

FINAL REPORT
on
**RESEARCH ON POWDERED ACTIVATED CARBON TO
REMOVE DISSOLVED OIL SPILL DISPERSANTS FROM
OHMSETT BASIN WATER**

for:

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SUMMARY

This project researched techniques to remove dissolved dispersant from Ohmsett water after the tank has been used for a series of chemical dispersant effectiveness experiments. The primary objective of the proposed study was to develop and test a simple, inexpensive system for expediently removing dissolved dispersant from Ohmsett tank water. The second objective of the study was to select and refine an analytical technique for determining the concentration of dissolved dispersant in the tank salt water.

Powdered activated carbon (PAC) was determined to be the best answer for the removal of dissolved dispersant from the tank water after chemical dispersant tests at Ohmsett. A series of lab- and bench-scale tests were undertaken to quantify the expected performance of PAC and design a full-scale removal system. These tests concluded that:

- The adsorptive capacity of PAC for Corexit 9500A dispersant (a commonly-stockpiled dispersant in the U.S.) is about 1 g/g;
- A dose of 50 ppm PAC could adsorb 20 ppm of dispersant, and 20 ppm of PAC could adsorb 10 ppm of dispersant, even in the presence of up to 10 ppm of dispersed oil in the water;
- The required contact time for the adsorption to proceed essentially to completion was 15 minutes.

The selected analytical technique was to measure the interfacial tension of the water against a highly refined mineral oil (USP or Technical grade) with a DuNouy ring tensiometer following procedures laid out in ASTM D971. The use of USP-grade mineral oil resulted in a much-expanded interfacial tension range compared to other crude or refined oils. Due to the very “clean” interface provided by the high-grade mineral oil this approach gave good discrimination at the sub-10 ppm dispersant concentration range with reliable measurements down to 2 ppm. For accuracy at low dispersant concentrations, it was important to pre-treat the water used to develop the calibration standards with the grade and type of activated carbon that would eventually be used in cleaning the tank water. It was also important to ensure that an adequate supply of one manufacturer’s mineral oil was available to cover all the analyses required. The calibration standards also need to equilibrate for several hours before measurement. It became clear, in order to efficiently prepare calibration curves and analyse samples accurately on an operational basis, that a more sophisticated, less operator-dependent DuNuoy ring apparatus was required for the lab at Ohmsett.

A prototype treatment system was built and tested following a series of cold-water dispersant experiments at Ohmsett in March 2003. The system consisted of three modified ISO shipping containers placed end to end in the tank, spanning its width. The containers held well-mixed slurry of activated carbon through which the tank water flowed from South to North under the influence of the filtration pumps.

The top and both sides of the three containers were cut away. The sides were replaced with an expanded metal mesh that supported a very fine filter cloth clamped around the edges of the opening. PVC air piping to bubble the slurry and maintain the PAC in suspension was laid along the bottom of the container. An additional air pipe was placed

directly at the bottom of the downstream, or exit, side to provide agitation to that filter cloth to ensure it did not become clogged by a cake of PAC. A square was cut in the bottom of the container and covered with mesh and cloth to permit the container to drain completely when removed from the tank. The intervening spaces between the ends of the containers were sealed with rubber wipers. Each container initially had 200 pounds of PAC added to it, and then the air bubbler system was activated. The head created by the filter pump moving water from the North end of the tank to the South end forced water through the filter cloth on the upstream (South) side of the containers and out the downstream (North) side at an average velocity of 0.5 ft/min. In the 15 minutes that the water spent inside the 8-foot wide containers it was thoroughly mixed with the PAC slurry. A second 200 pounds of PAC was added to each container on the second day of operation, for a total of 1200 lbs. The system was operated unattended for 72 hours.

Grab samples of the tank water taken before and after installation of the prototype system showed that it reduced the concentration of Corexit 9527 dispersant in the water (about 20 gallons had been sprayed during the testing program) from a range of 5.5 to 9 ppm before to below the detection limit (1 ppm) after. Combining the three to five days required to operate the leaf filter to reduce dispersed oil concentrations to below 10 ppm (SL Ross 2000b) and a two- to three-day period of treatment with PAC, it should be possible to return the tank to a quality that meets the standard for equipment testing in one week following a dispersant test series.

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1. Introduction

This report describes the activities conducted to research techniques to remove dissolved dispersant from Ohmsett water after the tank has been used for chemical dispersant effectiveness experiments.

1.1 Background

Two studies were recently completed of the feasibility of conducting dispersant effectiveness testing at Ohmsett (SL Ross 2000a and 2000b). It was concluded that realistic dispersant effectiveness testing at Ohmsett is viable. More specifically it was found that:

- the Ohmsett facility produces results during dispersant testing that are consistent with observations and measurements made at field trials;
- a suitable protocol can be devised that would enable a systematic investigation of test variables such as oil viscosity, dispersant treatment rate and sea conditions;
- the use of a 50/50 mix of diatomaceous earth and cellulose in the leaf filter allows for effective removal of dispersed oil from the tank water in a period of approximately one week;
- activated carbon will remove dissolved dispersant from the tank water, but it was not possible to determine how quickly, or how efficiently, it would work; and,
- the analytical technique to detect dissolved dispersant, measuring the surface tension of the water in the tank, did not prove to be effective and will need to be replaced.

A traditional activated carbon (AC) system was used during the feasibility studies but did not provide an effective dispersant removal solution for several reasons.

1. The system was large, expensive and time-consuming to set up and operate.
2. Even with the large capacity of the system (20,000 lb of activated carbon in two large vessels), it was only possible to treat the tank water at flow rates of 400 gpm, considerably less than the 1800 gpm flow rate normally used for suspended solids filtration, and too low to allow the tank contents (2.6 million gallons) to be processed in a reasonable period of time. This low flow rate was the default specified by the supplier of the system, and was not based on lab-scale or pilot-scale studies of the removal of dispersant from Ohmsett water. Doing such tests may allow the design of an AC system with a much higher flow rate.
3. It was not possible to process the tank water through the AC system until most of the dispersed oil had been filtered out in order to avoid fouling the activated carbon with oil. This meant waiting for the better part of a week (until the leaf

filter had reduced the dispersed oil concentrations) before beginning the activated carbon treatment.

Overall, this meant that the Ohmsett tank water could not be returned to a quality acceptable for testing for more than two to three weeks after the completion of a series of dispersant tests. This was obviously unacceptable to MMS given the busy testing schedule at Ohmsett.

For example, as a result of the positive conclusions from the feasibility study, one series of dispersant experiments for a commercial client was conducted in the fall of 2000. Much larger volumes of oil and dispersant than had been envisioned in the feasibility studies were used during these tests. An activated carbon system was not utilized to process the tank water after these tests. Subsequently, it was apparent that considerable concentrations of dispersant remained in the water after the tests. This caused problems for an emulsion formation research program scheduled after the dispersant experiments. This experience led to the decision to restrict dispersant testing at Ohmsett to the end of the season, after which the water would be treated to remove dispersed oil down to acceptable levels and the tank drained for winter maintenance. This means that dispersant testing could only be conducted in the tank when the water is relatively cool (about 40 to 45°F in November and December). In addition, there was concern about discharging the tank water if it contained dispersant components.

Recently, interest has been expressed by other commercial clients in testing their dispersant at Ohmsett in warm water. This would mean that the dispersant test program would have to be conducted in the summer months (when the tank water temperature is on the order of 75°F) and would require that the water be treated to remove dispersed oil and dissolved dispersant in a reasonable time period (i.e., approximately one week).

1.2 Objective and Goals

Objective: The primary objective of the research project was to develop and test a simple, inexpensive system for expediently removing dissolved dispersant from Ohmsett tank water. The second objective of the study was to develop an analytical technique for determining the concentration of dissolved dispersant in the tank salt water.

Goals: More specifically, the goals of the work proposed here were to:

- Conduct a review of potential dissolved dispersant removal processes and design laboratory tests to assess the most likely candidate(s). The dispersant removal process should not significantly extend the time presently required to remove dispersed oil droplets from the water;
- Carry out lab-scale tests on the efficacy of the selected dispersant removal technique;
- Conduct scale model tests at Ohmsett to quantify the design parameters for a full-scale system; and,
- Design and test a full-scale dissolved dispersant removal system.

2. Study Approach

Based on the earlier research on the feasibility of testing dispersants at Ohmsett, it was proposed that activated carbon, in some form, was the answer to removing dissolved dispersant from the tank water. What was required was the quantification of the parameters of the treatment problem through a series of small-scale laboratory tests and pilot-scale testing at Ohmsett; development of a simple, inexpensive, safe, technique for contacting the tank water with activated carbon at a high rate; and, testing of the proposed system.

Another aspect of dissolved dispersant removal was the need for an analytical technique to determine the concentration of dissolved dispersant components in the tank salt water. It is proposed that two types of analysis are required: a simple, day-to-day operational technique, and a more accurate certification analysis technique. Part of this project related to developing and refining the simple, inexpensive method for monitoring dispersant concentrations in the tank water using equipment and skills in place at Ohmsett. Another, concurrent project at SAIC Canada in Ottawa involved the development of a more sophisticated chemical analytical technique to quantify dispersant concentrations for certification of the water's quality for disposal (Cooper et al. 2003). The standard for equipment testing in the tank is that the interfacial tension between the test oil and the tank salt water be at least 18 dynes/cm.

3. Operational Analysis of Dissolved Dispersant

There were several challenges in finding an effective technique for measuring the concentration of dispersant dissolved in Ohmsett water. One of the most difficult was that the water contains dissolved and dispersed oil in addition to the dissolved dispersant. This is the main reason that the initially proposed technique, measuring changes in the surface tension of the water, did not work satisfactorily, despite this being an effective technique for measuring dissolved dispersant in tap water in the laboratory (SL Ross 2000b). The presence of dispersed oil droplets in the tank water samples that coalesced on the surface during the measurement process affected the surface tension measurement. It was proposed that the measurement of oil/water interfacial tension (IFT) be used as the day-to-day analytical technique for monitoring dispersant concentrations in the tank water. This approach also had the advantage that the standard that must be met for mechanical equipment testing in the tank is an oil/water interfacial tension of at least 18 dynes/cm, which can be confirmed directly with this measurement technique.

The first series of tests involved measuring the IFT of virgin 10W30 motor oil (virgin meaning containing no additives) against solutions of Corexit 9500A in Ohmsett water that had salt added to bring its salinity to 35 ppt. Virgin motor oil was initially selected because it does not vary in properties from batch to batch, and does not evaporate. The measurements were made with a CSC DuNouy Ring Tensiometer following procedures laid out in ASTM D971. Figure 1 shows the results. All test data may be found in

Appendix A.

