuse. This limited the reusability testing to AquaFlex[®]. The project team elected to test with diesel fuel and Hydrocal 300 to represent both light and medium oil types that are often recovered with sorbents.

RDC and BSEE OSPD recognize the need for independent evaluations of emerging pollution response technologies. BSEE OSPD recently began its new Testing of Oil Spill Technologies (TOST) initiative to evaluate oil pollution mitigation technologies to provide performance data to stakeholders, including technology developers. It was logical for RDC and BSEE OSPD to collaborate on this effort. Independent evaluations help to ensure that Federal On-Scene Coordinators (FOSCs) and other oil spill responders have access to reliable data about certain technologies before or during an active spill response. This would allow FOSCs to feel more comfortable about implementing newer technologies that may improve the overall response efficiency. The project team consulted with sponsors and stakeholders, and all agreed that evaluating promising Type I sorbents in 2022 would provide useful data to the spill-response community.

This work is a follow-on to RDC's project in 2017 that developed the Oil Spill Response Technology Evaluation Process. Available to the public, it describes a process that oil spill response subject matter experts can follow to thoroughly evaluate a new and emerging mechanical pollution response technology and summarize useful information for FOSCs. The project team used the evaluation guidelines to help select the four sorbents out of eight proposed by vendors.

This report summarizes test results of each sorbent with diesel fuel and Hydrocal 300. The report presents conclusions and recommendations, but is not intended to be guidance for Government procurement strategies as the sorbents' TRL were between 6 and 8. Instead, the report can be used as a knowledge product to inform FOSCs, Oil Spill Removal Organizations (OSROs), Area Committees, Federal/state agencies, and other spill responders on sorbent effectiveness and potential limitations for certain spill scenarios. This report will also help manufacturers better understand technology limitations and encourage product improvement.

¹ BSEE defines TRL 6 as "Full scale prototype demonstrated in relevant environments", TRL 7 as "Integrated technology tested on a large scale or in open water", and TRL 8 as "Final integrated system tested in real or relevant environment" (Panetta & Potter, 2016).



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LIST OF ACRONYMS, ABBREVIATIONS, AND SYMBOLS

| °C | Degree Celsius |
|-------------------------|--|
| ASTM | American Society for Testing and Materials |
| BSEE | Bureau of Safety and Environmental Enforcement |
| CG-MER | Coast Guard Office of Marine Environmental Response Policy |
| cm | Centimeter(s) |
| cm ³ | Cubic centimeter(s) |
| cP | Centipoise(s) |
| EPA | Environmental Protection Agency |
| FOSC | Federal On-Scene Coordinator |
| ft | Feet (imperial measurement) |
| ft^2 | Square feet |
| g | Gram(s) |
| h | Hour(s) |
| i.e., | "Id est"/in other words |
| in | Inch(es) |
| lb | Pound |
| m | Meter(s) |
| min | Minute(s) |
| Ohmsett | National Oil Spill Response & Renewable Energy Test Facility |
| OHM Sponge [™] | Oleophilic, Hydrophobic, Magnetic Sponge TM |
| OSPD | Oil Spill Preparedness Division |
| OSRO | Oil Spill Removal Organization |
| PPE | Personal protective equipment |
| RDC | Research and Development Center |
| RFI | Request for Information |
| S | Second(s) |
| TET | Technical Evaluation Team |
| TOST | Testing of Oil Spill Technologies |
| TRL | Technology Readiness Level |
| USCG | United States Coast Guard |
| | |



1 INTRODUCTION

Mechanical pollution-response technologies are constantly improving, along with tactics and techniques in using them, for a wide range of oil spill incidents in different environments. With better knowledge of new and emerging mechanical-response technologies, U.S. Coast Guard (USCG) Federal On-Scene Coordinators (FOSCs) could respond to oil spills more effectively and with greater efficiency. Currently, USCG FOSCs lack an independent evaluation of these new and emerging technologies, which limits response effectiveness and prevents new tactics from being implemented with a high degree of confidence. Overcoming this could mitigate environmental damage during spills and allow for quicker, return-to-normal operations in the Marine Transportation System.

During large scale responses, spill-response equipment manufacturers frequently approach Incident Commanders with the hope that responders will use their products during an active spill. This is both helpful and burdensome to the command structure and could require responders to first evaluate these technologies to determine the veracity of manufacturers' claims. This diverts the Incident Commanders' attention from the response at hand. It would be cost effective and forward-leaning to have an independent evaluation of these technologies prior to the time of need. In 2017, USCG Research and Development Center (RDC) conducted a project to develop an Oil Spill Response Technology Evaluation Process. Available to the public, the process is a tool that oil spill response experts can follow to thoroughly evaluate new and emerging mechanical pollution response technologies and summarize information for FOSCs.

In a parallel effort, the Bureau of Safety and Environmental Enforcement (BSEE) Oil Spill Preparedness Division (OSPD) began its Testing of Oil Spill Technologies (TOST) effort. The initiative allows BSEE OSPD to evaluate oil pollution mitigation technologies and provide performance data to stakeholders. Through systematic and unbiased testing, BSEE OSPD aims to collect data to facilitate decision making for oil spill preparedness and response operations.

With similar project objectives, RDC and BSEE OSPD formed a partnership and agreed to conduct an independent evaluation of one specific type of new and innovative mechanical recovery equipment. After consulting with their respective stakeholders, RDC and BSEE OSPD agreed that for 2022, the technology of interest would be "Type I" adsorbents (or sorbents²). There is a significant amount of existing sorbent research targeting the use of advanced materials to enhance adsorption rate and/or capacity, the use of natural or recycled materials to reduce the use of plastics, the multiple reuses of sorbents that can reduce secondary waste, and the development of biodegradable materials to provide an environmental benefit. According to ASTM F726-17 ("Sorbent Performance of Adsorbents for use on Crude Oil and Related Spills"), Type I sorbents are defined as roll, film, sheet, pad, blanket, and web sorbents.

Type I sorbents are used during most oil spills, typically in nearshore or other smaller spill sites. For oil spills in marsh environments, using sorbents is often the only feasible response option (Michel, 2022). Sorbent products are some of the most common products that equipment manufacturers approach FOSCs or

 $^{^2}$ In the oil spill response community, a sorbent is an interchangeable term that can be used for adsorbent or absorbent. In this report, the term "sorbent" is used to represent adsorbent only. Adsorption is the process that occurs when a liquid adheres or accumulates on the surface of a solid and the solid does not swell more than 50% in excess liquid. Absorption relies on capillary attraction; capillary force allows liquid to be taken up by the molecular structure within the solid material that causes the solid to swell by 50% or greater. There are many absorbent products currently available to oil spill responders, but they are not the focus of this technology evaluation.



Oil Spill Removal Organizations (OSROs) with during active spills. The RDC/BSEE team determined it would be cost-effective and forward-leaning to have a Government evaluation of these new technologies prior to the time of need.

2 PURPOSE, EXPERIMENTAL SETUP, AND EVALUATION METHODS

From October 17 to 28, 2022, the project team conducted a series of experiments at Ohmsett's chemistry lab and high bay area in accordance with a defined test plan (see APPENDIX B). The project team used the chemistry lab to conduct ASTM F726-17 experiments to collect the sorbents' maximum oil capacity and adsorption rate data for 24 hours (Figure 1).



Figure 1. Ohmsett's chemistry lab used for ASTM F726-17 tests.

The project team used Ohmsett's high bay area and two metal fluid trays to measure volume of water uptake and retrievability (Figure 2). Ohmsett staff equipped one tray with an electric motor and the other without one to assess sorbent performance in mix-energy and static conditions. This area was also used to test the reusability of the AquaFlex®'s Open-Cell® foam sorbent.



Figure 2. Ohmsett's high bay area with two fluid test trays.



The project team tested all sorbent products with diesel fuel and Hydrocal 300 (see 7APPENDIX A for Safety Data Sheet of each fuel). Hydrocal 300 is a naphthenic base oil used extensively at Ohmsett for testing oil spill response products due to its stable properties. Table 1 lists properties of each test fluid.

| Test Fuel | Temperature (°C) | Viscosity (cP) | Density (g/cm ³) | |
|--------------|------------------|----------------|------------------------------|--|
| Hydrocal 300 | 20 | 199.7 | 0.9023 | |
| Diesel | 20 | 9.52 | 0.8460 | |

| Table 1. | Test fuel | characteristics. |
|----------|-----------|------------------|
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The project team considered testing with a more viscous oil, but due to the fact that sorbents are often used in spills of light to medium oil types, the project team determined that diesel fuel and Hydrocal 300 were appropriate test oils.

2.1 Purpose of Sorbent Evaluation

The primary purpose of this effort was to provide performance data on how a sorbent might perform during typical field use. RDC and BSEE OSPD designed the experiments to determine the following characteristics of each sorbent:

- <u>Maximum oil capacity</u>: A volume measurement of oil adsorbed, where sorbent contact with additional oil, or a longer exposure time, will not add to the volume. In field use, it is not likely a sorbent will have sufficient contact time with oil to reach its maximum capacity, but this measurement provides an indication of potential oil recovery. For these tests maximum oil capacity was tested up to 24 hours of adsorption.
- <u>Adsorption rate</u>: A measurement of the time required for a sorbent to reach maximum oil capacity. (See Section 2.3.1.)
- <u>Buoyancy</u>: The ability of the sorbent to remain afloat indicated typically by a portion of the sorbent having freeboard.
- <u>Sorbent water uptake</u>: The volume of water adsorbed when deployed onto an oil-on-water slick.
- <u>Field retrieval</u>: A measure of sorbent tear strength indicating the ability to retrieve it from a spill when fully saturated.
- <u>Reusability</u>: The ability of the sorbent to retain its initial performance after repeated adsorption/desorption cycles using a manual/electric wringer system. *Note: This evaluation parameter applies to AquaFlex*® 's product only.

2.2 Sorbent Selection Process

In late 2021, RDC issued a Request for Information (RFI) as part of its market research effort to collect information on new and innovative sorbents with a technology readiness level (TRL) of 6 to 8 that would be of interest to spill responders. TRLs were defined in the RFI based on those developed specifically for oil spill response technologies and equipment (Panetta and Potter, 2016). The project team (RDC and BSEE OSPD project managers) was interested in sorbents that can provide any advantage over those traditionally used in response operations. The RFI focused both on sorbent innovation as well as how the sorbent would be used in the field, i.e., how it will be deployed and retrieved and how reusable sorbents will be processed. Reusable sorbents would be considered for testing only if the sorbent provider also provided its own sorbent



recovery system. Other requested information included effectiveness with different types of oil, its estimated TRL, previous test data, previous use with actual spills (if any), ease of use, and other criteria related to technical/background information about the sorbent, and the manufacturer's qualifications in the field of oil spill response. The project team received eight white papers from different sorbent manufacturers.

The project established a Technical Evaluation Team (TET) and it executed an evaluation of each white paper against the criteria in USCG's Oil Spill Response Technology Evaluation Process (available at <u>https://discover.dtic.mil</u>, report number CG-D-05-20). The objective was to identify the most promising sorbents (i.e., those that rated highly against the evaluation process criteria) for testing at BSEE's National Oil Spill Response Research & Renewable Energy Test Facility (Ohmsett). The TET selected four sorbent products (Section 2.2.1). Each manufacturer accepted an invitation to have their products tested using the ASTM F726-17 guidelines and the recently developed sorbent field scale test protocol (Section 2.3.1). They also consented to allow RDC and BSEE OSPD to share the Ohmsett test data in this publicly available report. BSEE OSPD funded all associated testing expenses.

2.2.1 Sorbent Products

The project team evaluated the following four sorbent products as shown in Figure 3:



AquaFlex® Open-Cell Foam



Imbibitive Technologies Imbiber Fiber™



Earthwise Sorbents Oil-Only Heavyweight Pads



MFNS Technologies OHM Sponge™

Figure 3. Selected sorbents for Ohmsett testing.

AquaFlex® is a sorbent manufacturer that previously tested its products multiple times at Ohmsett. The Open-Cell foam has been previously used in spills, including Deepwater Horizon in 2010 and thus its TRL is 9. However, its reusability performance had not been independently evaluated and was of interest to the



Acquisition Directorate Research & Development Center project team. The product itself is manufactured from an elastomeric, medical grade polymer (Smith, 2022) and was provided to the project team in a 20" wide roll. AquaFlex® states that it can adsorb a maximum of 32 times its weight in oil, remains buoyant when fully saturated, and performs well in many different environmental scenarios and across a wide range of fuel types (Smith, 2022). AquaFlex® also provided its electric wringer system for the project team's use to evaluate its product's reusability (Figure 4).



Figure 4. Sorbent hand wringer system provided by AquaFlex® to test reusability.

Earthwise Sorbents manufactures products based on recycled and repurposed foam/fabric materials. It repurposes polyurethane wastes from surfboards into sorbent pillows. They also recycle polypropylene wastes from other vendors into melt-blown pads that can be sized to many dimensions (Mullen, 2022). Earthwise Sorbents does not intend for their sorbent pads to be reusable. Based on Earthwise Sorbents' white paper, TET members estimated their product to be TRL 7 as they did not find evidence that Earthwise Sorbents previously tested their products with crude oil. Although their products appeared to be common sorbent pads, the base materials intrigued the TET members enough to warrant Ohmsett testing. Earthwise Sorbents provided its sample to the project team in a 36" wide roll.

Imbibitive Technologies originally submitted a white paper for its Imbiber Beads® product. However, as a loose sorbent, the TET determined it was not qualified as a Type I sorbent. In a resubmittal, Imbibitive Technologies proposed Imbiber FiberTM for their consideration. Imbiber FiberTM is a prototype that has Imbiber Beads® entrained into non-woven melt-blown polypropylene fabric pads sized 12" x 15" (Brinkman, 2022a). It has many similar properties as Imbiber Beads® and the beads, made of solid polymer structure, are claimed to "swell" two to three times their original size (Brinkman, 2022b)³. Imbibitive Technologies also indicates that their product can perform in a wide variety of environmental conditions and across several oil types (Brinkman, 2022b). TET members estimated its TRL to be 6 to 7.

Imbibitive Technologies sent three different prototypes to Ohmsett, each containing different concentrations of Imbiber Beads[®]. The available concentrations were 30 grams of Imbiber Beads[®] per square feet (ft²) of polypropylene fabric pad, 56 g/ft², and 70 g/ft². Since the project team requested only one type of sorbent sample from each manufacturer, Imbibitive Technologies' representative selected their 30-g/ft² sample to be tested.

³ TET members accepted Imbibitive Technologies' resubmittal on the basis that Imbiber Beads®, an *absorbent*, would be fully entrained within polypropylene fabric pads. TET members determined this new design warranted further consideration and testing at Ohmsett along with other adsorbents.



MFNS Technologies developed its Oleophilic, Hydrophobic, and Magnetic Sponge (OHM Sponge[™]) to be able to selectively adsorb oil from any mixture of oil and water. MFNS Technologies prepares the OHM Sponge[™] by coating a nanocomposite (made of Fe₃O₄ magnetic nanostructures and graphite) layer on a base polyurethane sponge, which allows it to adsorb up to 30 times its weight in oil according to MFNS Technologies (Dravid et al., 2022). This dip-coating technology is compatible with any size/shape of sponge. Currently, MFNS Technologies is considering manufacturing "typical sorbent sizes" to be used for cleanup (e.g., 12" x 12" x 0.5"). They also claim that OHM Sponge[™] can adsorb all types of oil in a variety of environmental conditions, including fresh and salt water. MFNS Technologies states that the OHM Sponge[™] is reusable and can be hand wrung or wrung with a mechanical wringer system (Dravid, 2023). However, reusability was not tested because a recovery system was not provided. TET members estimated its TRL to be 6. MFNS Technologies provided multiple samples sized at 6.0" x 11.5" x 0.5".

2.3 Evaluation Methods

2.3.1 ASTM F726-17 Standard (Maximum Oil Capacity and Buoyancy)

The RDC and BSEE OSPD team used the ASTM F726-17 standard to evaluate each sorbent's maximum oil capacity and buoyancy. The standard includes the Oil Adsorption-Short test (15-minute adsorption) and the Oil Adsorption-Long test (24-hour adsorption). The sorbent's oil capacity at the 24-hour mark represents its maximum oil capacity. The standard does not include quantitative testing of sorbents in oil and water. In previous sorbent experiments following the ASTM 726-17 standard, BSEE OSPD noted that most sorbents reached near-maximum oil capacity in a few hours or less. In response, the project team added a 3-hour adsorption test, not included in the standard, to provide additional data about the sorbent's oil capacity between 15 minutes and 24 hours.

With three data points about the sorbent capacity over 24 hours, the project team could determine each sorbent's *adsorption rate category*. BSEE OSPD developed adsorption rate categories (Table 2) to make it easier for responders or researchers to understand how fast the sorbent can be expected to adsorb different types of oil or fuel. RDC and BSEE OSPD project managers discussed how the adsorption rate category should be applied to a sorbent based on three data points. They agreed that a sorbent is considered to have reached near-maximum adsorption when the change in the average ratio of oil mass to dry sorbent mass between subsequent measurements is 10% or less. For example, a sorbent that receives Category I has less than 10% increase in the average oil mass to dry sorbent mass ratio between 0.25 hour and 3-hour oil capacity measurements.

| Adsorption Rate Category | Time to Near-Maximum Oil Capacity (hours) |
|--------------------------|---|
| 1 | 0.25 or less |
| II | Greater than 0.25 and less than 3.0 |
| III | Greater than 3.0 |

| Table 2. | Adsorption | rate categories. |
|----------|------------|------------------|
|----------|------------|------------------|

The categories are newly defined and not standardized. While the adsorption rate category is not specifically called out in ASTM F726-17, it is a useful metric to determine how quickly an adsorbent reaches near-maximum oil capacity.



The project team tested each sorbent sample in aluminum trays, one filled with pure diesel and the other filled with Hydrocal 300.The project team followed ASTM F726-17 procedures including proper sorbent-sample sizing (13 cm x 13 cm) (Figure 5), conditioning of the sorbents, 30-second drip time, and triplicate testing.



Figure 5. Cutting sorbent sample to proper ASTM size (13 cm x 13 cm).

From ASTM 726-17, the sorbent oil capacity (or oil adsorbency⁴) is calculated as the ratio of the total mass of oil adsorbed to the sorbent's dry weight as shown in Equation 1 and is measured as gram over gram (g/g).

Equation 1. Oil adsorbency

$$oil \ adsorbency = \frac{S_{ST} - S_0}{S_0}$$

where S_{ST} is the weight of sorbent sample at the end of each test run and S_0 is the initial dry sorbent weight.

Throughout the ASTM F726-17 tests as well as sorbent water uptake tests (which is not included in the standard) the project team recorded qualitative observations about the buoyancy of each sorbent sample. In real-world spill scenarios, the sorbent's ability to maintain buoyancy during oil uptake is important to its effectiveness and for responders to be able to locate and retrieve it.

2.3.2 Sorbent Water Uptake

Another aspect of this sorbent evaluation was to determine each sample's ability to selectively adsorb oil in the presence of water. There is no existing ASTM test standard to quantify a sorbent's oil capacity when exposed to both oil and water. Several years ago, Ohmsett/BSEE developed a field-scale test method called

⁴ Oil capacity and oil adsorbency are used interchangeably in ASTM F726-17



"Sorbent Water Uptake Tests with Oil" that intentionally calls for a relatively thin oil slick on the water surface. This setup exposes a sorbent sample to an oil volume that is less than the sorbent's known maximum capacity, ensuring that the sorbent contacts both oil and water. The project team used filtered water from Ohmsett's main tank with a measured salinity of 27 parts per thousand.

For each test, Ohmsett staff calculated the initial oil volume by halving the sorbent's maximum oil capacity (by volume) as determined by the ASTM F726 Oil Adsorption – Long Test (24-hours) result. This volume was then scaled to appropriately match each sorbent size tested during this set of tests. Ohmsett staff added water and this oil volume to two aluminum sheet metal test trays (1 m x 1 m x 0.2 m), creating a thin surface slick. Ohmsett staff then placed dry sorbent samples on a sample rack made of aluminum frames and thin gauge wire mesh. This was connected to a load cell (Figure 6) to record their weight, and then lowered it into the tray with oil and water. Once 15 minutes passed, the team lifted the rack from the tray and allowed the sorbents to drip for 30 seconds. After recording the sorbents' total fluid weight, the team lowered the rack back into the tray for another 15-minute, 30-second drip cycle. After recording weight for the second time, the team returned the sorbent to the oil/water mixture for 30 additional minutes for a total oil/water adsorption time of 60 minutes.



