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TEST TANK INTER-CALIBRATION FOR DISPERSANT EFFICIENCY

REPORT 2.2
Test tank inter-calibration for dispersant efficiency

ABOUT THE JIP

Over the past four decades, the oil and gas industry has made significant advances in being able to detect, contain and clean up spills in Arctic environments. To further build on existing research, increase understanding of potential impacts of oil on the Arctic marine environment, and improve the technologies and methodologies for oil spill response, in January 2012, the international oil and gas industry launched a collaborative four-year effort – the Arctic Oil Spill Response Technology Joint Industry Programme (JIP).

Over the course of the programme, the JIP will carry out a series of advanced research projects on six key areas: dispersants, environmental effects, trajectory modeling, remote sensing, mechanical recovery and in situ burning. Expert technical working groups for each project are populated by the top researchers from each of the member companies.

JIP MEMBERS

The JIP is managed under the auspices of the International Association of Oil and Gas Producers (OGP) and is supported by nine international oil and gas companies – BP, Chevron, ConocoPhillips, Eni, ExxonMobil, North Caspian Operating Company (NCOC), Shell, Statoil, and Total – making it the largest pan-industry programme dedicated to this area of research and development.

TABLE OF CONTENTS

ABSTRACT

The objective of the inter-basin calibration study was to determine consistent test protocols and natural energy conditions as a start of Task 2. Dispersant effectiveness of one oil-dispersant combination has been performed using the pre-weathered Norwegian crude oil Grane and Corexit 9500 as the dispersant. Three different energy conditions (low, medium, high) were established. Triplicate dispersant effectiveness tests have been conducted at each energy condition.

CEDRE, SINTEF and SL Ross have the same type of meso scale flume basin, of which the flumes at SINTEF and SL Ross have exactly the same dimensions, while CEDRE's flume is larger. For a test basin to be acceptable, the standard deviation of the triplicates at each energy condition should be no more than ± 20% of the average for the test basins. The dispersant efficiency in all energy levels performed in the flumes at SINTEF, SL Ross, and Cedre were within the ±20% of the average dispersant efficiency.

The results have shown that there was a very good correlation between the dispersant efficiency in the flumes at SINTEF and SL Ross at all three energy levels. Although the Cedre flume is different than the flumes at SINTEF/SL Ross, the correlation in dispersant efficiency was good, especially a low and high energy conditions. It is recommended that all three flumes are accepted for further testing of dispersant/OMA efficiency in Task 2.

CHAPTER 1. INTRODUCTION

CEDRE, SINTEF and SL Ross have the same type of meso scale flume basin, of which the flumes at SINTEF and SL Ross have exactly the same dimensions, while CEDRE's flume is larger. As a first phase of Task 2 of the project "Dispersant testing under realistic conditions", OGP has requested that a calibration between the three flumes be conducted. The SINTEF flume was used in the meso scale studies on dispersant testing in the SINTEF led Oil-in-ice JIP (Brandvik et al., 2010).

The objective of the inter-basin calibration study was to determine consistent test protocols and natural energy conditions as a start of Task 2. Dispersant effectiveness of one oil-dispersant combination has been performed using the pre-weathered Norwegian crude oil Grane and Corexit 9500 as the dispersant (dispersant to oil ratio of 1 to 25). Three different energy conditions (low, medium, high) were established. Triplicate dispersant effectiveness tests have been conducted at each energy condition at each laboratory, at least nine tests. Similar experiments are reported in Brandvik et al. (2010), using three energy levels with increasing wave height (swells, non-breaking waves and breaking waves). Generation of wave energy in the tanks was done by use of an oscillation wave generator.

There will always be a challenge to establish three energy levels that give deviations in dispersant effectiveness for the given oil and dispersant to oil ratio. As mentioned above, CEDRE 's tank is larger and has a somewhat different shape, so it may be difficult to get similar energy conditions to those achieved in the tanks at SINTEF and SL Ross, which are of similar design. Therefore, all future experiments involving dispersant testing will be performed at SINTEF and SL Ross, while the mineral fines experiments will be conducted at CEDRE.

SINTEF and SL Ross are expected to be able to apply relatively similar settings in their flumes, due to the similarity of the flumes. Water current, wave height and wind speed parameters have been measured and are reported as part of the calibration.

CHAPTER 2. MATERIALS AND METHODS

2.1 Oil and dispersant

The Norwegian asphaltenic oil Grane was used in the inter-calibration. Two barrels of Grane crude were submitted to SINTEF from Statoil, and artificially weathered in the large scale topping tank at SINTEF to a weathering degree representing approximately 200 °C+. The criteria for termination of the evaporation is based on sampling of oil for measuring increase in density (correlated to the calibrated relationship between evaporation and increase in densities) and verified by GC-analysis. Usually a bench scale weathering is performed first to estimate this relationship, but as the water content in the new batch of oil was too high, no bench scale topping could be performed, and data from a previous batch of Grane topped to 200°C+ was used for comparison (Strøm et al., 2012). Oil properties of both batches are given in Table 2.1 and their GC chromatograms of the topped oil residues in Figure 2.1.

Table 2.1 Properties of Grane crude oil. Bench scale topped 200 °C+ from Strøm et al. (2012), and the large scale topped oil that has been used in the inter-calibration.

PROPERTY	200°C+ bench scale	200°C+ large scale topped
Volume topped (%)	2	-
Specific gravity (g/l)	0,948	0,954
Pour point (°C)	-9	-12
Viscosity at 13°C (cP)	1288	1602

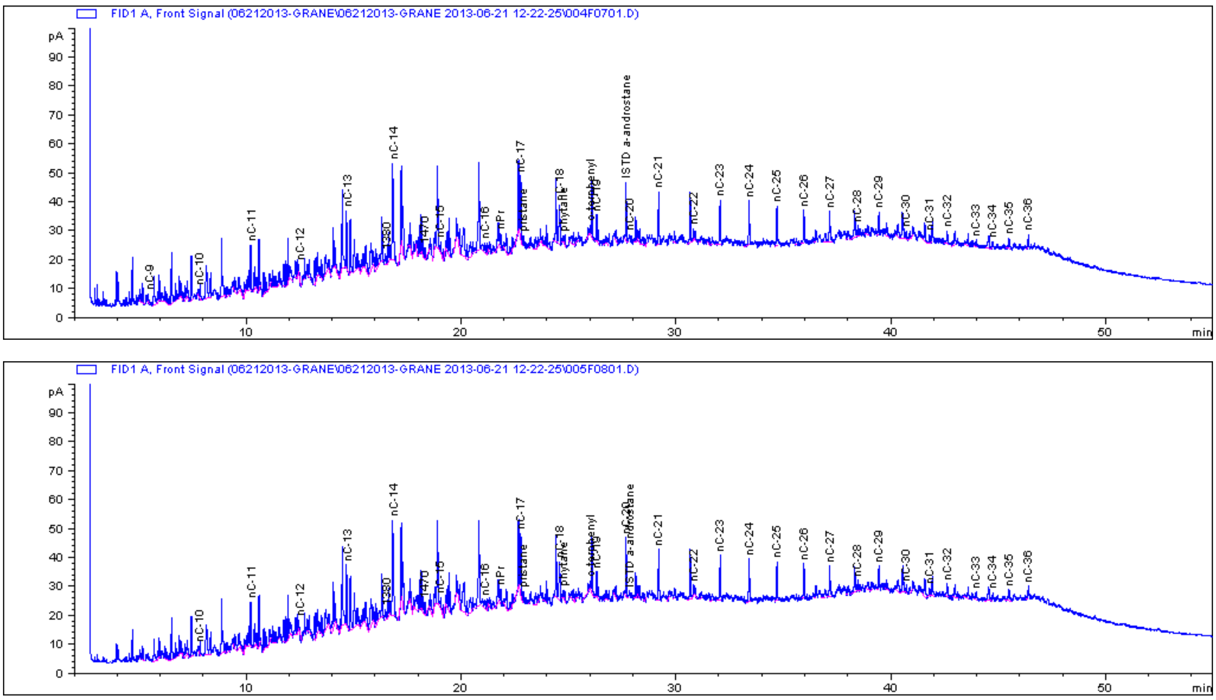


Figure 2.1 Laboratory weathered Grane from 2012 (upper chromatogram), and the large scale weathered Grane used in the inter calibration (bottom chromatogram)

The same batch of the chemical dispersant Corexit 9500 was used in all laboratories, and was supplied from common stock by SL Ross. However, the dispersant was from 2001, so its efficiency was compared to a newer stock at SINTEF (2013). The dispersant efficiency was tested for the batches using a standard crude oil and the IFP-test. Efficiency for the "old" Corexit 9500 was 48 ± 8% and for the "new" Corexit 9500 50 ± 5 %. It was concluded that the dispersant efficiency of the Corexit 9500 from 2001 is similar to the new batch.

2.2 SINTEF and SL Ross meso-scale flume

SINTEF and SL Ross have flumes of the same configuration and size. Key figures for the flumes, including Cedre's, are given in Table 2.2 and pictures of all flumes are shown on the front page. A sketch of the SINTEF/SL Ross flume is shown in Figure 2.2, which also includes the measuring locations.

Approximately 4.8 m^3 of seawater is circulated in the 10 meter long flumes. The SINTEF flume is located in a temperature controlled room (0°C – 20°C). The SL Ross tank sides and surface are insulated to maintain the water and air temperature during the testing. The water in both flumes is cooled by a refrigeration system connected to a cooling coil placed in the tank water. Two fans placed in a covered wind tunnel allow for control of the wind speed. The wind has been calibrated against data from field trials, real incidents, and bench scale testing to simulate an evaporation rate corresponding to a wind speed of 5-10 m/s at the sea surface.

Table 2.2 Key figures for the flumes

	SINTEF and SL Ross	Cedre
Flume (circulation) length inner wall	10,2 m	16,4 m
Flume (circulation) length outer wall	16,6 m	20,2 m
Flume height	1,5 m	1,4 m
Flume width	0,5 m	0,6 m
Seawater depth	1 m	0,9 m
Seawater volume	4,8 m^3	7,2 m^3
Seawater temperature	13 °C	13 °C
Dispersant applicator	Wagner 450	Wagner 450
Nozzle size applicator	0,8 mm	0,8 mm
Oil volume	1 L	1 L
Containment ring for oil and dispersant application	0,25 m^2	0,25 m^2
Oil film thickness (1 L oil) in containment ring	4 mm	4 mm
Dispersant to oil ratio (DOR)	1:25	1:25
Particle size analyzer	LISST*	Malvern
Position of particle size analyzer	Vertically, 37 cm depth, 25 cm from wall	40 cm depth, 30 cm from wall

**LISST: Laser In-Situ Scattering and Transmissiometry (Sequoia Scientific, Inc.)*

The wave generator consists of a triangular "plunger" and a wave breaking board. The "plunger" is driven by an electrical motor mounted on top of the "plunger", attached with an arm. The amplitude can be adjusted by the length of the arm and the frequency by a frequency converter.