Calibration of Interfacial Tension vs. Concentration of Corexit 9500A

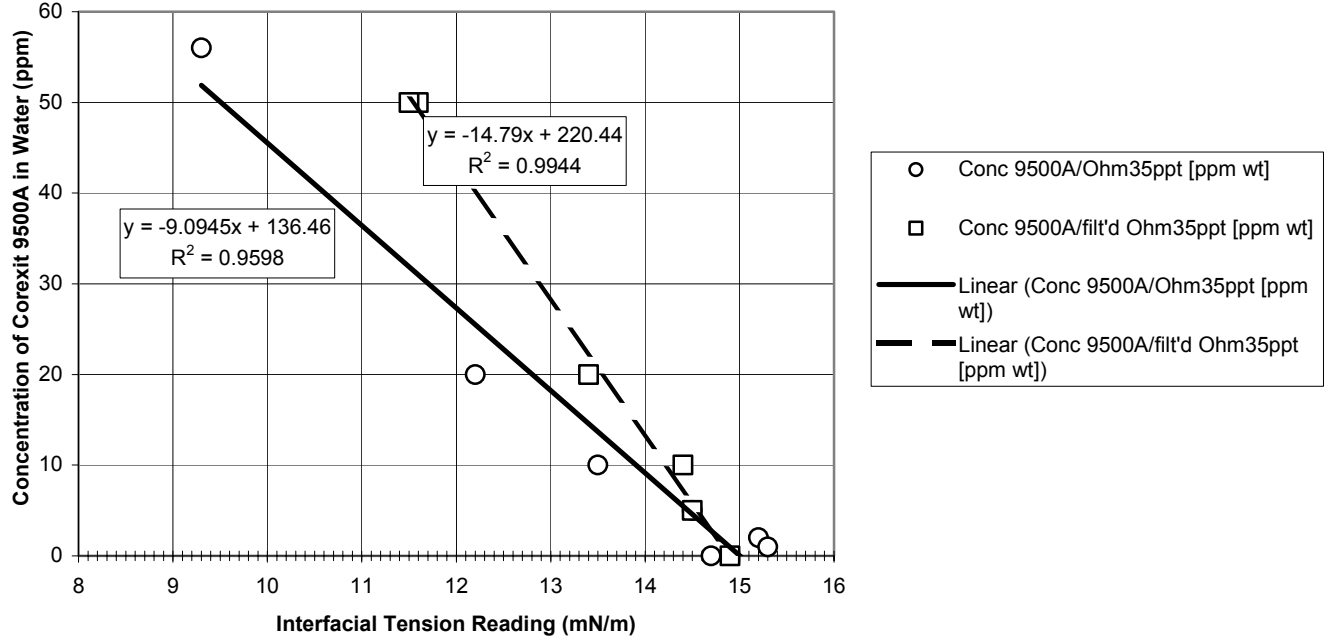


Figure 1: IFT of virgin 10W30 motor oil against solutions of Corexit 9500A in Ohmsett water.

Two curves are given, one for Ohmsett water as received, the other for Ohmsett water that had been dosed with activated charcoal, then filtered. This latter curve was used for the measurements in the lab to determine adsorption isotherms, since treatment of the water with activated carbon changed the interfacial tension, presumably by removing some dissolved materials in the water. The technique did allow reasonable determination of concentrations of Corexit 9500A in the water at concentrations of 10 ppm or higher, sufficient for the carbon adsorption isotherm determination, but could not discriminate lower concentrations in the 1 ppm range required to monitor dispersant removal from the Ohmsett tank itself. This was addressed by substituting a highly refined mineral oil (USP [i.e., pharmaceutical] or Technical grade) as the test oil. Figure 2 shows the results.

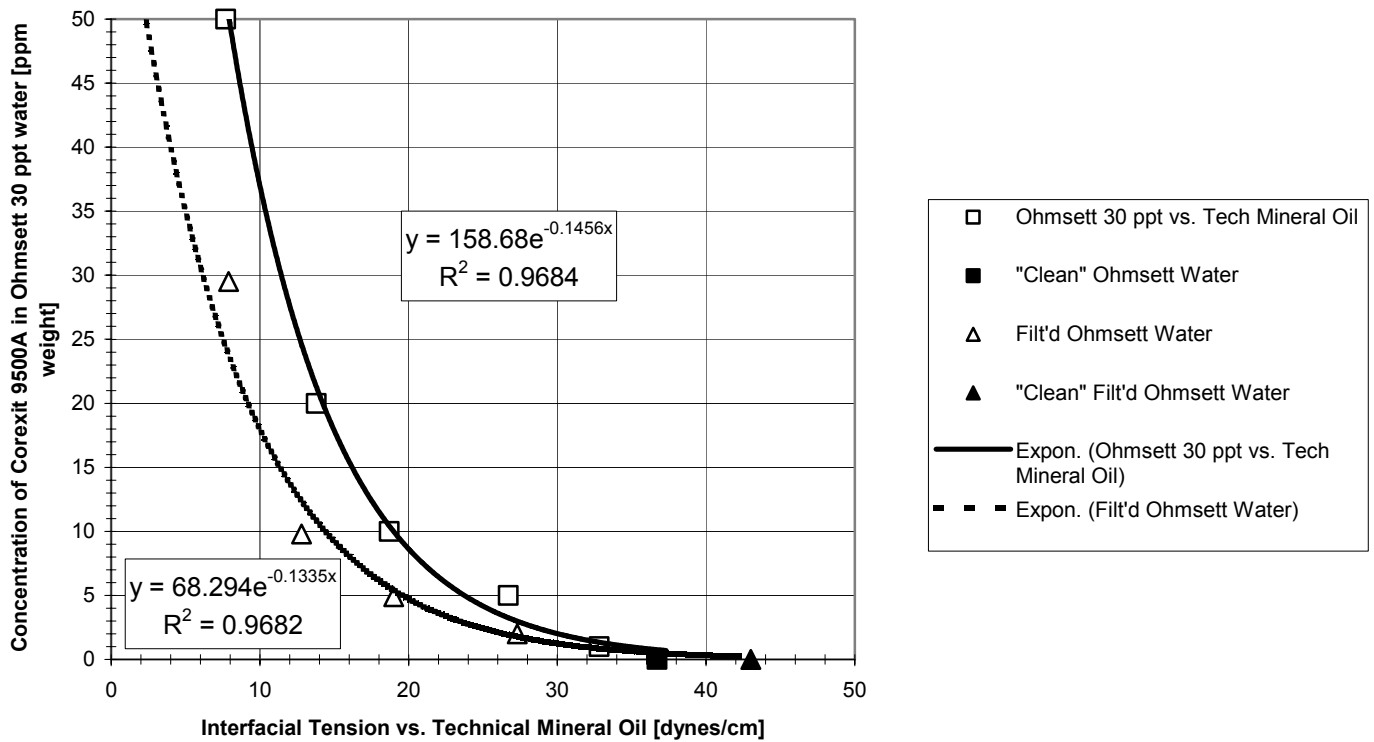


Figure 2: IFT of Technical grade mineral oil against solutions of Corexit 9500A in Ohmsett water.

As with Figure 1 it can be seen that pre-treating the water with activated carbon results in a slightly different calibration curve. The range of measured IFT is greatly extended (from 9 to 15 dynes/cm in Figure 1 to 9 to 50 dynes/cm in Figure 2) due to the much “cleaner” interface provided by the high-grade mineral oil. The results offer good discrimination at the sub-10 ppm range with reliable measurements down to 2 ppm achievable. It was discovered that it was necessary to let the mixture of Corexit 9500A and Ohmsett water sit for several hours before using it to develop a calibration curve: the IFT at high (>30 ppm) concentrations of dispersant would change considerably over the first hour after mixing the solution. This was presumed to be due to the rearrangement and equilibration of surfactants from the highly structured mixed-micelles that exist at high surfactant concentrations in the dispersant to the less structured arrangement that exist at diluted, lower surfactant concentrations in the Ohmsett water.

Figure 3 shows the different calibration curves developed during the prototype testing at Ohmsett. Note that the axes are transposed, compared to Figures 1 and 2 and that the curves are for a different, though closely related, dispersant. The curves were prepared for different types (mesh sizes) of activated carbon used to pre-filter the water (denoted as PAC in the graph legend) and different suppliers’ mineral oil (USP grade). The main differences between the best-fit curves are at the low concentrations of dispersant, where 1 ppm of Corexit 9527 gave IFTs ranging from 22 to 34 dynes/cm. It is clear that, for

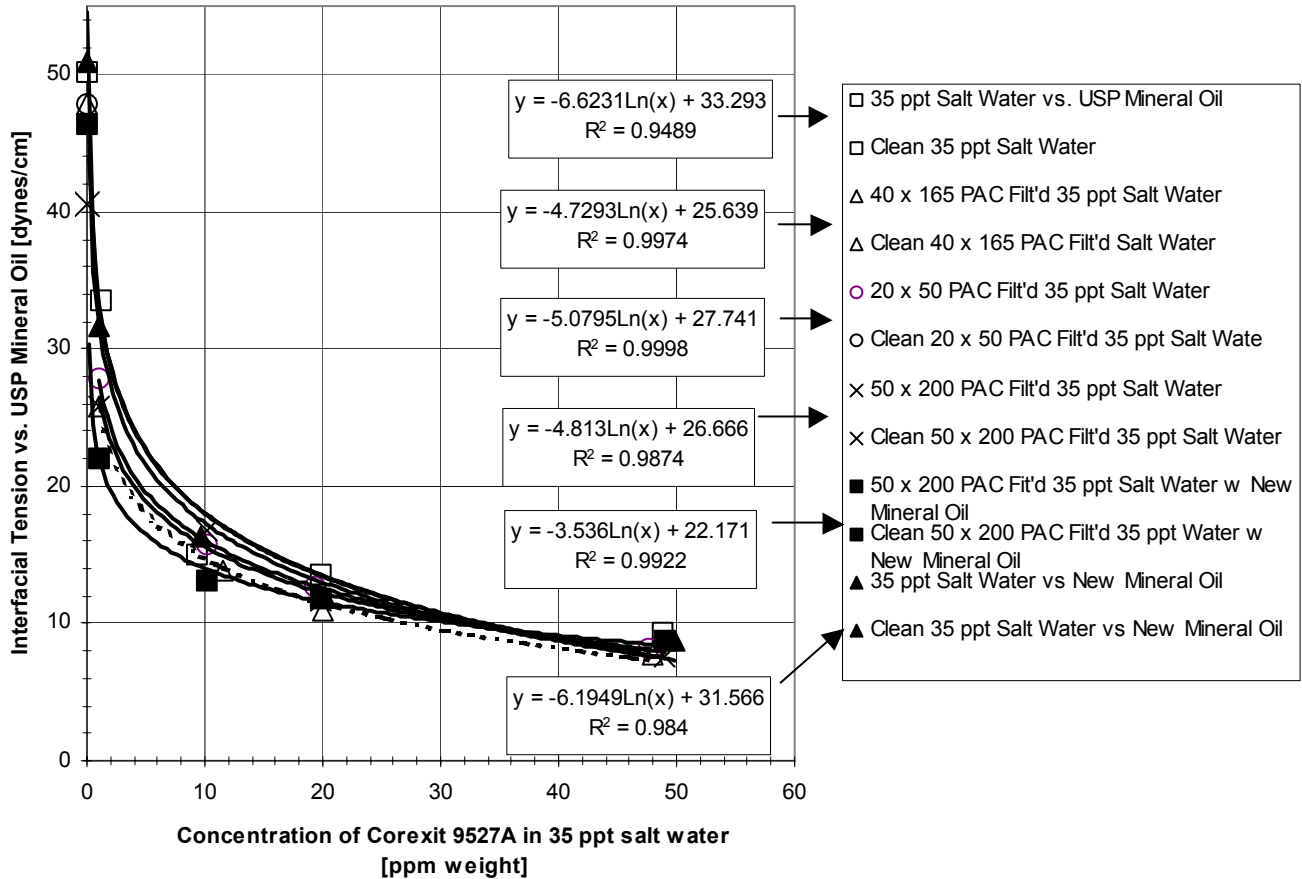


Figure 3: Calibration curves constructed for prototype tests at Ohmsett.

accuracy at low concentrations, it is important to develop the calibration curve with the grade and type of activated carbon added to the tank and to ensure that an adequate supply of one manufacturer's mineral oil is available to cover all the analyses required. It also became clear that, in order to efficiently prepare calibration curves and analyse samples accurately on an operational basis that a more sophisticated, less operator-dependant DuNuoy ring IFT apparatus is required for the lab at Ohmsett.