Figure 6. Left: Hoist-mounted load cell read-out configuration for raising/lowering the support rack. Right: Digital read-out display.

If the change in total fluid weight between the 30-minute and 60-minute marks was greater than 5 percent, the project team returned the sorbents back into the tray for an additional 30 minutes. It repeated this process until the change between measurements was less than 5 percent. The project team agreed that 5 percent was a reasonable value to indicate that the sorbent had reached maximum fluid capacity given the time it took to conduct each test.

The project team was interested in learning how each sorbent sample performed in the water uptake tests in both static/quiescent and mix-energy conditions (Figure 7). To achieve mix-energy conditions, the team connected one of the test trays to an eccentric drive, a variable speed right-angle gear reduction that allows for controlled speed in the range of 30-80 cycles per minute. The drive roller with an eccentricity of ½-inch provided a 1-inch overall stroke distance. During testing, Ohmsett staff maintained the speed at approximately 60 cycles per minute, yielding surface waves approximately ½ inch high. The other test tray was left undisturbed.





Figure 7. Left: Two test trays for water uptake tests. Right: Eccentric drive used for mix-energy conditions.

The project team was interested in learning about each sorbent's ratio of oil adsorbed to the sorbent's dry weight as well as the amount of water adsorbed. Calculating both outcomes required the project team to measure the volume of oil not adsorbed (i.e., the oil that remained in the test tray at the very end of a test run) (Coolbaugh and McKinney, 2021). Equation 2 shows the simple calculation.

Equation 2. Volume of oil in sorbent.

$$V_S = V_i - V_T$$

Where V_S is volume of oil in the sorbent, V_i is initial oil volume, and V_T is oil volume measured in the tray.

The project team calculated the oil weight in the sorbent using the oil/diesel fuel's known densities from Table 1. With the sorbent's dry weight, weight of oil adsorbed by the sorbent, and final weight of all fluid adsorbed, the team calculated the water weight adsorbed (Equation 3).

Equation 3. Weight of water in sorbent.

$$S_W = S_f - S_O - S_d$$

Where S_W is the weight of water in the sorbent, S_f is the final measured weight of the sorbent with oil and water, S_0 is the weight of oil adsorbed by the sorbent, and S_d is the sorbent's dry weight.

Once the project team determined the weight of water in the sorbent, it could then calculate the ratio of the adsorbed oil to the sorbent's dry weight, which indicates its performance in the presence of water. The project team was also able to compare the amount of oil and water picked up by the same sorbent to determine how oleophilic or hydrophobic it was. This represents a more realistic adsorption value given that responders are concerned with responding to oil spills that enter natural water bodies.

2.3.3 Retrievability

The ability of oil spill responders to easily retrieve sorbents from the field is an important consideration for effective response operations. Responders need the sorbents to be highly visible during recovery (i.e., floating) and to be able to remain structurally intact during the retrieval process. Using a pitchfork is a common approach for retrieving sorbents; responders may use one to either pick up a sorbent from underneath or by penetration.



At Ohmsett, the project team designed the retrievability test to be destructive. It decided to impose the most stringent yet practical approach of retrieving fully saturated sorbents (with water and oil) by using a single, typical pitchfork prong formed into a hook and suspended on a load cell (Figure 8). The objective was to first assess if a fully adsorbed sorbent would tear under its own weight when lifted out of the fluid on the hook. If a sorbent did not remain intact under its own weight it was considered to fail this test. If it remained intact, the project team recorded the load at which the sorbent would fail by pulling on it straight down with both hands.



Figure 8. Retrievability test with load cell measuring tear force.

2.3.4 Reusability (AquaFlex® only)

The final aspect of the project team's evaluation was to determine a sorbent's reusability, or how well it can recover, retain, and allow oil extraction through a wringer system after repeated uses. However, this test was limited to AquaFlex®'s Open-Cell Foam only since it was the only manufacturer that elected to provide its own wringer system called the ECO HydroFlexTM (Figure 9). As part of an agreement between RDC and AquaFlex®, RDC did not allow other samples to be tested with the ECO HydroFlexTM.



Figure 9. ECO HydroFlexTM, an electric wringer system provided by AquaFlex[®].



The project team measured the Open-Cell Foam's reusability performance by taking a similar approach with the sorbent water uptake tests. The team put the sample into the aluminum test tray with oil and water in static conditions to allow uptake of oil over 15 to 20 minutes. After a 30-second drip time, Ohmsett staff weighed the sample, ran it through the ECO HydroFlex[™] system twice (Figure 10), and then weighed the sample again. The team used a squeegee to collect all fluids wrung from the sample in the wringer tray and measured both oil and water volume. The team then poured the extracted fluids back into the test tray for another adsorption/desorption cycle. The project team aimed for at least 10 cycles per sample with diesel fuel and water, Hydrocal 300 and water, pure diesel fuel, and pure Hydrocal 300.



Figure 10. AquaFlex® sorbent sample tested for reusability performance.

The project team also made qualitative observations about ECO HydroFlexTM's ease of use and whether it was feasible to use in the field during an actual spill response incident.

3 FINDINGS

3.1 ASTM F726-17 Standard (Maximum Oil Capacity and Buoyancy)

3.1.1 AquaFlex®

AquaFlex®'s Open-Cell Foam was successfully tested in accordance with the ASTM standard without any samples needing repeat tests. Table 3 and Table 4 show AquaFlex®'s adsorption results over 15 minutes, 3 hours, and 24 hours and its adsorption rate category for both diesel and Hydrocal 300.



| | 15 MINUTE - SHORT TEST | | | | | | | | |
|-----------------|------------------------|-------------------------------|------------------------------|--------------------------------------|--------------------------------------|------------------|--------------------|--|--|
| OIL TYPE | OIL TEMP (°C) | SORBENT SAMPLE MASS (g) | SORBENT & OIL MASS (g) | OIL MASS / SAMPLE MASS (g / g) | THREE- TEST AVERAGE (g / g) | DEVIATION (%) | FLOATING / SUNK | | |
| Hydrocal 300 | 21.8 | 4.14 | 51.81 | 11.51 | 12.14 | -5.16 | Floating | | |
| Hydrocal 300 | 21.8 | 4.19 | 55.71 | 12.30 | 12.14 | 1.28 | Floating | | |
| Hydrocal 300 | 21.8 | 4.12 | 56.08 | 12.61 | 12.14 | 3.88 | Floating | | |
| Diesel | 22.4 | 4.24 | 90.47 | 20.34 | 20.35 | -0.07 | Floating | | |
| Diesel | 22.4 | 4.27 | 89.80 | 20.03 | 20.35 | -1.58 | Floating | | |
| Diesel | 22.4 | 4.20 | 91.08 | 20.69 | 20.35 | 1.64 | Floating | | |
| | | 3 H | OUR - CUSTC | M INTERVA | L TEST | | | | |
| OIL TYPE | OIL TEMP (°C) | SAMPLE MASS (g) | SORBENT & OIL MASS (g) | OIL MASS / SAMPLE MASS (g / g) | THREE- TEST AVERAGE (g / g) | DEVIATION (%) | FLOATING / SUNK | | |
| Hydrocal 300 | 22.7 | 4.14 | 87.61 | 20.16 | 20.65 | -2.36 | Floating | | |
| Hydrocal 300 | 22.7 | 4.19 | 94.95 | 21.66 | 20.65 | 4.90 | Floating | | |
| Hydrocal 300 | 23.0 | 4.12 | 87.03 | 20.12 | 20.65 | -2.54 | Floating | | |
| Diesel | 24.3 | 4.24 | 95.34 | 21.49 | 21.65 | -0.75 | Floating | | |
| Diesel | 24.3 | 4.27 | 95.79 | 21.43 | 21.65 | -0.99 | Floating | | |
| Diesel | 24.3 | 4.20 | 96.71 | 22.03 | 21.65 | 1.74 | Floating | | |
| | | | 24 HOUR - | LONG TEST | | | | | |
| OIL TYPE | OIL TEMP (°C) | SAMPLE MASS (g) | SORBENT & OIL MASS (g) | OIL MASS / SAMPLE MASS (g / g) | THREE- TEST AVERAGE (g / g) | DEVIATION (%) | FLOATING / SUNK | | |
| Hydrocal 300 | 21.1 | 4.14 | 93.63 | 21.62 | 22.13 | -2.30 | Floating | | |
| Hydrocal 300 | 21.1 | 4.19 | 101.69 | 23.27 | 22.13 | 5.17 | Floating | | |
| Hydrocal 300 | 20.9 | 4.12 | 92.66 | 21.49 | 22.13 | -2.87 | Floating | | |
| Diesel | 21.2 | 4.24 | 98.99 | 22.35 | 22.52 | -0.75 | Floating | | |
| Diesel | 21.2 | 4.27 | 99.38 | 22.27 | 22.52 | -1.08 | Floating | | |
| Diesel | 21.2 | 4.20 | 100.50 | 22.93 | 22.52 | 1.83 | Floating | | |

Table 3. ASTM F726-17 test results for AquaFlex®.



| OIL TYPE | MAXIMUM OIL CAPACITY (g/g) | CHANGE BETWEEN 15- MIN AND 3-HR AVERAGES (%) | CHANGE BETWEEN 3- HR AND 24-HR AVERAGES (%) | NEAR- MAXIMUM OIL CAPACITY (g/g) | TIME TO "NEAR- MAXIMUM" OIL CAPACITY (hour) | ADSORPTION RATE CATEGORY |
|-----------------|-------------------------------------|--|---|--|--|--------------------------------|
| Hydrocal 300 | 22.13 | 70.1 | 7.17 | 20.65 | >0.25 and ≤3.0 | II |
| Diesel | 22.52 | 6.39 | 4.02 | 20.35 | ≤0.25 | I |

Table 4. Adsorption rate category for AquaFlex® samples by oil type.

AquaFlex® showed similar oil capacity for both Hydrocal 300 and diesel fuel, except that in the first 15 minutes, there was slower adsorption rate of Hydrocal 300 compared with that of diesel fuel (12.14 g/g and 20.35 g/g, respectfully). AquaFlex®'s samples' capacity at the 3-hour mark with Hydrocal 300 averaged 20.65 g/g. Between 3 and 24 hours, the average capacity of the samples increased by only 7.17% to a maximum oil capacity of 22.13 g/g. AquaFlex®'s adsorption rate category with Hydrocal 300 is II.

AquaFlex®'s samples recovering diesel reached 20.35 g/g capacity after just 15 minutes and adsorbed an additional 6.39% between 15 minutes and 3 hours making them an adsorption rate category I for diesel recovery. The average capacity increased by an additional 4.02% to a maximum oil capacity of 22.52 g/g between 3 hours and 24 hours. Figure 11 shows the AquaFlex® sample floating in the tray with diesel fuel.

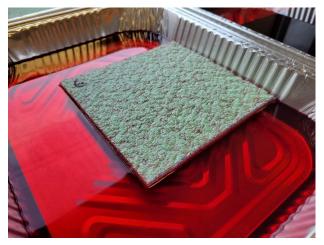


Figure 11. AquaFlex® sample in diesel oil for ASTM F726-17 test.

The project team noted that AquaFlex® samples remained buoyant for the entire 24-hour test period in both Hydrocal 300 and diesel fuel.

3.1.2 Earthwise Sorbents

Table 5 and Table 6 show the ASTM results of the six Earthwise Sorbents Oil-Only Heavyweight Pad samples and their adsorption rate categories for both diesel and Hydrocal 300. The tables do not include the results of Earthwise Sorbents' first test run with Hydrocal 300 because the adsorption capacity of one sample pad deviated by more than 15% (16.05% in this case) from the mean value of the three runs. If the deviation value is greater than 15%, the ASTM standard requires a replicate test with three new samples. During the second ASTM test with Hydrocal 300, all three samples' adsorption capacities were within the



Acquisition Directorate Research & Development Center 15% deviation and thus acceptable. Earthwise Sorbents did not need a repeat test with diesel fuel as all values were within acceptable range, although one sample showed higher oil capacity than the other two.

| 15 MINUTE – SHORT TEST | | | | | | | | | | |
|------------------------|------------------|-------------------------------|---------------------------|--------------------------------------|--------------------------------------|------------------|--------------------|--|--|--|
| OIL TYPE | OIL TEMP (°C) | SORBENT SAMPLE MASS (g) | SORBENT & OIL MASS (g) | OIL MASS / SAMPLE MASS (g / g) | THREE- TEST AVERAGE (g / g) | DEVIATION (%) | FLOATING / SUNK | | | |
| Hydrocal 300 | 20.2 | 4.86 | 47.86 | 8.85 | 8.97 | -1.34 | Floating | | | |
| Hydrocal 300 | 20.2 | 4.60 | 47.15 | 9.25 | 8.97 | 3.14 | Floating | | | |
| Hydrocal 300 | 20.2 | 5.23 | 51.29 | 8.81 | 8.97 | -1.80 | Floating | | | |
| Diesel | 22.4 | 4.78 | 39.93 | 7.35 | 6.71 | 9.54 | Floating | | | |
| Diesel | 22.4 | 4.45 | 33.04 | 6.42 | 6.71 | -4.29 | Floating | | | |
| Diesel | 22.0 | 4.83 | 35.55 | 6.36 | 6.71 | -5.25 | Floating | | | |
| | • | 3 H | IOUR - CUST | OM INTERVAL | TEST | | | | | |
| OIL TYPE | OIL TEMP (°C) | SAMPLE MASS (g) | SORBENT & OIL MASS (g) | OIL MASS / SAMPLE MASS (g / g) | THREE- TEST AVERAGE (g / g) | DEVIATION (%) | FLOATING / SUNK | | | |
| Hydrocal 300 | 22.3 | 4.86 | 48.10 | 8.90 | 8.92 ⁵ | -0.22 | Floating | | | |
| Hydrocal 300 | 22.3 | 4.60 | 47.32 | 9.29 | 8.92 | 4.15 | Floating | | | |
| Hydrocal 300 | 22.6 | 5.23 | 50.03 | 8.57 | 8.92 | -3.93 | Floating | | | |
| Diesel | 23.4 | 4.78 | 45.63 | 8.55 | 7.60 | 12.45 | Floating | | | |
| Diesel | 23.4 | 4.45 | 35.76 | 7.04 | 7.60 | -7.42 | Floating | | | |
| Diesel | 23.2 | 4.83 | 39.69 | 7.22 | 7.60 | -5.03 | Floating | | | |
| | - | - | 24 HOUR - | - LONG TEST | - | - | | | | |
| OIL TYPE | OIL TEMP (°C) | SAMPLE MASS (g) | SORBENT & OIL MASS (g) | OIL MASS / SAMPLE MASS (g / g) | THREE- TEST AVERAGE (g / g) | DEVIATION (%) | FLOATING / SUNK | | | |
| Hydrocal 300 | 20.1 | 4.86 | 49.77 | 9.24 | 9.28 | -0.47 | Floating | | | |
| Hydrocal 300 | 20.1 | 4.60 | 48.41 | 9.52 | 9.28 | 2.58 | Floating | | | |
| Hydrocal 300 | 20.0 | 5.23 | 52.76 | 9.09 | 9.28 | -2.11 | Floating | | | |
| Diesel | 21.2 | 4.78 | 46.89 | 8.81 | 7.89 | 11.68 | Floating | | | |
| Diesel | 21.2 | 4.45 | 37.55 | 7.44 | 7.89 | -5.70 | Floating | | | |
| Diesel | 21.1 | 4.83 | 40.65 | 7.42 | 7.89 | -5.98 | Floating | | | |

Table 5. ASTM F726-17 test results for Earthwise Sorbents.

⁵ Between 15 minutes and 3 hours, one sample's mass decreased from 51.29 g to 50.03 g. One possible explanation is that the drip time at the 3-hour mark may have been slightly longer than the drip time at the 15-minute mark by a few seconds. However, all samples were within 15% of the mean and thus acceptable according to ASTM F726-17.



| OIL TYPE | MAXIMUM OIL CAPACITY (g/g) | CHANGE BETWEEN 15- MIN AND 3-HR AVERAGES (%) | CHANGE BETWEEN 3- HR AND 24-HR AVERAGES (%) | NEAR- MAXIMUM OIL CAPACITY (g/g) | TIME TO "NEAR- MAXIMUM" OIL CAPACITY (hour) | ADSORPTION RATE CATEGORY |
|-----------------|-------------------------------------|---|--|--|--|--------------------------------|
| Hydrocal 300 | 9.28 | -0.56 | 4.04 | 8.97 | ≤0.25 | I |
| Diesel | 7.89 | 13.3 | 3.82 | 7.60 | >0.25 and ≤3.0 | II |

Table 6. Adsorption rate category for Earthwise Sorbents' samples by oil type.

With Hydrocal 300 and diesel fuel, the Oil-Only Heavyweight Pad samples showed an average maximum oil capacity of 9.28 g/g and 7.89 g/g, respectively. With Hydrocal 300, Earthwise Sorbents appeared to reach near-maximum capacity in the first 15 minutes, with an average value of 8.97 g/g. Between 15 minutes and 24 hours, this value increased by 3.46% to 9.28 g/g. Earthwise Sorbents' adsorption rate category is I for Hydrocal 300 (Figure 12).



Figure 12. Oil-Only Heavyweight Pad sample floating in tray during ASTM F726-17 tests with Hydrocal 300.

The Oil-Only Heavyweight Pad samples showed lower oil capacity and slightly slower adsorption rate with diesel fuel, with an average value of 6.71 g/g after the first 15 minutes. After 24 hours, the samples averaged 7.89 g/g. The slightly slower adsorption rate gave the Oil-Only Heavyweight Pads an adsorption rate category II for diesel.

