Application of the Field Scale Test Protocol for Type I Sorbents Recovering Oil on Water

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Abstract

There presently exists no industry recognized test protocol for the evaluation of a sorbent product within a simulated environment fundamentally analogous to typical field conditions. Of particular note, existing standards do not offer a method for quantifying sorbent performance when deployed for the recovery of oil on water or evaluating sorbents at full or meso-scale size. While the overall qualification of one sorbent's ability to perform as compared to another sorbent may be determined by testing methods established by the ASTM standard F726-17 *Standard Test Method for Sorbent Performance of Adsorbents for use on Crude Oil and Related Spills*, this method serves only to quantify the maximum adsorption capacity of a sample sorbent in a laboratory environment.

Under this existing standard, sorbents are exposed to a bath of oil and left undisturbed for a prescribed duration in order to obtain maximum adsorption values. As such, a more insightful method with which to better differentiate the functional characteristics of a sorbent product as seen in a non-ideal field environment is required. In 2018, a joint initiative between Ohmsett and the Bureau of Safety and Environmental Enforcement (BSEE) sought to explore the existing ASTM standard, and to expand upon it. The result of this ongoing development culminated in the successful demonstration of a variety of sorbent materials in August of 2019, as evaluated by 27 field scale tests and 46 ASTM F726-17 tests.

In support of this effort, a new test apparatus was designed and fabricated over subsequent testing phases to accommodate typical sorbent pads at their full size, as well as similarly sized samples of other configurations. Techniques were designed for determining the maximum uptake volumes and ratios of oil and water for a given sorbent, while continuously striving to maintain a broad reproducibility. Data resultant from testing performed with a selection of varying sorbent materials revealed significant performance variances, as well as highlighted unique behaviors and characteristics. These findings provide sorbent performance data critical to the evaluation and the direction of oil spill response and manufacturing efforts.

1.0 Introduction

There presently exists no industry recognized test protocol for the evaluation of a sorbent product within a simulated environment fundamentally analogous to typical field conditions. Of particular note, existing standards do not offer a method for quantifying sorbent performance when deployed for the recovery of oil on water or for evaluating sorbents at full or meso-scale size. The overall qualification of a sorbent's ability to adsorb as compared to another sorbent

may presently be assessed by testing methods established by the ASTM standard F726-17 *Standard Test Method for Sorbent Performance of Adsorbents for use on Crude Oil and Related Spills*. (ASTM, 2017) This test method serves to quantify the maximum adsorption capacity of a sample sorbent in a laboratory environment. Under this existing standard, sorbent samples sized to 13 cm x 13 cm (or larger if necessary to meet a minimum weight of 4 g) are exposed to a bath of oil and left undisturbed for a prescribed duration (15 minutes or 24 hours) in order to obtain maximum adsorption values. It further provides a qualitative assessment of a sorbent's ability to adsorb oil in the presence of water.

ASTM F726-17 provides valid and necessary performance data for sorbents; however, it does not offer an insightful method with which to differentiate the functional characteristics of one sorbent material from another as would be seen in a non-ideal field environment. Additionally, criticisms levied in a 2015 paper titled "Standardization of Oil Sorbent Performance Testing", published in the *Journal of Testing and Evaluation*, concluded that the majority of published tests on oil sorbents did not make use of any uniform standards, and crucial test parameters such as drip times were not consistent which precluded any meaningful data comparison between data sets. The paper also stated that a sample size of 13 cm x 13 cm may not be large enough to account for variations in manufacturing process of the full-size pads, potentially resulting in misrepresentative test data (Bazargan et al., 2015).

In 2018, a joint initiative between Ohmsett and the U.S. Department of the Interior's Bureau of Safety and Environmental Enforcement (BSEE) sought to explore the existing ASTM F726-17 standard and to expand upon it with the development of a Field Scale Test Protocol (denoted test protocol.) In the initial stages of the investigation, the test protocol was developed and refined to provide suitable field use characteristics beyond the limiting sample size and approach of ASTM F726-17, while still affording manageable mobility and control. This previous BSEE study, "*Development of a Field Scale Test Protocol for Type I Sorbents Recovering Oil on Water, Continuation of Project 7024*," was conducted at Ohmsett in April 2018, and February and March 2019 (DeVitis et al., 2019). The test protocol was further evolved, and used as the basis for an external customer test conducted in August 2019 in which multiple sorbents were tested in 27 field scale tests and 46 ASTM F726-17 tests.

In support of the test protocol development effort, a new test apparatus was designed and fabricated to accommodate typical sorbent pads at their full size, typically 38 cm (15 in) x 51 cm (20 in), and sorbent roll and blanket samples up to 91 cm (36 in) x 91 cm (36 in). Methods for determining the maximum oil uptake volumes and ratios of oil and water established during test protocol development were evolved with the continuing objective of maintaining broad reproducibility. Data resultant from testing performed with a selection of varying sorbent materials revealed significant performance variances and highlighted unique behaviors and characteristics. These findings provide sorbent performance data critical to the evaluation and direction of oil spill response and manufacturing efforts.

2.0 Operations at Ohmsett

Ohmsett is the National Oil Spill Response & Renewable Energy Test Facility located in Leonardo, New Jersey. Ohmsett has facilitated the testing of a wide variety of spill countermeasures in a controlled, repeatable, and safe environment since its construction in 1974. BSEE manages the facility as part of its mandated requirements by the Oil Pollution Act of 1990 (OPA, 1990).

3.0 August 2019 Customer Testing

Evaluations performed in August 2019 focused on several sorbent materials designed to be reusable. Testing involved variables including sorbent material, oil viscosity, and static and mix energy scenarios. The sorbents were tested to the ASTM F726-17 standard to assess maximum oil capacity. They were then tested to assess their performance in an oil-on-water environment. Section 5 describes test methods developed for the test protocol as well as an evolved test method used during the August 2019 tests. In addition, the customer was interested in assessing sorbent reusability, and several tests were conducted to assess this characteristic.

4.0 Field Scale Test Protocol Development

The field scale test protocol development effort included the design, fabrication and evaluation of the test apparatus, and development of methods to assess sorbent performance characteristics of interest as described below.

4.1 Performance Characteristics of Interest

The primary performance characteristics considered important to define the practical effectiveness of a sorbent were as follows:

a. Maximum Oil Capacity: A volumetric measurement of oil adsorbed by a sorbent taken at a point where neither contact with additional oil, nor a longer exposure time would result in an increase to the volume.
b. Oil Adsorbed: A volumetric measurement of oil adsorbed by a sorbent when deployed onto an oil slick floating on water, taken at prescribed time intervals.
c. Water Uptake: A volumetric measurement of water adsorbed by a sorbent when deployed onto an oil slick floating on water, taken at prescribed time intervals.