4. Review of Dissolved Dispersant Removal Processes

In this task, Web searches were used to identify possible techniques for removing dispersant components from Ohmsett water. As a result of this review, small-scale laboratory experiments were designed to ascertain the efficacy of the most promising technique.

The search for potential dispersant removal technologies was not restricted to activated carbon. The Water Quality Association lists several techniques for treating water containing foaming agents (such as soaps, etc. which are surfactants somewhat like dispersants) to meet the U.S. National Secondary Drinking Water Standards; these

include chlorination, activated carbon, ozonation, reverse osmosis and distillation. Obviously some of these (i.e., distillation) do not apply to the present problem, but all possibilities were assessed and screened. A concurrent project by SAIC Canada on the use of membrane technology to remove dissolved dispersant from Ohmsett water reviewed these separation processes (Cooper et al. 2003).

The review concluded for this study that the best technology available to remove dissolved dispersant was the application of Powdered Activated Carbon (PAC) to the tank water to adsorb the dispersant components, followed by filtration to remove the PAC from the water. PAC has been used for Taste and Odour removal from drinking water for many years, and is known to effectively remove many organic chemicals from water, including synthetic organics, pesticides, herbicides, color-compounds. It is relatively inexpensive (ca. \$1 per pound, cheaper than granular activated carbon), requires minimal capital expenditure for mixing and removal equipment, and can be applied only when needed (Najm et al. 1991). Many types and grades are available from a number of manufacturers in the U.S. Another major advantage of using PAC is that simple, standardized laboratory tests are available from ASTM to determine the potential effectiveness of PAC as a dispersant removal technique and to estimate quantities required to treat a specific dispersant. Additional challenges specific to removing dissolved dispersant from Ohmsett tank water included the fact that the water is salty (nominally 35 ppt) and chlorinated (with sodium hypochlorite) and would contain both dissolved and dispersed (i.e., very small droplets) oil.

Based on this review, it was decided to conduct a series of small-scale laboratory tests to ascertain the potential for PAC to remove dissolved dispersant from Ohmsett tank water.

5. Lab-scale Tests of PAC Dispersant Removal

These tests were carried out at the SL Ross lab in Ottawa. Standardized lab techniques (Freundlich isotherms to determine mass transfer characteristics of activated carbon in removing dispersant following ASTM D3860-98 – see Appendix B) were used to assess PAC as a removal process.

Figure 4 shows the results of the lab tests with two types of PAC (chosen because their specific purpose is to adsorb organics from drinking water) to remove 50 ppm of dissolved Corexit 9500A from Ohmsett water. The average particle size of both PAC's was about 45 μm (325 mesh). The water had salt added to it to bring its salinity to approximately 35 ppt, but did not contain any dispersed oil. For this series of tests the IFT analytical technique involved 10W30 motor oil (see Figure 1 in Section 3 above) and was thus only accurate to an estimated $\pm 25\%$. The tests gave very encouraging results, with the ultimate capacity of the PAC being on the order of 1g "equivalent" Corexit 9500A removed per gram of PAC. It was decided to use "equivalent" because it cannot be proven, using IFT, that the carrier solvent in the dispersant is also being removed (although it is very likely that it is). On this basis, a PAC dose of 50 ppm (equivalent to 1000 lbs of PAC in the 2.6 million gallon Ohmsett tank contents) would appear to be

PAC Adsorption Isotherm
(concentration of Corexit 9500 based on IFT readings)

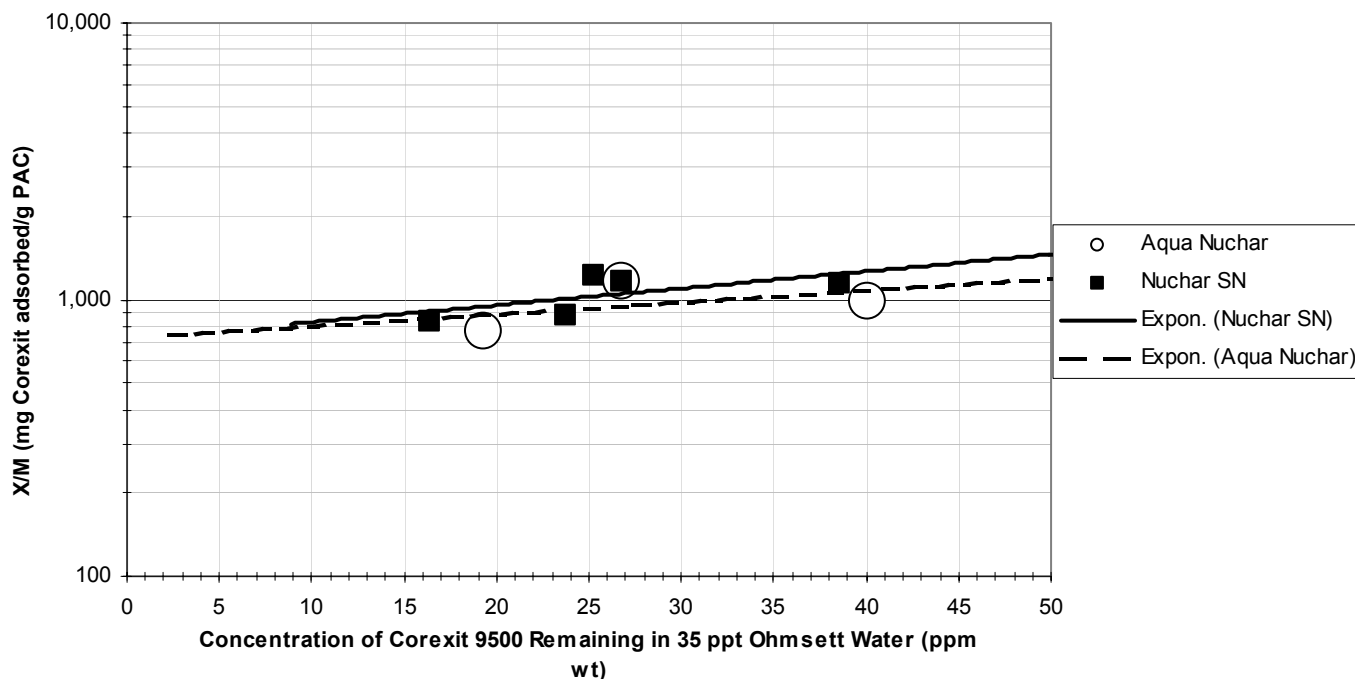


Figure 4: Adsorption isotherm (ASTM 3860) for Corexit 9500A in Ohmsett water on two grades of PAC.

capable of removing up to 50 ppm of dissolved dispersant.

Some of the data points from the tests (see Appendix B) were not used in the construction of the isotherm shown in Figure 4. The ASTM procedure calls for the isotherm to be constructed with data points with between 10% and 85% adsorbate (i.e., dispersant) removal. Although the IFT's for the samples treated with 50 and 100 ppm PAC indicate that the removal is less than 85% (see Appendix A), it was felt that this was an error due to the difficulty in reliably measuring concentrations of less than 10 ppm Corexit 9500A using IFT against the 10W30 motor oil. Since the IFT's measured for the 60 and 100 ppm PAC values were very close to that measured for the blank in the case of the Aqua Nuchar data, it is very likely that more than 85% of the dispersant has been removed from these samples. In the case of the Nuchar SN data the 50 and 100 ppm samples also appear to have had most of the Corexit 9500A removed from them.

It was noted that, after the agitation of the samples was stopped, both types of PAC settled to the bottom of the test flasks.

The next step was to conduct larger, bench scale tests at Ohmsett to examine adsorption kinetics, effects of low background dispersed oil concentrations, application/mixing of

PAC into the tank and efficiency of a DE/Cellulose mix on the leaf filter in removing suspended PAC from water.

6. Bench-Scale Tests

This task involved first conducting a series of bench-scale tests with the PAC removal concept at Ohmsett. The approach of conducting these tests on-site was not only more efficient, but also transferred the analytical and removal technology to the Ohmsett staff.

The tests involved adding salt (to 35 ppt), Corexit 9500A dispersant and dispersed crude oil to 20L samples of the tank water in a plastic carboy (Figure 5). The rate of removal of the dispersant by PAC was determined by measuring the IFT of water samples taken from the carboy and immediately filtered to remove the PAC at 1, 2, 5, 15 and 30 minutes after the PAC addition. The carboy was stirred by a paint mixer attachment to an electric drill for the entire test time. The same Aqua Nuchar PAC as used in the lab in Ottawa was employed at Ohmsett. Full data and lab notes may be found in Appendix C. The use of a highly refined mineral oil (technical grade for these tests) permitted the interfacial tension (IFT) method to reliably detect concentrations of Corexit 9500A in Ohmsett water down to 2 ppm (see Figure 2 in Section 3).

Figure 6 shows the adsorption of Corexit 9500A from Ohmsett water onto 50 ppm of PAC. It appears that 50 ppm of dispersant cannot be completely removed by 50 ppm of PAC, but that 20 ppm of dispersant can, with 90% efficiency. A 15-minute contact time appears to be sufficient for the adsorption to be essentially complete. The presence of up to 10 ppm of dispersed oil in the water does not appear to affect the adsorption.