3.1.3 Imbibitive Technologies

The project team tested with Imbibitive Technologies' Imbiber Fiber[™] prototype at the 30 g/ft² concentration of Imbiber Beads[®]. Table 7 does not include results from the first run with Hydrocal 300 due to two samples deviating from the mean oil capacity at the 15-minute and 3-hour marks by approximately 20%. This is greater than the 15% deviation allowed in the ASTM F726-17 standard. The second run yielded acceptable results. Table 8 shows the adsorption rate categories for both diesel and Hydrocal 300.



| | Table 7. ASTMT720-17 test results for minibilitive recimiologies (30 g/t pad). | | | | | | | | | | |
|-----------------|--|-------------------------------|---------------------------|--------------------------------------|--------------------------------------|------------------|--------------------|--|--|--|--|
| | 15 MINUTE - SHORT TEST | | | | | | | | | | |
| OIL TYPE | OIL TEMP (°C) | SORBENT SAMPLE MASS (g) | SORBENT & OIL MASS (g) | OIL MASS / SAMPLE MASS (g / g) | THREE- TEST AVERAGE (g / g) | DEVIATION (%) | FLOATING / SUNK | | | | |
| Hydrocal 300 | 20.2 | 10.68 | 60.58 | 4.67 | 4.52 | 3.43 | Floating | | | | |
| Hydrocal 300 | 20.2 | 11.99 | 62.43 | 4.21 | 4.52 | -6.88 | Floating | | | | |
| Hydrocal 300 | 20.2 | 10.86 | 61.61 | 4.67 | 4.52 | 3.45 | Floating | | | | |
| Diesel | 20.0 | 11.18 | 122.56 | 9.96 | 9.62 | 3.54 | Sunk | | | | |
| Diesel | 20.0 | 13.86 | 140.20 | 9.12 | 9.62 | -5.26 | Sunk | | | | |
| Diesel | 20.0 | 12.64 | 136.35 | 9.79 | 9.62 | 1.72 | Sunk | | | | |
| | | 3 HO | UR - CUSTO | M INTERVAL | TEST | | | | | | |
| OIL TYPE | OIL TEMP (°C) | SAMPLE MASS (g) | SORBENT & OIL MASS (g) | OIL MASS / SAMPLE MASS (g / g) | THREE- TEST AVERAGE (g / g) | DEVIATION (%) | FLOATING / SUNK | | | | |
| Hydrocal 300 | 22.6 | 10.68 | 66.67 | 5.24 | 5.22 | 0.43 | Floating | | | | |
| Hydrocal 300 | 22.6 | 11.99 | 73.11 | 5.10 | 5.22 | -2.34 | Floating | | | | |
| Hydrocal 300 | 22.7 | 10.86 | 68.63 | 5.32 | 5.22 | 1.91 | Floating | | | | |
| Diesel | 21.0 | 11.18 | 138.76 | 11.41 | 11.11 | 2.71 | Floating | | | | |
| Diesel | 21.0 | 13.86 | 161.31 | 10.64 | 11.11 | -4.25 | Floating | | | | |
| Diesel | 21.0 | 12.64 | 155.23 | 11.28 | 11.11 | 1.54 | Floating | | | | |
| | | | 24 HOUR - | LONG TEST | | | | | | | |
| OIL TYPE | OIL TEMP (°C) | SAMPLE MASS (g) | SORBENT & OIL MASS (g) | OIL MASS / SAMPLE MASS (g / g) | THREE- TEST AVERAGE (g / g) | DEVIATION (%) | FLOATING / SUNK | | | | |
| Hydrocal 300 | 20.1 | 10.68 | 106.31 | 8.95 | 8.92 | 0.41 | Floating | | | | |
| Hydrocal 300 | 20.1 | 11.99 | 114.65 | 8.56 | 8.92 | -3.98 | Floating | | | | |
| Hydrocal 300 | 20.1 | 10.86 | 111.16 | 9.24 | 8.92 | 3.57 | Floating | | | | |
| Diesel | 20.3 | 11.18 | 142.70 | 11.76 | 11.57 | 1.67 | Floating | | | | |
| Diesel | 20.3 | 13.86 | 172.28 | 11.43 | 11.57 | -1.21 | Floating | | | | |
| Diesel | 20.3 | 12.64 | 158.21 | 11.52 | 11.57 | -0.46 | Floating | | | | |

Table 7. ASTM F726-17 test results for Imbibitive Technologies (30 g/ft² pad).



| OIL TYPE | MAXIMUM OIL CAPACITY (g/g) | CHANGE BETWEEN 15- MIN AND 3-HR AVERAGES (%) | CHANGE BETWEEN 3- HR AND 24-HR AVERAGES (%) | NEAR- MAXIMUM OIL CAPACITY (g/g) | TIME TO "NEAR- MAXIMUM" OIL CAPACITY (hour) | ADSORPTION RATE CATEGORY |
|--------------|-------------------------------------|---|--|--|--|--------------------------------|
| Hydrocal 300 | 8.92 | 15.5 | 70.9 | 8.92 | >3.0 | III |
| Diesel | 11.57 | 15.5 | 4.14 | 11.11 | >0.25 and ≤3.0 | II |

Table 8. Adsorption rate category for Imbibitive Technologies samples by oil type.

The results show that the Imbiber Fiber[™] prototype had greater affinity for diesel fuel than Hydrocal 300, and that there was relatively slow oil uptake rate over the 24-hour period. With Hydrocal 300, the Imbiber Fiber[™] prototype's oil capacity averaged 4.52 g/g in the first 15 minutes. Between 15 minutes and 3 hours, the prototype samples' capacity increased by 15.5% to 5.22 g/g. At the 24-hour mark, the maximum oil capacity average was 8.92 g/g, an increase of 97.3% from the 15-minute result making the Imbiber Fiber[™] sorbent a category III adsorption rate when recovering Hydrocal 300.

With diesel fuel, the Imbiber FiberTM prototype appeared to reach near-maximum capacity after 3 hours with 11.11 g/g making it a category II adsorption rate. Between 3 hours and 24 hours, the oil capacity increased by only 4.14% from 11.11 g/g to 11.57 g/g.

The project team noted that in the first 15 minutes with diesel fuel, the Imbiber FiberTM prototype appeared to have sunk (Figure 13) but regained buoyancy at the 3-hour and 24-hour marks. In Hydrocal 300, the samples remained floating for the entire 24-hour period.



Figure 13. Imbiber Fiber[™] prototype sample undergoing 30-second drip time before being weighed in accordance with ASTM F726-17 standard.

3.1.4 MFNS Technologies

MFNS Technologies' OHM Sponge[™] appeared to perform very well with Hydrocal 300 but less so with diesel fuel. In the first 15 minutes, the OHM Sponge[™] adsorbed an average of 36.40 g/g of Hydrocal 300 (Table 9). After 24 hours, the oil capacity increased by only 1.73% to a value of 37.03 g/g, indicating that the sorbent reached near-maximum capacity in the first 15 minutes (Table 10). The project team also noted



that OHM Sponge[™] reached near-maximum capacity with diesel fuel after 15 minutes although it adsorbed much less compared to Hydrocal 300 with 11.99 g/g. After 24 hours, the sorbent's capacity with diesel fuel increased by 6.76% to a maximum oil capacity of 12.80 g/g. At the 3-hour mark, OHM Sponge[™] in both Hydrocal 300 and diesel fuel were similar to the adsorption numbers at the 24-hour mark. OHM Sponge[™] is classified as a category I adsorption rate for both diesel and Hydrocal 300.

| 15 MINUTE - SHORT TEST | | | | | | | | | | |
|------------------------|------------------|-------------------------------|---------------------------|--------------------------------------|--------------------------------------|------------------|--------------------|--|--|--|
| OIL TYPE | OIL TEMP (°C) | SORBENT SAMPLE MASS (g) | SORBENT & OIL MASS (g) | OIL MASS / SAMPLE MASS (g / g) | THREE- TEST AVERAGE (g / g) | DEVIATION (%) | FLOATING / SUNK | | | |
| Hydrocal 300 | 21.8 | 4.68 | 176.19 | 36.65 | 36.40 | 0.67 | Sunk | | | |
| Hydrocal 300 | 21.8 | 4.66 | 175.69 | 36.70 | 36.40 | 0.82 | Sunk | | | |
| Hydrocal 300 | 21.8 | 4.94 | 182.11 | 35.86 | 36.40 | -1.48 | Sunk | | | |
| Diesel | 22.4 | 4.91 | 61.61 | 11.55 | 11.99 | -3.72 | Sunk | | | |
| Diesel | 22.4 | 4.66 | 62.60 | 12.43 | 11.99 | 3.67 | Sunk | | | |
| Diesel | 22.7 | 4.59 | 59.67 | 12.00 | 11.99 | 0.05 | Sunk | | | |
| | | 3 H | IOUR - CUSTO | M INTERVAL | TEST | | | | | |
| OIL TYPE | OIL TEMP (°C) | SAMPLE MASS (g) | SORBENT & OIL MASS (g) | OIL MASS / SAMPLE MASS (g / g) | THREE- TEST AVERAGE (g / g) | DEVIATION (%) | FLOATING / SUNK | | | |
| Hydrocal 300 | 23.2 | 4.68 | 175.60 | 36.52 | 36.37 | 0.41 | Sunk | | | |
| Hydrocal 300 | 23.2 | 4.66 | 176.36 | 36.85 | 36.37 | 1.30 | Sunk | | | |
| Hydrocal 300 | 23.4 | 4.94 | 181.56 | 35.75 | 36.37 | -1.71 | Sunk | | | |
| Diesel | 24.3 | 4.91 | 62.68 | 11.77 | 12.02 | -2.15 | Sunk | | | |
| Diesel | 24.3 | 4.66 | 60.91 | 12.07 | 12.02 | 0.39 | Sunk | | | |
| Diesel | 24.3 | 4.59 | 60.75 | 12.24 | 12.02 | 1.76 | Sunk | | | |
| | | | 24 HOUR - | LONG TEST | • | | | | | |
| OIL TYPE | OIL TEMP (°C) | SAMPLE MASS (g) | SORBENT & OIL MASS (g) | OIL MASS / SAMPLE MASS (g / g) | THREE- TEST AVERAGE (g / g) | DEVIATION (%) | FLOATING / SUNK | | | |
| Hydrocal 300 | 20.9 | 4.68 | 179.01 | 37.25 | 37.03 | 0.60 | Sunk | | | |
| Hydrocal 300 | 20.9 | 4.66 | 178.62 | 37.33 | 37.03 | 0.82 | Sunk | | | |
| Hydrocal 300 | 20.7 | 4.94 | 185.24 | 36.50 | 37.03 | -1.43 | Sunk | | | |
| Diesel | 21.2 | 4.91 | 68.28 | 12.91 | 12.80 | 0.85 | Sunk | | | |
| Diesel | 21.2 | 4.66 | 65.29 | 13.01 | 12.80 | 1.66 | Sunk | | | |
| Diesel | 21.2 | 4.59 | 61.86 | 12.48 | 12.80 | -2.51 | Sunk | | | |

Table 9. ASTM F726-17 test results for MFNS Technologies.



| OIL TYPE | MAXIMUM OIL CAPACITY (g/g) | CHANGE BETWEEN 15- MIN AND 3-HR AVERAGES (%) | CHANGE BETWEEN 3- HR AND 24-HR AVERAGES (%) | NEAR- MAXIMUM OIL CAPACITY (g/g) | TIME TO "NEAR- MAXIMUM" OIL CAPACITY (hour) | ADSORPTION RATE CATEGORY |
|-----------------|-------------------------------------|---|--|--|--|--------------------------------|
| Hydrocal 300 | 37.03 | -0.082 | 1.81 | 36.4 | ≤0.25 | I |
| Diesel | 12.8 | 0.25 | 6.49 | 11.99 | ≤0.25 | Ι |

Table 10. Adsorption rate category for MFNS Technologies samples by oil type.

In all ASTM tests, the OHM Sponge[™] samples were either partially submerged or fully submerged as shown in Figure 14.



Figure 14. MFNS Technologies' OHM Sponge[™] partially sunken in tray filled with Hydrocal 300.

3.2 Sorbent Water Uptake

Table 11 and Figure 15 show the main findings of all sorbents' water uptake tests with diesel fuel in both static and mix-energy conditions. The table lists each sample's dry weight, the weight of diesel fuel dispensed, the weight of diesel and water that each sorbent took in, the percent water in the total fluid adsorbed, and the ratio of oil adsorbed to the dry sorbent weight. The sorbent samples are listed in alphabetical order. Figure 16 shows a sorbent (Earthwise Sorbents' Oil-Only Heavyweight Pads) undergoing a typical water uptake test in static conditions and Figure 17 a typical sorbent water uptake test with mix-energy conditions (with OHM SpongeTM).



Acquisition Directorate Research & Development Center

| SORBENT VENDOR | TEST CONDITION | SORBENT DRY WEIGHT (lb) | OIL DISPENSED (Ib) | OIL IN SORBENT (Ib) | WATER IN SORBENT (Ib) | PERCENT WATER IN SORBENT (%) | OIL / DRY SORBENT WEIGHT (Ib/Ib) |
|----------------------------|-------------------|-------------------------------|--------------------------|------------------------|--------------------------|------------------------------------|--|
| AquaFlex® | Static | 0.35 | 3.94 | 3.88 | 1.57 | 28.9 | 11.09 |
| AquaFlex® | Mix- Energy | 0.38 | 4.28 | 4.21 | 6.41 | 60.3 | 11.08 |
| Earthwise Sorbents | Static | 0.48 | 1.89 | 1.88 | 0.92 | 33 | 3.92 |
| Earthwise Sorbents | Mix- Energy | 0.46 | 1.82 | 1.8 | 0.34 | 16 | 3.91 |
| Imbibitive Technologies | Static | 0.64 | 3.70 | 3.64 | 3.34 | 47.9 | 5.69 |
| Imbibitive Technologies | Mix- Energy | 0.65 | 3.76 | 3.66 | 4.37 | 54.4 | 5.63 |
| MFNS Technologies | Static | 0.33 | 2.11 | 2.1 | 0.89 | 29.8 | 6.36 |
| MFNS Technologies | Mix- Energy | 0.33 | 2.11 | 2.11 | 4.18 | 66.5 | 6.39 |

Table 11. Amount of diesel fuel and water uptake by each sorbent.

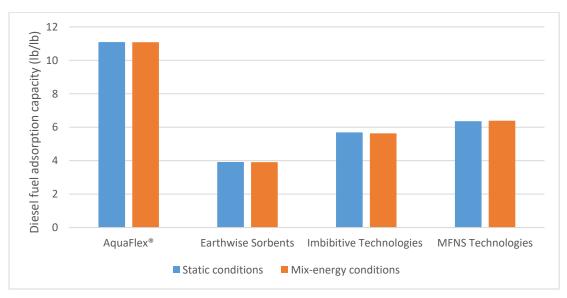


Figure 15. Oil capacity with diesel fuel in water for static and mix-energy conditions.





Figure 16. Oil-Only Heavyweight Pads in static conditions during a water uptake test.



Figure 17. OHM Sponge[™] in mix-energy conditions during a water uptake test.

The project team noted that with diesel fuel, AquaFlex®'s Open-Cell Foam had oil capacities of 11.09 lb/lb and 11.08 lb/lb for static and mix-energy conditions, respectively. For all sorbents the amount of oil adsorbed was at least 97% or more of the oil dispensed, indicating that all sorbents recovered diesel effectively in the presence of water. Sorbent samples by AquaFlex®, Imbibitive Technologies, and MFNS Technologies picked up greater amount of water in mix-energy conditions compared to static conditions. Earthwise Sorbents' Oil-Only Heavyweight Pads collected more water in static conditions than mix-energy conditions. Section 4 of this report provides additional analyses of sorbent water uptake results.

Table 12 and Figure 18 show the sorbents' findings with Hydrocal 300.



| SORBENT VENDOR | TEST CONDITION | SORBENT DRY WEIGHT (lb) | OIL DISPENSED (lb) | OIL IN SORBENT (Ib) | WATER IN SORBENT (Ib) | PERCENT WATER IN SORBENT (%) | OIL / DRY SORBENT WEIGHT (Ib/Ib) |
|----------------------------|-------------------|-------------------------------|--------------------------|------------------------|--------------------------|------------------------------------|--|
| AquaFlex® | Static | 0.35 | 3.87 | 3.81 | 5.12 | 57.4 | 10.89 |
| AquaFlex® | Mix- Energy | 0.34 | 3.76 | 3.64 | 5.38 | 59.7 | 10.71 |
| Earthwise Sorbents | Static | 0.49 | 2.27 | 2.06 | 0.2 | 8.8 | 4.2 |
| Earthwise Sorbents | Mix- Energy | 0.48 | 2.23 | 2.02 | 0.54 | 20.9 | 4.21 |
| Imbibitive Technologies | Static | 0.63 | 2.81 | 2.68 | 0.06 | 2.2 | 4.25 |
| Imbibitive Technologies | Mix- Energy | 0.65 | 2.88 | 2.7 | 0.09 | 3.2 | 4.15 |
| MFNS Technologies | Static | 0.34 | 6.29 | 6.22 | 0.19 | 2.9 | 18.29 |
| MFNS Technologies | Mix- Energy | 0.35 | 6.48 | 6.45 | 5.28 | 45 | 18.43 |

Table 12. Amount of Hydrocal 300 and water uptake by each sorbent.

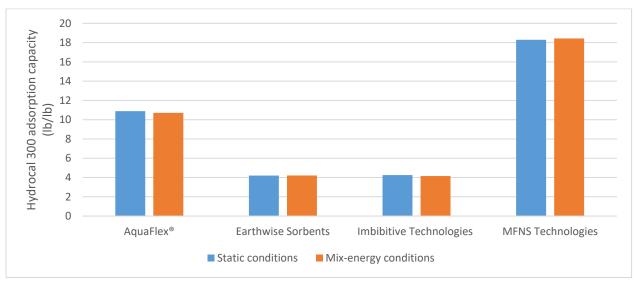


Figure 18. Oil capacity with Hydrocal 300 in water for static and mix-energy conditions.

With Hydrocal 300, OHM Sponge[™] had oil capacities of 18.29 g/g and 18.43 g/g for static and mix-energy conditions, respectively. All sorbents recovered 90% or more of the dispensed oil. Section 4 also discusses these findings in detail.

For the final sorbent water uptake test, the project team used water without oil to determine each sorbent's hydrophobicity. The team placed all four sorbents in two test trays without disturbance for approximately three full days (Figure 19 and Figure 20).





Figure 19. Water-only uptake test with AquaFlex® and MFNS Technologies' sorbents.



Figure 20. Water-only uptake test with Imbiber Fiber[™] and Oil-Only Heavyweight Pads.

Table 13 shows the results from the 3-day, water-only uptake tests with all four sorbents.

| SORBENT VENDOR | DRY WEIGHT (Ib) | WATER IN SORBENT (Ib) | WATER (Ib) / SORBENT (Ib) |
|-------------------------|-----------------|--------------------------|------------------------------|
| AquaFlex® | 0.26 | 0.48 | 1.85 |
| Earthwise Sorbents | 0.16 | 0.22 | 1.38 |
| Imbibitive Technologies | 0.16 | 0.17 | 1.06 |
| MFNS Technologies | 0.11 | 0.18 | 1.64 |

Table 13. Amount of water uptake by each sorbent.



3.3 Retrievability

Table 14 lists results from the retrievability test in alphabetical order. The project team was unable to determine the tear weight for OHM SpongeTM in diesel fuel since the water/diesel uptake test took longer than expected, preventing the tear test from taking place.

| SORBENT VENDOR | TEST OIL | LOADED WEIGHT (Ib) | TEAR WEIGHT (Ib) | NOTES |
|----------------------------|--------------|-----------------------|---------------------|---|
| AquaFlex® | Diesel | 1.96 | 7.76 | After reusability test with 100% diesel |
| AquaFlex® | Hydrocal 300 | 5.29 | 17.16 | - |
| AquaFlex® | Hydrocal 300 | 3.24 | 10.30 | After reusability test with 100% Hydrocal 300 |
| Earthwise Sorbents | None | 1.34 | 3.76 | Tested with dry sample because two tear weight values showed big difference |
| Earthwise Sorbents | Diesel | 3.90 | 24.48 | - |
| Earthwise Sorbents | Hydrocal 300 | 3.55 | 4.87 | - |
| Imbibitive Technologies | Diesel | 2.24 | 6.25 | - |
| Imbibitive Technologies | Hydrocal 300 | 1.68 | 8.48 | - |
| MFNS Technologies | Diesel | N/A | N/A | Ran out of time after 150 minute test |
| MFNS Technologies | Hydrocal 300 | 1.25 | 4.86 | - |

Table 14. Retrievability test results for all sorbents.

With Earthwise Sorbents, the project team noted a big difference in the tear weight for the oil pads between diesel and Hydrocal 300. It was decided to execute another test with a dry sample as a third data point. AquaFlex® also had additional data points since the project team was interested in the retrievability of the Open-Cell Foam after its reusability tests with diesel fuel and Hydrocal 300. Section 4 of this report discusses data from Table 14 in detail.

3.4 Reusability (AquaFlex® only)

The project team noted that AquaFlex®'s Open-Cell Foam generally performed better with diesel fuel compared with Hydrocal 300. Figure 21 shows the wrung sample weight by cycle number for all four reusability tests (diesel fuel with water, pure diesel fuel, Hydrocal 300 with water, and pure Hydrocal 300).



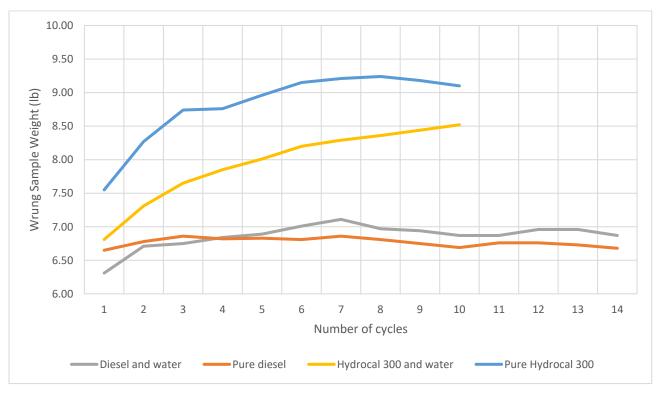


Figure 21. AquaFlex®'s Open-Cell Foam wrung sample weight per cycle for all reusability tests.