Additional secondary performance characteristics considered to define the practical effectiveness of a sorbent were as follows:

d. Oil Viscosity Range: Consideration given towards acceptable oil recovery by the sorbent over a range of viscosities.
e. Buoyancy: The ability of the sorbent to remain afloat indicated typically by a portion of the sorbent having freeboard.
f. Field Retrieval: A measure of sorbent strength indicating the ability to retrieve it from a spill when fully saturated.
g. Manual Oil Recovery: A volumetric measurement of oil manually recovered from a sorbent by way of a wringer roller following adsorption test. Note: this performance characteristic was not initially considered during the test protocol development but was added for the August 2019 tests.

4.2 Test Apparatus Design

The overarching requirement during the test protocol development process was to provide test methods and a test apparatus that could be duplicated and implemented to produce replicable test results at a reasonable cost at Ohmsett as well as other test facilities. With that objective in mind, two test apparatuses were fabricated, identical except for the added capability of the second apparatus to impart surface energy during testing. Accounting for typical commercially available sorbent sizes while keeping the design within a manageable footprint area, the apparatuses were designed to accommodate sorbents up to 0.92 m (3 ft) x 0.92 m (3 ft).

Each test apparatus was comprised of the following:

1. Fluid Tray: The tray to contain test fluids was designed and fabricated using 0.48 cm (3/16 in) aluminum sheet material 102 cm (40 in) square x 20 cm (8 in) deep. To aid in visual identification within the test matrix, the trays were labeled as tray #1 (static) and tray #2 (with surface energy capability). Each tray had a bottom mounted 3.2 cm (1¼ in) drainpipe to facilitate draining and cleaning. Guide rods along the side walls of each tray allowed for a controlled removal of a sorbent sample from the test fluid by way of a hoist.
2. Sorbent Support Rack: The sorbent support rack was designed to support the sorbent while it was lowered into the test fluid and to extract the sorbent from the test fluid after exposure. Prior testing assessed the best method for orienting the sorbent while it dripped off the excess oil. Sorbents hung in a vertical orientation demonstrated the effects of gravity as fluid moved down through the sorbent and drained out the bottom corner, potentially leading to artificially low fluid volume retention when testing full or meso-scale samples. When supported horizontally as shown in Figure 1, the sorbent reached a point of no dripping in a relatively short time. To minimize the effects of gravity and to better facilitate a reproducible lift method, the use of a horizontal support rack was adopted. The support rack was designed to be lightweight and horizontally supportive of the sorbent while minimizing contact with the sorbent. Each support rack was fabricated using 1.9 cm (3/4 in) x 0.32 cm (1/8 in) aluminum angle topped with a thin gauge wire mesh.
3. Load Cell: The weight measurement device, key to obtaining accurate test data, was selected for its stated measurement accuracy within the desired weight range. A 0-45 kg S-Beam load cell with an accuracy of 0.02 % (0.01 kg) with an analog to digital converter was used in conjunction with a continuous data logging application, shown in Figure 2. The 0.02% accuracy corresponds to an approximate volume accuracy of 5 ml of oil. A calibration check was performed daily on the instrumentation system by placing a known weight on the load frame.

   In addition, the test apparatus with surface energy capability (tray #2) was equipped with the following:
4. Eccentric Drive: A variable speed motor, shown in Figure 3, was used to move the fluid tray back and forth to create surface energy. A right-angle gear reduction allowed for a controlled speed of 30-80 cycles per minute. The fluid tray supported on the linkage was able to freely move respective to the motor. The drive roller with an eccentricity of 1.3 cm (½ in) provided a 2.5 cm (1 in) overall stroke distance. During testing, speed was maintained at 60 cycles per minute. This setup attained surface waves approximately 1.3 cm (½ in) high with a period of 15.2 cm (6 in).

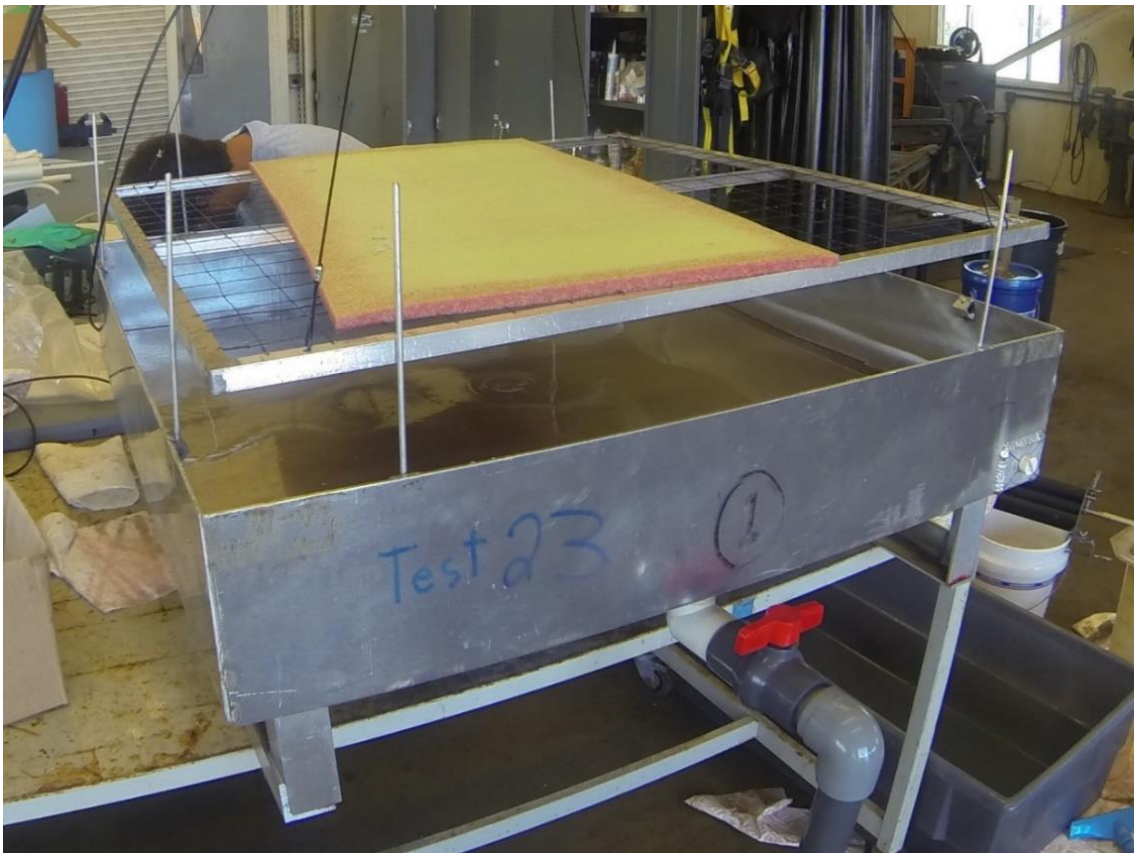

Figure 1 Static test apparatus with saturated sorbent pad positioned on the sorbent support rack suspended over the fluid tray