The flume was filled with seawater the day before the experiments. SINTEF used natural seawater from the Trondheimsfjord (35 psu), while SL Ross used artificial seawater with a salinity of 32-35 psu. It was planned to perform the inter calibration at 0 °C. However, the inter-calibrations were conducted at 13 °C to minimize the time required to bring the water temperature down to 0 °C, which will be the standard test condition in Task 2. The seawater temperature in the North Sea at summer is approximately 13 °C.

After the experiment was finalized, remaining surface oil was skimmed off and the flume emptied. The seawater contains dispersed oil droplets and it should be treated according to regulations and permits. The flume was thoroughly cleaned using a high pressure washer and clean seawater filled in for the next experiment. In the SL Ross testes the water was re-used after filtering through both a sand and activated carbon filter to clean the water prior to the next test.

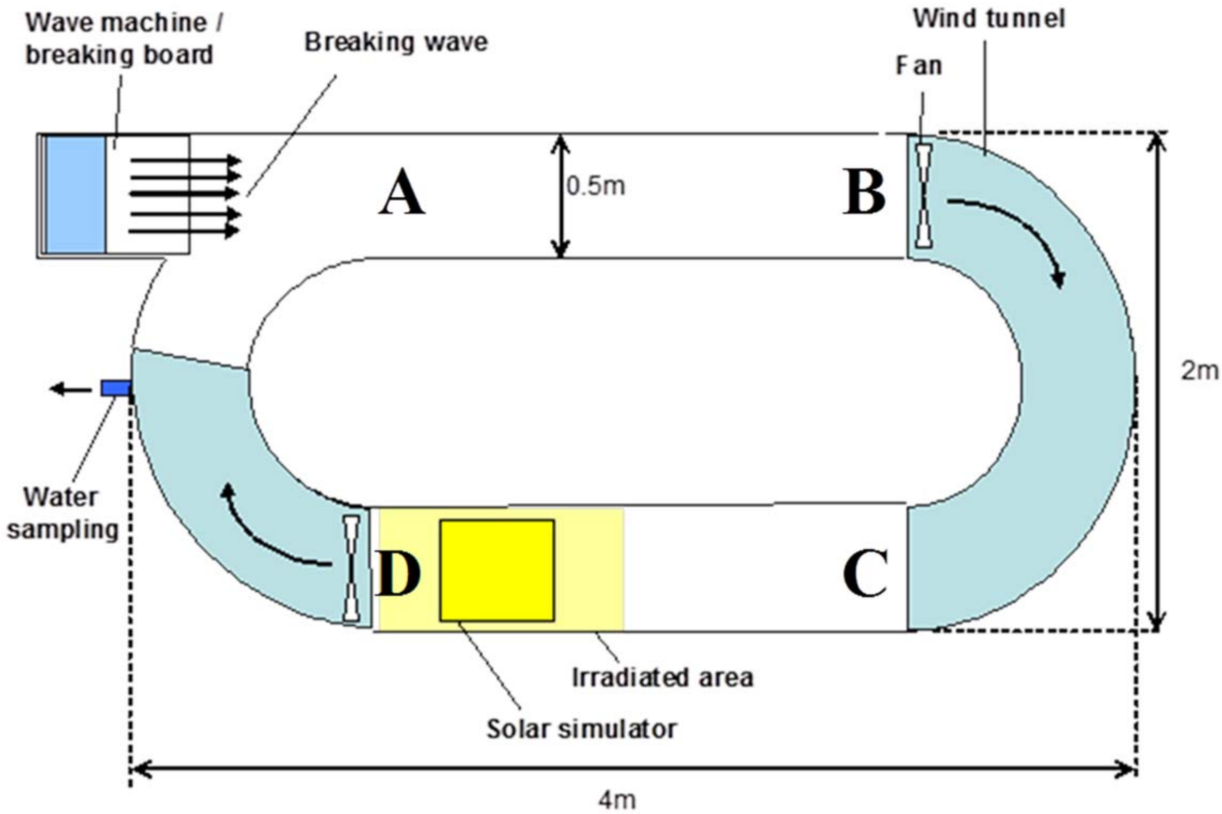


Figure 2.2 Meso scale flume tank at SINTEF and SL Ross with measuring points A, B, C and D.

2.2.1 Wave energy settings

SINTEF and SL Ross have used similar settings to simulate different energy levels in the flume (given in Table 2.3). Parameters like wind speed (Table 2.4), water currents (Table 2.5), and wave height are important in order to achieve comparable results. These parameters were measured at different pre-defined places in the flumes, as indicated in Figure 2.2.

Table 2.3 Wave energy settings used in the SINTEF and SL Ross flumes

Energy level	Conditions	Wave conditions	Amplitude Wave maker	Frequency Wave maker	Current propeller
High (setting 1)	Open water	Breaking	20 cm	49 rpm (34*)	None
Medium (setting 2)	Open water	Non-breaking	16 cm	29 rpm (20*)	None
Low (setting 3)	Open water	Swells	12 cm	24 rpm (17*)	None/Yes**

** Settings on SINTEF frequency converter. **SINTEF used a propeller in the low energy tests*

2.2.2 Wind speed

At SINTEF the wind speed was measured with a Kimo-Vane Probe Thermo-anemometer. The wind speed was measured as average over two minutes (1 Hz) in the centre of the flume a few centimetres above the water surface at each measuring position (A, B, C, D) This value was reported as wind speed (m/s). SL Ross measured wind speed using a hand held hot-wire anemometer. Wind speeds were measured in the centre of the tank a few cm above the water surface.

The wind speed measurements in the SL Ross and SINTEF flumes match quite well. SL Ross values at B and D are slightly lower likely due to safety shrouds around the fan blades blocking the air flow.

Table 2.4 Wind speed given in m/s. Measuring locations given in Figure 2.2.

	SINTEF				SL Ross			
Energy level	A	B	C	D	A	B	C	D
High	1,9	2,1	2,1	2,0	1,9	1,8	2,2	1,9
Medium	2,0	2,2	2,5	2,4	2,0	1,9	2,4	1,8
Low	2,0	2,3	2,3	2,2	2,1	1,8	2,5	1,9

2.2.3 Water currents

SINTEF measured water currents with a Vane Wheel sensor and a Flowtherm NT logging device (Höntzsch). Measurements were done at 3 different depths - 20, 30 and 50 cm below the surface. Measurements (1 Hz) were done for minimum 200 seconds at each depth. Measurements for each depth were then averaged and these 3 values were averaged again. This value was reported as water currents in m/s.

SL Ross measured water currents using small drogues placed in the water at three depths (20, 30 and 50 cm) and their movements were timed across AB and CD sections of the tank. The results were then averaged and the speeds provided in the table below.

The water velocities for high and medium energy are very similar, but SL Ross values at low energy setting are somewhat higher than the SINTEF data.

Table 2.5 Water current given in m/s. Measuring locations given in Figure 2.2.

	SINTEF				SL Ross	
Energy level	A	B	C	D	A-B	C-D
High	0,10	0,14	0,08	0,09	0,15	0,08
Medium	0,11	0,16	0,09	0,10	0,14	0,09
Low	0,01	0,03	0,01	0,02	0,05	0,04

2.2.4 Wave height

SL Ross measured wave heights using three wave probes mounted at locations B, C and D that measured wave heights and period over a 10 minute test duration. The average, maximum and H 1/3 wave heights and average wave periods are provided in Appendix A (Tables 1 and 2 for the first 2 minutes of each test and for the full 10 minute duration, respectively). Measurements were repeated at least three times for each energy level.

SINTEF has purchased the same wave probes as SL Ross, but due to delayed delivery and complications with the software, wave height was initially measured visually. An operator observed the wave through the window of the flume at measuring points B, C and D. The crest of the wave was determined by observing the largest wave through the flume window over 2-3 minutes (see Figure 2.3). The largest waves typically appeared at the same point for each individual window. The operator used a marker to mark the crest of single waves, then estimated the average height and used a marker to mark the average crest height on the flume window. The same procedure was then used to determine the through of the wave. A ruler was used to determine the distance between the marks and this value was reported as wave height (cm). There were difficulties in measuring the wave height, due to the changing nature of the wave and the subjective operator-dependent measuring method. After the inter-calibration tests were finalized, SINTEF performed wave height measurements with the wave gauge, but with only one measurement for each energy level (data not shown).

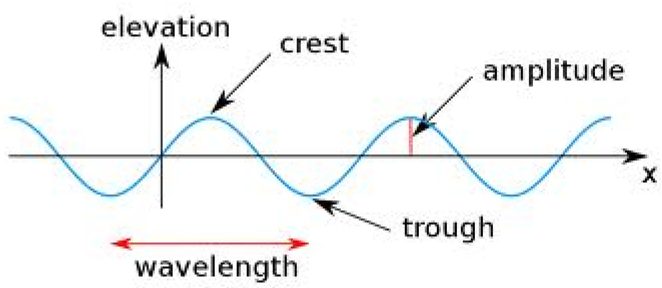


Figure 2.3 Wave description

A summary of the wave height measurements are given in Table 2.6. No measurements were performed at location A due to too much interference from the wave maker.

The SL Ross wave probe height data recorded consistently smaller wave heights with reduced wave energy levels. SINTEF's visual height measurements fall between the average and maximum wave height recorded by SL Ross with the exception of the medium energy test where the SINTEF visually recorded values are consistently higher than the maximum wave height values recorded by the wave probe in the SL Ross tests.

Table 2.6 Average wave height (H ave) and maximum wave height (H max), given in cm, with standard deviation from three measurements (SL Ross data is from measurements with wave probe over 10 minutes, SINTEF data from visual observations over 2-3 minutes).

Energy level	Location B		
	SL Ross		SINTEF
	H ave	H max	H ave
High	6,4 (0,7)	13 (1,5)	11 (0,6)
Medium	3,4 (0,1)	6,0 (1,1)	8,1 (0,2)
Low	1,2 (0,3)	2,6 (0,6)	2,0 (1,0)
Energy level	Location C		
	SL Ross		SINTEF
	H ave	H max	H ave
High	7,4 (0,4)	15 (2,3)	9,4 (0,5)
Medium	3,0 (0,2)	4,8 (0,3)	6,0 (0,3)
Low	0,8 (0,2)	1,1 (0,1)	2,0 (0,2)
Energy level	Location D		
	SL Ross		SINTEF
	H ave	H max	H ave
High	5,6 (0,4)	11 (0,5)	8,2 (0,4)
Medium	4,2 (0,3)	6,9 (0,4)	7,6 (0,1)
Low	1,2 (0,3)	2,6 (0,6)	1,9 (0,2)

2.3 Cedre meso scale flume

CEDRE 's tank is larger and has a somewhat different shape (Figure 2.4) than the tanks at SINTEF and SL Ross. Key figures for the flume are included in Table 2.2. In Cedre's flume the energy level is varied only by changing the wave generator speed.