From the figure, the wrung sample weight in diesel and water and pure diesel appears to be similar. The weight value remains consistent per cycle at approximately 6.80 pounds, even after 14 adsorption/desorption cycles. There is slightly higher wrung sample weight with diesel and water. Table 15 and Table 16 list those values for each cycle.

| NUMBER OF CYCLES | WRUNG SAMPLE WEIGHT (Ib) | RECOVERED OIL VOLUME (L) | RECOVERED WATER VOLUME (L) |
|------------------|-----------------------------|-----------------------------|-------------------------------|
| 1 | 6.31 | 0.78 | 0.25 |
| 2 | 6.71 | 0.69 | 0.28 |
| 3 | 6.75 | 0.90 | 0.20 |
| 4 | 6.84 | 0.78 | 0.32 |
| 5 | 6.89 | 0.92 | 0.34 |
| 6 | 7.01 | 0.92 | 0.35 |
| 7 | 7.11 | 1.12 | 0.45 |
| 8 | 6.97 | 1.30 | 0.37 |
| 9 | 6.94 | 1.19 | 0.37 |
| 10 | 6.87 | 1.23 | 0.32 |
| 11 | 6.87 | 1.21 | 0.36 |
| 12 | 6.96 | 1.32 | 0.39 |
| 13 | 6.96 | 1.58 | 0.40 |
| 14 | 6.87 | 1.76 | 0.44 |
| AVERAGE | 6.86 | 1.12 | 0.35 |

Table 15. Wrung sample weight, recovered oil and water volumes for reusability test with diesel and water.



| NUMBER OF CYCLES | WRUNG SAMPLE WEIGHT (Ib) | RECOVERED OIL VOLUME (L) |
|------------------|--------------------------|--------------------------|
| 1 | 6.65 | 2.79 |
| 2 | 6.78 | 2.45 |
| 3 | 6.86 | 2.80 |
| 4 | 6.82 | 3.03 |
| 5 | 6.83 | 2.85 |
| 6 | 6.81 | 3.05 |
| 7 | 6.86 | 3.25 |
| 8 | 6.81 | 3.25 |
| 9 | 6.75 | 3.25 |
| 10 | 6.69 | 3.30 |
| 11 | 6.76 | 3.30 |
| 12 | 6.76 | 3.40 |
| 13 | 6.73 | 3.35 |
| 14 | 6.68 | 3.55 |
| AVERAGE | 6.77 | 3.12 |

Table 16. Wrung sample weight and recovered oil volume for reusability test with pure diesel fuel.

The project team was not able to do more than 10 adsorption/desorption cycles with Hydrocal 300 because the increased oil viscosity made the wringer system significantly harder to use, even with the wringer's electric-powered foot pedal operation. The thick, sticky oil on the rollers from the previous wringing cycle necessitated Ohmsett staff to "push" the sorbent sample into the wringer entry area and then pull the sorbent hard on the other side to complete the wringing. The rollers did not rotate as fast as they did with diesel fuel. Figure 21 also shows the wrung sample weight with Hydrocal 300 and water and pure Hydrocal 300. In both test runs, the sorbent weight appeared to increase per cycle before somewhat leveling out after the seventh or eighth cycle. The wrung sample weight was also higher for the pure Hydrocal 300 test compared to Hydrocal 300 and water, which is different from the diesel fuel tests. Table 17 and Table 18 list the wrung sample weight values for the Hydrocal 300 tests.

Table 17. Wrung sample weight, recovered oil and water volumes for reusability test with Hydrocal 300 and water.

| NUMBER OF CYCLES | WRUNG SAMPLE WEIGHT (Ib) | RECOVERED OIL VOLUME (L) | RECOVERED WATER VOLUME (L) | |
|------------------|-----------------------------|-----------------------------|-------------------------------|--|
| 1 | 6.81 | 0.17 | 0.00 | |
| 2 | 7.31 | 0.32 | 0.03 | |
| 3 | 7.65 | 0.29 | 0.05 | |
| 4 | 7.85 | 0.27 | 0.05 | |
| 5 | 8.01 | 0.23 | 0.07 | |
| 6 | 8.20 | 0.10 | 0.08 | |
| 7 | 8.29 | 0.15 | 0.08 | |
| 8 | 8.36 | 0.14 | 0.10 | |
| 9 | 8.44 | 0.15 | 0.08 | |
| 10 | 8.52 | 0.20 | 0.09 | |
| AVERAGE | 7.94 | 0.20 | 0.06 | |



| NUMBER OF CYCLES | WRUNG SAMPLE WEIGHT (Ib) | RECOVERED OIL VOLUME (L) |
|------------------|--------------------------|--------------------------|
| 1 | 7.55 | 0.41 |
| 2 | 8.27 | 0.50 |
| 3 | 8.74 | 0.69 |
| 4 | 8.76 | 0.89 |
| 5 | 8.96 | 1.00 |
| 6 | 9.15 | 0.89 |
| 7 | 9.21 | 1.07 |
| 8 | 9.24 | 1.10 |
| 9 | 9.18 | 1.26 |
| 10 | 9.10 | 1.20 |
| AVERAGE | 8.82 | 0.90 |

Table 18. Wrung sample weight and recovered oil volume for reusability test with pure Hydrocal 300.

Figure 22 and Figure 23 show the comparison in the total amount of oil and water recovered by the wringer system for each cycle.

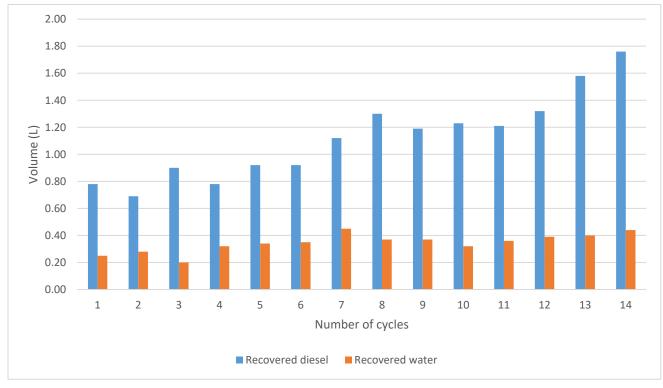


Figure 22. Total volume recovered per cycle after running the Open-Cell Foam through wringer system twice with diesel fuel.



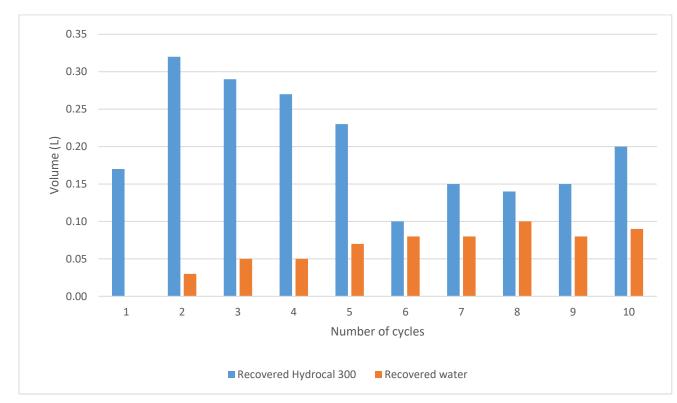
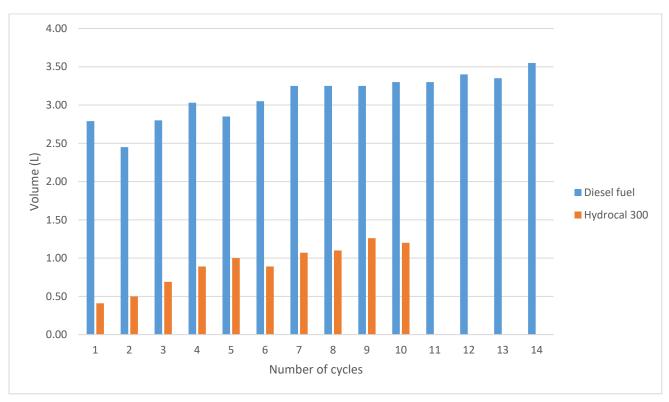
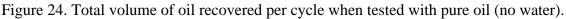


Figure 23. Total volume recovered per cycle after running the Open-Cell Foam through wringer system twice with Hydrocal 300.

The two figures show that the wringer system extracted a greater amount of both oil and water from the sorbent sample with diesel fuel compared to that of Hydrocal 300. Figure 24 also shows the amount of oil recovered from the sorbent samples during each cycle with the wringer system for both pure diesel fuel and Hydrocal 300 tests.







Section 4 of this report discusses all reusability performance findings in greater detail.

The project team noted that with the wringer system, it was important to wear proper personal protective equipment (PPE), including splash protection for the eyes. When Ohmsett staff wrung out the sorbent pads, especially as the end of the diesel fuel sample went through the rollers, there were "squirts" of oil that flew toward the people handling the wringer. Ohmsett staff needed to carefully monitor the speed of the rollers to ensure a clean and safe operation.

4 DISCUSSION AND ANALYSIS

4.1 ASTM F726-17 Standard (Maximum Oil Capacity and Buoyancy)

Table 19 summarizes the maximum oil capacity results from the ASTM F726-17 tests for each sorbent with diesel and Hydrocal 300. It also includes the "custom" near-maximum oil capacity values. The adsorption rate category allows for an easy analysis of how quickly a sample adsorbs a certain type of oil. When the average oil capacity for three samples at a measured time is within 10% of the average oil capacity from the previous measurement, the sample is considered to have reached near-maximum oil adsorption.



| SORBENT VENDOR | OIL TYPE | MAXIMUM OIL CAPACITY (g/g) | NEAR-MAXIMUM OIL CAPACITY (g/g) | TIME TO "NEAR- MAXIMUM" OIL CAPACITY (hour) | ADSORPTION RATE CATEGORY |
|----------------------------|--------------|----------------------------------|---------------------------------------|---|--------------------------------|
| AquaFlex® | Diesel | 22.52 | 20.35 | ≤0.25 | I |
| AquaFlex® | Hydrocal 300 | 22.13 | 20.65 | >0.25 and ≤3.0 | II |
| Earthwise Sorbents | Diesel | 7.89 | 7.60 | >0.25 and ≤3.0 | II |
| Earthwise Sorbents | Hydrocal 300 | 9.28 | 8.97 | ≤0.25 | I |
| Imbibitive Technologies | Diesel | 11.57 | 11.11 | >0.25 and ≤3.0 | II |
| Imbibitive Technologies | Hydrocal 300 | 8.92 | 8.92 | >3.0 | 111 |
| MFNS Technologies | Diesel | 12.80 | 11.99 | ≤0.25 | I |
| MFNS Technologies | Hydrocal 300 | 37.03 | 36.40 | ≤0.25 | I |

Table 19. Adsorption rate category for each sorbent sample by oil type.

Earthwise Sorbents and Imbibitive Technologies have their sorbent samples rated as II with diesel fuel since they reached near-maximum capacity within 3 hours. For the same fuel type, MFNS Technologies and AquaFlex® were rated as I. MFNS Technologies had its sorbent samples rated as I with Hydrocal 300 as did Earthwise Sorbents. AquaFlex®'s samples were rated as II for Hydrocal 300, and those of Imbibitive Technologies were rated as III since they needed longer than 3 hours to reach near-maximum oil capacity.

Imbibitive Technologies' samples had low oil capacity with Hydrocal 300 at 8.92 g/g after 24 hours. With diesel fuel, it also had low near-maximum oil capacity at 11.11 g/g during the 3-hour mark. The project team noted that in the first 15 minutes with diesel fuel, Imbiber FiberTM initially sank to the bottom of the tray, but floated again at the 3-hour and 24-hour marks. With Hydrocal 300, Imbiber FiberTM samples stayed afloat from the beginning to the end of the 24-hour test period.

AquaFlex®'s samples showed consistent adsorption values for both diesel and Hydrocal 300, coming in with near-maximum oil capacity values of 20.35 g/g and 20.65 g/g, respectively. With Hydrocal 300, the Open-Cell Foam experienced a 70.1% increase in oil capacity between 15 minutes and 3 hours before slowing down to a 7.17% increase between 3 hours and 24 hours. For both fuel types, AquaFlex®'s samples remained floating for the full 24-hour test periods.

For Imbibitive Technologies' first ASTM test run with Hydrocal 300, two of the three 13 cm x 13 cm samples had their adsorption values deviate by more than 20% of the average, which necessitated a repeat test. Upon closer inspection, the project team noted that the Imbiber Beads® were not evenly distributed throughout the 12" x 15" polypropylene fabric pad. The project team typically cut out three 13 cm x 13 cm samples from a single pad but realized that for some Imbiber FiberTM pads, one section was noticeably thicker than the other. It may be that the Imbiber Beads® inside the pads settled to one section during product shipping to Ohmsett. Investigating further, the project team weighed a 13 cm x 13 cm sample from the "thin" section and compared to a 13 cm x 13 cm sample from the "thick" section of the same pad. The sample on the "thin" section weighed approximately 9-10 grams while the "thick" sample weighed approximately 23 grams. After this, the project team was careful to ensure that Imbiber FiberTM samples were generally the same weight before testing.



The project team also noted that all Imbiber FiberTM pads shed Imbiber Beads[®] with very minimal handling (Figure 25). Before Ohmsett testing, Imbibitive Technologies made clear that their Imbiber FiberTM was still considered a prototype. Despite the amount of Imbiber Beads[®] shed by the Imbiber FiberTM pads, the project team continued testing with the understanding that the final product would likely have design improvements to contain the beads and keep them evenly distributed in the pads. With the current Imbiber Beads[®] injection method, the fabric pad frequently delaminated (Figure 26), especially at the 24-hour mark of the ASTM tests.



Figure 25. Close view of Imbiber Beads[®] released from Imbiber Fiber[™] after minimal handling. White beads are shown distributed on black paper.

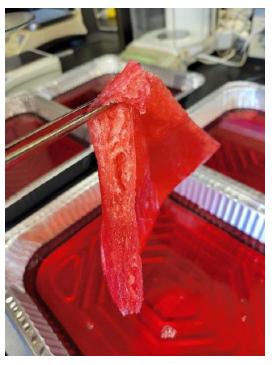


Figure 26. Imbiber Fiber[™] sample experiencing delamination at 24-hour mark of ASTM F726-17 test with diesel fuel.



Earthwise Sorbents also required a repeat ASTM test for its sorbent pads with Hydrocal 300, with one sample deviating by approximately 16% from the average. The project team was not able to discern the reason for the deviation, but the next test run yielded numbers that were within 15% of the average. With diesel fuel, the Oil-Only Heavyweight Pads had a near-maximum oil capacity of 7.60 g/g. Its performance with Hydrocal 300 was slightly improved with 8.97 g/g. The pads remained floating in both oil types for the entire duration of the 24-hour test periods.

Although MFNS Technologies' OHM Sponge[™] showed good adsorption performance with Hydrocal 300 (near-maximum oil capacity of 36.40 g/g within the first 15 minutes), all their samples sank and remained at the bottom of the trays during each time interval (15 minutes, 3 hours, and 24 hours). This was also true for their samples with diesel fuel. In addition, OHM Sponge[™] showed considerably less maximum oil capacity with diesel fuel with 12.80 g/g compared to 37.03 g/g for Hydrocal 300 after 24 hours.

4.2 Sorbent Water Uptake

From Table 11, it is evident that all sorbents recovered essentially all the diesel that was dispensed. Therefore, the sorbents that had access to greater volumes of diesel showed higher adsorption performance. AquaFlex® showed good adsorption performance with diesel fuel in water with an oil to dry weight ratio of 11.09 lb/lb and 11.08 lb/lb for static and mix-energy conditions, respectively. The AquaFlex® Open-Cell Foam picked up 28.9% water by weight in static conditions and 60.3% in mix-energy conditions. The AquaFlex® recovered greater than 98% of the diesel dispensed, for both static and mix-energy conditions.

MFNS Technologies' OHM SpongeTM exhibited good performance in diesel with 6.36 lb/lb and 6.39 lb/lb for static and mix-energy, respectively. The OHM SpongeTM picked up 29.8% water in static conditions and 66.5% water in mix-energy conditions. The OHM SpongeTM recovered greater than 99% of the diesel dispensed.

With diesel fuel, the Oil-Only Heavyweight Pads showed an oil capacity of 3.92 lb/lb and 3.91 lb/lb for static and mix-energy conditions respectively. It recovered greater than 99% of the diesel dispensed for both conditions. The Oil-Only Heavyweight Pads picked up 16.0% water in mix-energy conditions.

Imbiber FiberTM showed oil capacities of 5.69 lb/lb and 5.63 lb/lb for static and mix-energy conditions, respectively, recovering 98% and 97% of the diesel dispensed in each condition. Imbiber FiberTM picked up 47.9% water in static conditions and 54.4% in mix-energy conditions.

With Hydrocal 300 (Table 12), OHM Sponge[™] had an adsorption performance of 18.29 lb/lb and 18.43 lb/lb for static and mix-energy conditions, respectively, and adsorbed greater than 99% of the oil dispensed in both conditions. For mix-energy, OHM Sponge[™] adsorbed 45.0% water by weight but in static conditions, it adsorbed only 2.9% water by weight.

AquaFlex® had 10.89 lb/lb and 10.71 lb/lb for static and mix-energy conditions. AquaFlex® adsorbed greater than 97% of the oil dispensed for both conditions, but also adsorbed 57.4% and 59.7% water by weight for both test conditions (static and mix-energy).

With Hydrocal 300, Imbiber FiberTM adsorbed 2.2% and 3.2% water by weight for static and mix-energy conditions, respectively. Imbiber FiberTM had oil to dry weight ratios of 4.25 lb/lb and 4.15 lb/lb for static and mix-energy conditions, respectively and adsorbed 95% of the oil dispensed in both conditions.



The Oil-Only Heavyweight Pads registered 4.20 lb/lb and 4.21 lb/lb for static and mix-energy, respectively and adsorbed 91% of the oil dispensed in both conditions. The Oil-Only Heavyweight Pads also showed low water uptake in static conditions with 8.8% and 20.9% for mix-energy conditions.

The project team noted a large discrepancy in the amount of water adsorbed by the OHM Sponge[™] samples (2.9% vs. 45.0% in static/mix-energy conditions with Hydrocal 300 and 29.8% vs. 66.5% in static/mixenergy conditions with diesel fuel). It is possible that the small sizes of the OHM Sponge[™] samples may have played a role in the mix-energy tests. Earthwise Sorbents and AquaFlex® provided their samples in rolls, which allowed the project team to cut to their preferred size for testing. Imbibitive Technologies' provided their samples in 12" x 15" pads, which is much larger in comparison to those of MFNS Technologies. MFNS Technologies provided more than 60 individual samples sized at 6.0" x 11.5" x 0.5". The project team used 12 OHM Sponge[™] samples for each test run (Figure 17). During the mix-energy tests, the team noticed much more splashing in the test tray compared to other samples that were larger and did not allow for separation between the sample pads. This may have contributed to a greater water uptake of water for OHM Sponge[™]. However, the many small OHM Sponge[™] samples appeared to have had more contact with oil due to the splash effect compared with other samples in mix-energy conditions, which should have allowed for greater oil uptake. The mix-energy test with diesel fuel for OHM Sponge[™] took 150 minutes; there was not less than 10% in total fluid change between measurements until the 150-minute mark.

The project team also executed water-only tests with all four sorbents, with two in each tray in static conditions for three full days. Imbibitive Technologies' Imbiber Fiber[™] had a water to dry weight ratio of 1.06 lb/lb. AquaFlex®'s Open-Cell Foam registered at 1.85 lb/lb, Earthwise Sorbents at 1.38 lb/lb and MFNS Technologies with 1.64 lb/lb. These results are consistent with water uptake results with Hydrocal 300 in static conditions; Imbiber Fiber[™] had a water uptake of 2.2% of the sorbent weight and AquaFlex® with 57.4%. With diesel fuel in static conditions, Imbiber Fiber[™] had a water uptake of 47.9% and AquaFlex® had 28.9%.

4.3 Retrievability

Each sorbent manufacturer had its samples tested for retrievability with a "tear test". All samples passed in that they did not tear on the hook from their own weight. In all cases additional force was required to tear the samples. From Table 14 the project team noted that the Oil-Only Heavyweight Pad had a tear weight of 24.48 lbs after it was evaluated for water uptake with diesel fuel. Due to the high number, the project team tested the pad again with a dry sample (and without diesel fuel). The load cell registered 3.76 lbs for its tear weight.