Figure 2 Screen showing load cell measurement in pounds (left) and Hoist-mounted load cell (right)

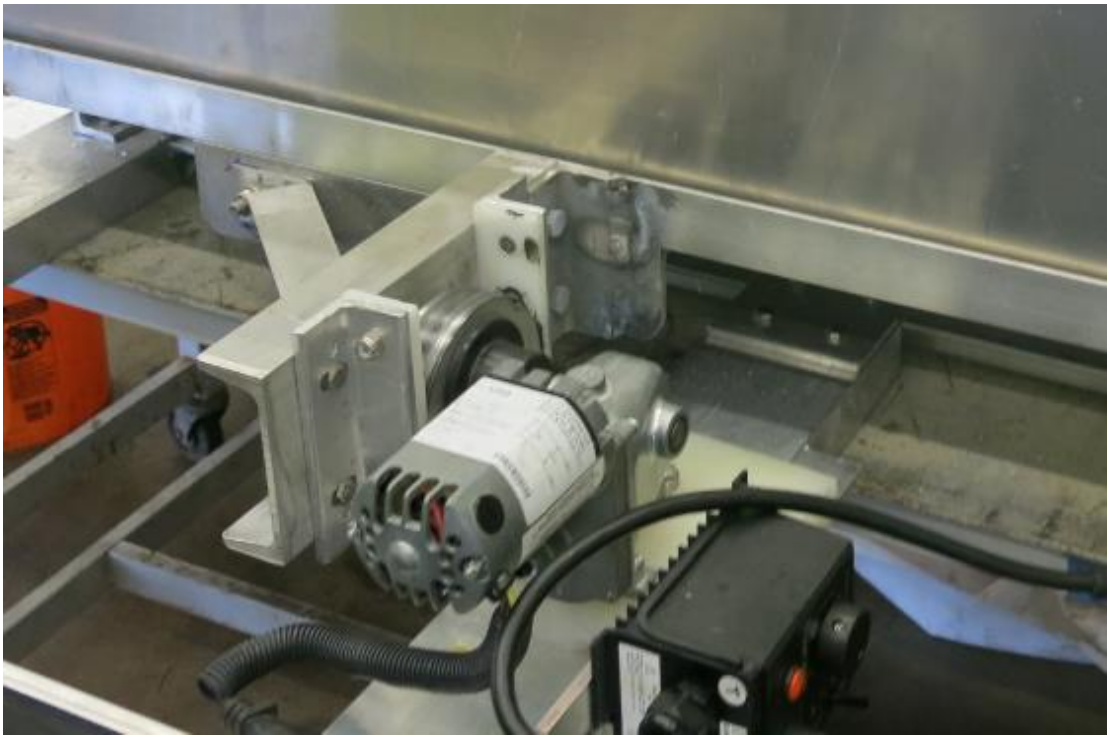

Figure 3 Variable speed motor used to horizontally move the fluid tray and induce surface energy to the test fluid

4.3 Test Fluid Selection:

The test protocol was designed such that it could be implemented at a reasonable cost at facilities besides Ohmsett. With that in mind, test fluids that would meet the following requirements were targeted:

- Readily available to anyone performing the sorbent tests
- Stable properties to allow for test repeatability
- Safe to use in typical test environments
- Properties closely aligned to oil properties and viscosity ranges defined in ASTM F726-17, shown in Table 1

Table 1 Oil properties and viscosity ranges as defined in ASTM F726-17

Oil Type	*Viscosity Range*	*Density Range*	*Example*
Light	1 to 10 cP	0.820 to 0.870 g/cm^3	Diesel fuel, mineral oil
Medium	200 to 400 cP	0.860 to 0.970 g/cm^3	Crude oil, canola oil, mineral oil
Heavy	1500 to 2500 cP	0.930 to 1.000 g/cm^3	Bunker C or residual fuel, mineral oil
Weathered	8000 to 10 000 cP	0.930 to 1.000 g/cm^3	Emulsified crude oil, mineral oil

Several fluids were used during the test protocol development phases including diesel fuel, Hydrocal 300 lube stock, commercially available hydraulic fluids, and commercially

available food grade mineral oils. An additional light refined oil, Hydrocal 38, was identified as a potential test fluid candidate but was not used for tests. One of the remaining questions is whether the use of test fluids such as mineral oils would accurately represent results that would be achieved by a sorbent adsorbing similar viscosity crude oils or other oils. This is an area that requires additional investigation.

For the August 2019 tests, the customer requested that diesel fuel #2 and FG 460 hydraulic oil be used for the tests. Water utilized in testing was obtained directly from the Ohmsett tank.

4.4 Sorbent Sample Group

Multiple sorbent products were used to fully investigate the unknown limitations of the test protocol during its development. Sorbents were chosen to represent a wide variety of products with respect to the sorbent base material and varying quality. This was important because of the difference in adsorption rates between sorbents. Single-ply polypropylene sorbents reached their maximum oil capacity in minutes, whereas a feather encapsulated mat sorbent required hours to reach its maximum oil capacity.

For the August 2019 customer tests, newly developed open-cell foam sorbent products were tested. Figure 4 shows a view of one of the open-cell foam sorbents tested.