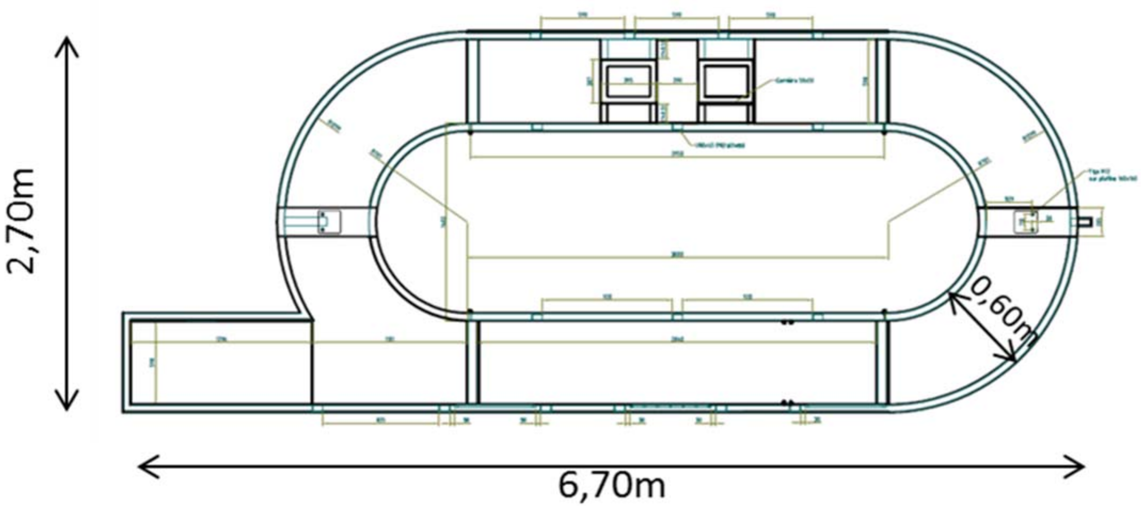


Figure 2.4 Meso scale flume tank at Cedre

The test conditions applied for the three energy levels are summarized in Table 2.7. More details describing the settings and experiments are given in Appendix C.

Table 2.7 Test conditions for inter-calibration testing at Cedre

	TEST CONDITIONS
LOW ENERGY AVERAGE ± STANDARD DEVIATION	*Temperature (oil & water) : 13,2 ± 1,0°C* *Oil Quantity : 956,4 ± 13,2g* *Wave amplitude : 3 to 4cm* *Wave generator speed : 14,6 ± 0,0 rpm*
MEDIUM ENERGY AVERAGE ± STANDARD DEVIATION	*Temperature (oil & water) : 13,2 ± 1,0°C* *Oil Quantity : 956,6 ± 10,6g* *Wave amplitude : 4 to 7cm* *Wave generator speed : 17,4 ± 0,1 rpm*
HIGH ENERGY AVERAGE ± STANDARD DEVIATION	*Temperature (oil & water) : 12,2 ± 0,3°C* *Oil Quantity : 955,5 ± 31,4g* *Wave amplitude : 5 to 10cm* *Wave generator speed: 19,6 ± 0,1 rpm*

2.4 Test methods

A 200°C+ residue of the asphaltenic Norwegian Grane crude oil and the dispersant Corexit 9500 has been used for this calibration.

The oil was placed in a containment ring on the wave maker side of position B then sprayed with dispersant using a Wagner 450 paint sprayer fitted with a 0.8 mm diameter nozzle (Figure 1, Appendix A). The treated oil was allowed to sit for 1 minute prior to lifting the containment ring. Once the ring was removed the wave maker and air fans were started. The wave and wind energy were continued for a 1 hour period with continuous LISST particle size and concentration measurements. The LISST uses the technique of laser diffraction to obtain particle size distribution of particles up to 500 μm, and was mounted vertically in the flume, as shown in Figure 2.5. Cedre used a Malvern for the same measurements (Appendix C).

Water grab samples were taken at 20, 40 and 60 minutes into the test. At SL Ross these samples were taken from a tube that was mounted on the side of the LISST with its opening positioned at the same location as the LISST's measurement cell. The tube was connected to a pump which was operated for a 10 second period to flush the line prior to each sample. SINTEF collected the water samples from the water sampling valve (50 cm depth) shown in Figure 2.2.

A summary of the major steps followed for each test is provided below:

1. Fill tank with 32 to 35 ppt salt water or natural seawater, adjust water temperature to 13°C, clean surface of tank.
2. Position LISST and water sampling tube in tank at position C with LISST measurement cell located 37 cm below water surface.
3. Start the LISST in real time mode and continuously collect concentration and particle size data throughout the test.
4. Place oil containment ring on wave maker side of position B.
5. Clean water surface on inside of ring with sorbent pad to facilitate oil spreading.
6. Weigh jug plus 1 litre of Grane 200+ and record.
7. Weight the Wagner 450 dispersant sprayer with dispersant and record.
8. Carefully place 1 Litre of Grane 200+ oil in ring by pouring onto an aluminium foil spill plate.
9. Apply 38 g (40ml) of Corexit 9500 dispersant from OGP supply using the Wagner sprayer (8 second spray at maximum setting with 0.8 mm nozzle).
10. Wait 1 minute.
11. Lift the ring and start the waves maker and fans at approximately 1.5 minutes after dispersant application. Record time of waves on.
12. Take water samples at 20 min, 40 min, and end of test (60 min.) for comparison to LISST concentration measurements.
13. Weight oil jug and Wagner sprayer to determine weight of oil and dispersant applied.

14. Determine concentration of oil in water samples by extracting oil with DCM solvent and colorimetric analysis of concentration using a response curve developed for the Grane 200+ crude oil.

Test method used in Cedre is different, especially:

1. Cedre used a Malvern particle size analyzer instead of a LISST

2. The dispersed oil concentration was recorded for the full duration of the test (approximately 1 hour), using a SFUV. At the end of the test, when the dispersed oil was homogeneously distributed in the water column in the whole flume a sample of the water passing through the SFUV was collected to calibrate the SFUV results. This dispersed oil concentration was kept as the concentration value for the test (as described in Annex C). In the same way, the droplet size distribution considered in the each test final result was the distribution obtained at the end of the test.

Figure 2.5 LISST located in position C in the flume

2.5 Measurements of dispersant effectiveness

To quantify the dispersant effectiveness, both SINTEF and SL Ross have good experience with the use of the combination of LISST and water sampling to measure oil droplet size and oil concentration in the water column. CEDRE has used a Malvern particle size analyser, SFUV (Turner design), as well as water sampling for the same measurements. The oil concentration in water samples were determined by liquid-liquid extraction with dichloromethane followed by colorimetric analysis of concentration using a response curve developed for the Grane 200+ crude oil.

CHAPTER 3. RESULTS AND DISCUSSIONS

A summary and comparison of the average results are presented here. More detailed description of the experiments and results are given for SL Ross in Appendix A, SINTEF in Appendix B, and Cedre in Appendix C.

All tests are conducted at least in triplicate for each energy condition. Water sampling and particle size distribution were measured after 20, 40 and 60 minutes for SINTEF and SL Ross. In

Table 3.1 the average of these three measurements with standard deviation are shown for all laboratories. At Cedre, the dispersed oil concentration kept as each test result, was the dispersed oil concentration when stable at the end of the test as observed with the SFUV, and measured.at the laboratory on a water sample taken from the water flowing from the SFUV.

Cedre used the SFUV measurements for monitoring the tests, and the SFUV readings where calibrated afterwards (with a water sample taken downstream from the water flowing through the SFUV at the end of the test when the reading was stable). This calibration carried out afterward showed the SFUV readings had to be multiplied by a factor x2 to obtain the actual dispersed oil concentration in the flume. Additionally, unlike LISST, the Malvern instrument used in Cedre, gives only a relative distribution of the droplet size, which does not allow calculating any dispersed oil concentration.

SINTEF observed a difference of approximately a factor of 2 between the in situ LISST concentration measurements in the flume, and the extracted water samples. SINTEF has moved the LISST around in the flume to check if there could be inhomogeneity between the location for water sampling and LISST measurements, or concentration shifts in the flume, but the differences within the flume was insignificant. In the comparison between the flumes, it is therefore suggested that the water sample concentrations from Cedre and SINTEF are used. The early SL Ross water sample concentration measurements were not consistent and a change in the oil extraction process was made late in test program to solve the problem. The LISST data from SL Ross match the water sample concentration quite well for the cases where the improved extraction process was implemented. Based on this, it was concluded that the LISST oil concentration data from the SL Ross tests is more reliable overall than the water sample concentration determinations.

The results given in

Table 3.1 suggest that the dispersant efficiency for Grane crude oil was high during medium and high energy conditions (more than 70 % in the SINTEF/SL Ross flumes). However, there was not a significant difference between dispersant efficiency in the medium and the high energy tests performed at SINTEF, so it is recommended that small adjustments are made for the medium energy prior to Task 2. It could be due to the wave energy frequencies applied for medium and high energy was too similar, or simply that the crude oil used has a higher dispersibility than assumed for the selected conditions. The difference between the high energy and low energy in the SINTEF/SL Ross flumes is insignificant, approximately 90 % and 40 %, respectively. Cedre has a larger flume, but used the same amount of oil and dispersant as SINTEF and SL Ross. They also experienced 90% dispersant efficiency in the high energy experiments, and approximately 55% and 35 % in the medium and low energy tests, respectively.

For a test basin to be acceptable, the standard deviation of the triplicates at each energy condition should be no more than ± 20% of the average for the test basins. Calculated average for all three flumes indicates that the SL Ross flume is the one closest to average, but the average for all flumes are used when evaluating the dispersant efficiency for the different energy levels.

The comparison of the results is given in Figure 3.1 and shows the difference in dispersant efficiency in the low and high energy tests performed in the flumes at SL Ross, SINTEF, and Cedre were small, with less than 7 % standard deviation. The dispersant efficiency in the medium energy test at SINTEF is in the same range as the high energy tests, while the SL Ross tests are lower, but still within the 20% deviation (Figure 3.1). However, the difference in dispersant efficiency between the high energy and medium energy tests in the SINTEF flume is small, so small adjustments in the wave frequency for medium energy will be performed prior to Task 2.