Figure 7 shows the effect of increasing dispersed oil concentration on the adsorption of 20 ppm of Corexit 9500A from Ohmsett water by 50 ppm PAC. Both the 20-ppm oil and 50-ppm oil solutions showed appreciably degraded adsorption kinetic curves compared with the lower oil concentration solutions. The filtrate from the 20-ppm oil tests was sent to an outside laboratory for Total Petroleum Hydrocarbon analysis by the IR method. The results indicated that: the tank water contained less than 1.5 ppm oil; and, only the 30-minute sample (of 2, 5, 15 and 30 minute sample submitted) gave a reading above the detection limit (6 to 8 mg/L) of 9.7 mg/L, resulting in an estimated 50% removal of the dispersed oil. This leads to the conclusion that the oil concentration in the tank should be below 10 ppm prior to commencing dissolved dispersant removal with PAC.

Figure 8 demonstrates that the adsorption kinetics does not appear to be a strong function of PAC concentration. A concentration of 50 ppm PAC adsorbs 20 ppm of dispersant at approximately the same rate as 20 ppm of PAC adsorbs 10 ppm of dispersant. Based on the kinetic studies of the adsorption of Corexit 9500A on PAC it appears that a 15-minute contact time is all that is required to remove about 90 % of the "equivalent" Corexit 9500A, even in the presence of 5 ppm dispersed ANS crude oil droplets. This was true for 50 ppm PAC treating a solution of 20 ppm Corexit 9500A and 20 ppm PAC treating a solution of 10 ppm Corexit 9500A. Further, dispersed oil does not appear to detract



Figure 5: Apparatus for dispersant removal by PAC bench tests.

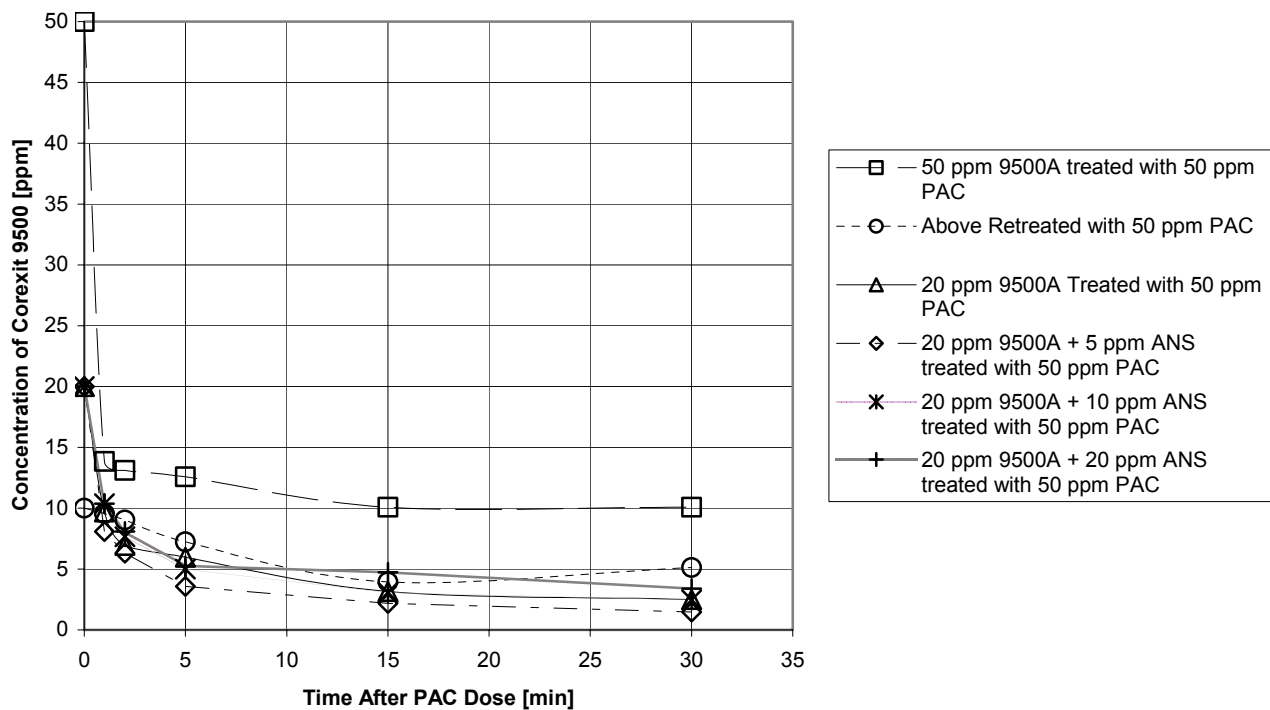


Figure 6: Results of bench-scale PAC kinetic adsorption tests.

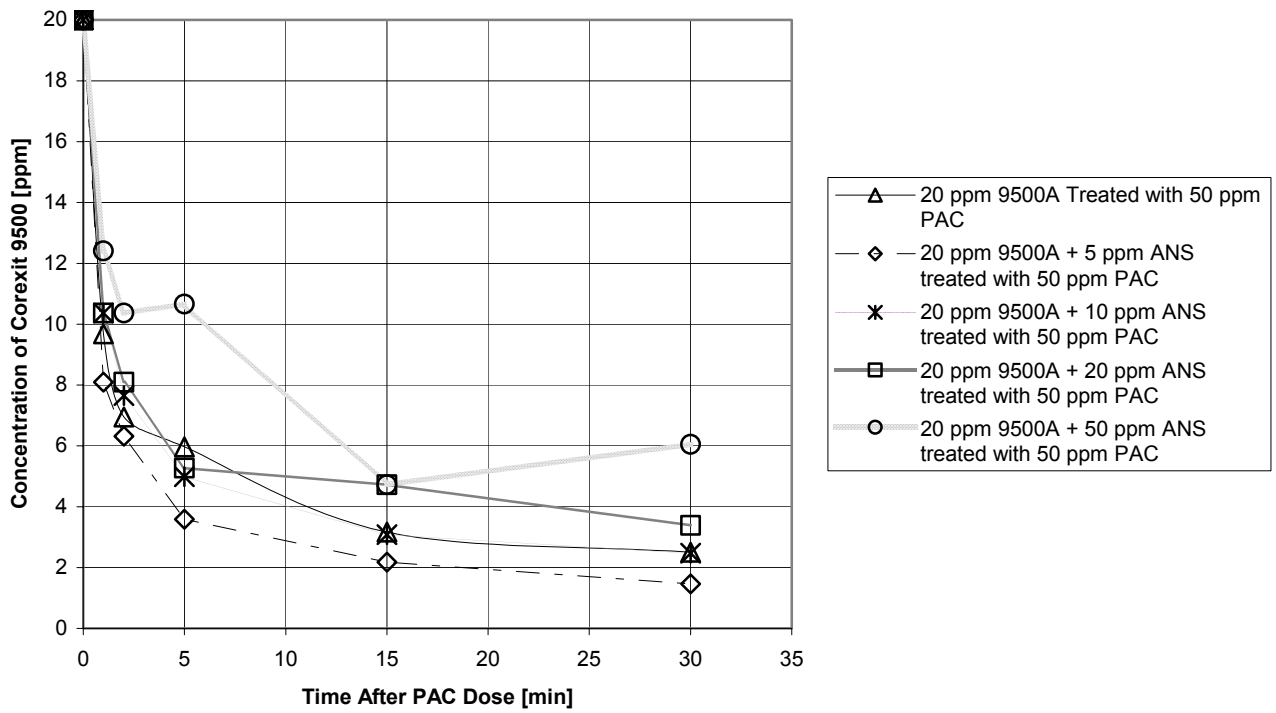


Figure 7: Effect of background dispersed oil on adsorption of Corexit 9500A by PAC.

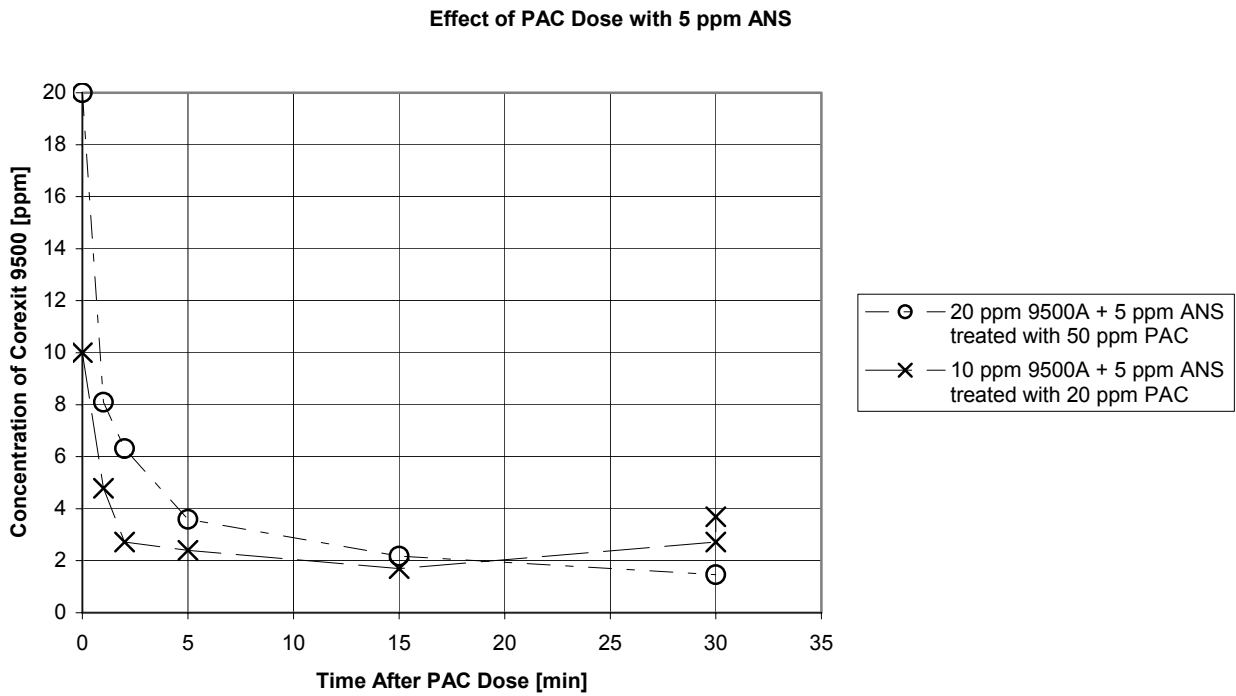


Figure 8: Adsorption of dispersant by different concentrations of PAC.

appreciably from the adsorption of dissolved Corexit 9500A, until dispersed oil concentrations exceed 10 ppm.

As another part of this task, tests were conducted to determine the ability of the leaf filter to remove PAC from the tank water. A slurry of Aqua Nuchar PAC in water was created in a portable tank (Figures 9 and 10) and slowly pumped over 10 minutes into the filter intake box at the bottom of the tank in the North-east corner at a rate designed to achieve a concentration of approximately 100 ppm in the filter influent. Samples of the water were taken simultaneously upstream and downstream of the filter (Figure 11) and subsequently filtered (Figure 12) to determine the carbon content. Weighing the samples revealed that over 80% of the carbon was removed, although this is believed to be a very conservative estimate because the filter papers were not oven-dried before use, making their tare weights very susceptible to humidity effects. It was concluded that the Ohmsett leaf filter could effectively remove the PAC from the water with high efficiency. Although not specifically monitored, an increase in the pressure drop across the leaf filter



Figure 9: Mixing PAC into water in portable tank.