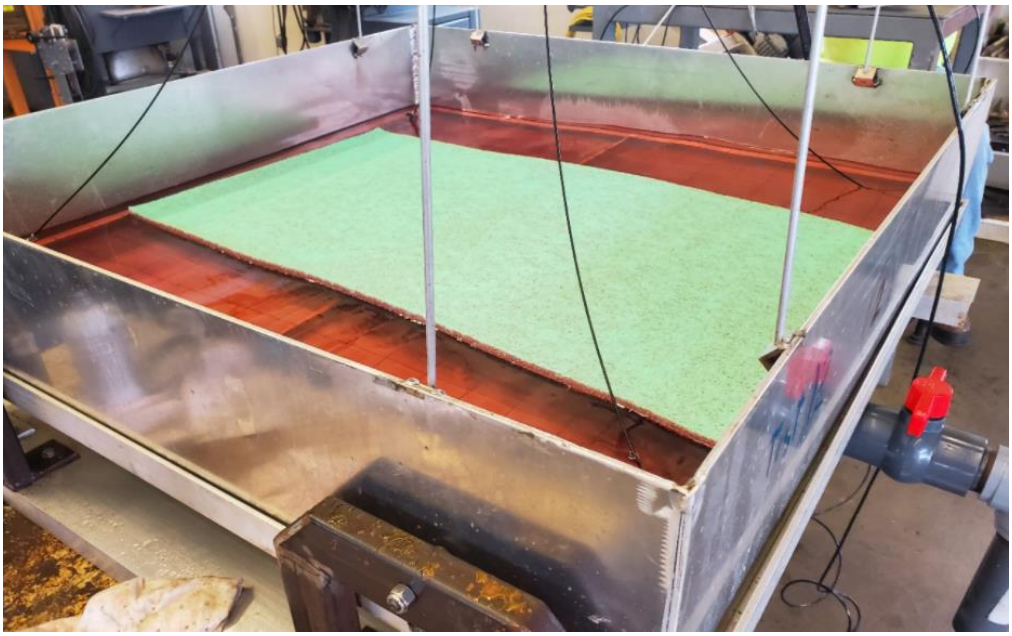

Figure 4 View of an open-cell foam sorbent sample undergoing testing

5.0 Areas of Investigation and Test Method Descriptions

The following subsections provide a description of the test methods developed for the test protocol, basis for approaches taken, and test method evolution for the August 2019 customer tests.

5.1 Effects of Fluid Adhering to the Sorbent Support Rack Study

The sorbent support rack, instrumental in the submersion and handling of sorbents, gains weight once coated in test fluid as shown in Figure 5. Of primary concern was the amount and

weight of oil that adhered to the support rack when raised, and the impact this would have on mass balancing and oil adsorption calculations. Although the added oil weight was small relative to the support rack, it was discovered to be significant relative to weight measurements for lightweight sorbents such as single-ply polypropylene sorbents.

In response to this, the test protocol includes a compensation for oil weight on the rack. Observing that oil retained on the support rack was consistent and stabilized to a point of no dripping within a short period of time, the approach for obtaining the pre-test net weight of the rack and sorbent sample utilized the following steps:

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

This procedure provides repeatable results and allows for continued minimization of external disruption or interference during the testing process.

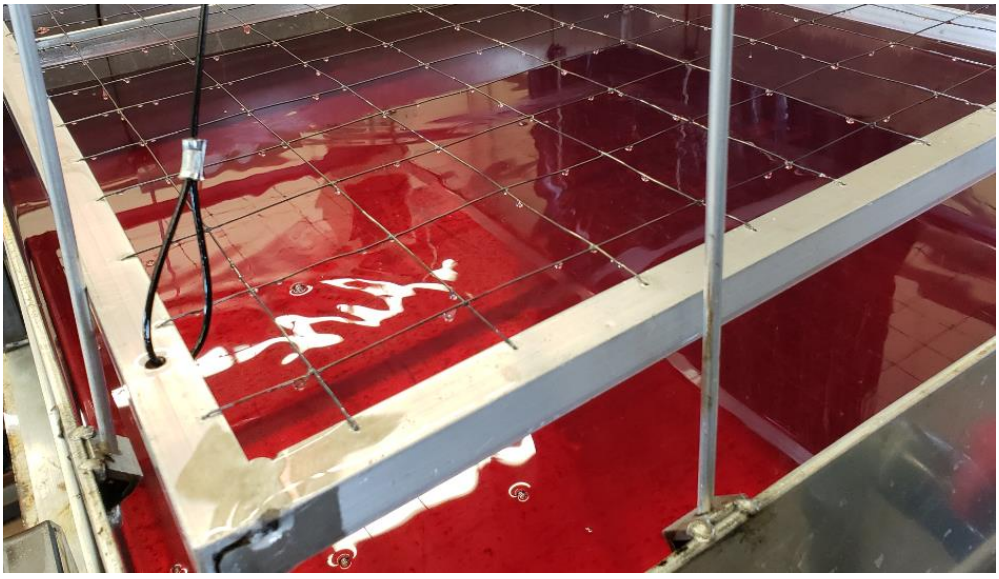

Figure 5 View of oil droplets adhering to the sorbent support rack

5.2 Sorbent Maximum Oil Capacity Test Method in Oil Only Environment

The purpose of this test method is to measure the maximum capacity of oil that a sorbent can adsorb in an oil-only environment. During the development of the test protocol it became apparent that the rate at which sorbents reached their maximum capacity varied significantly. Light weight sorbents adsorbing light oil could reach maximum capacity in less than 1 minute; heavy duty sorbents adsorbing viscous oil took multiple hours to reach capacity. Based on testing with a wide variety of sorbents, a time frame of 15 minutes was chosen for a sorbent's first exposure to oil, after which the sorbent support rack with sorbent was raised, allowed to drip while weight measurements were collected, and then redeployed onto the slick. The sorbent was then allowed to adsorb for 45 minutes after which it was raised, and the same process followed for dripping and collecting weight measurements. For the third increment until the end of the test, the time for oil exposure was one hour. This process was repeated until the sorbent did not

adsorb additional oil within the increment. This required that the sorbent be exposed to the oil for a minimum of two increments and a minimum of one hour. Test steps were as follows:

1. Perform load cell calibration
2. Measure sorbent dimensions
3. Obtain and record pre-test net weight of the rack and sorbent sample (initial weight)
4. Place sorbent sample onto the support rack and lower rack, allowing sorbent to float freely on oil surface
5. Start timer and video documentation
6. After 15 minutes begin load cell data collection and raise support rack with sorbent.
7. Monitor change in gross weight and observe for oil dripping to tapering off to point of no dripping. Calculate the weight-of-oil to weight-of-sorbent ratio (oil/sorbent ratio).
8. Lower sorbent back into fluid. After 45 minutes begin load cell data collection and raise support rack with sorbent. Repeat step 7. If the second measurement for oil/sorbent ratio repeats, that serves as confirmation the sorbent has reached its maximum capacity. If the measurement does not repeat, then the process is repeated using one-hour intervals until the oil/sorbent ratio repeats.