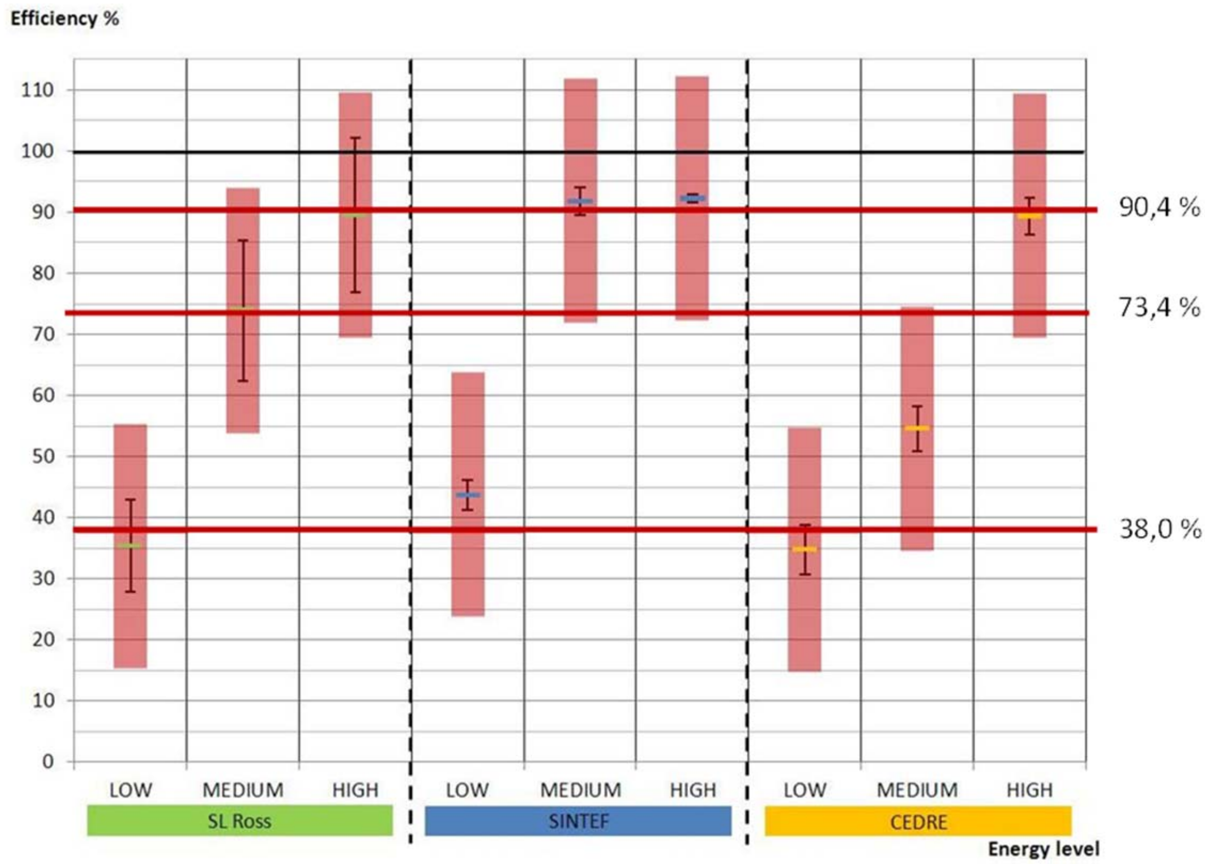


Figure 3.1 Comparison of dispersant efficiency at three energy levels. The average of three tests and the 20% deviation (pink bars) are shown. The red vertical lines indicate the average dispersant efficiency for all three laboratories. More details in

Table 3.1. Note that the flume at Cedre is larger than the flumes at SINTEF and SL Ross, which have the same shape and size.

Table 3.1 Dispersant efficiency testing, more details in Appendices A, B and C.

Test	by LISST / Malvern					by Water Sample Analysis			
	Drop	Ave.	Con	Efficien	Efficien	Ave.	Con	Efficien	Efficien
	d50	ppm	StDe	%	StDev	ppm	StDe	%	StDev
SL ROSS									
Low Energy 1	51	85,8	7,5	42,0		108	10,0	55,4	
Low Energy 3	56	75,1	10,3	37,0		147	12,0	75,2	
Low Energy 4	62	57,2	11,2	27,2		72	10,0	35,9	
Average low energy SL	56	72,7	9,7	35,4	7,5	109		45,7	19,7
Mid Energy 1	20	120	6,5	61,7		122	9,0*	65,6	
Mid Energy 2	11	171	3,3	84,6		115	4,0*	59,5	
Mid Energy 4	11	155	3,0	75,3		172	16,0	87,4	
Average mid energy SL	14	149	4,3	73,9	11,5	136		76,5	14,7
High Energy 1	12	165	15,5	83,3		159	13,0	84,1	
High Energy 2	11	201	19,6	104		96	13,0	52,1	
High Energy 3	13	164	17,1	81,0		172	5,0*	89,5	
Average high energy SL	12	177	17,4	89,5	12,5	142		86,8	20,2
SINTEF									
Low Energy 1	104	33,7	7,0	18,0		78,6	14,2	41,9	
Low Energy 2	104	45,1	7,8	23,5		82,4	2,3*	42,9	
Low Energy 3	104	37,9	9,0	19,9		88,8	18,6	46,6	
Average low energy	104	38,9	7,9	20,4	2,8	83,3		43,8	2,5
Mid Energy 2	14	95,2	5,6	48,1		180	2,8*	91,2	
Mid Energy 4	17	81,9	3,5	42,4		183	5,6*	94,3	
Mid Energy 5	12	87,8	2,4	47,3		167	2,5*	90,0	
Average mid energy	14	88,3	3,8	45,9	3,1	177		91,8	2,3
High Energy 1	20	129	4,1	66,6		178	1,9*	91,7	
High Energy 3	24	86,4	5,3	47,7		167	0,2*	92,1	
High Energy 4	24	94,6	4,4	47,1		187	3,5*	93,0	
Average high energy	22	103	4,6	53,8	11,1	177		92,3	0,7
Cedre									
Low Energy 1	38	19,3				40,6		31,0	
Low Energy 2	44	21,7				45,6		34,3	
Low Energy 3	40	25,0				52,5		39,0	
Average low energy	40,7	22,0				46,2	6,0**	34,8	4,0
Mid Energy 1	44	35,2				73,9		56,2	
Mid Energy 2	27	32,2				67,7		50,4	
Mid Energy 3	27	36,2				76		57,2	
Average mid energy	32,7	34,5				72,5	4,3**	54,6	3,7
High Energy 1	20	53,2				112		87,5	
High Energy 2	24	60,3				127		93,0	
High Energy 3	15	56,2				118		87,0	
Average high energy	19,7	56,6				119	7,9**	89,4	3,0
All flumes***									
Low energy								38	6,2
Mid energy								73	17
High energy								90	6,7

**Standard deviation of SL Ross and SINTEF calculated for each tests based on the samples collected after 20, 40, and 60 minutes.*

*** Cedre calculated the standard deviation based on the 3 tests of each energy level, using only the concentration obtained at the end of the test*

****Efficiency from LISST for SL Ross, and water samples for SINTEF and Cedre*

CHAPTER 4. CONCLUSIONS

CEDRE, SINTEF and SL Ross have the same type of meso scale flume basin, of which the flumes at SINTEF and SL Ross have exactly the same dimensions, while CEDRE's flume is larger. The SINTEF flume was used in the meso scale studies on dispersant testing in the SINTEF led Oil-in-ice JIP and is defined as the standard test basin. The objective of the inter-basin calibration study was to determine consistent test protocols and natural energy conditions as a start of Task 2. Dispersant effectiveness of one oil-dispersant combination has been performed using the pre-weathered Norwegian crude oil Grane and Corexit 9500 as the dispersant (dispersant to oil ratio of 1 to 25). Three different energy conditions (low, medium, high) were established. Triplicate dispersant effectiveness tests have been conducted at each energy condition at each laboratory, at least nine tests.

For a test basin to be acceptable, the standard deviation of the triplicates at each energy condition should be no more than ± 20% of the average for the test basins. The dispersant efficiency in all energy levels performed in the flumes at SINTEF, SL Ross, and Cedre were within the ±20% of the average dispersant efficiency.

The results have shown that there was a very good correlation between the dispersant efficiency in the flumes at SINTEF and SL Ross at all three energy levels. Although the Cedre flume is different than the flumes at SINTEF/SL Ross, the correlation in dispersant efficiency was good, especially a low and high energy conditions. It is recommended that all three flumes are accepted for further testing of dispersant/OMA efficiency in Task 2. However, the difference in dispersant efficiency between the high energy and medium energy tests in the SINTEF flume is small, so small adjustments in the wave frequency for medium energy will be performed prior to Task 2.

CHAPTER 5. REFERENCES

Brandvik, P.J., J.L.M. Resby, P.S. Daling, F. Leirvik, and J. Fritt-Rasmussen (2010). Meso scale weathering of oil as a function of ice conditions. Oil properties, dispersibility and in situ burnability of weathered oil as a function of time. SINTEF oil in ice JIP report no. 19. SINTEF A15563, ISBN 978-82-14-04772-1. *www.sintef.no/Projectweb/JIP-Oil-In-Ice/Publications/*.

Strøm, T. (2013). Re-check weathering properties for Oseberg A, Oseberg C and Grane crude oil. SINTEF report A24284, ,ISBN 978-82-14-05538-2.

APPENDIX A. SL ROSS WAVE TANK INTER-CALIBRATION MEASUREMENT

1 Basic Tank Response Measurements

Wind Speeds

Measured at locations A, B, C and D using a hand held hot-wire anemometer. Wind speeds were measured in the centre of the tank a few cm above the water surface. The values match the Sintef results quite well. Our values at B and D are slightly lower likely due to safety shrouds around the fan blades blocking the air flow.

Wind Speeds (m/s)				
	A	B	C	D
setting 1	1.9	1.8	2.2	1.9
setting 2	2.0	1.9	2.4	1.8
setting 3	2.1	1.8	2.5	1.9

Water Currents

Water currents were measured using small drogues placed in the water at three depths (20, 30 and 50 cm) and their movements were timed across AB and CD sections of the tank. The results were then averaged and the speeds provided in the table below. Our vane wheel sensor is not capable of measuring the low flow velocities occurring in these tests so the simple float timing method was used. The water velocities for settings 1 and 2 are very similar to those reported by Sintef but our setting 3 values are somewhat higher than the Sintef data.

Water Currents		
	A-B	C-D
setting 1	0.15	0.08
setting 2	0.14	0.09
setting 3	0.05	0.04

Wave Heights

Wave heights were measured using three wave probes mounted at locations B, C and D that measured wave heights and period over a 10 minute test duration. The average, maximum and H 1/3 wave heights and average wave periods are provided in Tables 1 and 2 for the first 2 minutes of each test and for the full 10 minute duration, respectively. Measurements were repeated at least three times for each energy level.