Figure 10: Samples from before and after filter.



Figure 10: Mixing slurry



Figure 11: Filter papers from samples.

from 30 to 40 psig was observed over the 10-minute duration of this test, leading to concerns that a continuous feed of PAC to the filter would require frequent backwashing of the filter, and subsequent high operator costs for the dispersant removal process.

7. Full-scale System Design

Using the results of the bench scale testing, two options for a full-scale system for the removal of dissolved dispersant from Ohmsett tank water were designed. Since the system would only be used following completion of a dispersant testing program, and the cost of dispersant removal would have to be borne by testing clients, the system design was guided strongly by the principles of simplicity and low cost. This meant that the existing Ohmsett filter system pumps and piping were to be used to the greatest extent possible, and any treatment vessels required would be “off-the-shelf”.

The first option was based on applying the bench-scale results directly to the problem. Based on the 15-minute residence time requirement, and an 1800 gpm nominal water filtration rate, a hold-up volume of 30,000 gallons (100 cubic metres) would be required to treat the water with PAC before filtration. This could be accomplished by installing a simple, temporary treatment channel 10 feet wide and 40 feet long along the North end of the West wall of the 8-foot deep tank that feeds directly into the inlet sump for the filter pump suction at the bottom of the tank beside the beaches. The channel would use a tarp, with the bottom weighted to seal with the tank bottom, suspended from wire cables between the Main and Auxiliary bridges, to form its other vertical wall. The purpose of the tarp wall would be to prevent PAC from getting out into the entire tank. Since PAC sinks when mixing energy is removed, everywhere that PAC is allowed to settle would have to be vacuumed. This channel would be open only at its south end to allow inflow from the tank. A box would be constructed to connect the channel directly to the inlet sump to the filter, so that the water is drawn only down the channel. PAC would be continuously injected into the upstream end of the channel at a rate based on the concentration of dispersant in the water. Air bubbler pipes (powered by the Main Bridge compressor) would be installed along the centerline of the channel to mix PAC into the water in the channel. The residence time of the water in the channel would be 15 minutes. This concept would minimize the capital cost of a system, but due to the continuous feed of PAC to the leaf filter, would require constant attention from a filter operator to backwash when the pressure drop across the leaf filter caused the system to shut down.

The second concept proposed was a treatment channel that spanned the width of the tank, and contained a well-mixed slurry of activated carbon through which the tank water flowed from South to North under the influence of the filtration pumps (the 1800 gpm nominal filtration rate equates to circulating the 2.6 million gallons of tank water once per day at an average rate of 0.5 ft/min. along the tank). In order to achieve a 15-minute hold-up, the treatment chamber would have to be 8 feet wide, and to span the width of the tank it would have to be 65 feet long. In order to seal along the bottom and extend above the normal surface level, it would have to be 8 feet high. The chamber would have to allow water to pass easily through it, yet retain the PAC slurry inside. The advantages of

this system over the first option were: first, that the PAC would be retained inside the chamber and prevented from entering the tank water, and subsequently the leaf filter, thus eliminating the cost of a filter operator to constantly attend the system.

After considerable discussion it was decided to produce a prototype chamber based on the second option, using modified 20'-ISO shipping containers (also known as Conex's). These were the appropriate height and width, and three placed end-to-end a short distance apart would span the 65-foot width of the tank. Figures 12 and 13 illustrate the basic concept of the modified shipping container. The top and two sides of the container are cut away. The sides are replaced with an expanded metal mesh that supports a very fine filter cloth clamped around the edges of the opening. On the upstream side of the box, the filter cloth is on the outside of the box, on the downstream side the cloth is on the inside. PVC air piping is laid along the bottom of the container on approximately 16-inch centers with $\frac{1}{8}$ -inch holes drilled in the bottom every 8 inches. An additional air pipe is placed directly at the bottom of the downstream, or exit, side to provide agitation to the filter cloth to ensure a cake of PAC does not clog it. A square is cut in the bottom of the container and covered with mesh and cloth to permit the container to drain completely when removed from the tank. Figures 14 and 15 show one of the finished containers beside the tank.

Figure 16 shows the intended operation of the system. The three modified containers are placed end to end across the tank, with the intervening spaces sealed with rubber wipers. The intention is that each container has 200 pounds of PAC added to it, and then the air bubbler system is connected to the compressor and activated. This agitates the slurry of PAC and prevents it from settling. The head created by the filter pump moving water from the North end of the tank to the South end forces water through the filter cloth on the upstream (South) side of the containers and out the downstream (North) side. In the 15 minutes that the water spends inside the containers it is thoroughly mixed with the PAC slurry. When the water has been cleaned, the containers are lifted very slowly out of the water (to prevent tearing the filter cloth) and allowed to drain back into the tank. The containers are then placed on the ground beside the tank to dry. The dried, spent PAC is removed by opening the doors and shovelling it out, for disposal with the spent DE. Figures 17 through 20 show the system being installed in the tank and operated.

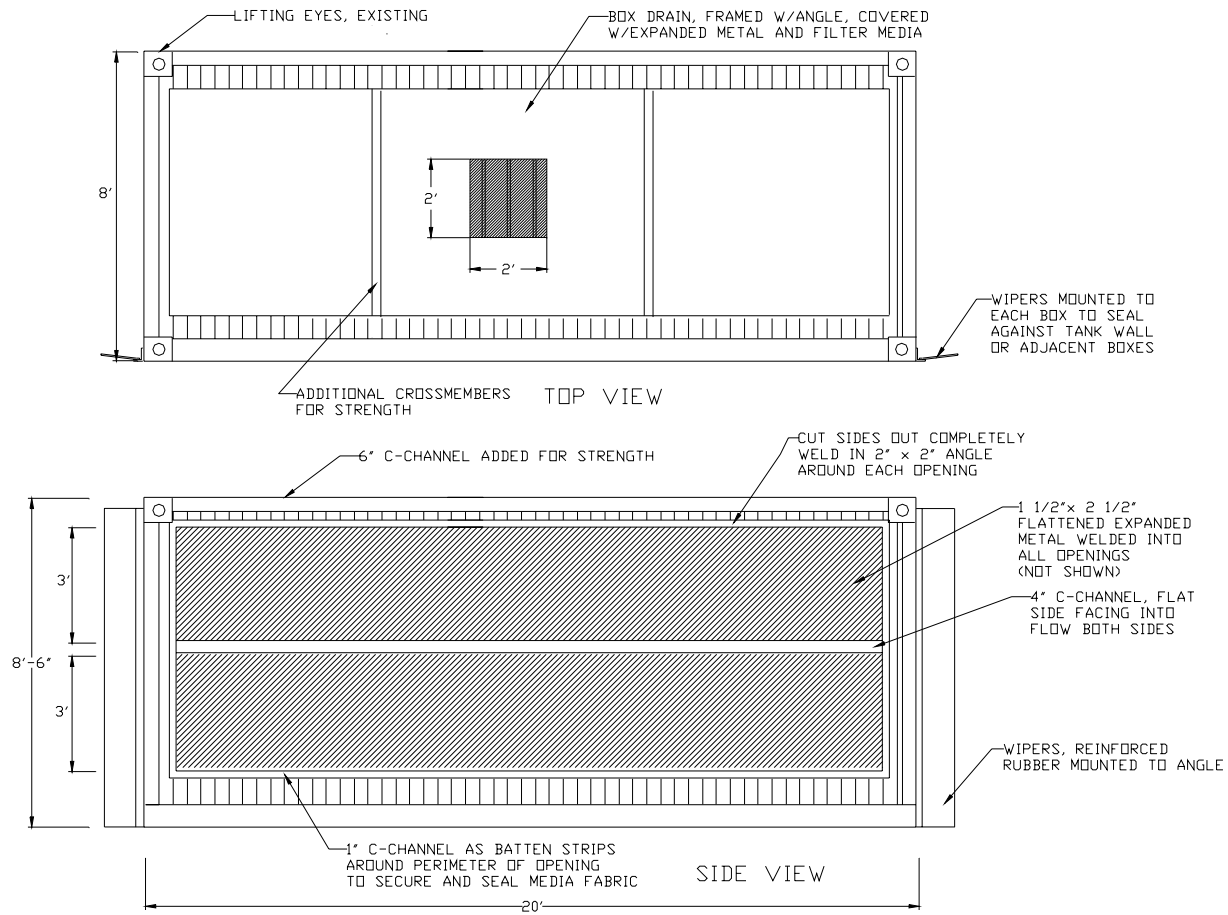


Figure 12: Modifications required to standard ISO shipping containers.

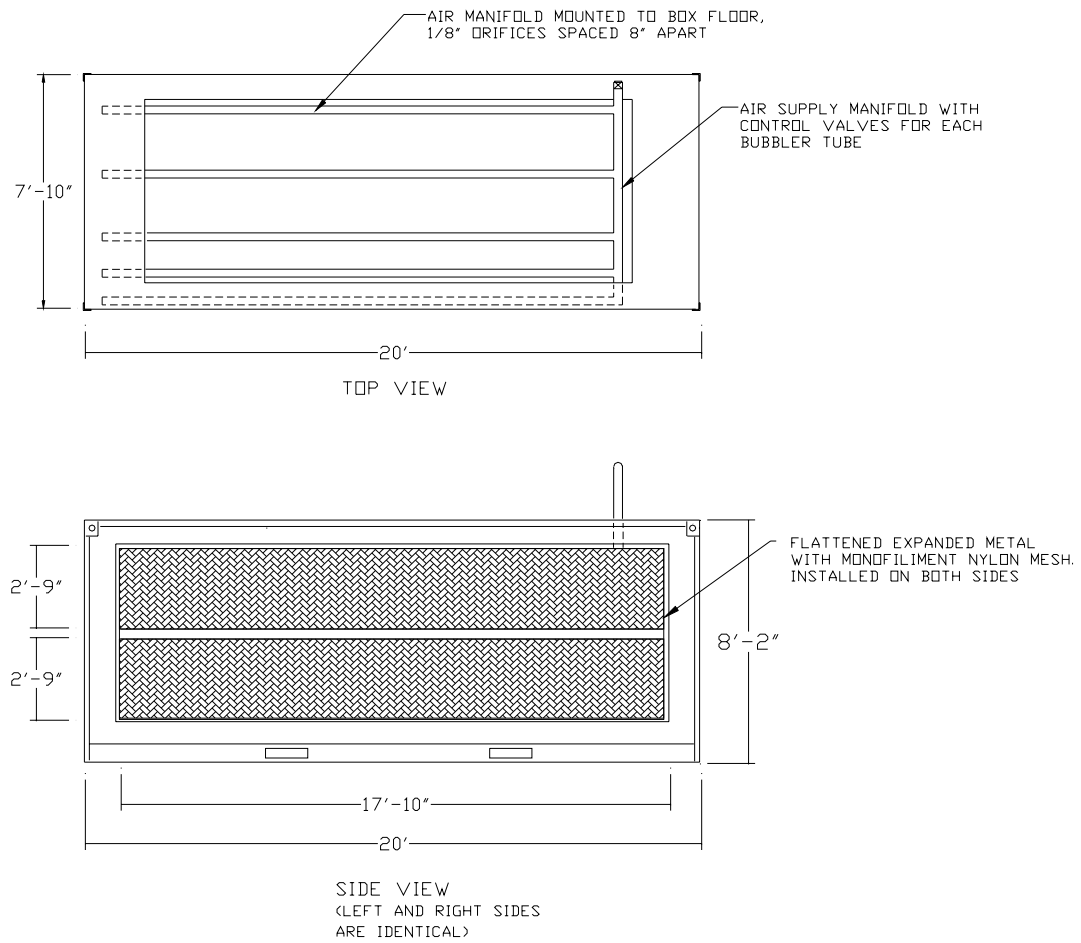


Figure 13: Plans for the addition of expanded metal mesh, filter cloth and air bubbler piping to modified containers.