Weight measurements were taken continuously once the sorbent was raised from the oil and allowed to drip. Initial measured gross weight of the sorbent and rack was erroneously high due to free oil dripping from the support rack and sorbent and diminished until it reached a point of little change over time. This data can be plotted as a drip curve, either as net oil weight versus time, or as an oil/sorbent ratio versus time as shown in Figure 6. Measurements obtained were later used to determine the actual weight or volume of oil adsorbed. Important to note is how much the oil/sorbent ratio changes over drip time. Reporting of oil/sorbent ratio should include additional information about drip time or should be presented as a drip curve.

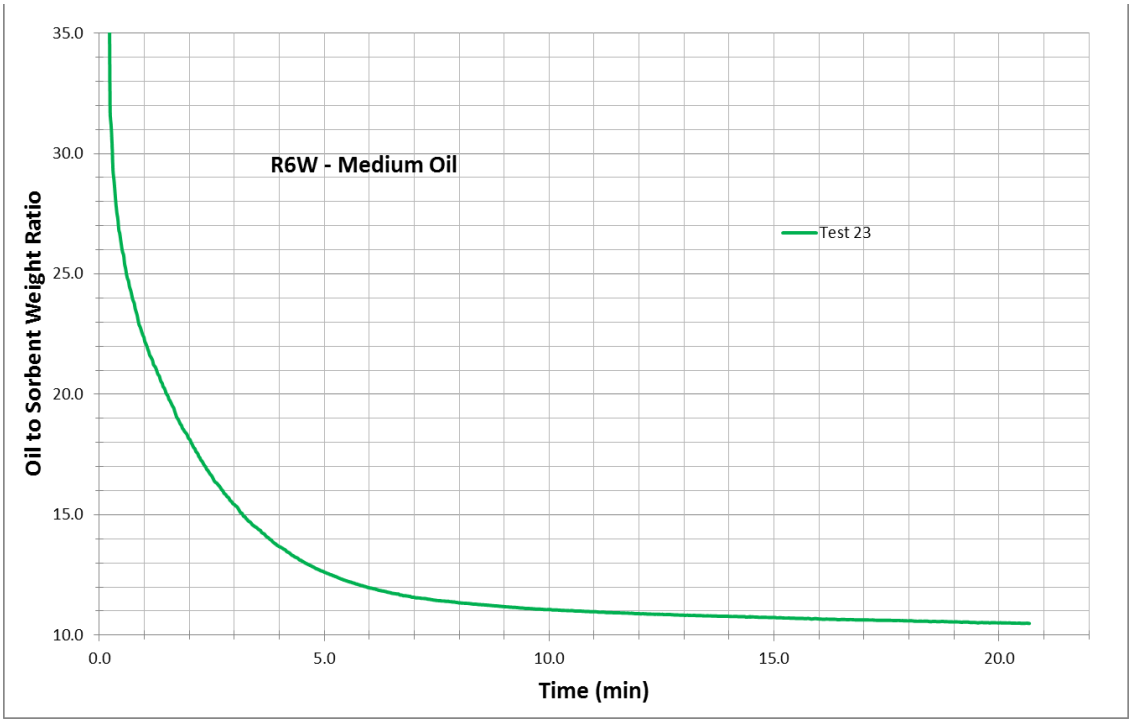

Figure 6 Example drip curve showing oil/sorbent weight ratio versus time

5.3 Sorbent Water Uptake Test in Oil on Water Environment

The intention of this test method was to determine if a sorbent sample would adsorb water when exposed to a relatively thin oil slick on water. The approach was designed to expose test sorbents to a volume of oil less than the predetermined maximum capacity, thereby ensuring contact with both oil and water. This condition allowed for the potential of a sorbent to adsorb water, possibly by way of wicking action or inferior hydrophobic properties. Once values were obtained for the weight of oil and water adsorbed, the amount of each fluid adsorbed could be represented as a ratio, hence Oil to Water Ratio.

The decision was made to use one half of the maximum oil capacity determined by the maximum oil capacity test method described in section 5.2 in order to provide a reasonable oil volume to simultaneously promote wicking and leave available capacity within the sorbent for water uptake. After deploying a half-max volume of oil onto the water surface the sorbent was lowered onto the fluid surface for a prescribed one-hour residence time, removed, and allowed to drip until the point of no dripping while weight measurements were recorded. The fluid remaining in the tray was then decanted of free water and the remaining oil was recovered and measured. Recorded values for the oil volume initially dispensed and the volume recovered allowed the volume remaining in the sorbent to be calculated. Once the volume of oil remaining in the sorbent was calculated, it was converted into a weight measurement, and subtracted from the overall weight gained by the sorbent in order to find the weight of water recovered. Test steps were as follows:

1. Place 2.5 cm of water into the tray
2. Place the appropriate volume of oil on the water surface (half of the sorbent's maximum capacity)
3. Obtain and record pre-test net weight of the rack and sorbent sample (initial weight)
4. Place the sorbent on the sorbent support rack and lower it into the fluid for one hour
5. Raise the sorbent support rack and allow it to drain to the point of no drip while recording weight
6. 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)
7. Decant free water from the fluid tray
8. Collect the oil and remaining water into graduated cylinders and allow it to separate
9. Calculate weight of oil recovered from tray by converting volume to weight using specific gravity
10. Subtract recovered oil weight from initial weight of oil dispensed to obtain weight of oil removed by sorbent
11. 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

This technique was verified and found to be an accurate approach based on conducting control tests. However, through testing it became apparent that applying a sorbent to a quiescent surface of oil and water did not promote water uptake. To further enhance the method and provide conditions more closely mimicking field use, comparative tests were performed with the addition of surface energy. The test parameters and method were replicated in each case, with the only change being the introduction of energy. In each case, the amount of water retained in the

sorbents increased significantly for the tests including energy. The test protocol thus currently includes the use of energy for this test method.

5.4 Evolution of Sorbent Water Uptake Test in Oil on Water Environment – August 2019 Tests

The test method described in section 5.3 was evolved for the August 2019 customer tests. Significant changes included the amount of oil initially added to the test tray, sorbent exposure times, and drip times. For these tests, the volume of oil used was determined based on ASTM F726-17 testing rather than sorbent maximum capacity tests. F726-17 testing produced maximum oil capacity which was scaled relative to the sorbent sample size. The relative amount of oil was then added on water in the fluid tray to create a surface slick. The sorbent was applied to the water surface, allowed to adsorb incrementally for a total time of 60 minutes, and weighed after a 30 second drip time. This test method utilized the following steps:

1. Perform load cell calibration
2. Measure sorbent dimensions
3. Obtain and record pre-test net weight of the rack and sorbent sample
4. Place sorbent sample onto rack and lower until sample floats freely on the fluid surface
5. Begin timer and video or time-lapse documentation
6. After 15 minutes begin load cell data collection and withdraw support rack and sorbent for 30 seconds before re-immersing sample in test fluid
7. After 30 minutes begin load cell data collection and withdraw support rack and sorbent for 30 seconds before re-immersing sample in test fluid
8. After 60 minutes begin load cell data collection and withdraw support rack and sorbent for 30 seconds concluding submersion
9. 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)
10. Decant free water from the fluid tray
11. Collect the oil and remaining water into graduated cylinders and allow it to separate
12. Calculate weight of oil recovered from tray by converting volume to weight using specific gravity
13. Subtract recovered oil weight from initial weight of oil dispensed to obtain weight of oil removed by sorbent
14. 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

Advantages of this test method included the ability to test without first conducting field scale maximum adsorption tests described in section 5.2. Disadvantages include the necessity to conduct ASTM F726-17 tests to determine the oil volume to use. This method provides useful data to consider as the test protocol is further evolved in the future.