AquaFlex®'s Open-Cell Foam had its tear test with Hydrocal 300 after the water uptake test with Hydrocal 300 and registered 17.16 lbs. After 10 cycles of adsorption and desorption with the same oil, its tear weight was lower at 10.30 lbs. The project team expected this because during the reusability test, an Ohmsett staff observed that they needed to be careful handling the Open-Cell Foam towards the end of the test cycle as they believed that they could easily poke a finger through the foam or rip it apart with minimal effort (Figure 27).





Figure 27. AquaFlex®'s Open-Cell Foam before (left) and after reusability test (right).

The foam was also noticeably thinner after running through the wringer system multiple times. With diesel fuel, the tear weight value was lower at 7.76 lbs after it was run through the wringer system 14 times.

Imbibitive Technologies' showed tear weight values of 6.25 lbs and 8.48 lbs for diesel fuel and Hydrocal 300, respectively. Despite issues with delamination, the fabric pad showed good strength in withstanding downward force.

The project team was not able to test MFNS Technologies' OHM Sponge[™]'s performance with diesel fuel but with Hydrocal 300, its tear weight was 4.86 lbs.

4.4 Reusability (AquaFlex® only)

The project team executed 14 adsorption/desorption cycles with pure diesel fuel and diesel fuel in water, but only 10 with Hydrocal 300. The higher oil viscosity made the wringer system more difficult to work with and thus the process took longer. With Hydrocal 300, the Open-Cell Foam appeared to retain more oil in the sorbent while desorbing less after each cycle. Figure 21 shows the wrung sample weight for both pure Hydrocal 300 and Hydrocal 300 and water; there is an upward curve for wrung sample weight that appears to level out for pure Hydrocal 300. Even after 10 cycles with Hydrocal 300 and water, the Open-Cell Foam appears to continue to retain more fluid volume per cycle albeit at a slower pace. However with pure diesel fuel and diesel fuel and water, the wrung sample weight was consistent per cycle, even between the first and the 14th cycle. This indicates that the Open-Cell Foam is able to adsorb and desorb roughly the same amount of diesel fuel and water volumes, showing good performance even after 14 cycles.

Figure 22 shows that generally, AquaFlex®'s Open-Cell Foam was able to adsorb a greater volume of diesel fuel per cycle while water uptake was relatively low and consistent at approximately 0.35 L. In the last 4 cycles, the Open-Cell Foam recovered 1.21 L, 1.32 L, 1.58 L, and 1.76 L of diesel fuel. With Hydrocal 300, Figure 23 show that results are less consistent. Generally, the Open-Cell Foam was able to adsorb more oil than water but towards the end of the test, water uptake volume somewhat increased while oil volume decreased. Figure 24 directly compares the Open-Cell Foam's reusability performance in pure oil tests. It is clear that the foam adsorbs and desorbs a greater volume of diesel fuel than Hydrocal 300. With Hydrocal



300, the volume of recovered oil appears to increase slightly per cycle while the recovered diesel fuel volume starts high with the first cycle, slowly increases until the 7th cycle where it begins to level out. With both fuel types, the tests prove that the foam displays consistent performance even after 10 or 14 cycles.

During the reusability tests, Ohmsett staff frequently carried the Open-Cell Foam sample between the test trays and the wringer system. Even when fully adsorbed, the sample retained the excess fluids (both oil and water) without excessively dripping. After the wringing process, there was even less dripping as the staff returned the foam samples to the test trays. Despite good retention of fluids at maximum adsorption, Ohmsett staff noticed that foam integrity was degraded and felt they could easily poke through it with their fingers or rip it apart with minimal force. They exercised extra caution when using the wringer system towards the end of the tests. However, test data showed good adsorption/desorption values and that performance at the end of the test was either improved or consistent with performance at the first cycle.

The ECO HydroFlex[™] wringer system was difficult to operate with Hydrocal 300, an oil with higher viscosity than diesel fuel. Even though it was electric-powered, the rollers were heavily coated with Hydrocal 300 and were very "sticky", which effectively reduced its rotational speed. Ohmsett staff had difficulty in getting the rollers to "catch" the sorbent pad upon entry and on the exit side, the staff needed to pull on the foam to help facilitate the wringing process. Because of this, the sorbent sample size was longer and narrower at the end of the reusability test compared to the original size before the test (approximately 5" longer and <1" narrower). With diesel fuel, the wringer was easier to operate but Ohmsett staff needed to be cognizant of the high rotational speed and the "squirting" effect, especially on the entry side when the last of the sorbent went through. Overall, using a wringer system requires proper PPE, especially eye protection and face mask/shield as it can make for a messy operation.

5 CONCLUSIONS

The project team successfully executed an independent evaluation of sorbent samples from four different manufacturers using two different oil types: diesel fuel to represent low viscosity oil and Hydrocal 300 to represent medium crude oil.

5.1 AquaFlex®

- AquaFlex®'s Open-Cell Foam was consistent in its performance with both diesel fuel and Hydrocal 300.
- The Open-Cell Foam adsorbed large amounts of water during the sorbent water uptake tests (at least 57% by weight except for the static test with diesel fuel). This indicates low selectivity for oil especially in mix-energy conditions. For the three-day, water-only uptake test, AquaFlex®'s water weight to dry sorbent weight was 1.85 lb/lb.
- With diesel fuel and water, AquaFlex®'s Open-Cell Foam recovered 98% of the dispensed diesel fuel in both static and mix-energy conditions. With Hydrocal 300 and water, it recovered 98% of the dispensed oil in static conditions and 97% in mix-energy conditions.
- AquaFlex® was rated as a category I adsorption rate for diesel and category II for Hydrocal 300, indicating that it would effectively recover these types of oil relatively quickly.
- The Open-Cell Foam demonstrated good retrievability with relatively high tear force.
- The Open-Cell Foam samples remained floating in all test events.
- With the ECO HydroFlex[™] wringer system, AquaFlex[®] demonstrated very good reusability value, providing consistent adsorption/desorption values between the first and last cycles. The Open-Cell



Foam showed better reusability performance with diesel fuel than Hydrocal 300. With Hydrocal 300, water made up of a large part of the total fluid recovered per cycle.

- Using qualitative observations, the Open-Cell Foam integrity appeared to degrade over time during the reusability tests, necessitating careful handling by Ohmsett staff to prevent damage.
- AquaFlex®'s Open-Cell Foam may be ideal for light to medium crude oils in calm water environments. Also, rain may increase water uptake.

5.2 Earthwise Sorbents

- The Oil-Only Heavyweight Pads showed slightly better performance with Hydrocal 300 than diesel fuel.
- For the three-day, water-only uptake test, Earthwise Sorbents' water weight to dry sorbent weight was 1.38 lb/lb. The pads also demonstrated low-to-average water uptake overall and its percent amount of water in the sorbent sample during the mix-energy test with diesel fuel was 16.0%. However, in static conditions with diesel fuel, its water uptake was more than double at 33.0%.
- With diesel fuel and water, the Oil-Only Heavyweight Pads recovered 99% of the dispensed diesel fuel in both static and mix-energy conditions. With Hydrocal 300 and water, it recovered 91% of the dispensed oil in both static and mix-energy conditions.
- The Oil-Only Heavyweight Pads were rated as category II adsorption rate for diesel and category I for Hydrocal 300, indicating that they recover these types of oil relatively quickly.
- Earthwise Sorbents' Oil-Only Heavyweight Pad had a tear weight of 24.48 lbs after testing with diesel fuel in the water uptake test, indicating good retrievability. However with Hydrocal 300, the tear weight was much less at 4.87 lbs. With a dry sample, the oil pad registered a tear weight of 3.76 lbs. Overall, the Oil-Only Heavyweight Pads demonstrated good retrievability and remained buoyant during all test events.
- Earthwise Sorbents' samples remained floating in all test events.

5.3 Imbibitive Technologies

- Imbibitive Technologies' Imbiber Fiber[™] showed better selectivity with diesel fuel than Hydrocal 300. The sorbent also showed very similar oil adsorption performance between static and mix-energy conditions. In terms of percent water in the sorbent by weight, it showed low water uptake when tested with Hydrocal 300 in both static and mix-energy conditions. However, with diesel fuel in both static and mix-energy conditions, the water uptake was much higher.
- Imbibitive Technologies showed low water uptake during the three-day, water-only uptake test with 1.06 lb/lb.
- With diesel fuel and water, Imbiber Fiber[™] recovered 98% of the dispensed diesel fuel in static conditions and 97% in mix-energy conditions. With Hydrocal 300 and water, it recovered 95% of the dispensed oil in static conditions and 94% in mix-energy conditions.
- Imbiber Fiber[™] was rated as a category II adsorption rate for diesel and category III for Hydrocal 300, indicating that for medium oils it would be most effective in a situation where it had sufficient exposure time to fully adsorb.
- Imbiber FiberTM demonstrated average tear weight results, indicating good strength for retrievability. However, Imbiber FiberTM was shown to easily delaminate over time when adsorbed with oil and water.
- Imbiber Fiber[™] samples remained buoyant for all test events except for the first 15 minutes with diesel fuel.
- When dry, Imbiber Beads® shed from the fiber very easily, with minimal handling.



5.4 MFNS Technologies

- MFNS Technologies' OHM SpongeTM was generally a very good performing sorbent during all testing. It showed very high selectivity for Hydrocal 300 and achieved maximum oil capacity within the first 15 minutes during the ASTM F726-17 test. It also had high oil adsorption values in static and mix-energy conditions with water.
- Although not at the level of its performance with Hydrocal 300, the OHM Sponge[™] still showed good results with diesel fuel.
- OHM Sponge[™] had relatively high water adsorbency with 1.64 lb/lb in the three-day, water-only uptake test. It also had high percent water in the sorbent by weight for both the diesel and Hydrocal 300 mix-energy test. There are big differences in water uptake between static and mix-energy conditions for OHM Sponge[™].
- With diesel fuel and water, OHM Sponge[™] recovered >99% of the dispensed diesel fuel in both static and mix-energy conditions. With Hydrocal 300 and water, it also recovered 99% of the dispensed oil in both static and mix-energy conditions.
- OHM Sponge[™] was rated as a category I adsorption rate for both diesel and Hydrocal 300, indicating that it quickly adsorbs both types of oil.
- Retrievability may be a challenge with OHM Sponge[™] as it showed low tear weight after being tested with Hydrocal 300. The samples also sank during the entire 24-hour period of ASTM tests with diesel fuel and Hydrocal 300. However, they remained floating during the full duration of the sorbent water uptake tests.
- OHM Sponge[™] may be ideal for light to medium crude oils in calm water environments. This is contingent on OHM Sponge[™] being able to ensure that their product can remain buoyant. Also, rain may increase water uptake.

6 **RECOMMENDATIONS**

RDC recommends the Coast Guard continue to stay engaged with Interagency Coordinating Committee on Oil Pollution Research (ICCOPR) partners and take advantage of opportunities to participate in testing and evaluating oil spill response technologies. Independent Government testing and evaluation of new and emerging response technologies well in advance of oil spill incidents allows FOSCs to have access to relevant, data on these technologies.

RDC recommends ICCOPR encourage further development and commercialization of reusable sorbent technologies. The potential for significantly reducing oil pollution clean-up waste-generation could directly affect overall disposal logistics concerns and subsequent costs.

USCG RDC and BSEE OSPD make the following recommendations for spill responders:

- When considering reusable sorbents, make sure logistics and planning support wringing operations.
- Wear proper PPE for any oil wringing operations. Expect challenging and messy operation with slip hazards, especially when working with oils with higher viscosity even if wringer is electrically operated. Decontamination zones need to be carefully considered for wringing operations, especially if taking place on a vessel.
- Place greater importance on sorbent water uptake adsorption values than ASTM F726-17 adsorption values, as they better reflect real world spill response. However, ASTM adsorption values can be useful to know if oil is especially thick in certain locations during real world oil spill response.



USCG RDC and BSEE OSPD make the following recommendations for sorbent manufacturers:

- Imbibitive Technologies:
 - Develop a method to keep the Imbiber Beads® fully encapsulated in the fabric pad without risk • of spilling into the environment.
 - Develop a method so as to keep the Imbiber Beads[®] more evenly distributed in the pad, even after shipping, handling, and deploying.
- MFNS Technologies:
 - Ensure that OHM Sponge[™] is fully capable of remaining buoyant after full adsorption for • retrievability in a variety of environmental conditions.
 - Test reusability of OHM Sponge[™] with a range of oil types and provide data to spill response community.
- AquaFlex®:
 - Continue to improve Open-Cell Foam hydrophobicity (reducing water uptake).
 - On ECO HydroFlexTM wringer system, enhance ease of feeding sorbent material through the rollers, especially with more viscous oil types.

7 REFERENCES

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APPENDIX A. TEST OIL SAFETY DATA SHEETS

A.1 Number Two (2) Diesel Fuel



Product Name: NO. 2 DIESEL FUEL Revision Date: 22 Oct 2019 Page 1 of 14

SAFETY DATA SHEET

SECTION 1

PRODUCT AND COMPANY IDENTIFICATION

PRODUCT

 Product Name:
 NO. 2 DIESEL FUEL

 Product Description:
 Hydrocarbons and Additives

 Product Code:
 123455-22, 123455-29, 152017-00

 Intended Use:
 Diesel engine fuel, Heating Oil

COMPANY IDENTIFICATION Supplier:

EXXON MOBIL CORPORATION 22777 Springwoods Village Parkway Spring, TX 77389 USA

24 Hour Health Emergency Transportation Emergency Phone Product Technical Information MSDS Internet Address

809-737-4411 800-424-9300 or 703-527-3887 CHEMTREC 800-662-4525 www.exxon.com, www.mobil.com

SECTION 2

HAZARDS IDENTIFICATION

This material is hazardous according to regulatory guidelines (see (M)SDS Section 15).

CLASSIFICATION:

Flammable liquid: Category 3.

Acute inhalation toxicant: Category 4. Skin irritation: Category 2. Carcinogen: Category 2. Specific target organ toxicant (repeated exposure): Category 2. Aspiration toxicant: Category 1.



Signal word: Dang

Hazard Statements:





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H226: Flammable liquid and vapor. H304: May be fatal if swallowed and enters airways. H315: Causes skin irritation. H332: Harmful if inhaled. H351: Suspected of causing cancer. H373: May cause damage to organs through prolonged or repeated exposure. Bone marrow, Liver, Thymus

Precautionary Statements:

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P101: If medical advice is needed, have product container or label at hand. P102: Keep out of reach of children. P103: Read label before use.P201: Obtain special instructions before use. P202: Do not handle until all safety precautions have been read and understood. P210: Keep away from heat/sparks/open flames/hot surfaces. -- No smoking. P233: Keep container tightly closed. P240: Ground / bond container and receiving equipment. P241: Use explosion-proof electrical, ventilating, and lighting equipment. P242: Use only non-sparking tools. P243: Take precautionary measures against static discharge. P260: Do not breathe mist / vapours. P264: Wash skin thoroughly after handling. P271: Use only outdoors or in a well-ventilated area. P273: Avoid release to the environment. P280: Wear protective gloves/protective clothing/eye protection/face protection.P301 + P310; IF SWALLOWED: Immediately call a POISON CENTER or doctor/physician. P302 + P352: IF ON SKIN: Wash with plenty of soap and water. P304 + P340: IF INHALED: Remove person to fresh air and keep comfortable for breathing. P308 + P313: IF exposed or concerned: Get medical advice/ attention. P312: Call a POISON CENTER or doctor/physician if you feel unwell. P331: Do NOT induce vomiting. P332 + P313: If skin irritation occurs: Get medical advice/ attention. P362 + P364: Take off contaminated clothing and wash it before reuse. P370 + P378: In case of fire: Use water fog, foam, dry chemical or carbon dioxide (CO2) to extinguish, P391: Collect spillage.P403 + P235: Store in a well-ventilated place. Keep cool. P405: Store locked up.P501: Dispose of contents and container in accordance with local regulations.

Contains: DIESEL OIL..C9-20

Other hazard information:

HAZARD NOT OTHERWISE CLASSIFIED (HNOC): None as defined under 29 CFR 1910.1200.

PHYSICAL / CHEMICAL HAZARDS

Material can accumulate static charges which may cause an ignition. Material can release vapors that readily form flammable mixtures. Vapor accumulation could flash and/or explode if ignited.

HEALTH HAZARDS

May cause central nervous system depression. High-pressure injection under skin may cause serious damage. Under conditions of poor personal hygiene and prolonged repeated contact, some polycyclic aromatic compounds (PACs) have been suspected as a cause of skin cancer in humans. May be irritating to the eyes, nose, throat, and lungs.

ENVIRONMENTAL HAZARDS

Expected to be toxic to aquatic organisms. May cause long-term adverse effects in the aquatic environment.

| NFPA Hazard ID: | Health: | 2 | Flammability: | 2 | Reactivity: | 0 |
|-----------------|---------|----|---------------|---|-------------|---|
| HMIS Hazard ID: | Health: | 2* | Flammability: | 2 | Reactivity: | 0 |

NOTE: This material should not be used for any other purpose than the intended use in Section 1 without expert advice. Health studies have shown that chemical exposure may cause potential human health risks which may vary from person to person.

SECTION 3

COMPOSITION / INFORMATION ON INGREDIENTS





Product Name: NO. 2 DIESEL FUEL Revision Date: 22 Oct 2019 Page 3 of 14

This material is defined as a mixture.

Hazardous Substance(s) or Complex Substance(s) required for disclosure

| Name | CAS# | | GHS Hazard Codes |
|-----------------|------------|----------------|---|
| | | Concentration* | |
| DIESEL OILC9-20 | 68334-30-5 | 80 - > 99% | H226, H304, H332, H351, H315, H373, H401, H411 |

Hazardous Constituent(s) Contained in Complex Substance(s) required for disclosure

| Name | CAS# | Concentration* | GHS Hazard Codes |
|---------------|----------|----------------|--------------------------|
| ETHYL BENZENE | 100-41-4 | 0.1 - 1% | H225, H304, H332, H373, |
| | | | H401, H412 |
| NAPHTHALENE | 91-20-3 | 0.1 - 1% | H228(2), H302, H351, |
| | | | H400(M factor 1), H410(M |
| | | | factor 1) |

* All concentrations are percent by weight unless material is a gas. Gas concentrations are in percent by volume.

NOTE: Composition may contain up to 0.5% performance additives and / or dyes.

As per paragraph (i) of 29 CFR 1910.1200, formulation is considered a trade secret and specific chemical identity and exact percentage (concentration) of composition may have been withheld. Specific chemical identity and exact percentage composition will be provided to health professionals, employees, or designated representatives in accordance with applicable provisions of paragraph (i).

SECTION 4 FIRST AID MEASURES

INHALATION

Remove from further exposure. For those providing assistance, avoid exposure to yourself or others. Use adequate respiratory protection. If respiratory irritation, dizziness, nausea, or unconsciousness occurs, seek immediate medical assistance. If breathing has stopped, assist ventilation with a mechanical device or use mouth-to-mouth resuscitation.

SKIN CONTACT

Remove contaminated clothing. Dry wipe exposed skin and cleanse with waterless hand cleaner and follow by washing thoroughly with soap and water. For those providing assistance, avoid further skin contact to yourself or others. Wear impervious gloves. Launder contaminated clothing separately before reuse. Discard contaminated articles that cannot be laundered. If product is injected into or under the skin, or into any part of the body, regardless of the appearance of the wound or its size, the individual should be evaluated immediately by a physician as a surgical emergency. Even though initial symptoms from high pressure injection may be minimal or absent, early surgical treatment within the first few hours may significantly reduce the ultimate extent of injury.

EYE CONTACT

Flush thoroughly with water. If irritation occurs, get medical assistance.

INGESTION

Seek immediate medical attention. Do not induce vomiting.

NOTE TO PHYSICIAN

If ingested, material may be aspirated into the lungs and cause chemical pneumonitis. Treat appropriately,





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PRE-EXISTING MEDICAL CONDITIONS WHICH MAY BE AGGRAVATED BY EXPOSURE

Contains hydrocarbon solvent/petroleum hydrocarbons; skin contact may aggravate an existing dermatitis.

SECTION 5

FIRE FIGHTING MEASURES

EXTINGUISHING MEDIA

Appropriate Extinguishing Media: Use water fog, foam, dry chemical or carbon dioxide (CO2) to extinguish flames.