Table 1 Wave Height Data for First 120 seconds of Wave Maker Action

Wave Heights (first 2 minutes of wave action)												
	Location B (WP1)				Location C (WP2)				Location D (WP3)			
	H ave (m)	H max (m)	H 1/3 (m)	T ave (s)	H ave (m)	H max (m)	H 1/3 (m)	T ave (s)	H ave (m)	H max (m)	H 1/3 (m)	T ave (s)
setting 1	0.080	0.115	0.096	1.327	0.079	0.119	0.099	1.433	0.070	0.115	0.091	1.460
setting 1	0.081	0.137	0.108	1.355	0.075	0.158	0.113	1.267	0.054	0.112	0.080	1.348
setting 1	0.078	0.144	0.106	1.332	0.082	0.160	0.118	1.281	0.066	0.106	0.084	1.353
setting 2	0.022	0.039	0.034	1.391	0.019	0.040	0.026	1.737	0.048	0.070	0.060	2.032
setting 2	0.030	0.049	0.043	1.521	0.026	0.050	0.038	1.770	0.04	0.066	0.057	1.775
setting 2	0.033	0.071	0.052	1.252	0.029	0.044	0.038	2.030	0.040	0.073	0.061	1.439
setting 2	0.028	0.059	0.042	1.425	0.025	0.046	0.033	1.885	0.042	0.068	0.062	1.732
setting 3	0.014	0.032	0.020	2.088	0.008	0.010	0.010	2.245	0.010	0.016	0.013	2.283
setting 3	0.009	0.024	0.014	2.124	0.007	0.011	0.009	2.408	0.007	0.012	0.010	2.286
setting 3	0.012	0.021	0.017	2.075	0.006	0.011	0.009	2.196	0.009	0.015	0.012	1.962

Table 2 Wave Height Data for Full 10 Minutes of Wave Maker Action

Wave Heights (over full 10 minute record)												
	Location B (WP1)				Location C (WP2)				Location D (WP3)			
	H ave (m)	H max (m)	H 1/3 (m)	T ave (s)	H ave (m)	H max (m)	H 1/3 (m)	T ave (s)	H ave (m)	H max (m)	H 1/3 (m)	T ave (s)
setting 1	0.071	0.115	0.083	1.353	0.070	0.119	0.083	1.333	0.059	0.115	0.069	1.375
setting 1	0.058	0.137	0.078	1.335	0.076	0.158	0.092	1.336	0.051	0.112	0.064	1.322
setting 1	0.064	0.144	0.081	1.328	0.077	0.160	0.094	1.316	0.058	0.106	0.069	1.377
setting 2	0.025	0.047	0.033	1.751	0.021	0.040	0.028	1.957	0.042	0.070	0.054	1.831
setting 2	0.035	0.049	0.043	1.851	0.032	0.051	0.042	1.789	0.045	0.066	0.053	1.921
setting 2	0.034	0.071	0.043	1.763	0.030	0.046	0.038	1.977	0.040	0.073	0.052	1.635
setting 2	0.034	0.059	0.042	1.857	0.029	0.048	0.038	1.873	0.041	0.068	0.052	1.762
setting 3	0.015	0.032	0.018	2.403	0.010	0.010	0.010	2.483	0.008	0.016	0.011	2.316
setting 3	0.010	0.024	0.014	2.315	0.007	0.011	0.009	2.468	0.009	0.013	0.010	2.420
setting 3	0.012	0.021	0.015	2.266	0.007	0.011	0.008	2.334	0.009	0.015	0.011	2.275

2 Dispersant Effectiveness Test Results: SL Ross

Test Conditions

All tests were completed with Grane 200+ crude oil that was supplied by Sintef and Corexit 9500 dispersant from a common stock supplied by SL Ross. The target Oil to Dispersant ratio was 25. Three wave energy levels were tested as specified by Sintef based on their previous experience with the flume tank (Highh: 44 rpm with 20 cm amplitude, Medium: 29 rpm and 16 cm and Low: 24 rpm and 12cm). All tests were conducted in 35 ppt salt water at 13 °C.

Test Methods

The oil was placed in a containment ring on the wave maker side of position B then sprayed with dispersant using a Wagner paint sprayer fitted with a 0.8 mm diameter nozzle (Figure 1). The treated oil was allowed to sit for 1 minute prior to lifting the containment ring. Once the ring was removed the wave maker and air fans were started. The wave and wind energy were continued for a 1 hour period with continuous LISST particle size and concentration measurements. Water grab samples were taken at 20, 40 and 60 minutes into the test. These samples were taken from a tube that was mounted on the side of the Lisst with its opening positioned at the same location as the Lisst's measurement cell. The tube was connected to a pump which was operated for a 10 second period to flush the line prior to each sample.

A summary of the major steps followed for each test is provided below.

1. Fill tank with 32 to 35 ppt salt water, adjust water temperature to 13 C, clean surface of tank.
2. Position LISST and water sampling tube in tank at position C with LISST measurement cell located 37 cm below water surface.
3. Start the LISST in real time mode and continuously collect concentration and particle size data throughout the test. Start Lisst "rocking motor" to provide a gentle movement of the Lisst to eliminate fouling of the horizontal glass surfaces (see Figure 2).
4. Place oil containment ring on wave maker side of position B.
5. Clean water surface on inside of ring with sorbent pad to facilitate oil spreading.
6. Weigh jug plus 1 litre of Grane 200+ and record.
7. Weight the Wagner dispersant sprayer with dispersant and record.
8. Carefully place 1 Litre of Grane 200+ oil in ring by pouring onto an aluminium foil spill plate.
9. Apply 38 g (40ml) of Corexit 9500 dispersant from OGP supply using the Wagner sprayer (8 second spray at maximum setting with 0.8 mm nozzle).
10. Wait 1 minute.
11. Lift the ring and start the waves maker and fans at approximately 1.5 minutes after dispersant application. Record time of waves on.
12. Take water samples at 20 min., 40 min. and end of test (60 min.) for comparison to LISST concentration measurements.
13. Weight oil jug and Wagner sprayer to determine weight of oil and dispersant applied.
14. Determine concentration of oil in water samples by extracting oil with DCM solvent and colorimetric analysis of concentration using a response curve developed for the Grane 200+ crude oil.

Figure 1. Oil in containment ring prior to and after dispersant application

Figure 2. Motor, offset wheel and pull cord used to rock the Lisst particle size analyzer

3 Test Results

Oil Concentrations and Dispersant Efficiency Estimates

Oil concentrations and drop size distributions were determined from the Lisst data at 20, 40 and 60 minutes into each test. In water oil concentrations were also determined by solvent extraction and analysis using a NovaSpec III spectrophotometer and 370 nm wavelength. A summary of the oil concentration results and estimated dispersant efficiencies are shown in Tables 3, 4 and 5 for the low, medium and high energy level tests, respectively.

Comments on Oil Concentration and Efficiency Estimates

During the early stages of the testing it was apparent that the LISST particle size analyser's horizontal glass surface was fouling with oil, especially during the low energy tests where the water movement in the test tank was minimal. The fouling was not pronounced or noticeable in the medium and high energy tests that were conducted earlier in the test program. To alleviate this problem the Lisst was slowly rocked using a pull-cord drawn through a small hole in the tank's cover and attached to the Lisst. The cord was operated manually for the low energy tests number 2 and 3 and using the motorized off-center wheel shown in Figure 2 for the low energy test #4. The Lisst concentration results for all but the fourth low energy test have inconsistent results and it is felt that the fourth test's numbers are likely the most representative of the low energy condition.

The results of the water grab sample analyses for the early tests were also not consistent. The oil extraction process was improved for the fourth low and medium energy test s and for the third high energy test. The in-water oil concentrations measured using the improved extraction procedure resulted in much better consistency in the results and much better correspondence with the Lisst concentration data.

Based on what we feel are the best data collected during the latter tests when the Lisst was rocked and the water extraction process was improved, the dispersant efficiencies for the low energy tests were in the 30 to 40% range, 80 to 85% for the medium energy tests, and 85 to 90% for the high energy tests. There was not a significant difference between the medium and high energy tests. These results are consistent with the data collected by Sintef for their water sample analyses shown at the bottom of Tables 1 through 3.

Table 3. Oil Concentrations and Dispersant Efficiency Estimates: Low Energy Tests

				by LISST			by Water Sample Analysis		
	Oil Wt	Disp. Wt	ODR	Ave. Conc.	Conc.	%	Ave. Conc.	Conc.	%
Test	g	g	by wt	ppm	StDev	Efficiency	ppm	StDev	Efficiency
Low Energy 1 @20 min	935	32	29.2	102.8	28	50	135	7	69
Low Energy 1 @40 min	935	32	29.2	172.6	16	85	116	7	60
Low Energy 1 @60 min	935	32	29.2	85.8	7.5	42	108	10	55
Low Energy 2 @20 min	937.8	31.9	29.4	133.0	19	65	172	9	88
Low Energy 2 @40 min	937.8	31.9	29.4	192.3	13.8	94	173	39	89
Low Energy 2 @60 min	937.8	31.9	29.4	108.8	13.6	53	147	12	75
Low Energy 3 @20 min	930.2	33.5	27.8	90.4	14.2	45	86	8	44
Low Energy 3 @40 min	930.2	33.5	27.8	178.6	17.6	88	94	7	49
Low Energy 3 @60 min	930.2	33.5	27.8	75.1	10.3	37	134	2	69
Low Energy 4 @20 min	962.4	33.2	29.0	77.9	13.5	37	82	6	41
Low Energy 4 @40 min	962.4	33.2	29.0	62.8	10.6	30	70	2	35
Low Energy 4 @60 min	962.4	33.2	29.0	57.2	11.2	27	72	10	36
Low Energy Sintef 2 @20 min	921.6	36	25.6	41.2	9.9	20	78.1		41
Low Energy Sintef 2 @40 min	921.6	36	25.6	50.5	10.7	25	82.2		43
Low Energy Sintef 2 @60 min	921.6	36	25.6	47.6	12.7	24	82.1		43

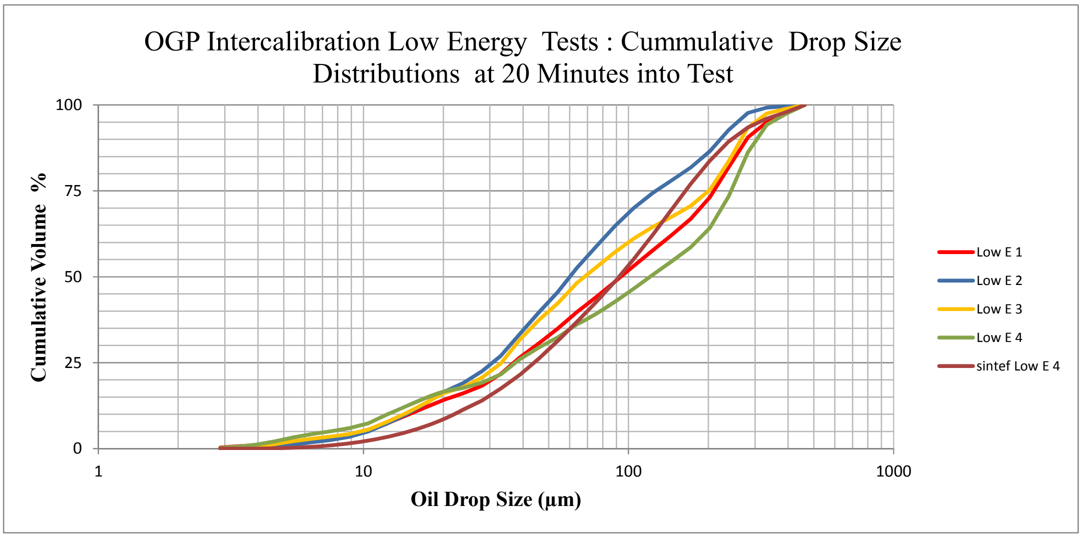
Table 4. Oil Concentrations and Dispersant Efficiency Estimates: Medium Energy Tests