5.5 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. A separate test method was not developed 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.

5.6 Field Retrieval

The ability of a fully saturated sorbent to be retrieved intact was determined to be an important functional characteristic for successful field use, and a method for assessing this was developed. 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. The hook was suspended on the load cell. The method was explored using the following steps:

1. A sorbent that had undergone maximum oil adsorption was placed on the fluid tray and lowered into test oil.
2. The sorbent was allowed five minutes to re-saturate.
3. The sorbent was attached to the pitchfork prong by puncturing it 15.2 cm (6 in) inboard shortest edge.
4. With load cell operating, the sorbent was raised from the oil bath.
5. The sorbent passed if it remained stable on the prong and failed if it tore from the prong.
6. Sorbents that passed were further subjected to a downward force until failure was reached and the force was recorded. Figure 7 shows a single-ply polypropylene sorbent undergoing the field retrieval test.

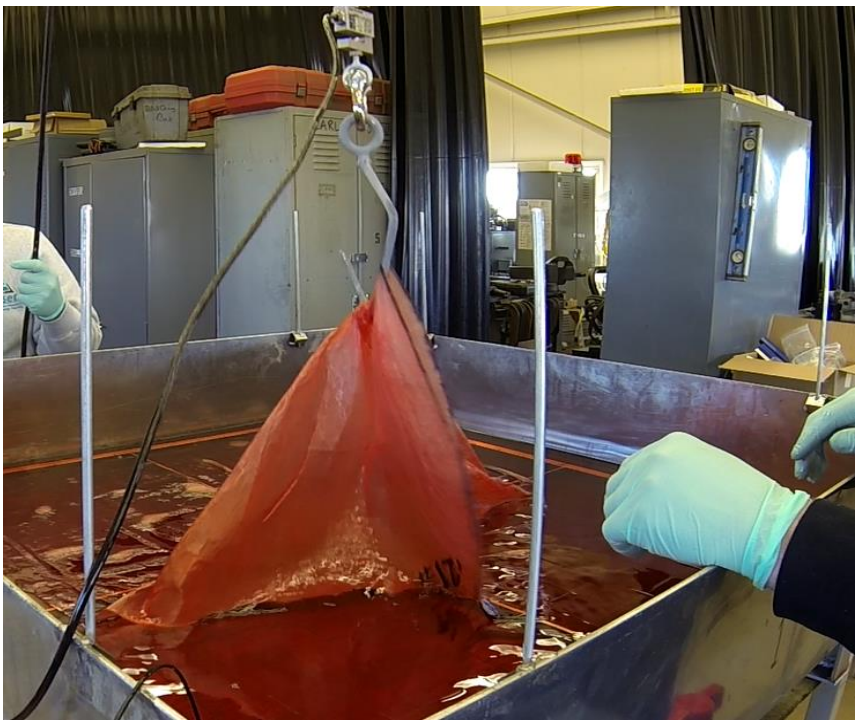

Figure 7 Single-ply sorbent undergoing a field retrieval test

5.7 Manual Oil Recovery

The field scale test protocol did not include a method for determining the amount of oil that could be recovered from a saturated sorbent. However, the customer involved in the August

2019 tests was interested in this data and a method for quantifying this was investigated. A manual roller system was employed to evenly apply pressure to saturated sorbent samples and to recover a portion of the oil adsorbed during the saturation test. Knowing the oil that was adsorbed as determined through mass balance calculations, the percentage of oil recovered from the sorbent and the oil remaining in the sorbent were calculated. This data could be useful in assessing sorbents for their reusability in response operations. Figure 8 shows a view of the manual roller system employed for this test. This test method will likely be further developed as the field scale test protocol continues to evolve.

Figure 8 Manual roller system employed for the manual oil recovery tests

6.0 Results and Discussion

The objectives of this study were to evolve the previously developed test protocol to meet a customer's needs and provide test methods that are reasonably simple to perform and fairly evaluates each sample in a time efficient manner. All pertinent testing parameters, raw data and notes were recorded and provided to the customer. Documented information included pertinent details defining the test method, amount of oil adsorbed in terms of weight, volume, and the adsorption ratio (g oil recovered/ g sorbent), etc. The following provides a discussion and overall

assessment of the test protocol status, observations, and potential improvements. Tables provided below include selected results to illustrate each test method within the test protocol.

6.1 Sorbent Maximum Oil Capacity Tests per ASTM F716-17

The ASTM F726-17 standard test method was used to assess maximum oil capacity of the sorbents. Oil capacity data was obtained under the "Oil Adsorption Short Test" and the "Oil Adsorption Long Test" for Type 1 sorbents. Tables 2 and 3 provide averaged results for each sorbent tested with diesel. Note that the ASTM test method requires three repeats with each result deviating no more than 15% from the mean of the three results. Observations on sorbent buoyancy were also recorded.