Figure 14: Finished container, with rubber wipers installed at ends.



Figure 15: View inside container through open doors.

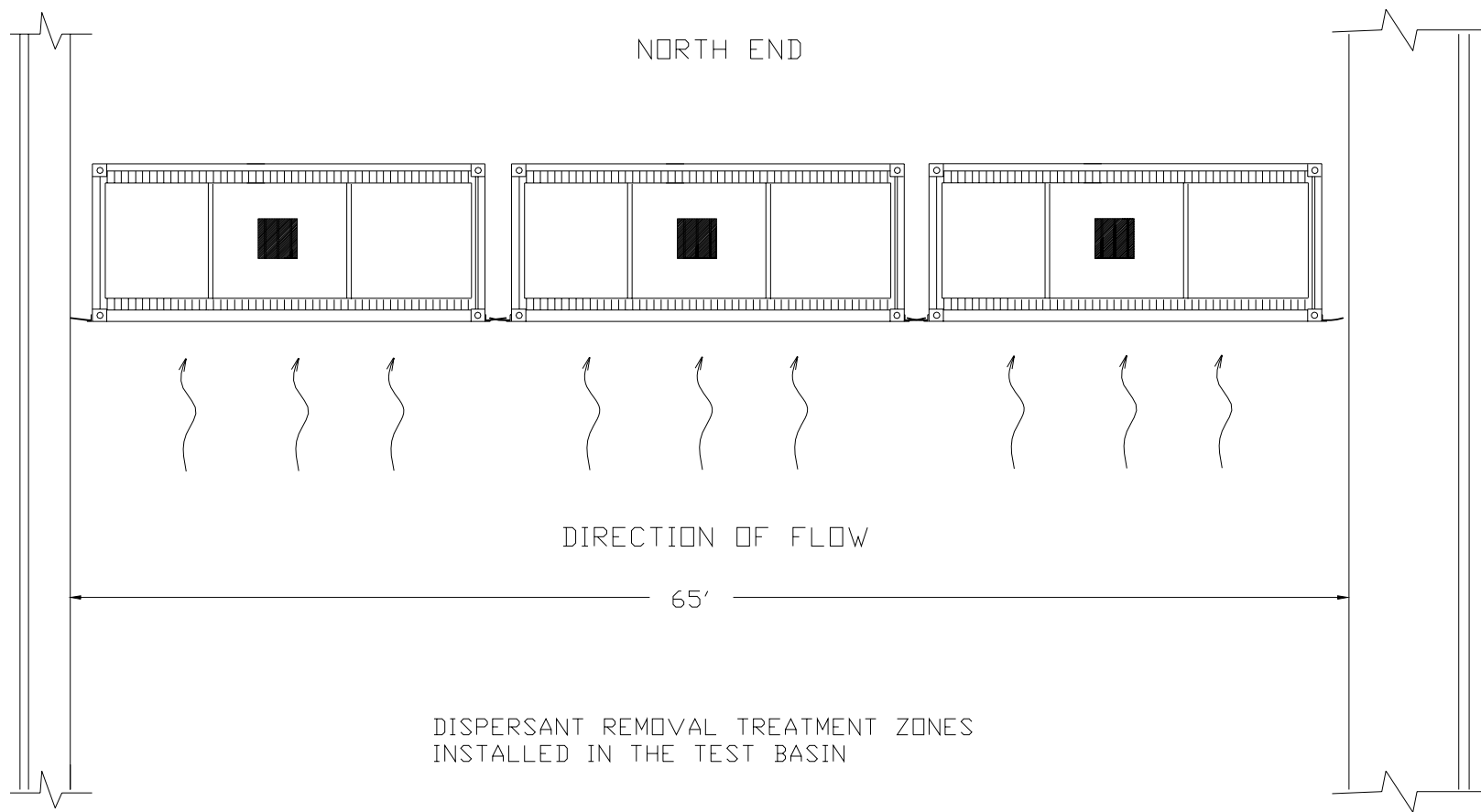


Figure 16: Intended operation of the dispersant removal system.



Figure 17: Containers being lifted into place across tank.



Figure 18: Checking operation of bubbler system.



Figure 19: Adding initial 200-lb load of PAC to each container.



Figure 20: System in operation.

8. Full-scale System Test

Prior to ordering the PAC for the full-scale system tests, another series of bench-scale adsorption kinetics tests was conducted. This was necessary because a different dispersant was used (Corexit 9527 as opposed to 9500A) and the smallest mesh size of filter cloth for the container sides that was available was 168 x 168 mesh (90 μm x 90 μm gaps). Thus, the PAC to be used would have to be of much larger particles than the 325 mesh (45 μm) mean particle size PACs originally tested. Two different sizes of PAC from a different supplier were tested: 40 x 165 mesh (particle sizes between 90 and 350 μm); and, 50 x 200 mesh (particle sizes between 75 and 300 μm). Figure 21 shows the results. The data may be found in Appendix D.

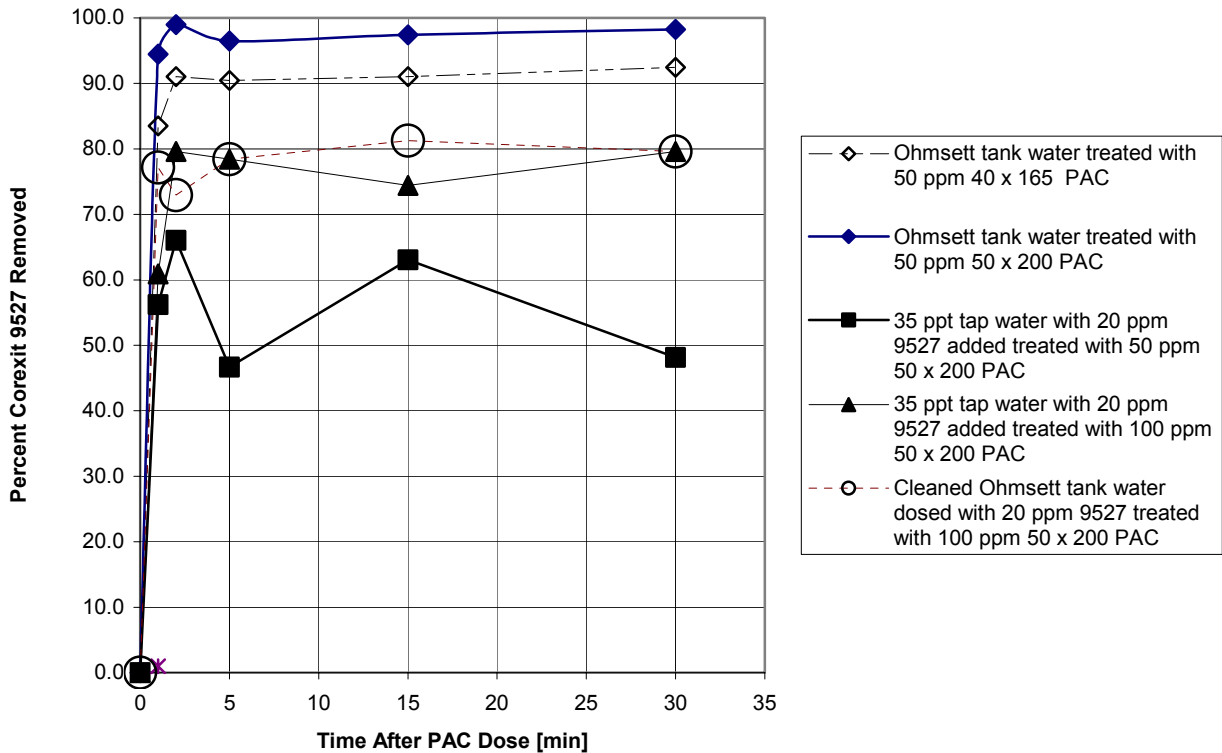


Figure 21: Adsorption kinetics for different size PAC.

The removal effectiveness is expressed as percent of the original concentration of dispersant because two types of samples were tested: tank water containing dispersant (5.5 to 9 ppm) from a series of effectiveness tests that had been filtered for two weeks to remove dispersed oil; and, tap and cleaned tank water dosed with Corexit 9527 to the 20 ppm level. In general the results were very similar to the previous bench-scale tests (Figures 6, 7 and 8 in Section 6). The 50 x 200 mesh PAC seemed to do a slightly better job on the actual tank water than the 45 x 165 PAC, but the overall adsorptive capacity of

the 50 x 200 PAC seemed to be slightly less on the Corexit 9527 than the PAC used in the laboratory and bench-scale tests was with Corexit 9500A. This points out the necessity to test each dispersant proposed for use at Ohmsett with the carbon to be utilized, to confirm that it will be effectively removed by the PAC system. Conducting an adsorption kinetic test as described in Section 6 should suffice.

The 50 x 200 mesh PAC was chosen for the full-scale tests, even though the finer portions would pass through the 168 x 168 mesh filter cloth on the containers. The initial charge of PAC was 200 lbs per container. An additional 200 lbs was added to each container after 24 hours to ensure that all the dispersant was adsorbed.

The full-scale tests were conducted in March 2003, two weeks after a series of cold-water dispersant effectiveness experiments had taken place at Ohmsett (SL Ross 2003). These tests employed about 20 gallons of Corexit 9527. If all this dispersant ended up in the water (some could remain with oil that is recovered off the water's surface after an inefficient test) the concentration of dispersant in the water would be 7.7 ppm (20 gallons in 2.6×10^6). Figure 22 shows the concentration of dispersant in grab samples of Ohmsett water taken from various positions around the tank before and after the PAC was added to the containers and the air bubblers started. The concentrations of Corexit 9527 measured prior to the start of the removal system range from 5.5 to 9 ppm; after the system was started all the samples were measured at < 1 ppm over the 72 hours that the system was operated.