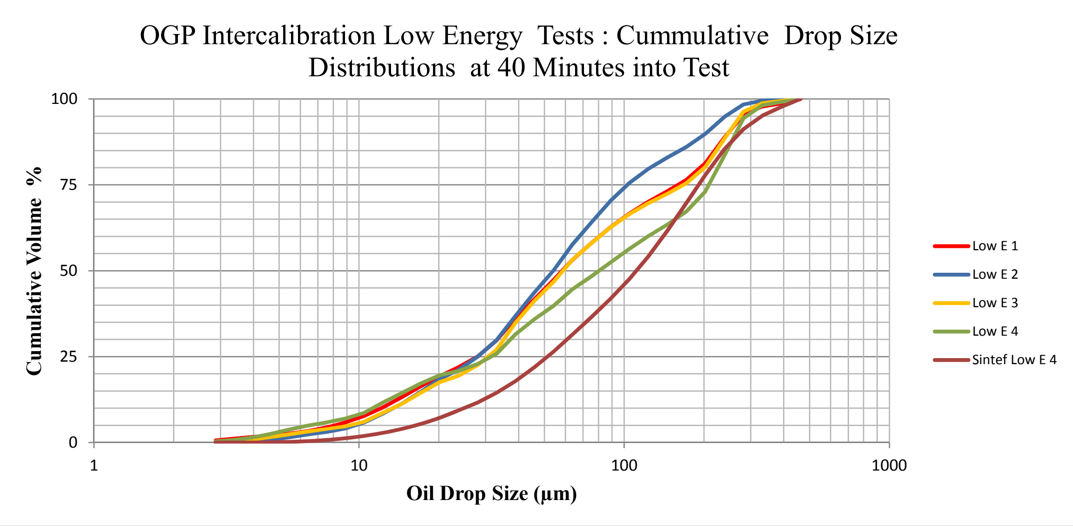
				by LISST			by Water Sample Analysis		
	Oil Wt	Disp. Wt	ODR	Ave. Conc.	Conc.	%	Ave. Conc.	Conc.	%
Test	g	g	by wt	ppm	StDev	Efficiency	ppm	StDev	Efficiency
Mid Energy 1 @20 min	893.1	35.1	25.4	123	10.9	63	133	12	71
Mid Energy 1 @40 min	893.1	35.1	25.4	128	9.4	66	124	19	67
Mid Energy 1 @60 min	893.1	35.1	25.4	120.4	6.5	62	122	9	66
Mid Energy 2 @20 min	927.2	35.8	25.9	171.6	4	85	165	7	85
Mid Energy 2 @40 min	927.2	35.8	25.9	170.6	2.8	84	125	14	65
Mid Energy 2 @60 min	927.2	35.8	25.9	171.3	3.3	85	115	4	60
Mid Energy 3 @20 min	897.4	31	28.9	185.9	12.1	95	125	26	67
Mid Energy 3 @40 min	897.4	31	28.9	195.1	13.3	100	89	23	48
Mid Energy 3 @60 min	897.4	31	28.9	188.1	12.3	96	77	6	41
Mid Energy 4 @20 min	944.3	32.9	28.7	179.9	8.9	87	172	5	87
Mid Energy 4 @40 min	944.3	32.9	28.7	161.5	4.2	78	165	9	84
Mid Energy 4 @60 min	944.3	32.9	28.7	155.4	3.0	75	172	16	87
Mid Energy Sintef 4 @20 min	928.6	38.8	23.9	104.1	5.1	51	185		96
Mid Energy Sintef 4 @40 min	928.6	38.8	23.9	105.3	4.0	52	179		92
Mid Energy Sintef 4 @60 min	928.6	38.8	23.9	106.8	4.9	53	174		90

Table 5. Oil Concentrations and Dispersant Efficiency Estimates: High Energy Tests

Test	Oil Wt g	Disp. Wt g	DOR by wt	by LISST Ave. Conc. ppm	by LISST Conc. StDev	by LISST % Efficiency	by Water Sample Analysis Ave. Conc. ppm	by Water Sample Analysis Conc. StDev	by Water Sample Analysis % Efficiency
High Energy 1 @20 min	907.9	36.7	24.7	170.9	16.9	86	159	6	84
High Energy 1 @40 min	907.9	36.7	24.7	171.1	22.8	86	165	27	87
High Energy 1 @60 min	907.9	36.7	24.7	165.1	15.5	83	159	13	84
High Energy 2 @20 min	885	36.9	24.0	194.1	16	100	116	16	63
High Energy 2 @40 min	885	36.9	24.0	212.2	16.1	110	116	9	63
High Energy 2 @60 min	885	36.9	24.0	200.9	19.6	104	96	13	52
High Energy 3 @20 min	922.9	32.5	28.4	174.6	19.7	87	173	2	90
High Energy 3 @40 min	922.9	32.5	28.4	171.1	21.5	85	172	8	89
High Energy 3 @60 min	922.9	32.5	28.4	163.9	17.1	81	172	5	89
High Energy Sintef 4 @20 min	964.2	38.6	25.0	102.1	6.5	48	186		92
High Energy Sintef 4 @40 min	964.2	38.6	25.0	102.7	6.7	49	186		92
High Energy Sintef 4 @60 min	964.2	38.6	25.0	102.2	5.4	49	180		89

Figures 3, 4 and 5 provide the oil drop size distributions measure at SL Ross for the low, medium and high energy dispersant effectiveness tests. Also included on these plots are drop size distributions for one of the Sintef tests at each mixing energy. The oil drop sizes in the low energy tests were significantly larger than both the medium and high energy tests. The volume median oil drop diameters in the low energy tests were in the 60 to 100 whereas the VMD for the medium and high energy tests were in the 10 to 20 μm range. The Sintef drop distribution for the medium energy test was very similar to the SL Ross data. The Sintef drop diameters were somewhat larger than the SLRoss data in the low and high energy tests.





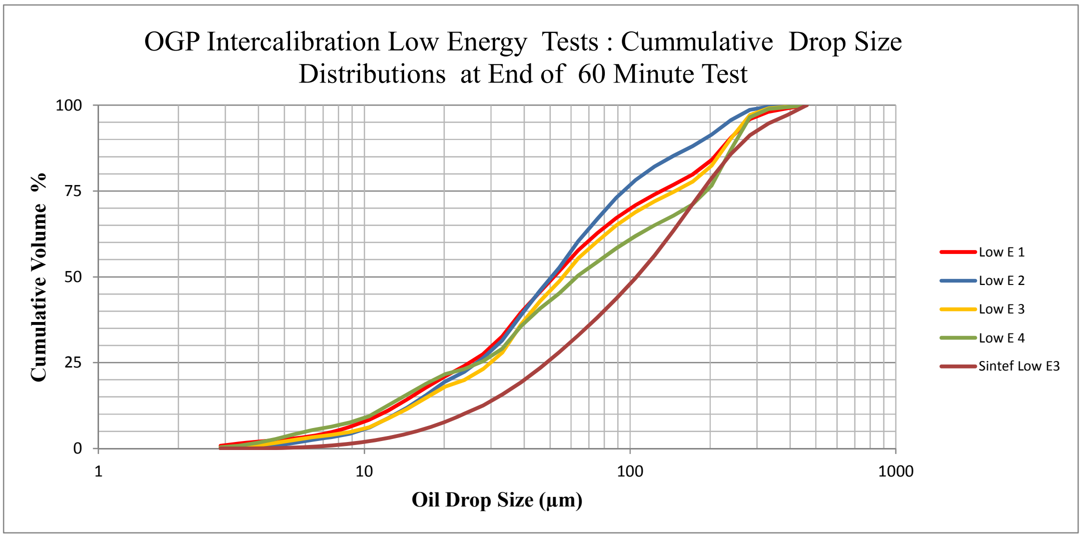
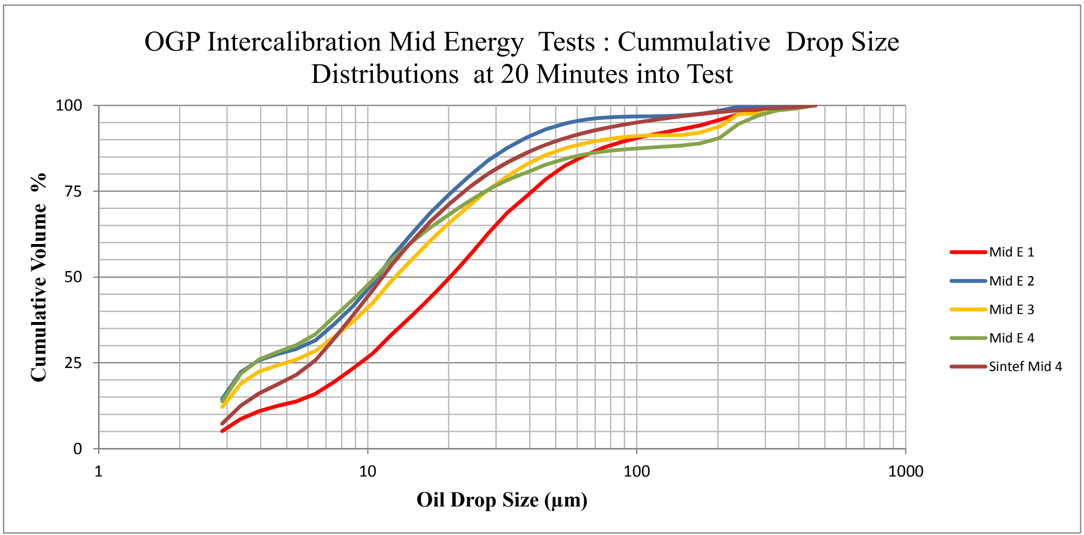
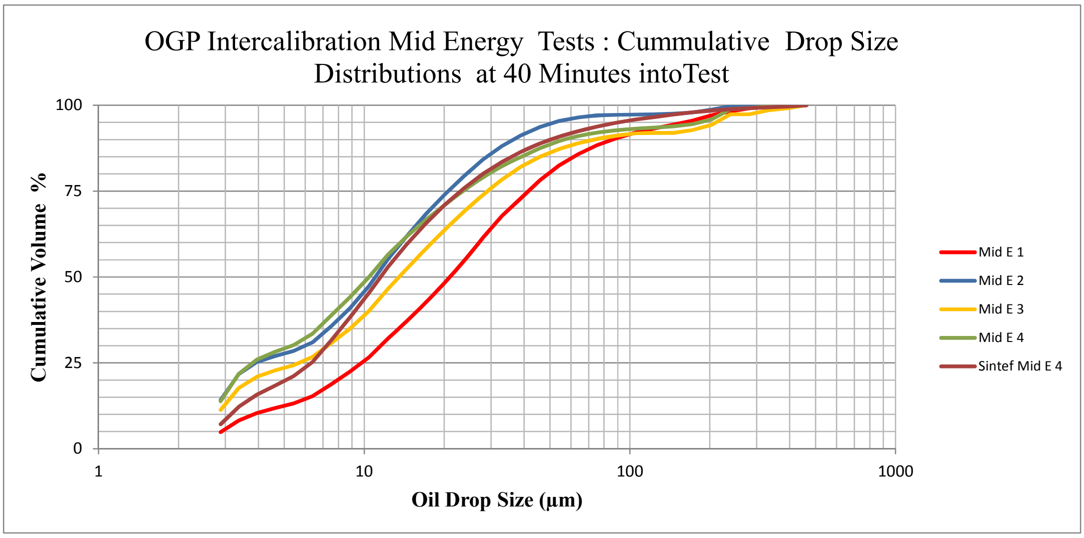