Table 2 ASTM F726-17 15-minute Oil Capacity Testing with Diesel

SORBENT SAMPLE	OIL TYPE	TIME IN OIL	AVERAGE ADSORBENCY, OIL(g) /SORBENT (g)	FLOATING OR SUNK
Manufacturer Sample A	DIESEL	15 min	32.2	FLOATING
Manufacturer Sample B	DIESEL	15 min	27.9	FLOATING
Manufacturer Sample C	DIESEL	15 min	24.0	FLOATING
Manufacturer Sample D	DIESEL	15 min	22.1	FLOATING
Manufacturer Sample E	DIESEL	15 min	21.0	FLOATING

Table 3 ASTM F726-17 24-hour Oil Capacity Testing with Diesel

SORBENT SAMPLE	OIL TYPE	TIME IN OIL	AVERAGE ADSORBENCY, OIL(g) /SORBENT (g)	FLOATING OR SUNK
Manufacturer Sample A	DIESEL	24 hr	35.0	FLOATING
Manufacturer Sample B	DIESEL	24 hr	30.6	FLOATING
Manufacturer Sample C	DIESEL	24 hr	27.5	FLOATING
Manufacturer Sample D	DIESEL	24 hr	28.3	FLOATING
Manufacturer Sample E	DIESEL	24 hr	22.5	FLOATING

Additional observations worthy of note and recommendations for possible improvements are provided below:

- ASTM F726-17 tests were used to obtain maximum adsorption of the sorbents for the August 2019 customer tests.
- During the test protocol development, data obtained using the ASTM F726-17 was compared to data obtained using the maximum capacity field scale test method data.
- Test results did not provide a consistent or clear trend. Further comparative maximum capacity tests should be conducted to determine the recommended approach for assessing a sorbent's maximum oil capacity.

6.2 Sorbent Water Uptake Tests in Oil on Water Environment (method 5.4)

Sorbent samples A through E were tested as described in section 5.4. The amount of oil that each sample was exposed to was determined by scaling the ASTM test results. The average weight of oil adsorbed and the average sample weight for the ASTM tests were calculated and scaled up by the same relative amount. For example, if an average sample weight was 10 grams and the sample adsorbed an average of 25 grams of oil during ASTM F726-17 testing, then a larger sample weighing 40 grams would be exposed to 100 grams of oil.

Results for these tests are illustrated in Tables 4 and 5. Subjecting the sorbent samples to an amount of oil equivalent to the maximum capacity volume allowed for an understanding of a sorbent's ability to perform in sub-optimal conditions. Although the sorbents were exposed to adequate oil, the availability of water and an incomplete exposure to oil expectedly hindered performance. The "% Oil in Sorbent" column describes what percentage of adsorbed fluid was oil (versus water), and the "Adsorbency Oil(g)/Sorbent(g)" column gives the ratio of oil weight to sorbent weight at the end of the test. Note that for Sample D in Table 4, an incongruity was observed between the amount of fluid adsorbed and the ratio of oil to water adsorbed. This was due to measurement error and should be explored further as the test protocol development continues.

Table 4 Static Maximum Oil Capacity Testing with Diesel

TEST TYPE	SORBENT SAMPLE	SORBENT DIMENSIONS (cm)	SORBENT WEIGHT (g)	% OIL IN SORBENT	ADSORBENCY, OIL (g) / SORBENT (g)
STATIC	Manufacturer Sample A	91 x 46	199.6	99.5%	30.3
STATIC	Manufacturer Sample B	91 x 49	122.5	85.3%	23.8
STATIC	Manufacturer Sample C	91 x 49	122.5	98.1%	20.3
STATIC	Manufacturer Sample D	59 x 59	136	100.9%	13.1
STATIC	Manufacturer Sample E	91 x 48	118	94.7%	19.0

The further introduction of surface energy to subsequent tests revealed performance characteristics as either hindered or enhanced by wave action. While the introduction of motion allowed all tested sorbents to access and adsorb more of the available oil, there are notable differences in the individual hydrophobic responses. The Manufacturer Sample B sorbent was found to adsorb significantly more oil, while dramatically increasing its hydrophobic response.

The Manufacturer Sample C and Manufacturer Sample E sorbents were observed to perform more consistently with and without an applied surface energy.

Table 5 Dynamic maximum oil capacity testing with Diesel

TEST TYPE	SORBENT SAMPLE	SORBENT DIMENSIONS (cm)	SORBENT WEIGHT (g)	% OIL IN SORBENT	ADSORBENCY, OIL (g) / SORBENT (g)
MIX ENERGY	Manufacturer Sample A	91 x 46	186.0	94.8%	31.3
MIX ENERGY	Manufacturer Sample B	91 x 49	117.9	96.7%	28.1
MIX ENERGY	Manufacturer Sample C	91 x 49	117.9	98.1%	22.5
MIX ENERGY	Manufacturer Sample D	60 x 59	99.8	93.4%	24.8
MIX ENERGY	Manufacturer Sample E	91 x 48	117.9	98.3%	21.2

The ratio of oil weight adsorbed relative to the sorbent sample weight was calculated as the Oil to Sorbent Weight Ratio after a drip time of 30 seconds, which may overestimate a sorbent's adsorption capacity. However, when considering this information for field use application, allowing the sorbent to drip for a protracted duration may not be realistic. The defined interval process for exposing the sorbent to oil was found to effectively optimize the overall testing time and simultaneously provide confirmation of field scale maximum capacity realization within a reasonable window of time. However, this defined interval may also underrepresent some sorbents that require a longer oil exposure to maximize their adsorption capacity.

Additional observations worthy of note and recommendations for possible improvements are provided below:

- Due to the volume of testing required, triplicate testing was not performed. A select set of tests should be repeated for verification of repeatability.
- During some tests, fluid was found to pool on top of the sorbent. To ensure sample weights were accurately measured, the rack was briefly tilted allowing free fluid to pour off. This was found to be necessary for lower viscosity oils. During the test protocol development an alternate method of flipping the sorbent over immediately after it was lifted out of the fluid was employed. Additional assessment to provide a repeatable method for handling this issue should be completed.
- Varying temperatures were observed in a warehouse testing environment with a range of 21-28°C. Comparative tests of oils at viscosities corresponding to the minimum and maximum should be further investigated to better represent a range of performance.
- Drip times and overall test times should be further investigated to ensure that the final test protocol provides a fair representation of sorbent performance that could be expected during field use while minimizing overall test time.

6.3 Sorbent Water Uptake Tests in Oil on Water Environment (half oil volume)

Tests were performed in which sorbents were placed on a quiescent water bath with an oil presence of one-half the maximum capacity for the sample. Results are shown in Table 6. In

comparing data from Tables 4 and 6 it is clear, although not unexpected, that the amount of oil dispensed on the water surface has a great effect on results when testing in a static environment. All sorbent samples ended up with a greater percentage of water adsorbed and a lower weight of oil adsorbed when exposed to a thinner oil layer.

Half-max oil capacity testing was also conducted with the application of surface energy. Results are presented in Table 7. For this set of tests, data for sample B was not collected. All sorbent samples for this test condition when compared to Table 5 data also adsorbed a lower overall weight of oil and a greater percentage of water. For a test method involving testing of a sorbent deployed in oil-on-water, it is clear that the amount of oil the sorbent is exposed to should be consistent in order to provide results that can be compared to one another.