When the air bubblers were first started, there was a short period in which a "cloud" of PAC moved away from both sides of the containers. This was presumably the fines from the PAC washing through the filter cloth. This cloud disappeared after a few hours operation. When the second load of PAC was added a similar phenomenon was observed. The bubblers seemed to perform effectively, even though the airflow valves were only cracked open. Opening the airflow valves too far could cause the filter cloth to rip or pull out from underneath its battens.

On removal from the tank, it was necessary to raise the containers very slowly, or the pressure of the water would tear the filter cloth and allow the PAC slurry to leak into the tank. The containers should have a second support system, such as the expanded metal mesh, added on both sides to better support the fragile filter cloth while the containers are being put into or removed from the tank. It was also noted that it was impossible to lift the containers from the tank level; the containers always tipped towards the door end, due to the extra weight of the doors, which trapped a considerable volume of water inside. It is recommended that the filter cloth-covered floor drains be relocated to the door end to allow complete drainage of the containers prior to swinging them out of the tank.

Considering the time required to operate the leaf filter to reduce dispersed oil concentrations to below 10 ppm after a dispersant test series in the tank (three to five days – SL Ross 2000b) and a two to three day period of treatment with PAC, it should be possible to return the tank to a quality that meets the standard for equipment testing in one week's time.

Tank Water Samples Before and After Removal System Startup

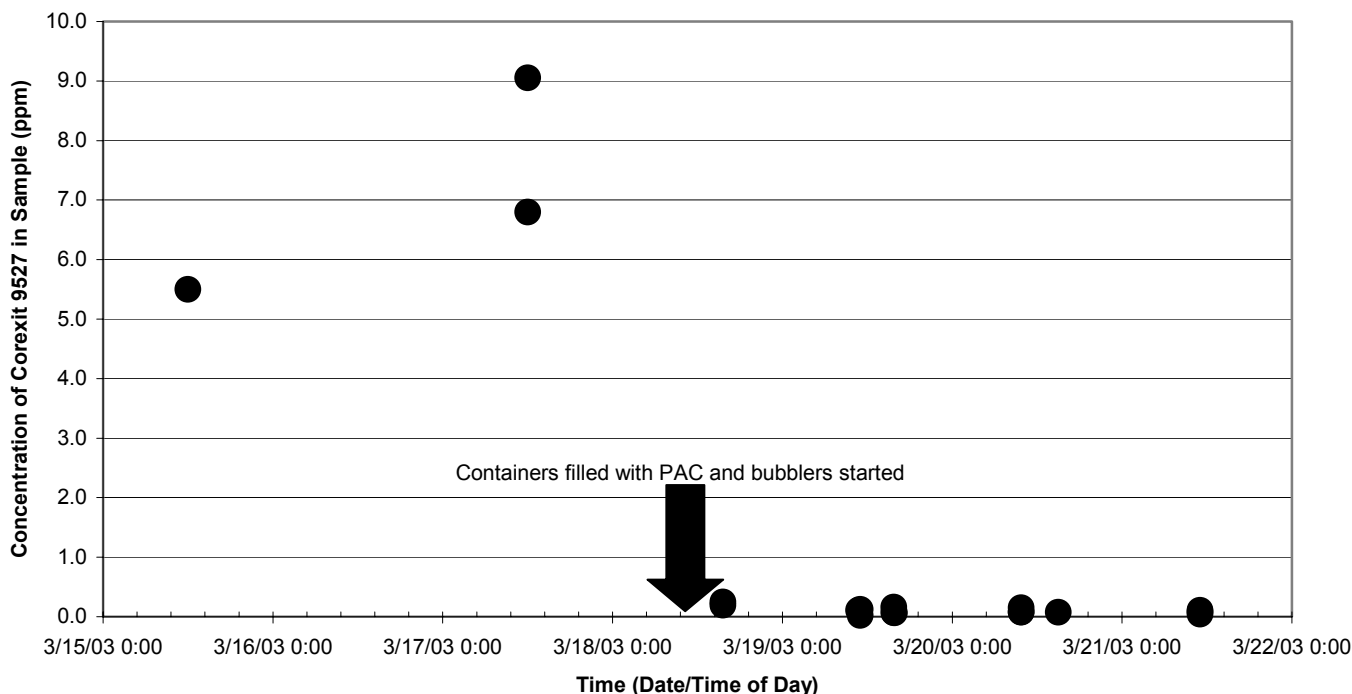


Figure 22: Concentrations of Corexit 9527 measured in Ohmsett tank water before and after system start-up.

9. Conclusions and Recommendations

1. Powdered activated carbon (PAC) was determined to be the best answer for the removal of dissolved dispersant from the tank water after chemical dispersant tests at Ohmsett. A series of lab- and bench-scale tests were undertaken to quantify the expected performance of PAC and design a full-scale removal system. These tests concluded that:
 - The adsorptive capacity of PAC for Corexit 9500A dispersant (a commonly-stockpiled dispersant in the U.S.) is about 1 g/g;
 - A dose of 50 ppm PAC could adsorb 20 ppm of dispersant, and 20 ppm of PAC could adsorb 10 ppm of dispersant, even in the presence of up to 10 ppm of dispersed oil in the water;
 - The required contact time for the adsorption to proceed essentially to completion was 15 minutes.
2. It was concluded that the best operational analytical technique to measure dissolved dispersant concentrations in the tank water was to measure the interfacial tension of the water against a highly refined mineral oil (USP or Technical grade) with a

DuNouy ring tensiometer following procedures laid out in ASTM D971. The use of USP-grade mineral oil resulted in a much-expanded interfacial tension range compared to other crude or refined oils. Due to the very “clean” interface provided by the high-grade mineral oil this approach gave good discrimination at the sub-10 ppm dispersant concentration range with reliable measurements down to 2 ppm. For accuracy at low dispersant concentrations, it was important to pre-treat the water used to develop the calibration standards with the grade and type of activated carbon that would eventually be used in cleaning the tank water. It was also important to ensure that an adequate supply of one manufacturer’s mineral oil was available to cover all the analyses required. The calibration standards also need to equilibrate for several hours before measurement. It became clear, in order to efficiently prepare calibration curves and analyse samples accurately on an operational basis, that a more sophisticated, less operator-dependent DuNuoy ring apparatus was required for the lab at Ohmsett.

3. A prototype treatment system was built and tested following a series of cold-water dispersant experiments at Ohmsett in March 2003. The system consisted of three modified ISO shipping containers (the sides were removed and replaced with fine filter cloth) placed end to end in the tank, spanning its width. The containers held well-mixed slurry of powdered activated carbon through which the tank water flowed from South to North under the influence of the filtration pumps.
4. Grab samples of the tank water taken before and after installation of the prototype system showed that it reduced the concentration of Corexit 9527 dispersant in the tank water from a range of 5.5 to 9 ppm before to below the detection limit (1 ppm) after.
5. Combining the three to five days required to operate the leaf filter to reduce dispersed oil concentrations to below 10 ppm (SL Ross 2000b) and a two- to three-day period of treatment with PAC, it should now be possible to return the tank to a quality that meets the standard for equipment testing in one week following a dispersant test series.
6. It is recommended that, prior to conducting dispersant effectiveness tests in the tank, each dispersant proposed for use at Ohmsett be tested with the PAC to be utilized, to confirm that it will be effectively removed by the PAC system. Preparing a dispersant concentration – IFT calibration curve and conducting an adsorption kinetic test as described in this report should suffice.
7. The containers should have a second support system, such as another expanded metal mesh added on both sides, to better support the fragile filter cloth while the containers are being put into or removed from the tank. It is further recommended that the filter cloth-covered floor drains be relocated to the door end to allow complete drainage of the containers prior to swinging them out of the tank

10. References

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Appendix A – Initial IFT vs. Concentration Data

Conc 9500A/Ohm35ppt [ppm wt]	IFT reading Ohm35/10W30
56	9.3
20	12.2
10	13.5
5	14.5
2	15.2
1	15.3
0	14.7
Conc. PAC in Filt'd Water	IFT reading Ohm35/10W30
1000	13.1
1000	13.9
100	15.5
50	15.6
Conc 9500A/filt'd Ohm35ppt [ppm wt]	IFT reading Ohm35/10W30
50	11.6
50	11.5
20	13.4
10	14.4
5	14.5
0	14.9
50	11.6
50	11.5
20	13.4
10	14.4
5	14.5
0	14.9

Notes:
1. Tensiometer re-calibrated before tests - surface tension of tap water was 70.8 dynes/cm at 20-25°C, within 2% of CRC value for distilled water
2. Ohmsett water as rec'd had a density of 1.015 g/cm ³ at 24.7°C thus added 0.006 g salt/g water to bring up to approx 35 ppt
3. Surface tension of 35 ppt Ohmsett water was 65 dynes/cm
4. Sample of Hydrocal was dyed and had IFT of 10.4, 11.2 and 11.8 dynes/cm thus was not used
5. Used virgin 10W/30 motor oil with IFT of 14.7 over Ohmsett 35 ppt water as test for conc of 9500.
6. Discovered that treating Ohmsett 35 ppt water with PAC then filtering with P8 Fisherbrand filter paper reduced IFT of water (see IFT-Conc Calibration chart).
7. Unfiltered Ohmsett 25 ppt had IFT of 14.7 dynes/cm, 1000 ppm PAC filtered had 13.1 dynes/cm and 100 and 50 ppm PAC filtered had 15.5 and 15.6 dynes/c
8. Used Ohmsett 35 ppt water treated with 100 ppm PAC then filtered to make up 9500 conc vs. IFT standards.