Figure 3 Low Energy Oil Drop Size Distribution Graphs





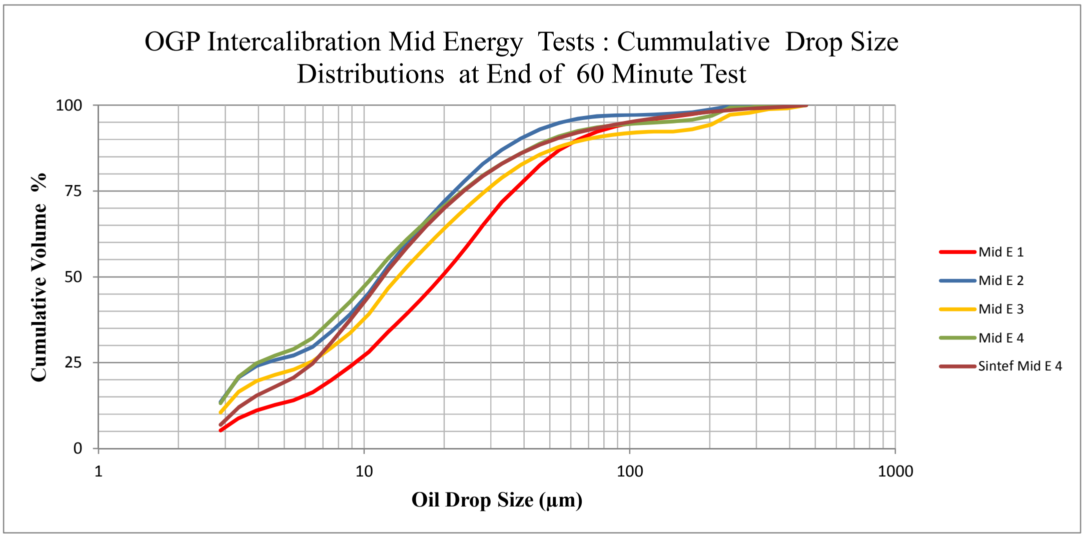
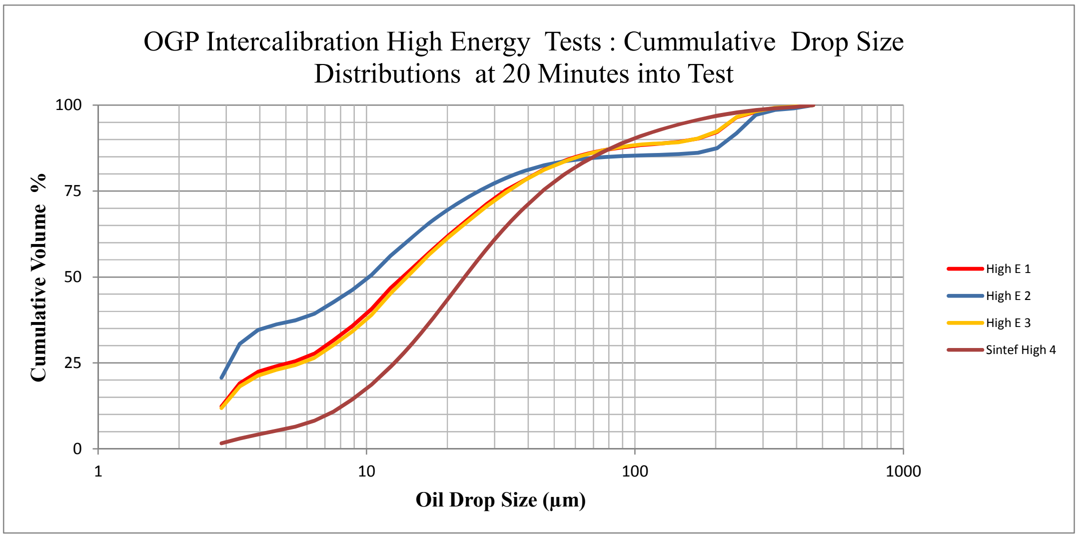
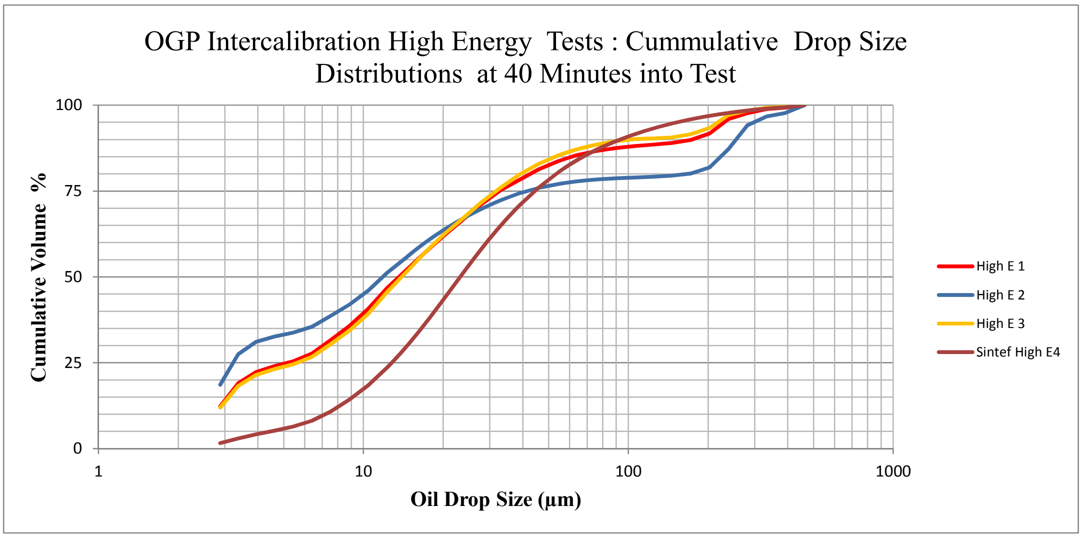


Figure 4 Medium Energy Oil Drop Size Distribution Graphs





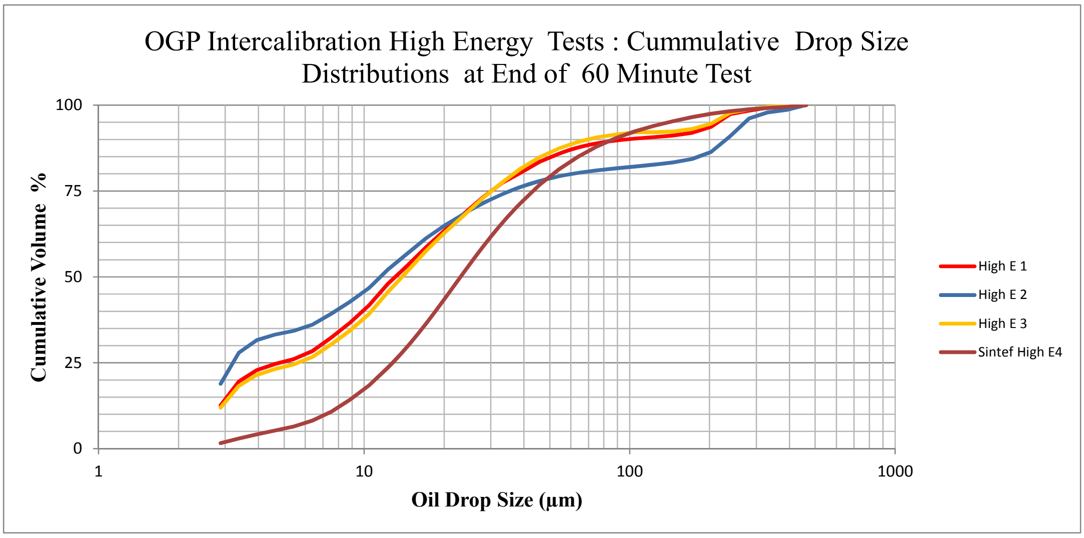


Figure 5 High Energy Oil Drop Size Distribution Graphs

APPENDIX B. SINTEF INTER-CALIBRATION RESULTS

Inter-calibration of flume – SINTEF

Thor-Arne Pettersen, Marius Johnsen, and Liv-Guri Faksness

1 Introduction

CEDRE, SINTEF and SL Ross have the same type of meso scale flume basin, of which the flumes at SINTEF and SL Ross have exactly the same dimensions, while CEDRE's flume is larger. The SINTEF flume was used in the meso scale studies on dispersant testing in the SINTEF led Oil-in-ice JIP (Brandvik et al., 2010), and is defined as the standard test basin. As a first phase of task 2 of the project "Dispersant testing under realistic conditions", OGP has requested that a calibration between the three flumes to be conducted. The objective of the inter-basin calibration study was to determine consistent test protocols and natural energy conditions as a start of Task 2.

Dispersant effectiveness of one oil-dispersant combination has been performed using the pre-weathered Norwegian crude oil Grane and Corexit 9500 as the dispersant (dispersant to oil ratio of 1 to 25). Three different energy conditions (low, medium, high) were established. Triplicate dispersant effectiveness tests have been conducted at each energy condition at each laboratory, at least nine tests. Similar experiments are reported in Brandvik et al. (2010), using three energy levels with increasing wave height (swells, non-breaking waves and breaking waves). Generation of wave energy in the tanks was done by use of an oscillation wave generator.

2 Basic settings and measurements

General

The flume was filled with seawater the day before the experiments. SINTEF used natural seawater from the Trondheimsfjord (35 psu). It was planned to perform the inter-calibration at 0 °C, so all initial measurements and testing of settings were done at 0-3°C. The inter-calibrations were conducted at 13 °C to minimize the time required to bring the water temperature down to 0 °C, which will be the standard test condition in Task 2. After the experiment was finalized, remaining surface oil was skimmed off and the flume emptied. The seawater contained dispersed oil droplets and it was treated according to regulations and permits. The flume was thoroughly cleaned using a high pressure washer and clean seawater filled in for the next experiment. The settings used in the inter-calibration are given in Table 1, and the SINTEF flume with sampling locations is shown in Figure 1.