Table 6 Static Half-max Oil Capacity Testing with Diesel

TEST TYPE	SORBENT SAMPLE	SORBENT DIMENSIONS (cm)	SORBENT WEIGHT (g)	% OIL IN SORBENT	ADSORBENCY, OIL (g) / SORBENT (g)
STATIC	Manufacturer Sample A	91 x 46	181.4	95.3%	15.4
STATIC	Manufacturer Sample B	91 x 49	117.9	82.8%	14.6
STATIC	Manufacturer Sample C	91 x 49	117.9	90.6%	11.7
STATIC	Manufacturer Sample D	58 x 58	104.3	93.4%	12.1
STATIC	Manufacturer Sample E	91 x 48	122.5	89.0%	10.6

Table 7 Dynamic Half-max Oil Capacity Testing with Diesel

TEST TYPE	SORBENT SAMPLE	SORBENT DIMENSIONS (cm)	SORBENT WEIGHT (g)	% OIL IN SORBENT	ADSORBENCY, OIL (g) / SORBENT (g)
MIX ENERGY	Manufacturer Sample A	91 x 46	181.4	93.5%	15.6
MIX ENERGY	Manufacturer Sample B	Test not conducted			
MIX ENERGY	Manufacturer Sample C	91 x 49	117.9	86.2%	12.0
MIX ENERGY	Manufacturer Sample D	59 x 59	108.9	84.0%	10.6
MIX ENERGY	Manufacturer Sample E	91 x 48	127.0	88.5%	11.2

During the series of water uptake tests, duration and surface energy setting were held as constants throughout the investigation. The effects of varying either duration or energy are unknown at this time. Although the current method demonstrates a quantifiable way for measuring relative water adsorption, additional refinement is possible upon further investigation of the given parameters. Furthermore, a study linking sorbent performance at a macro scale with generated waves with results obtained using the test apparatus could further confirm validity. Tests performed on the energy table inherently enable a more complete exposure of the sorbent to available oil, while maintaining a low enough rate so as not to incur fluid mixing. Additional

observations worthy of note and recommendations for possible improvements are provided below:

- Investigate the effects of varying duration or surface energy
- Refine method for quantifying water adsorption
- Conduct comparative testing with macro scale (tank) tests to validate the test apparatus
- Buoyancy

Currently buoyancy is considered a result determined by observation in this group of methods. Although samples tested on the custom apparatus did not sink, it should remain a consideration as testing did not simulate particulate and debris that might accumulate and add weight to the surface oil and sorbent. As this method stands now, a noted observation is warranted at the conclusion of the maximum oil capacity and water uptake tests.

6.4 Test Oils and Oil Viscosity Range

Test fluids were targeted that were readily available, safe to use in typical test environments, aligned to oil properties and viscosity ranges defined in ASTM F726-17, and had relatively stable properties to allow for test repeatability. However, in the August 2019 tests the customer required testing to be conducted with diesel which does evaporate over time. The test protocol could consider including control testing to account for a fluid's change in properties over time, especially for such test fluids.

One question that needs to be further explored is how test results using fluids such as mineral oils would compare to testing with crude oils of similar viscosities. This is an area of future investigation.

Of continued consideration is the effectiveness of a sorbent over a range of oil viscosity. Individual materials may be found to perform better in different situations for recovery of different fluids. Classifying available sorbents accordingly would be beneficial for improving emergency response times and effectiveness. Time constraints and difficulty working with higher viscosity oil prevented the investigation of this characteristic during this study.

6.5 Manual Oil Recovery

This test was performed following the prescribed saturation of a sorbent in order to determine the amount of fluid that can be successfully recovered from a spent sorbent. These numbers may hint at the further ability of a sorbent to adsorb additional oil. The limiting size of the manual roller necessitated the reduction of a spent sorbent to a smaller strip for wringing. The destructive nature of this wringer-based process prevented further testing at this time. If deemed to be beneficial, an alternate wringer-based system could be designed for this test.

7.0 Conclusions

Development of this test protocol was initiated in response to criticisms of ASTM F726-17 and to evaluate sorbents in manner that was more representative of field performance. Information provided by ASTM F726-17 testing is valuable both as a standalone assessment and as a baseline to begin the testing outlined here. In order to more accurately assess and predict the behavior of a sorbent under varying field conditions, additional information is required. Expanding upon the results obtained, this new method may validate/invalidate that same data, while exploring and revealing more advanced behaviors that might influence performance under diverse environmental conditions.

In particular, this test protocol demonstrates the hydrophobic capabilities of a sorbent when deployed directly over a body of water. The ability of a sorbent to adsorb oil while exposed to both water and oil may become compromised which will be uncovered in this test protocol. The further introduction of mixing energy changes the way that both oil and water interact with a sorbent.

Buoyancy is generally considered a necessary feature in a sorbent. In addition to the adsorption performance of a sorbent, the ability to float can greatly impact the retrieval process. While the buoyant nature of a saturated sorbent was observed, the potential effects of sedimentary and other environmental influences were not taken into account.

Throughout the course of this study, unexpected and unique behaviors in sorbents were identified that would otherwise not be demonstrated by the ASTM tests. While it was anticipated that the introduction of energy to the water's surface might impede a sample's hydrophobic properties, the opposite effect was also witnessed. Additionally, while temperature and viscosity were taken into account with each test, performance within an expected field range could not be further investigated. While the test methodology is notably more complex, the resultant data and observations are equally more informative.

The methods employed by this study are intended to become constituent components of a BSEE-Ohmsett developed protocol for assessing sorbent performance characteristics considered essential for successful use in the field. Upon finalization, consideration should be given to how values and results are reported. From the perspective of a manufacturer, values such as maximum capacity, water uptake, and adsorption rate will likely serve for marketing purposes. Conversely, overall effectiveness as ascertained by the performance characteristics studied will likely be of more value to a user. Furthermore, performance qualities characterized by way of a range may be more indicative of expected results than a solitary maximum value.

8.0 Acknowledgements

The overall intent of this protocol is to allow suppliers and users to refer to a standardized set of performance characteristics that correlates the capability expected and needed in field use with a proven level of performance rated by these defined methods. Development of the methods outlined was initially sponsored by the U.S. Department of the Interior's Bureau of Safety and Environmental Enforcement (BSEE). Subsequent testing phases were funded by AquaFlex Holdings, LLC. Testing result data is provided courtesy of AquaFlex Holdings, LLC.

The authors would like to gratefully acknowledge the staff of Applied Research Associates, Inc., who operate Ohmsett under contract to BSEE, and David DeVitis, the former Test Director at Ohmsett during the initial development of the Field Scale Sorbent Protocol.

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