Appendix B – PAC Adsorption Isotherm Data

Conc Aqua Nuchar PAC [ppm wt]	IFT reading Ohm35/10W30	Calc'd Conc 9500 from IFT reading (ppm)	Corexit 9500 Remaining (IFT) [mg]	Corexit 9500 Adsorbed (IFT) [mg]
blank (no 9500)				
		19.296	2.412	
	100	17.817	2.227125	4.022875
	60	20.775	2.596875	3.653125
	40	19.296	2.412	3.838
	20	26.691	3.336375	2.913625
	10	40.002	5.00025	1.24975
Conc Nuchar SN PAC [ppm wt]	IFT reading Ohm35/10W30			
	100	14.1	1.1901	3.8099
	50	14.3	0.8943	4.1057
	40	13.8	1.6338	3.3662
	30	13.3	2.3733	2.6267
	20	13.2	2.5212	2.4788
	20	13.1	2.6691	2.3309
	10	12.3	3.8523	1.1477

9. Used ASTM D 3860-98 to construct adsorption isotherms for two grades of PAC (Aqua Nuchar and Nuchar SN).
10. Error estimates on Isotherm_IFT chart are : + 25% for y values, based on error in weighing PAC and + 8 ppm for x values based on repeatability with DuNouy Ring Tensiometer.
11. Estimate that approx. 1g of Corexit 9500 can be adsorbed by 1 g of PAC in concentration range of interest (20 ppm = one drum of Corexit mixed into Ohmsett tank contents).
12. Effective treatment dose seems to be 50 ppm (1000 lbs PAC mixed into 2.6 million gallons of tank water).
13. PAC settled to bottom of test flasks in lab tests when mixing energy removed - Aqua Nuchar settled slower than Nuchar SN.
14. Need to examine adsorption kinetics, effect of background dispersed oil concentrations, application/mixing of PAC into tank by firehose/wave action and efficiency of DE/Cellulose mix on leaf filter in removing suspended PAC from

Appendix C – Bench-scale Data

Conc 9500A in Ohmsett 30 ppt [ppm wt]	IFT reading [dynes/cm]				IFT reading [dynes/cm]	Time After 50 ppm PAC (min)	IFT reading [dynes/cm]	Calc. Conc. 9500A Remaining [ppm]	% Removed
0	35.8				36.7		0	50	0
1	35.1				32.8		1	13.8544011	72.2911978
5	19.4				26.7		2	13.11082397	73.7783521
10	9.9				18.7		5	12.57945016	74.8410997
20	9.9				13.8		15	10.08859243	79.8228151
50	1.5 after 10 minutes				7.7		30	10.08859243	79.8228151
50	1.1 after 20 minutes								
50	3.7 after 1 hour					Time After Add1 50 ppm PAC (min)	IFT reading [dynes/cm]	Calc. Conc. 9500A Remaining [ppm]	
50	4.6 next morning					0	0	10	0
Conc 9500A in Filt'd Ohmsett 30 ppt [ppm wt]									
0	43						1	9.547129368	80.9057413
29.5	7.9 after 1 hour						2	9.034727054	81.9305459
9.8	12.8 after 1 hour (dilut'd from above)						5	7.245760174	85.5084797
4.9	19 dilut'd from above						15	3.94955787	92.1008843
2	27.3 dilut'd from above						30	5.132723751	89.7345525
						Time After 50 ppm PAC in 20 ppm 9500A (min)	IFT reading [dynes/cm]	Calc. Conc. 9500A Remaining [ppm]	
29.5	1.6 after 5 minutes					0	0	20	0
29.5	1.4 after 10 minutes					1	14.7	9.679707847	51.6014608
						2	17.1	6.952093871	65.2395306
						5	18.2	5.973539766	70.1323012
						15	22.8	3.167505864	84.1624717
						30	24.5	2.505514245	87.4724288
Leaf Filter PAC Removal Test Data									
Sample #	Volume [mL]	Filter Paper Wt. [g]	PAC Conc. [mg/L]	Corrected PAC Conc [mg/L]					
1-up		470							
						Time After 50 ppm PAC in 20 ppm 9500A + 5 ppm ANS (min)	IFT reading [dynes/cm]	Calc. Conc. 9500A Remaining [ppm]	
						0	0	20	0
1-down		440	103.8297872	88.93617021					
						1	16	8.090949603	59.545252
						2	17.8	6.312327592	68.438362
			32.5	16.59090909		5	21.9	3.586099897	82.0695005
				81.34515007		15	25.5	2.182742227	89.0862889
						30	28.4	1.463216586	92.6839171
2-up		463							
						Time After 50 ppm PAC in 20 ppm 9500A + 10 ppm ANS (min)	IFT reading [dynes/cm]	Calc. Conc. 9500A Remaining [ppm]	
						0	0	20	0
2-down		385	117.9265659	102.8077754					
						1	14.2	10.37073322	48.1463339
						2	16.4	7.656701666	61.7164917
			36.1038961	17.92207792		5	19.5	4.993085531	75.0345723
				82.56739059		15	23	3.081332148	84.5933393
						30	24.6	2.471197373	87.6440131
3-up		459							
						Time After 50 ppm PAC in 20 ppm 9500A + 20 ppm ANS (min)	IFT reading [dynes/cm]	Calc. Conc. 9500A Remaining [ppm]	
						0	0	20	0
3-down		410	143.7908497	128.540305					
						1	14.2	10.37073322	48.1463339
						2	16	8.090949603	59.545252
			33.17073171	16.09756098		5	19.1	5.276267139	73.6186643
				87.47664324		15	19.9	4.725102513	76.3744874
						30	22.3	3.393630959	83.0318452
Tap water control									
						Time After 50 ppm PAC in 20 ppm 9500A + 50 ppm ANS (min)	IFT reading [dynes/cm]	Calc. Conc. 9500A Remaining [ppm]	
						0	0	20	0
						1	12.9	12.40715524	37.9642238
						2	14.2	10.37073322	48.1463339
						5	14	10.66076445	46.6961777
						15	19.9	4.725102513	76.3744874
						30	18.1	6.056492751	69.7175362
No Water Control									
						Time After 20 ppm PAC in 10 ppm 9500A + 5 ppm ANS (min)	IFT reading [dynes/cm]	Calc. Conc. 9500A Remaining [ppm]	
						0	0	10	0
						1	19.8	4.790718776	52.0928122
						2	23.9	2.721657877	72.7834212
Upstream Control		420							
						5	24.8	2.403967259	75.9603274
						15	27.3	1.70291309	82.9708691
			19.04761905			30	23.9	2.721657877	72.7834212
						30	21.7	3.686389909	63.1361009
Downstream Control		400							
						0	0	10	0
						1	19.8	4.790718776	52.0928122
						2	23.9	2.721657877	72.7834212
						5	24.8	2.403967259	75.9603274
						15	27.3	1.70291309	82.9708691
			15.25			30	23.9	2.721657877	72.7834212
						30	21.7	3.686389909	63.1361009

Appendix D – Full-scale Data

Conc 9527A in 35 ppt Salt Water (ppm wt)	IFT reading [dynes/cm]		Time After 50 ppm 40 x 165 PAC added to tank water drawn 15/03/03 (min)	IFT reading [dynes/cm]	Calc. Conc. 9527A Remaining (ppm)	% Removed		IFT reading [dynes/cm]	Calc. Conc. 9527A Remaining (ppm)	% Removed				
0	60.2			0	22.0	5.5	0	CLEAN TANK WATER	47.6	0.0				
1.2	33.6			1	26.1	0.9	83.5066579							
9.4	15			2	29	0.5	91.0670553	Sample from upstream of filter on 15/03/03	22.0	5.5		3/15/03 12:00	37695.50	
19.8	13.6			5	28.7	0.5	90.482041	Sample from upstream of filter on 17/03/03	20.6	6.8		3/17/03 12:00	37697.50	
48.8	9.4			15	29	0.5	91.0670553	ditto	18.7	9.1		3/17/03 12:01	37697.50	
Conc 9527A in 40 x 165 PAC Filtr'd 35 ppt Salt Water (ppm wt)			30	29.8	0.4	92.4572422		Sample from mid-tank, East side at 1535 hrs 18/03/03	28	0.2	98.9092055	3/18/03 15:35	37698.65	
0	47.8		Time After 50 ppm 50 x 200 PAC added to tank water drawn am 18/03/03 (min)					ditto	27.1	0.2	98.6009742	3/18/03 15:36	37698.65	
1	25.8			0	25.4	2.7	0	Sample from mid-tank, East side at 1045 19/03/03	30	0.1	99.3639084	3/19/03 10:45	37699.45	
11.6	13.9			1	28.9	0.1	94.4770028	Sample from pre-filter sample tube at 1100 19/03/03	29.6	0.1	99.3101199	3/19/03 11:00	37699.46	
20	11			2	34.9	0.0	98.9878385	Sample from inside West container at 1100 19/03/03	35.7	0.0	99.8770959	3/19/03 11:00	37699.46	
47.9	7.8			5	30.5	0.1	96.4671348	Sample from mid-tank, East side at 1545 19/03/03	31.7	0.1	99.6190659	3/19/03 15:45	37699.66	
Conc 9527A in 20 x 60 PAC Filtr'd 35 ppt Salt Water (ppm wt)				15	31.6	0.1	97.4262952	Sample from pre-filter sample tube at 1550 19/03/03	32	0.1	99.6600519	3/19/03 15:50	37699.66	
0	47.9			30	32.9	0.0	98.2180666	Sample from West side, just South of containers at 1545 19/03/03	28.6	0.2	99.0846347	3/19/03 15:45	37699.66	
1	27.8		Time After 50 ppm 50 x 200 PAC added to 35 ppt tap water dosed with 20 ppm 9527 (min)					Sample from mid-tank, East side at 0945 20/03/03	28.9	0.1	99.1590925	3/20/03 9:45	37700.41	
10.2	15.8			0		20	0	Sample from pre-filter sample tube at 0945 20/03/03	31.2	0.1	99.5612064	IFT against Calsol = 21.9 dynes/cm	3/20/03 9:45	37700.41
19.3	12.7			1	14.5	8.8	66.234827	Sample from West side, just South of containers at 0945 20/03/03	31	0.1	99.5366725	3/20/03 9:45	37700.41	
47.7	8.2			2	15.4	6.8	66.0695284	Sample from pre-filter sample tube at 1500 20/03/03	31.3	0.1	99.5734419	3/20/03 15:00	37700.41	
Conc 9527A in 50 x 200 PAC Filtr'd 35 ppt Salt Water (ppm wt)				5	13.8	10.7	46.653831	Sample from mid-tank, East side at 1100 21/03/03	33.4	0.0	99.7644656	3/21/03 11:00	37700.41	
0	40.5			15	15.1	7.4	63.0651664	Sample from pre-filter sample tube at 1100 21/03/03	30	0.1	99.3839084	3/21/03 11:00	37700.41	
1.1	25.8			30	13.9	10.4	48.1413665	Sample from West side, just South of containers at 1100 21/03/03	31.1	0.1	99.5486199	3/21/03 11:00	37700.41	
10.2	16.8		Time After 100 ppm 50 x 200 PAC added to 35 ppt tap water dosed with 20 ppm 9527 (min)											
19.9	11.7			0		20	0							
49	7.6			1	14.9	7.8	60.9159821							
Conc 9527A in 50 x 200 PAC Filtr'd 35 ppt Salt Water w New Mineral Oil (ppm wt)				2	17.2	4.1	79.6055317							
0	46.4			5	17	4.3	78.418752							
1.1	22.1			15	16.4	5.1	74.4277431							
10.2	13.2			30	17.2	4.1	79.6055317							
19.9	11.8													
49	8.7		Time After 100 ppm 50 x 200 PAC added to cleaned tank water dosed with 20 ppm 9527 (min)											
Conc 9527A in 35 ppt Salt Water w New Mineral Oil (ppm wt)				0		20	0							
0	50.9			1	16.8	4.6	77.1629121							
1.1	31.7			2	16.2	5.4	72.9396614							
9.7	16.4			5	17	4.3	78.418752							
20.1	11.9			15	17.5	3.7	81.2644634							
49.8	8.8			30	17.2	4.1	79.6055317							