Inappropriate Extinguishing Media: Straight Streams of Water

FIRE FIGHTING

Fire Fighting Instructions: Flammable. Evacuate area. Prevent runoff from fire control or dilution from entering streams, sewers, or drinking water supply. Firefighters should use standard protective equipment and in enclosed spaces, self-contained breathing apparatus (SCBA). Use water spray to cool fire exposed surfaces and to protect personnel.

Unusual Fire Hazards: Vapors are flammable and heavier than air. Vapors may travel across the ground and reach remote ignition sources causing a flashback fire danger. Hazardous material. Firefighters should consider protective equipment indicated in Section 8.

Hazardous Combustion Products: Aldehydes, Incomplete combustion products, Oxides of carbon, Smoke, Fume, Sulfur oxides

FLAMMABILITY PROPERTIES

 Flash Point [Method]: >38°C (100°F) [ASTM D-93]

 Flammable Limits (Approximate volume % in air): LEL: 0.6 UEL: 7.0

 Autoignition Temperature: >200°C (392°F)

SECTION 6

ACCIDENTAL RELEASE MEASURES

NOTIFICATION PROCEDURES

In the event of a spill or accidental release, notify relevant authorities in accordance with all applicable regulations. US regulations require reporting releases of this material to the environment which exceed the applicable reportable quantity or oil spills which could reach any waterway including intermittent dry creeks. The National Response Center can be reached at (800)424-8802.

PROTECTIVE MEASURES

Avoid contact with spilled material. Warn or evacuate occupants in surrounding and downwind areas if required due to toxicity or flammability of the material. See Section 5 for fire fighting information. See the Hazard Identification Section for Significant Hazards. See Section 4 for First Aid Advice. See Section 8 for advice on the minimum requirements for personal protective equipment. Additional protective measures may be necessary, depending on the specific circumstances and/or the expert judgment of the emergency responders.

For emergency responders: Respiratory protection: half-face or full-face respirator with filter(s) for organic vapor and, when applicable, H2S, or Self Contained Breathing Apparatus (SCBA) can be used depending on the size of spill and potential level of exposure. If the exposure cannot be completely characterized or an oxygen deficient atmosphere is possible or anticipated, SCBA is recommended. Work gloves that are resistant to aromatic hydrocarbons are recommended. Note: gloves made of polyvinyl acetate (PVA) are not water-resistant and are not suitable for emergency use. Chemical goggles are recommended if splashes or contact





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> with eyes is possible. Small spills: normal antistatic work clothes are usually adequate. Large spills: full body suit of chemical resistant, antistatic material is recommended.

SPILL MANAGEMENT

Land Spill: Eliminate all ignition sources (no smoking, flares, sparks or flames in immediate area). Stop leak if you can do it without risk. All equipment used when handling the product must be grounded. Do not touch or walk through spilled material. Prevent entry into waterways, sewer, basements or confined areas. A vapor suppressing foam may be used to reduce vapors. Use clean non-sparking tools to collect absorbed material. Absorb or cover with dry earth, sand or other non-combustible material and transfer to containers. Large Spills: Water spray may reduce vapor; but may not prevent ignition in closed spaces.

Water Spill: Stop leak if you can do it without risk. Eliminate sources of ignition. Warn other shipping. If the Flash Point exceeds the Ambient Temperature by 10 degrees C or more, use containment booms and remove from the surface by skimming or with suitable absorbents when conditions permit. If the Flash Point does not exceed the Ambient Air Temperature by at least 10C, use booms as a barrier to protect shorelines and allow material to evaporate. Seek the advice of a specialist before using dispersants.

Water spill and land spill recommendations are based on the most likely spill scenario for this material; however, geographic conditions, wind, temperature, (and in the case of a water spill) wave and current direction and speed may greatly influence the appropriate action to be taken. For this reason, local experts should be consulted. Note: Local regulations may prescribe or limit action to be taken.

ENVIRONMENTAL PRECAUTIONS

Large Spills: Dike far ahead of liquid spill for later recovery and disposal. Prevent entry into waterways, sewers, basements or confined areas.

SECTION 7

HANDLING AND STORAGE

HANDLING

Avoid all personal contact. Do not siphon by mouth. Do not use as a cleaning solvent or other non-motor fuel uses. For use as a motor fuel only. It is dangerous and/or unlawful to put fuel into unapproved containers. Do not fill container while it is in or on a vehicle. Static electricity may ignite vapors and cause fire. Place container on ground when filling and keep nozzle in contact with container. Do not use electronic devices (including but not limited to cellular phones, computers, calculators, pagers or other electronic devices, etc.) during safety critical tasks, such as bulk fuel loading or unloading operations, or in storage areas where vapors may be present, unless the devices are certified intrinsically safe by an approved national testing agency and to the safety standards required by national and/or local laws and regulations. Prevent small spills and leakage to avoid slip hazard. Material can accumulate static charges which may cause an electrical spark (ignition source). Use proper bonding and/or ground procedures. However, bonding and grounds may not eliminate the hazard from static accumulation. Consult local applicable standards for guidance. Additional references include American Petroleum Institute 2003 (Protection Against Ignitions Arising out of Static, Lightning and Stray Currents) or National Fire Protection Agency 77 (Recommended Practice on Static Electricity) or CENELEC CLC/TR 50404 (Electrostatics - Code of practice for the avoidance of hazards due to static electricity).

Static Accumulator: This material is a static accumulator. A liquid is typically considered a nonconductive, static accumulator if its conductivity is below 100 pS/m (100x10E-12 Siemens per meter) and is considered a semiconductive, static accumulator if its conductivity is below 10,000 pS/m. Whether a liquid is nonconductive or semiconductive, the precautions are the same. A number of factors, for example liquid temperature, presence of contaminants, anti-static additives and filtration can greatly influence the conductivity of a liquid.



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STORAGE

The type of container used to store the material may affect static accumulation and dissipation. Keep container closed. Handle containers with care. Open slowly in order to control possible pressure release. Store in a cool, well-ventilated area. Storage containers should be grounded and bonded. Fixed storage containers, transfer containers and associated equipment should be grounded and bonded to prevent accumulation of static charge. Keep away from incompatible materials.

SECTION 8

EXPOSURE CONTROLS / PERSONAL PROTECTION

EXPOSURE LIMIT VALUES

Exposure limits/standards (Note: Exposure limits are not additive)

| Substance Name | Form | Limit / Standard | | | NOTE | Source |
|---|------------------------------------|------------------|-----------|---------|------|------------|
| DIESEL OILC9-20 | Stable Aerosol. | TWA | 5 mg/m3 | | Skin | ExxonMobil |
| DIESEL OILC9-20 | Vapor. | TWA | 200 mg/m3 | | Skin | ExxonMobil |
| DIESEL OILC9-20 [total hydrocarb, vapor&aerosol] | Inhalable fraction and vapor | TWA | 100 mg/m3 | | Skin | ACGIH |
| ETHYL BENZENE | | TWA | 435 mg/m3 | 100 ppm | N/A | OSHA Z1 |
| ETHYL BENZENE | | TWA | 20 ppm | | N/A | ACGIH |
| NAPHTHALENE | | TWA | 50 mg/m3 | 10 ppm | N/A | OSHA Z1 |
| NAPHTHALENE | | TWA | 10 ppm | | Skin | ACGIH |

NOTE: Limits/standards shown for guidance only. Follow applicable regulations.

Biological limits

| Substance | Specimen | Sampling Time | Limit | Determinant | Source |
|---------------|---------------------------------------|---------------|-----------------|---|----------------------|
| ETHYL BENZENE | Creatinine in urine | End of shift | 0.15 g/g | Sum of mandelic acid and phenylolyoxylic acid | ACGIH BELs (BEIs) |
| NAPHTHALENE | No Biological Specimen provided | End of shift | Not Assigned | 1-Naphthol, with hydrolysis + 2-Naphthol, with hydrolysis | ACGIH BELS (BEIS) |

ENGINEERING CONTROLS

The level of protection and types of controls necessary will vary depending upon potential exposure conditions. Control measures to consider:

Use explosion-proof ventilation equipment to stay below exposure limits.

PERSONAL PROTECTION

Personal protective equipment selections vary based on potential exposure conditions such as applications, handling practices, concentration and ventilation. Information on the selection of protective equipment for use



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with this material, as provided below, is based upon intended, normal usage.

Respiratory Protection: If engineering controls do not maintain airborne contaminant concentrations at a level which is adequate to protect worker health, an approved respirator may be appropriate. Respirator selection, use, and maintenance must be in accordance with regulatory requirements, if applicable. Types of respirators to be considered for this material include:

Half-face filter respirator

For high airborne concentrations, use an approved supplied-air respirator, operated in positive pressure mode. Supplied air respirators with an escape bottle may be appropriate when oxygen levels are inadequate, gas/vapor warning properties are poor, or if air purifying filter capacity/rating may be exceeded.

Hand Protection: Any specific glove information provided is based on published literature and glove manufacturer data. Glove suitability and breakthrough time will differ depending on the specific use conditions. Contact the glove manufacturer for specific advice on glove selection and breakthrough times for your use conditions. Inspect and replace worn or damaged gloves. The types of gloves to be considered for this material include:

Chemical resistant gloves are recommended. If contact with forearms is likely wear gauntlet style gloves.

Eye Protection: If contact with material is likely, chemical goggles are recommended.

Skin and Body Protection: Any specific clothing information provided is based on published literature or manufacturer data. The types of clothing to be considered for this material include: Chemical/oil resistant clothing is recommended.

Specific Hygiene Measures: Always observe good personal hygiene measures, such as washing after handling the material and before eating, drinking, and/or smoking. Routinely wash work clothing and protective equipment to remove contaminants. Discard contaminated clothing and footwear that cannot be cleaned. Practice good housekeeping.

ENVIRONMENTAL CONTROLS

Comply with applicable environmental regulations limiting discharge to air, water and soil. Protect the environment by applying appropriate control measures to prevent or limit emissions.

SECTION 9

PHYSICAL AND CHEMICAL PROPERTIES

Note: Physical and chemical properties are provided for safety, health and environmental considerations only and may not fully represent product specifications. Contact the Supplier for additional information.

GENERAL INFORMATION

Physical State: Liquid Color: Clear (May Be Dyed) Odor: Petroleum/Solvent Odor Threshold: N/D

IMPORTANT HEALTH, SAFETY, AND ENVIRONMENTAL INFORMATION

Relative Density (at 15 °C): 0.81 - 0.87 Density (at 15 °C): 810 kg/m³ (6.76 lbs/gal, 0.81 kg/dm³) - 876 kg/m³ (7.31 lbs/gal, 0.88 kg/dm³) Flammability (Solid, Gas): N/A





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> Flash Point [Method]: >38°C (100°F) [ASTM D-93] Flammable Limits (Approximate volume % in air): LEL: 0.6 UEL: 7.0 Autoignition Temperature: >200°C (392°F) Boiling Point / Range: 145°C (293°F) - 370°C (698°F) Decomposition Temperature: N/D Vapor Density (Air = 1): > 2 at 101 kPa Vapor Pressure: 0.067 kPa (0.5 mm Hg) at 20 °C Evaporation Rate (n-butyl acetate = 1): N/D pH: N/A Log Pow (n-Octanol/Water Partition Coefficient): > 3.5 Solubility in Water: Negligible Viscosity: 1.7 cSt (1.7 mm2/sec) at 40 °C - 4.1 cSt (4.1 mm2/sec) at 40 °C Oxidizing Properties: See Hazards Identification Section.

OTHER INFORMATION

Freezing Point: N/D Melting Point: N/A Pour Point: <-6°C (21°F)

SECTION 10

STABILITY AND REACTIVITY

REACTIVITY: See sub-sections below.

STABILITY: Material is stable under normal conditions.

CONDITIONS TO AVOID: Avoid heat, sparks, open flames and other ignition sources.

MATERIALS TO AVOID: Halogens, Strong Acids, Strong Bases, Strong oxidizers

HAZARDOUS DECOMPOSITION PRODUCTS: Material does not decompose at ambient temperatures.

POSSIBILITY OF HAZARDOUS REACTIONS: Hazardous polymerization will not occur.

SECTION 11

TOXICOLOGICAL INFORMATION

INFORMATION ON TOXICOLOGICAL EFFECTS

| Hazard Class | Conclusion / Remarks | |
|--|---|--|
| Inhalation | | |
| Acute Toxicity: (Rat) 4 hour(s) LC50 4100 mg/m3 (Vapor and aerosol) | Moderately toxic. Based on test data for structurally similar materials. Test(s) equivalent or similar to OECD Guideline 403 | |
| Irritation: No end point data for material. | Elevated temperatures or mechanical action may form vapors, mist, or fumes which may be irritating to the eyes, nose, throat, or lungs. | |
| Ingestion | | |
| Acute Toxicity (Rat): LD50 > 5000 mg/kg | Minimally Toxic. Based on test data for structurally similar materials. Test(s) equivalent or similar to OECD Guideline 401 | |
| Skin | | |
| Acute Toxicity (Rabbit): LD50 > 5000 mg/kg | Minimally Toxic. Based on test data for structurally similar materials. Test(s) equivalent or similar to OECD Guideline 434 | |
| Skin Corrosion/Irritation (Rabbit): Data | Irritating to the skin. Based on test data for structurally similar | |



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| available. | materials. Test(s) equivalent or similar to OECD Guideline 404 | | |
|---|---|--|--|
| Eye | | | |
| Serious Eye Damage/Irritation (Rabbit): Data available. | May cause mild, short-lasting discomfort to eyes. Based on test data for structurally similar materials. Test(s) equivalent or similar to OECD Guideline 405 | | |
| Sensitization | | | |
| Respiratory Sensitization: No end point data for material. | Not expected to be a respiratory sensitizer. | | |
| Skin Sensitization: Data available. | Not expected to be a skin sensitizer. Based on test data for structurally similar materials. Test(s) equivalent or similar to OECD Guideline 406 | | |
| Aspiration: Data available. | May be fatal if swallowed and enters airways. Based on physical chemical properties of the material. | | |
| Germ Cell Mutagenicity: Data available. | Not expected to be a germ cell mutagen. Based on test data for structurally similar materials. Test(s) equivalent or similar to OE0 Guideline 471 475 | | |
| Carcinogenicity: Data available. | Caused cancer in laboratory animals, but the relevance to huma is uncertain. Based on test data for structurally similar materials. Test(s) equivalent or similar to OECD Guideline 451 | | |
| Reproductive Toxicity: Data available. | Not expected to be a reproductive toxicant. Test(s) equivalent or similar to OECD Guideline 414 | | |
| Lactation: No end point data for material. | Not expected to cause harm to breast-fed children. | | |
| Specific Target Organ Toxicity (STOT) | | | |
| Single Exposure: No end point data for material. | Not expected to cause organ damage from a single exposure. | | |
| Repeated Exposure: Data available. | Concentrated, prolonged or deliberate exposure may cause organ damage. Based on test data for structurally similar materials. Test(s) equivalent or similar to OECD Guideline 410 413 | | |

TOXICITY FOR SUBSTANCES

| NAME | ACUTE TOXICITY | |
|---------------|---|--|
| ETHYL BENZENE | Inhalation Lethality: 4 hour(s) LC50 17.8 mg/l (Vapor) (Rat); Oral Lethality: LD50 3.5 g/kg (Rat) | |
| NAPHTHALENE | Inhalation Lethality: 4 hour(s) LC50 > 0.4 mg/l (Max attainable vapor conc.) (Rat); Oral Lethality: LD50 533 mg/kg (Mouse) | |

OTHER INFORMATION

For the product itself:

Target Organs Repeated Exposure: Bone marrow, Liver, Thymus

Vapor concentrations above recommended exposure levels are irritating to the eyes and the respiratory tract, may cause headaches and dizziness, are anesthetic and may have other central nervous system effects.

Small amounts of liquid aspirated into the lungs during ingestion or from vomiting may cause chemical pneumonitis or pulmonary edema.

Diesel fuel: Caused cancer in animal tests. Caused mutations in vitro. Repeated dermal exposures to high concentrations in test animals resulted in reduced litter size and litter weight, and increased fetal resorptions at maternally toxic doses. Dermal exposure to high concentrations resulted in severe skin irritation with weight loss and some mortality. Inhalation exposure to high concentrations resulted in respiratory tract irritation, lung changes/infiltration/accumulation, and reduction in lung function.

Diesel exhaust fumes: Carcinogenic in animal tests. Inhalation exposures to exhaust for 2 years in test animals resulted in lung tumors and lymphoma. Extract of particulate produced skin tumors in test animals. Caused mutations



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Product Name: NO. 2 DIESEL FUEL Revision Date: 22 Oct 2019 Page 10 of 14

in vitro.

Contains:

NAPHTHALENE: Exposure to high concentrations of naphthalene may cause destruction of red blood cells, anemia, and cataracts. Naphthalene caused cancer in laboratory animal studies, but the relevance of these findings to humans is uncertain.

ETHYLBENZENE: Caused cancer in laboratory animal studies. The relevance of these findings to humans is uncertain.

The following ingredients are cited on the lists below:

| Chemical Name | CAS Number | List Citations | |
|---------------|------------|----------------|--|
| ETHYL BENZENE | 100-41-4 | 5 | |
| NAPHTHALENE | 91-20-3 | 2, 5 | |

| | REGULATORY LISTS SE | ARCHED |
|--------------|---------------------|---------------|
| 1 = NTP CARC | 3 = IARC 1 | 5 = IARC 2B |
| 2 = NTP SUS | 4 = IARC 2A | 6 = OSHA CARC |

SECTION 12 ECOLOGICAL INFORMATION

The information given is based on data for the material, components of the material, or for similar materials, through the application of bridging principals.

ECOTOXICITY

Material -- Expected to be toxic to aquatic organisms. May cause long-term adverse effects in the aquatic environment.

MOBILITY

More volatile component - Highly volatile, will partition rapidly to air. Not expected to partition to sediment and wastewater solids.

High molecular wt. component -- Low solubility and floats and is expected to migrate from water to the land. Expected to partition to sediment and wastewater solids.

PERSISTENCE AND DEGRADABILITY Biodegradation:

Material -- Expected to be inherently biodegradable Atmospheric Oxidation:

More volatile component -- Expected to degrade rapidly in air

ECOLOGICAL DATA





Product Name: NO, 2 DIESEL FUEL Revision Date: 22 Oct 2019 Page 11 of 14

| Ecotoxicity | | | |
|----------------------------|------------|------------------------------------|---|
| Test | Duration | Organism Type | Test Results |
| Aquatic - Acute Toxicity | 48 hour(s) | Daphnia magna | EL50 1 - 1000 mg/l: data for similar materials |
| Aquatic - Acute Toxicity | 96 hour(s) | Fish | LL50 1 - 100 mg/l: data for similar materials |
| Aquatic - Acute Toxicity | 72 hour(s) | Pseudokirchneriella subcapitata | EL50 1 - 100 mg/l: data for similar materials |
| Aquatic - Chronic Toxicity | 72 hour(s) | Pseudokirchneriella subcapitata | NOELR 1 - 10 mg/l: data for similar materials |

Persistence, Degradability and Bioaccumulation Potential

| Media | Test Type | Duration | Test Results |
|-------|------------------------|-----------|-------------------------|
| Water | Ready Biodegradability | 28 day(s) | Percent Degraded < 60 : |
| | | | similar material |

SECTION 13

DISPOSAL CONSIDERATIONS

Disposal recommendations based on material as supplied. Disposal must be in accordance with current applicable laws and regulations, and material characteristics at time of disposal.

DISPOSAL RECOMMENDATIONS

Product is suitable for burning in an enclosed controlled burner for fuel value or disposal by supervised incineration at very high temperatures to prevent formation of undesirable combustion products.

REGULATORY DISPOSAL INFORMATION

RCRA Information: Disposal of unused product may be subject to RCRA regulations (40 CFR 261). Disposal of the used product may also be regulated due to ignitability, corrosivity, reactivity or toxicity as determined by the Toxicity Characteristic Leaching Procedure (TCLP). Potential RCRA characteristics: IGNITABILITY.

Empty Container Warning Empty Container Warning (where applicable): Empty containers may contain residue and can be dangerous. Do not attempt to refill or clean containers without proper instructions. Empty drums should be completely drained and safely stored until appropriately reconditioned or disposed. Empty containers should be taken for recycling, recovery, or disposal through suitably qualified or licensed contractor and in accordance with governmental regulations. DO NOT PRESSURISE, CUT, WELD, BRAZE, SOLDER, DRILL, GRIND, OR EXPOSE SUCH CONTAINERS TO HEAT, FLAME, SPARKS, STATIC ELECTRICITY, OR OTHER SOURCES OF IGNITION. THEY MAY EXPLODE AND CAUSE INJURY OR DEATH.

| SECTION 14 | TRANSPORT INFORMATION | |
|------------|-----------------------|--|
| | | |

LAND (DOT)

Proper Shipping Name: DIESEL FUEL Hazard Class & Division: COMBUST ID Number: NA1993 Packing Group: III Marine Pollutant: Yes ERG Number: 128 Label(s): NONE

SEL FUEL COMBUSTIBLE LIQUID





Product Name: NO. 2 DIESEL FUEL Revision Date: 22 Oct 2019 Page 12 of 14

> Transport Document Name: NA1993, DIESEL FUEL, COMBUSTIBLE LIQUID, PG III, MARINE POLLUTANT

Footnote: The flash point of this material is greater than 100 F. Regulatory classification of this material varies. DOT: Flammable liquid or combustible liquid. OSHA: Combustible liquid. IATA/IMO: Flammable liquid. This material is not regulated under 49 CFR in a container of 119 gallon capacity or less when transported solely by land, as long as the material is not a hazardous waste, a marine pollutant, or specifically listed as a hazardous substance.

LAND (TDG)

Proper Shipping Name: GAS OIL Hazard Class & Division: 3 UN Number: 1202 Packing Group: III Special Provisions: 88, 150

SEA (IMDG)

| A (IMDG) | |
|--------------------------|---|
| Proper Shipping Name: | GAS OIL |
| Hazard Class & Division: | 3 |
| EMS Number: F-E, S-E | |
| UN Number: 1202 | |
| Packing Group: III | |
| Marine Pollutant: Yes | |
| Label(s): 3 | |
| Transport Document Nam | e: UN1202, GAS OIL, 3, PG III, (>38°C c.c.), MARINE POLLUTANT |
| | |

AIR (IATA) Proper Shipping Name: GAS OIL Hazard Class & Division: 3 UN Number: 1202 Packing Group: III Label(s) / Mark(s): 3 Transport Document Name: UN1202, GAS OIL, 3, PG III

SECTION 15

REGULATORY INFORMATION

OSHA HAZARD COMMUNICATION STANDARD: This material is considered hazardous in accordance with OSHA HazCom 2012, 29 CFR 1910.1200.

Listed or exempt from listing/notification on the following chemical inventories: AICS, DSL, IECSC, KECI, PICCS, TSCA

SARA 302: No chemicals in this material are subject to the reporting requirements of SARA Title III, Section 302

CERCLA: This material is not subject to any special reporting under the requirements of the Comprehensive Environmental Response, Compensation and Liability Act (CERCLA). Contact local authorities to determine if other reporting requirements apply.



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SARA (311/312) REPORTABLE GHS HAZARD CLASSES: Acute Toxicity (any route of exposure), Aspiration Hazard, Carcinogenicity, Flammable (gases, aerosols, liquids, or solids), Skin Corrosion or Irritation, Specific Target Organ toxicity (single or repeated exposure)

SARA (313) TOXIC RELEASE INVENTORY:

| Chemical Name | CAS Number | Typical Value | |
|---------------|------------|---------------|--|
| ETHYL BENZENE | 100-41-4 | 0.1 - 1% | |
| NAPHTHALENE | 91-20-3 | 0.1 - 1% | |

The following ingredients are cited on the lists below:

| Chemical Name | CAS Number | List Citations | |
|-----------------|------------|------------------|--|
| DIESEL OILC9-20 | 68334-30-5 | 1, 18 | |
| ETHYL BENZENE | 100-41-4 | 1, 4, 10, 17, 19 | |
| NAPHTHALENE | 91-20-3 | 1, 4, 10, 17, 19 | |

--REGULATORY LISTS SEARCHED--

| 1 = ACGIH ALL | 6 = TSCA 5a2 | 11 = CA P65 REPRO | 16 = MN RTK |
|---------------|------------------|-------------------|-------------|
| 2 = ACGIH A1 | 7 = TSCA 5e | 12 = CA RTK | 17 = NJ RTK |
| 3 = ACGIH A2 | 8 = TSCA 6 | 13 = IL RTK | 18 = PA RTK |
| 4 = OSHA Z | 9 = TSCA 12b | 14 = LA RTK | 19 = RI RTK |
| 5 = TSCA 4 | 10 = CA P65 CARC | 15 = MI 293 | |

Code key: CARC=Carcinogen; REPRO=Reproductive

| SECTION 16 | OTHER INFORMATION | |
|------------|-------------------|--|
| | | |

WARNING: Cancer and Reproductive Harm - www.P65Warnings.ca.gov. Chemicals known to the State of California to cause cancer, birth defects, or other reproductive harm are created by the combustion of this product.

This warning is given to comply with California Health and Safety Code 25249.6 and does not constitute an admission or a waiver of rights.

N/D = Not determined, N/A = Not applicable

KEY TO THE H-CODES CONTAINED IN SECTION 3 OF THIS DOCUMENT (for information only):

- H225: Highly flammable liquid and vapor; Flammable Liquid, Cat 2
- H226: Flammable liquid and vapor; Flammable Liquid, Cat 3
- H302: Harmful if swallowed; Acute Tox Oral, Cat 4
- H304: May be fatal if swallowed and enters airways; Aspiration, Cat 1
- H315: Causes skin irritation; Skin Corr/Irritation, Cat 2
- H332: Harmful if inhaled; Acute Tox Inh, Cat 4
- H351: Suspected of causing cancer; GHS Carcinogenicity, Cat 2
- H373: May cause damage to organs through prolonged or repeated exposure; Target Organ, Repeated, Cat 2
- H400: Very toxic to aquatic life; Acute Env Tox, Cat 1
- H401: Toxic to aquatic life; Acute Env Tox, Cat 2





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H410: Very toxic to aquatic life with long lasting effects; Chronic Env Tox, Cat 1 H411: Toxic to aquatic life with long lasting effects; Chronic Env Tox, Cat 2 H412: Harmful to aquatic life with long lasting effects; Chronic Env Tox, Cat 3

THIS SAFETY DATA SHEET CONTAINS THE FOLLOWING REVISIONS:

Composition: Component Table information was modified. Section 07: Handling and Storage - Handling information was modified. Section 12: information was modified.

Section 14: Special Provisions information was added.

THIS MSDS COVERS THE FOLLOWING MATERIALS: DIESEL EFFICIENT | DIESEL NO. 2 | ESSO DIESEL FUEL | EXXON DIESEL FUEL | EXXON SYNERGY DIESEL EFFICIENT | LOW SULFUR DIESEL | MARINE DIESEL FUEL | MOBIL DIESEL EFFICIENT | MOBIL DIESEL FUEL | MOBIL SYNERGY DIESEL EFFICIENT | ULTRA LOW SULFUR DIESEL | WINTERIZED DIESEL FUEL

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Hydrocal 300 A.2



MATERIAL SAFETY DATA SHEET

| AND DED TO REACTLY TO PRIME HERE, LP | | DISTRIBUTED BY |
|---------------------------------------|---|---|
| 1. Product and Company Identification | | R. E. CARROLL, INC. |
| Material name | Hydrocal 300 | 1570 NORTH OLDEN AVENUE TRENTON, NJ 08638-3204 |
| Version # | 01 | 609-695-6211 / 800-257-9365 |
| Revision date | 03-18-2011 | 005-050-02117 000-207-0000 |
| CAS # | Mixture | |
| Manufacturer information | Calumet Specialty Products Partners, L.P. 2780 Waterfront Pkwy E. Dr. Suite 200 Indianapolis, IN 46214 US www.calumetfubricants.com Technical Services 317-328-5660 CHEMTREC International 703-527-3887 | |
| 2. Hazards Identification | | |
| Potential health effects | | |
| | | |

| Eyes | Health injuries are not known or expected under normal use. |
|---------------------------------|---|
| Skin | Health injuries are not known or expected under normal use. |
| Ingestion | Health injuries are not known or expected under normal use. |
| Potential environmental effects | Ecological injuries are not known or expected under normal use. |

3. Composition / Information on Ingredients

| Non-hazardous components | | CAS # | Percent |
|---|--|------------|----------|
| Heavy Naphthenic Clay Treated Distillates (petroleum) | | 64742-44-5 | 90 - 100 |
| Heavy Hydrotreated Naphthenic Distillates (petroleum) | | 54742-52-5 | 2.5 - 10 |
| 4. First Aid Measures | | | |
| First ald procedures | | | |
| Eye contact | Rinse with water. Get medical attention if irritation develops and persists. | | |
| | | | |

Skin contact Rinse skin with water/shower. Get medical attention if imitation develops and persists. Inhalation If breathing is difficult, remove to fresh air and keep at rest in a position comfortable for breathing. Call a physician if symptoms develop or persist. Ingestion Rinse mouth. If ingestion of a large amount does occur, call a poison control center immediately. 5. Fire Fighting Measures Flammable properties Not flammable by OSHA criteria. Not combustible by OSHA criteria. Extinguishing media Suitable extinguishing Water fog. media Unsuitable extinguishing Water. Do not use water jet as an extinguisher, as this will spread the fire. media Protection of firefighters Protective equipment and Wear suitable protective equipment. precautions for firefighters Fire fighting Move containers from fire area if you can do so without risk. equipment/instructions

6. Accidental Release Measures

Keep unnecessary personnal away. Do not touch damaged containers or spilled material unless wearing appropriate protective clothing. Keep people away from and upwind of apil/leak. Keep upwind. Keep out of low areas.

Material name: Hydrocal 300

Personal precautions

Version #: 01 Revision date: 03-18-2011 Print date: 03-18-2011

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DIGTOR PEO DV



Acquisition Directorate Research & Development Center 1/4

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| filow of material, fit his is without risk. Dike the splited mataral, where this is possible. Correcting up possible. Correcting page to an eminor ensulue containers. Following product recovery, flush area with water. Small Splits: Wipe up with absorbant material (e.g. cloth, fleece). Clean surface throughly to monove resulue containers. Following product recovery, flush area with water. Small Splits: Wipe up with absorbant material (e.g. cloth, fleece). Clean surface throughly to monove resulue containers. Following product recovery, flush area with water. Small Splits: Wipe up with absorbant material (e.g. cloth, fleece). Clean surface throughly to material from direct surfage. Keep new from heat, sparks and open flame, sources of hest or sources of ignition. Prote material from direct surfage. Keep new from heat, sparks and open flame. 8. Exposure Controls / Personal Protection Occupational exposure limits US. AGGIH Throshold Limit Values Components Type Value Form Heavy Hydrotreated NWA 5.0000 mg/m3 Inheliable fraction, Naphthenic Jobilisties (petroloum) (6474-52-5). US. OSHA Table Z-1 Limits for Air Contaminants (29 CFR 1910.1000) Components Type Value Form Heavy Hydrotreated PEL 500.0000 ppm Mash Henic Jobilisties (petroleum) (6474-52-5). S.0000 mg/m3 Mist. 2000,0000 mg/m3 Mist. 2000 | | | | - Incomentation and a Description |
|--|--|---|---|---|
| posible. Cover with plastic sheet to prevent spreading. Absorb in vermicatile, any sand or earth and place into containers. Following product recovery, flush area with water. Small Spills: Wipe up with absorbant material (e.g. doth, fleece). Clean surface thoroughly to remove residual containmation. Near return spills in original containers for re-use. For waste disposal, see section 13 of the MSDS. 7. Handling DD NOT handle, store or open near an open flame, sources of hest or sources of ignition. Prote material from direct surfight. Avoid prolonged exposure. Storage Keep away from heat, sparks and open flame, 8. Exposure Controls / Personal Protection Occupational exposure limits US. ACell Threshold Limit Values <u>Components Type Value Form</u> Heavy Hydrotreated Nay Hydrotreated Protection Components Type Value Form Heavy Hydrotreated Protection Components Type Value Form Heavy Hydrotreated PEL 500000 ppin Maphthenic Distillates (petroloum) (64742-52-5) US. OSEN Table Z-1 Limits for Air Contaminante (29 CFR 1910.1000) Components Type Value Form Heavy Hydrotreated PEL 500.0000 ppin Source spreader of the set or sources, local exhaust ventilation rate and how the set of up of the set or sources and acceptable level. Petronal protective equipment Eye / face protection Skin protection Normal work cothing (cong sleeved shifts and long pants) is recommended. When workers are facing concentrations above the exposure limits. If exposure limits have not been established, maintain airborned. Skin protection Normal work cothing (cong sleeved shifts and long pants) is recommended. Skin protection Normal work cothing (cong sleeved shifts and long pants) is recommended. Skin protection Normal work cothing (cong sleeved shifts and long pants) is recommended. Skin protection Normal work cothing (cong sleeved shifts and long pants) is recommended. Skin protection Normal work cothing (cong sleeved shifts and long pants) is recommended. When workers are facing concentrations above the exposure limits. If exposure limits the Liquid | Methods for containment | flow of material, if this is without risk. Dike | the spilled material, where t | n immediate area). Stop the his is possible. Prevent |
| remove residual contamination. Never return apilis in original containers for re-use. For waste disposal, see section 13 of the MSDS. 7. Handling and Storage Handling DO NOT handle, store or open near an open fisme, sources of hest or sources of ignition. Prote material from direct surfight. Avoid prolonged exposure. Storage Keep away from heat, sparks and open fisme. 8. Exposure Controls / Personal Protection Occupational exposure limits US. ACGIH Threshold Limit Values Components Type Value Form Heavy Hydrotreated TWA 5.0000 mg/m3 Inheiable fraction. Naphtheric Distillates (petroleum) (64742-52-6) US. OSHA Table Z-1 Limits for Air Contaminante (29 CFR 1910.1000) Components Type Value Form Heavy Hydrotreated PEL 500.0000 ppm Naphtheric Distillates (petroleum) (64742-52-6) S.0000 mg/m3 Mist. 2000.0000 mg/m3 Engineering controls Good general ventilistion (typically 10 air changes per hour) should be used. Ventilation rates should be matched to conditions. If applicable, use process endosures, local exhaust ventilation or other engineering controls Eye wash fountain is recommended. Skin protection Eye wash fountain is arbonal wind long pents) is recommended exposure limits. Physical & Chemical Properties Appearance Product is water-white to pale straw liquid. Form Liquid. Color Mot available. Odor Mot available. Odor Mot available. Metting point/Freezing point Not available. Metting point/Freezing point Not available. Metting point/Freezing point Not available. | Methods for cleaning up | possible. Cover with plastic sheet to prevent spreading. Absorb in vermiculite, dry sand or | | miculite, dry sand or earth |
| MSDS. 7. Handling and Storage Handling DO NOT handle, store or open near an open flame, sources of hest or sources of ignition. Prote material from direct sunlight. Avoid protongod exposure. Storage Keep away from heat, sparks and open flame. 8. Exposure Controls / Personal Protection Occupational exposure limits US. ACGIN Threshold Limit Values Components Type Value Form Heavy Hydrotreated Naphtheat (29 CFR 1910.1000) Components Type US. ACGIN Expland TWA Naphthenic Distillates (petroloum) (6474-25-25) Type US. OSHA Table 2-1 Limits for Air Contaminants (29 CFR 1910.1000) Components Components Type Value Form Heavy Hydrotreated (petroloum) (6474-25-25) 5.0000 mg/m3 Mist. 2000,0000 mg/m3 (petroloum) (6474-25-25) S.0000 mg/m3 Mist. 2000,0000 mg/m3 Engineering controls Good general ventilation (typically 10 air change per hour) should be used. Ventilation rates should be matched to conditions. If applicable, use process enclosures, local exhaust ventilation ar other angineering controls Good general ventilation (typically 10 air change per hour) should be used. Ventilation rates should be matched to conditions. If applicable, use process enclosures, local exhaust ventilatin a coroanue wich dobin | | | rial (e.g. cloth, fleece). Clea | n surface thoroughly to |
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| material from direct sunlight. Avoid protorigad exposure. Storage Keep away from heat, sparks and open flame. 8. Exposure Controls / Personal Protection Occupational exposure limits US. ACGIH Threshold Limit Values Components Type Value Form Measy Hydrotreated (betroloum) (64742-52-5) TWA 5.0000 mg/m3 Inhelable fraction. Components Type Value Form Components Type Value Form Heavy Hydrotreated (betroloum) (64742-52-5) PEL 500.0000 ppm 5.0000 mg/m3 Mist. 2000.0000 Components Type Value Form | 7. Handling and Storage | | | |
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| Cocupational exposure limits US. ACGIH Threshold Limit Values Type Value Form Heavy Hydrotreated Naprithenic Distillates (betroleum) (64742-52-5) TWA 5.0000 mg/m3 Inheisble fraction. Components Type Value Form Components Type Value Form Heavy Hydrotreated Naprithenic Distillates (petroleum) (64742-52-5) PEL 500.0000 ppm Components Type Value Form Heavy Hydrotreated (petroleum) (64742-52-5) PEL 500.0000 ppm Scood general ventilation (typically 10 air changes per hour) should be used. Ventilation rates should be matched to conditions. If applicable, use process enclosures, local exhaust ventilation or other engineering controls to maintain airborne levels below recommended exposure limits. If exposure limits have not been established, maintain airborne levels to an acceptable level. Personal protective equipment Eye / face protection Respiratory protection Kye wash fourtain is recommended. Skin protection Respiratory protection Kye wash fourtain is recommended. Skin protection Respiratory protection When work clothing (long sleeved shifts and long pents) is recommended. Shipaical & Chemical Properties Liquid. Color Not | Storage | Keep away from heat, sparks and open fla | ime. | |
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| General hygiene considerationsHandle in accordance with good industrial hygiene and safety practice.9. Physical & Chemical ProtectiesAppearanceProduct is water-white to pale straw liquid.Physical stateLiquid.FormLiquid.ColorNot available.Odor thresholdNot available.pHNot available.Vapor pressureNot available.Vapor densityNot available.Water point/Freezing pointNot available. | Respiratory protection | | above the exposure limit the | y must use appropriate |
| Appearance Product is water-white to pale straw liquid. Physical state Liquid. Form Liquid. Color Not available. Odor Not available. Odor threshold Not available. pH Not available. Vapor pressure Not available. Vapor density Not available. Metting point/Freezing point Not available. | | | hygiene and safety practice | 3. |
| Physical state Liquid. Form Liquid. Color Not available. Odor Not available. Odor threshold Not available. pH Not available. Vapor pressure Not available. Vapor density Not available. Melting point/Freezing point Not available. | 9. Physical & Chemical Pr | operties | | |
| Physical state Liquid. Form Liquid. Color Not available. Odor Not available. Odor threshold Not available. pH Not available. Vapor pressure Not available. Vapor density Not available. Melting point/Freezing point Not available. | Appearance | Product is water-white to pale straw liquid | | |
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| Odor Not available. Odor threshold Not available. pH Not available. Vapor pressure Not available. Vapor density Not available. Melting point/Freezing point Not available. | Form | | | |
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| pH Not available. Vapor pressure Not available. Vapor density Not available. Melting point/Freezing point Not available. | Odor | Not available. | | |
| Vapor pressure Not available. Vapor density Not available. Melting point/Freezing point Not available. | Odor threshold | Not available. | | |
| Vapor density Not available. Melting point/Freezing point Not available. | pH | Not available. | | |
| Melting point/Freezing point Not available. | Vapor pressure | Not available. | | |
| | Vapor density | Not available. | | |
| Solubility (water) Not available. | Melting point/Freezing point | Not available. | | |
| | Solubility (water) | Not available. | | |
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| | 0.9087 |
|--|---|
| elative density | Not available. |
| lash point | 350 °F (176.7 °C) Cleveland Open Cup |
| lammability limits in air, upper, 6 by volume | Not avsilable. |
| lammability limits in air, lower, 6 by volume | Not available. |
| uto-ignition temperature | > 600 °F (> 315.6 °C) estimated |
| liscosity | 300 SUS |
| our point | -26 °F (-32.2 °C) ASTM D97 |
| ither data | |
| Density | 0.9025 g/cm3 |
| Flammability class | Combustible IIIB |
| Viscosity temperature | 100 °F (37.8 °C) |
| 0. Chemical Stability & Re | eactivity Information |
| chemical stability | Material is stable under normal conditions. |
| conditions to avoid | Heat, flamos and sparks. Avoid temperatures exceeding the flash point. |
| ncompatible materials | Not available. |
| lazardous decomposition roducts | No hazardous decomposition products are known. |
| 1. Toxicological Informat | ion |
| Carcinogenicity | This product is not considered to be a carcinogen by IARC, ACGIH, NTP, or OSHA. |
| 2. Ecological Information | 1 |
| cotoxicity | This product has no known eco-toxicological effects. |
| Persistence and legradability | Not evailable. |
| 3. Disposal Consideratio | ns |
| Disposal instructions | Collect and reclaim or dispose in sealed containers at licensed waste disposal site. This product, in its present state, when discarded or disposed of, is not a hazardous waste according to Federal regulations (40 CFR 261.4 (b)(4)). Under RCRA, it is the responsibility of the user of the product to determine, at the time of disposal, whether the product meets RCRA criteria for hazardous waste. Dispose in accordance with all applicable regulations. |
| Contaminated packaging | Empty containers should be taken to an approved waste handling sits for recycling or disposal. |
| 14. Transport Information | |
| тот | |
| Not regulated as dangerous goods | 8. |
| 15. Regulatory Information | |
| JS federal regulations | All components are on the U.S. EPA TSCA Inventory List. |
| 8 | |
| Drug Enforcement Adminis | CERCLA/SARA Hazardous Substances - Not applicable. tration (DEA). List 2, Essential Chemicals (21 CFR 1310.02(b) and 1310.04(f)(2) |
| Not regulated | nanon femoly mar y' essential quemicals for our renoration and renordities |
| DEA Essential Chemical Co | de Number |
| Not regulated Drug Enforcement Adminis | tration (DEA). List 1 & 2 Exempt Chemical Mixtures (21 CFR 1310.12(c)) |
| Not requiated | annen feinich mer im einender annennen minnen eine fei eini reinitefelt |
| DEA Exempt Chemical Mixt | ures Code Number |
| Not regulated | |
| CERCLA (Superfund) reportable None |) quantity |



| Hazard categories | Immediate Hazard - No | |
|---|--|----------------------------|
| The second se | Delayed Hazard - No | |
| | Fire Hazard - No | |
| | Pressure Hazard - No Réactivity Hazard - No | |
| Section 302 extremely hazardous substance | No | |
| Section 311 hazardous chemical | No | |
| Inventory status | | |
| Country(s) or region | Inventory name | On inventory (yes/no) |
| Australia | Australian Inventory of Chemical Substances (AICS) | Yes |
| Canada | Domestic Substances List (DSL) | Yes |
| Canada | Non-Domestic Substances List (NDSL) | No |
| China | Inventory of Existing Chemical Substances in China (IECSC) | Yes |
| Europe | European Inventory of Existing Commercial Chemical Substances (EINECS) | Yes |
| Europe | European List of Notified Chemical Substances (ELINCS) | No |
| Japan | Inventory of Existing and New Chemical Substances (ENCS) | No |
| Korea | Existing Chemicals List (ECL) | Yes |
| New Zealand | New Zealand Inventory | Yes |
| Philippines | Philippine Inventory of Chemicals and Chemical Substances (PICCS) | Yes |
| United States & Puerto Rico | Toxic Substances Control Act (TSCA) Inventory | Yes |
| *A "Yes" indicates that all compo | ments of this product comply with the inventory requirements administered by the | governing country(s) |
| State regulations | This product does not contain a chemical known to the State of Califo defects or other reproductive harm. | mia to cause cancer, birth |
| 16. Other Information | | |
| Further information | HMIS® is a registered trade and service mark of the NPCA. | |
| HMIS® ratings | Heatth: 0 Flammability: 1 Physical hazard: 0 | |
| NFPA ratings | Health: 0 Fiammability: 1 Instability: 0 | |
| Disclalmer | The information in the sheat was written based on the best knowledge available. | and experience currently |
| ssue date | 03-18-2011 | |
| This data sheet contains changes from the previous version in section(s): | This document has undergone significant changes and should be revi | ewed in its entirely, |

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APPENDIX B. OHMSETT TEST PLAN

Type I Sorbent Testing at Ohmsett – BSEE Project 1128 and USCG RDC Project 1011

1. Introduction

The purpose of this effort is for the Bureau of Safety and Environmental Enforcement (BSEE) and the United States Coast Guard Research and Development Center (USCG RDC) to test Type I sorbents at Ohmsett to provide performance data that will predict how a sorbent may perform during typical field use. Type I sorbents are specified in the ASTM F726-17 standard as roll, film, sheet, pad, and blanket sorbents.

For a sorbent to be considered effective in the field there are only a few functional characteristics of interest including:

- Recoverability of surface oil given a range of viscosities
- The volume of oil recoverable by the sorbent (maximum oil capacity)
- The rate at which the sorbent will adsorb (adsorption rate)
- The amount of water the sorbent may adsorb
- Buoyancy
- Ability to retrieve the sorbent when it is fully saturated
- Reusability for those sorbents who provide means of processing for reuse

These tests will attempt to characterize sorbents based on the characteristics of interest. Maximum oil capacity and adsorption rate will be determined by testing the sorbents to the ASTM standard ASTM F726-17. Sorbents will be tested using the Oil Adsorption-Short test (15-minute adsorption) and the Oil Adsorption-Long test (24-hour adsorption). In addition, they will be tested using the Oil Adsorption test with a 3-hour adsorption duration. These three data points will quantify the sorbents' maximum oil capacity and also allow the sorbents to be categorized based on their time to maximum sorption as detailed in Section 3 Task 1.

Sorbent samples will then be tested using the recently developed Field Scale Sorbent Test Method "Sorbent Water Uptake Test with Oil" to determine how much oil and water the sorbents will adsorb when subjected to both. During this test buoyancy and field retrievability will also be assessed.

Reusability will be tested for one of the sorbents. The manufacturer will supply the manual wringer system equipment (ECO HydroFlexTM) needed to process the sorbent for reuse along with written instructions and potentially a video. USCG RDC will coordinate with the manufacturer for delivery of this system to Ohmsett.



Testing will occur over a two-week time period with four sorbents and two different oils. All sorbents will be provided by the manufacturers. Hydrocal 300 and diesel will be used for the tests.

2. Background

BSEE recently initiated a program for evaluating oil pollution mitigation technologies to provide performance data to stakeholders to facilitate decision making for oil spill preparedness and response operations (TOST program). USCG RDC also has a project titled, "Emerging Pollution Response Technology Evaluation" with objectives that align with those of BSEE. During independent evaluation at Ohmsett, BSEE and USCG RDC will collect data through systematic and unbiased testing. They will develop a summary of test results, findings and recommendations for each sorbent product in a report. It will be used to inform Coast Guard Federal On-Scene Coordinators and other spill responders about what sorbents would be most effective for certain spill scenarios and understand potential limitations. The report will also help manufacturers better understand technology limitations and potentially re-design to be more effective. This report will be disseminated to stakeholders and to the public. This program is envisioned to be an annual effort where different technologies of interest will be assessed.

BSEE and USCG RDC plan to conduct testing in October 2022. For this effort, innovative sorbent products will be tested. sorbent information was solicited through a Request for Information announcement and sorbents submittals were evaluated using the RDC's recently developed technology evaluation process. Technologies that were highly rated were invited to submit their products for testing at Ohmsett using ASTM F726 testing and the recently developed sorbent field scale test protocol.

3. Test Plan

The evaluation will be conducted with the following sorbents:

1. Earthwise Sorbents Oil-Only Heavyweight Pads - Pad made from recycled polypropylene waste.

Product dimensions: 15" x 18" www.earthwisesorbents.com



2. MFNS Technologies OHM Sponge – Commercial sponge treated with a nanocomposite coating. Product dimensions: 12" X 12" x .5" www.mfns-tech.com

3. AquaFlex® Pad – Open-Cell Foam Technology Product dimensions: TBD



www.aqflx.com

4. Imbibitive Technologies Imbiber Fiber Product dimensions: TBD www.Imbiberbeads.com

Sorbents will be evaluated for the following characteristics:

- <u>Maximum Oil Capacity</u>: A volume measurement of oil adsorbed by a sorbent in which contact with additional oil or a longer exposure time will not add to the volume. (In field use it is not likely a sorbent will have sufficient time on the oil to recover its maximum capacity but this measurement provides an indication as to the potential oil recovery.)
- <u>Adsorption Rate:</u> For the purposes of this study the adsorption rate is proposed as the time necessary for a sorbent to reach maximum oil capacity. For this study sorbents will be categorized based on their adsorption rate. Additional details are provided in Task 1 detail.
- <u>Water Uptake:</u> The volume of water adsorbed into a sorbent when deployed onto an oil on water slick.
- <u>Buoyancy:</u> The ability of the sorbent to remain afloat indicated typically by a portion of the sorbent having freeboard.
- <u>Field Retrieval:</u> A measure of sorbent strength indicating the ability to retrieve it from a spill when fully saturated. (For this method, the approach taken is based on discussions with vendors and spill response personnel who have identified various recovery methods. Resultantly, the most challenging retrieval technique identified is the use of a standard pitch fork which is best practice recommended by vendor and used by oil spill response technician.
- <u>Reusability</u>: The ability of the sorbent to retain its initial performance after repeated adsorption/desorption cycles using a manual wringer system. *Note: This applies to AquaFlex*®'s *product only*.

Two test oils will be used for the sorbent evaluation. Test oils include diesel and Hydrocal 300.

Task 1. Maximum Oil Capacity and Adsorption Rate

Multiple adsorption tests will be conducted in parallel using ASTM F726-17 for Type I sorbents in the Ohmsett laboratory. These tests will be conducted to determine the maximum volume of oil that can be recovered by the sorbent. Sorbents samples will be tested using the ASTM F726-17 Oil Adsorption-Short test (15-minute adsorption) and the Oil Adsorption-Long test (24-hour adsorption). However, previous tests with various sorbents indicated that of the samples tested, the majority reached full adsorption in a few hours or less. Therefore, a 3-hour adsorption time increment will be added. This will allow the sorbents to be grouped into adsorption rate categories. For purposes of this test the categories will include:



| Adsorption Rate Category | Time for Full Adsorption (hours) |
|--------------------------|-------------------------------------|
| 1 | 0.25 or less |
| 11 | Greater than 0.25 and less than 3.0 |
| III | Greater than 3.0 |

Note that these categories are newly defined and as such are not standardized.

Standard ASTM procedure will be followed including conditioning of the sorbents, standard drip times, and triplicate testing.

Task 2. Sorbent Water Uptake

The ASTM F726 test method does not include a means to quantify the amount of oil and water a sorbent will adsorb when exposed to both and oil and water. The sorbents will therefore be tested using the "Sorbent Water Uptake Tests with Oil" test method developed recently by Ohmsett/BSEE. The intention of this test method is to determine if a sorbent sample will adsorb water when exposed to a relatively thin oil slick on water. The approach logic is to expose test sorbents to "some" oil, a volume less than the known capacity thereby ensuring contact with both oil and water. This condition allows for the sorbent to potentially adsorb water which may be due to wicking action or possibly inferior hydrophobic properties. Once values have been obtained for the weight of oil and water adsorbed, the amount of each fluid adsorbed can be represented as a ratio, hence "Oil to Water Ratio."

Sorbents will be exposed to a scaled volume of oil equal to half their maximum oil capacity determined by the ASTM F726 Oil Adsorption – Long Test (24-hours) results. The relative amount of oil will be added on water in the test tray to create a surface slick. The sorbent will be applied to the water surface, allowed to adsorb incrementally for a total time at which they do not further adsorb fluid, and weighed after a 30-second drip time. This test method will utilize the steps developed in the field scale test protocol including the following:

- 1. Perform load cell calibration
- 2. Measure sorbent dimensions
- 3. Conduct Procedure for Compensating for Oil Weight on the Rack (described at the end of section 3).
- 4. Document initial weight of rack and sorbent sample
- 5. Place sorbent sample onto rack and lower until sample floats freely on the fluid surface
- 6. Begin timer and video or time-lapse documentation
- 7. After 15 minutes begin load cell data collection and withdraw support rack and sorbent for 30 seconds before re-immersing sample in test fluid
- 8. After 30 minutes begin load cell data collection and withdraw support rack and sorbent for 30 seconds before re-immersing sample in test fluid
- 9. After 60 minutes begin load cell data collection and withdraw support rack and sorbent for 30 seconds



- 10. If the sorbent continues to increase weight at the 60-minute adsorption increment, the sorbent may be subjected to additional incremental re-immersions with a goal of reaching maximum adsorption.
- 11. Swing the sorbent and sorbent support rack away from the fluid tray and dispose of the sorbent (to prevent any fluid from dripping back into the fluid tray during sorbent removal)
- 12. Decant free water from the fluid tray
- 13. Collect the oil and remaining water into graduated cylinders and allow it to separate
- 14. Calculate weight of oil recovered from tray by converting volume to weight using specific gravity
- 15. Subtract recovered oil weight from initial weight of oil dispensed to obtain weight of oil removed by sorbent
- 16. Subtract weight of oil in sorbent and sorbent tare weight from post-test net sorbent and fluid weight to obtain weight of water in sorbent

Each sorbent will be tested to this method in both quiescent conditions and in simulated wave conditions.

Task 3: Buoyancy

It is important that sorbents maintain buoyancy during field use for effective adsorption and to be able to locate and retrieve them after use. Sorbents will not be tested specifically for buoyancy. However, during the maximum capacity and water uptake tests, the testers should note observations related to buoyancy and report any loss of buoyancy as part of the test results.

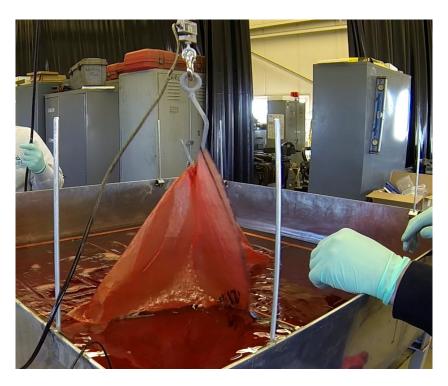
Task 4: Field Retrieval

The ability of a fully saturated sorbent to be retrieved intact is an important characteristic for successful field use. The field retrieval test is a destructive test designed to measure the load at which a sorbent might fail during typical field retrieval.

Discussions with responders identified an ordinary pitchfork as a commonly used method of retrieval for sorbents. For this reason, the retrieval test was designed to impose the most stringent yet practical approach of retrieving fully saturated sorbents by employing a single typical pitchfork prong formed into a hook and suspended on a load cell. Steps include the following:

- 1. A sorbent that has undergone the full maximum oil/water adsorption is placed on a fluid tray and lowered back into the test fluid.
- 2. The sorbent is allowed five minutes to re-saturate.
- 3. The sorbent is attached to the pitchfork prong by puncturing it 15.2 cm (6 in) inboard shortest edge.
- 4. With load cell operating, the sorbent is raised from the fluid.
- 5. The sorbent passes if it remains stable on the prong and fails if it tears from the prong.
- 6. Sorbents that passed may be further subjected to a downward force until failure is reached. In this case the force will be recorded. The figure below shows a single-ply polypropylene sorbent undergoing the field retrieval test.





Task 5: Reusability (with AquaFlex® sorbent only)

AquaFlex®'s Open-Cell Foam has been previously tested at Ohmsett, but the focus this time around will be its reusability, or how well it is able to recover, retain, and allow extraction of oil through a wringer system after repeated uses. The sorbent's maximum oil capacity, adsorption rate, water uptake and its buoyancy will be monitored after at least five (5) adsorption/desorption cycles. BSEE and USCG RDC will also evaluate the ease of use of using ECO HydroFlexTM and determine whether or not it is feasible to use in the field during an actual spill response incident, including observations about transportability and portability.

Procedure for Compensating for Oil Weight on the Rack

The sorbent support rack, instrumental in the submersion and handling of sorbents, gains weight once coated in test fluid. Although the added oil weight is typically small relative to the support rack, it may be significant relative to weight measurements for lightweight sorbents such as single-ply polypropylene sorbents.

This procedure will allow compensation for oil weight on the rack. This procedure provides repeatable results and allows for continued minimization of external disruption or interference during the testing process.

- 1. Obtain sorbent sample pre-test weight (typically measured on a dry support rack)
- 2. Immerse the support rack without sorbent sample into the test fluid for approximately two minutes
- 3. Raise the rack from the test fluid while recording load cell readings and obtain a "wet rack" tare weight once a stable weight is reached
- 4. Place sorbent sample on support rack and document net weight as initial weight



4. Pre-Test Activities

- Ohmsett will prepare the two field scale test apparatuses and ensure they are ready for testing.
- Ohmsett will procure equipment needed to conduct ASTM F726 tests including sample trays.
- Ohmsett will procure 300 gallons of diesel and Hydrocal 300.
- BSEE will process visitors for needed base access.
- Ohmsett will assist the with the development of the detailed test plan.
- Ohmsett will develop the appropriate forms, logs, and checklists to assure the appropriate documentation of data, notes, QA/QC during testing.
- BSEE/USCG RDC will arrange for delivery of the sorbents to be tested. Ohmsett will receive the sorbents and ensure that the stated quantities/types were received.

5. Other Requirements

- Ohmsett shall perform health, safety and quality assurance monitoring of personnel, equipment and methods during testing.
- All materials handling equipment (forklifts, cranes, etc.) and Ohmsett infrastructure shall be operated by ARA/Ohmsett personnel.
- ARA/Ohmsett shall manage all test fluids and Ohmsett based instrumentation used in this test program.
- Ohmsett will assist BSEE/Serco in resolving any unexpected issues during the experiment as needed
- Ohmsett shall perform test fluids sampling and laboratory analyses of physical properties. Upon completion of testing, Ohmsett shall de-rig/decon equipment; assist with demobilization, equipment removal and packing as necessary.
- Ohmsett shall unpack, use, repack and ship the manual wringer system (ECO HydroFlex[™]) back to AquaFlex[®] using the pre-paid shipping label.
- Ohmsett shall provide copies of all notes and recorded observations, as well as other data as appropriate in a Data Summary Binder for submission to BSEE and USCG RDC representatives.

6. Deliverables/Documentation

Ohmsett shall maintain accurate and complete files documenting all work performed. Documentation includes all calculations, assumptions, sources of information, work papers and other data that may be present. All information developed or collected is Government property and will be available for Government inspection upon request. These records will be maintained until the contract is completed, at which time all records will be transferred to the Bureau of Safety and Environmental Enforcement without



delay. The contractor is required to maintain all test information as proprietary for a period of five (5) years after issuance.

All deliverables produced by Ohmsett shall be submitted to the BSEE COR and to the USCG RDE Project Manager. An original copy should be maintained at the Ohmsett Facility. A copy of the cover letter documenting distribution of deliverables shall be sent to the BSEE COR, and USCG RDC Project Manager under separate cover.

1. Data Summary Binder/Technical Report

Ohmsett will compile all manually and computer-generated data for incorporation into the Data Summary Binder. Two copies will be provided and will contain the following: Statement of Work, Executive Summary, Engineer Notes, QC Checklist, Oil Properties Data, Environmental Data, Daily Test logs, Photo/Video documentation. **Due:** Within 45 calendar days of completion of testing

2. Visual Documentation

Ohmsett will provide video documentation of all pertinent aspects of the test on digital media. Digital still photographs of selected events will also be provided on digital media, with Thumbnail Prints of select photos (where applicable). **Due:** Within 45 calendar days of completion of testing

3. Monthly Status Report

Task Order current and planned activities, major purchases, schedule, documentation and milestones will be included in a monthly status report deliverable to BSEE. **Due:** Fifteen (15) calendar days after the end of each month.

4. Quality Control Documentation

Quality control checks will be performed on all test instrumentation and documented by the designated QA/QC representative and included in deliverable documentation. All data that is collected and analyzed; all calculations associated with the data; instrumentation and equipment used; laboratory procedures; and results for a project will be maintained and identified for the specific project.

Due: As noted above - incorporated in Data Summary, where necessary