Table 1: Settings

Setting no.	Conditions	Wave conditions	Amplitude Wave maker	Frequency Wave maker	Current propeller
1 (high energy)	Open water	Breaking	20 cm	49 rpm (34*)	None
2 (medium energy)	Open water	Non-breaking	16 cm	29 rpm (20*)	None
3 (low energy)	Open water	Swells	12 cm	24 rpm (17*)	Yes

** Settings on SINTEF frequency converter.*

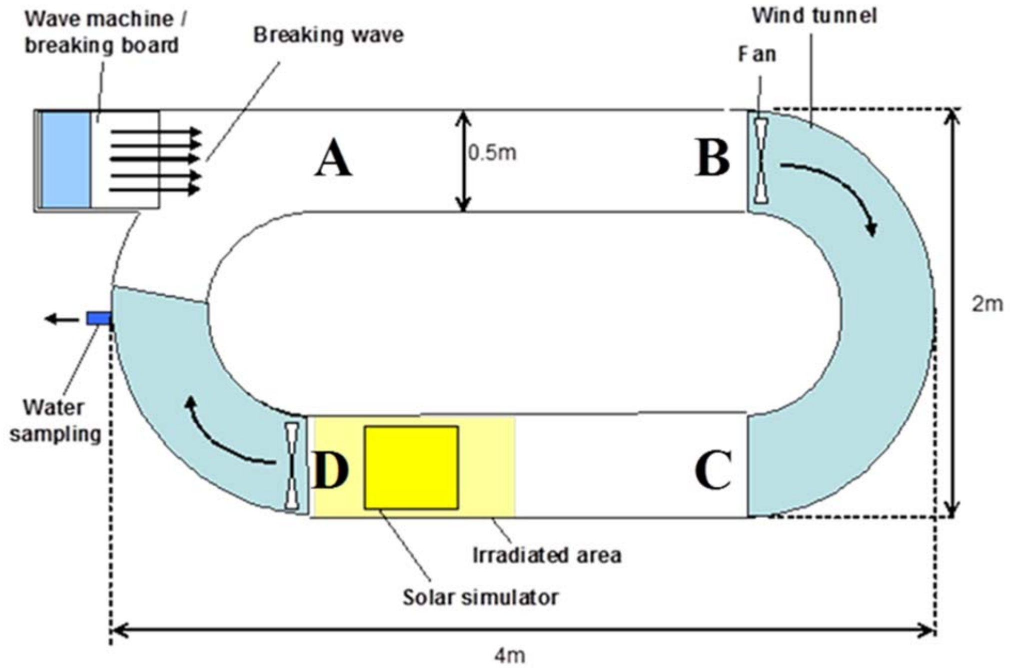


Figure 1: Flume schematic with measuring points A,B,C and D

Wave height

Wave height was measured visually. An operator observed the wave through the window of the flume at measuring points B, C and D. The crest (Figure 2) of the wave was determined by observing the largest wave through the flume window over 2-3 minutes. The largest waves typically appeared at the same point for each individual window. The operator used a marker to mark the crest of single waves, then estimated the average height and used a marker to mark the average crest height on the flume window. The same procedure was then used to determine the through of the wave. A ruler was used to determine the distance between the marks and this value was reported as wave height (cm). The visual measurements are given in Table 2.

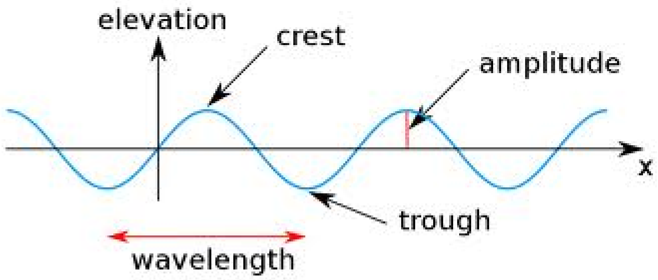


Figure 2: Wave

There were difficulties in measuring the wave height, due to the changing nature of the wave and the subjective operator-dependent measuring method. After the inter-calibration tests were finalized, SINTEF performed wave height measurements with the same type of wave gauge as SL Ross. More testing and calibration of the wave probe must be performed at SINTEF prior to further tests in Task 2, but the preliminary testing indicated that a few adjustments on the wave generator are needed for especially the setting for high energy, but also low energy.

Table 2: Wave height (cm), average of three visual measurements with standard deviation.

Measuring point	B	C	D
Setting 1	11 (0,6)	9,4 (0,5)	8,2 (0,4)
Setting 2	8,1 (0,2)	6,0 (0,3)	7,6 (0,1)
Setting 3	2,0 (1,0)	2,0 (0,2)	1,9 (0,2)

Wind speed

Wind speed was measured as average over two minutes (1 Hz) with a Kimo - Vane Probe Thermo-anemometer. Wind speed was measured in the center of the flume a few centimeters above the water surface at each measuring position (A, B, C, D) This value was reported as wind speed (m/s) in Table 3.

Table 3: Windspeed (m/s)

Measuring point	A	B	C	D
Setting 1	1,9	2,1	2,1	2,0
Setting 2	2,0	2,2	2,5	2,4
Setting 3	2,0	2,3	2,3	2,2

Water current

Water current was measured with a Vane Wheel sensor and a Flowtherm NT logging device (Höntzsch). Measurements were done at 3 different depths (20, 30 and 50 cm below the surface). Measurements (1 Hz) were done for minimum 200 seconds at each depth. Measurements for each depth were then averaged and these 3 values were averaged again. This value was reported as water current (m/s) in Table 4..

Table 4: Water current (m/s)

Measuring point	A	B	C	D
Setting 1	0,10	0,14	0,08	0,09
Setting 2	0,11	0,16	0,09	0,10
Setting 3	0,01	0,03	0,01	0,02

3 Test method

A 200°C+ residue of the asphaltenic Norwegian Grane crude oil and the dispersant Corexit 9500 has been used for this calibration.

The oil was placed in a containment ring on the wave maker side of position B then sprayed with dispersant using a Wagner 450 paint sprayer fitted with a 0.8 mm diameter nozzle. The treated oil was allowed to sit for 1 minute prior to lifting the containment ring. Once the ring was removed the wave maker and air fans were started. The wave and wind energy were continued for a 1 hour period with continuous LISST particle size and concentration measurements. The LISST uses the technique of laser diffraction to obtain particle size distribution of particles up to 500 µm, and was mounted vertically in the flume, as shown in Figure 2.5 in the main report.

Water samples (approximately 1 L) for oil-in-water measurements were collected after 20, 40 and 60 minutes using the water sampling valve (50 cm depth) shown in Figure 1. The water samples were extracted using dichloro methane (DCM), and the concentration was determined measuring absorbance at 410 nm with a Shimadzu UV-1800 spectrophotometer using a response curve developed for the Grane 200+ crude oil.

In situ measurements of the oil concentration and particle size distributions were performed using a LISST-100x or a LISST-Deep. Due to high concentrations, an 80 % path reduction module (PRM) was installed in the LISST-100x. All experiments were run for at least 60 minutes.

4 Results and discussion

There were a few problems with the LISST (Sequoia Scientific, Inc) in situ measurements, so totally 12 experiments were performed. In two of the experiments (high energy 1 and medium energy 1), a LISST-Deep was used, without the PRM. The data for the high energy experiment was acceptable, while there were problems with the medium energy experiment (too high oil concentration in the flume). Therefore, for the remaining experiments, a LISST-100x with 80% PRM was used. Installing the PRM will reduce the laser transmission energy.

In the experiments medium energy 3 and high energy 2, there were observed fouling of oil on the LISST glass window during the run (see Figures 4 and 5), so the collected data sets from the LISST were limited. The results from these three experiments were therefore ignored when calculating the average values and standard deviation (Table 5).

A propeller with very low frequency was used during the low energy experiments to keep the water circulation in the flume and to limit the risk of oil fouling on the LISST window. In experiment 1 and 3 the propeller was turned off for 10 to 20 minutes, and the oil concentration increased, as illustrated in Figure 3. These data was also ignored when calculating the oil concentration.

Figure 6 shows the cumulative droplet size distribution for all experiments at 20, 40 and 60 minutes. There was no variation in droplet size versus time in the high energy and low energy experiment. The droplet size in the low energy experiments was significantly larger than the medium and high energy experiments, as the volume median oil droplet diameter (d50) was 104 µm in the low energy experiments and 14 and 22 µm for the medium and high energy, respectively. The results also show that the cumulative droplet size in the medium energy experiments is slightly smaller than in the high energy experiments. This is also indicated in Figure 7, which illustrates the average droplet size distribution. As the variation related to time was insignificant, just data after 60 minutes are shown.

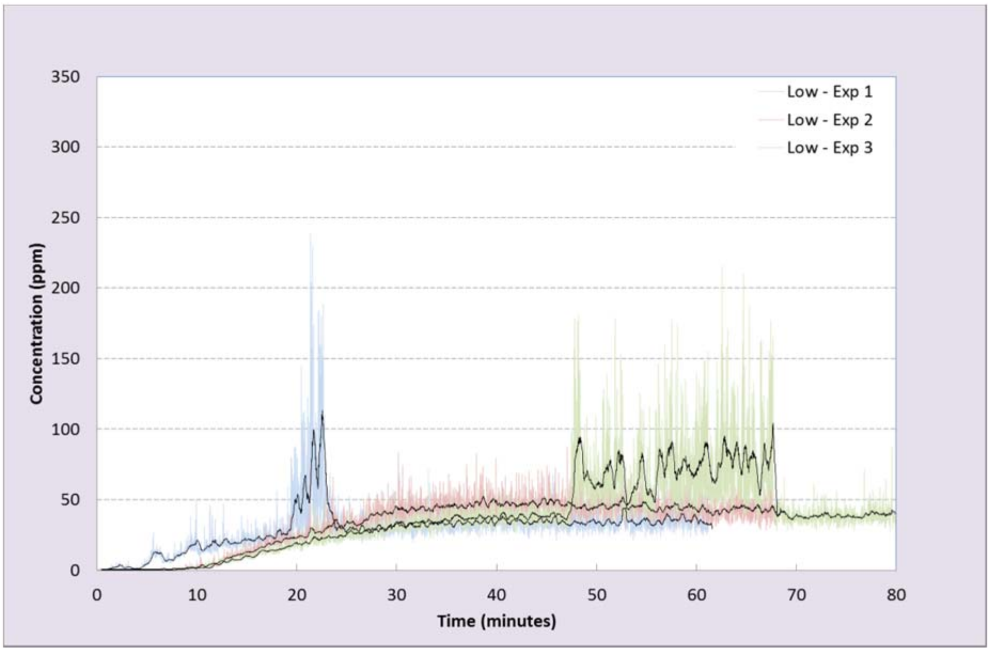


Figure 3. Low energy: Oil concentration (in ppm) from the in situ LISST measurements. Propeller turned off from approximately 18-25 minutes in exp 1, and from approximately 48 to 68 minutes in exp 3.

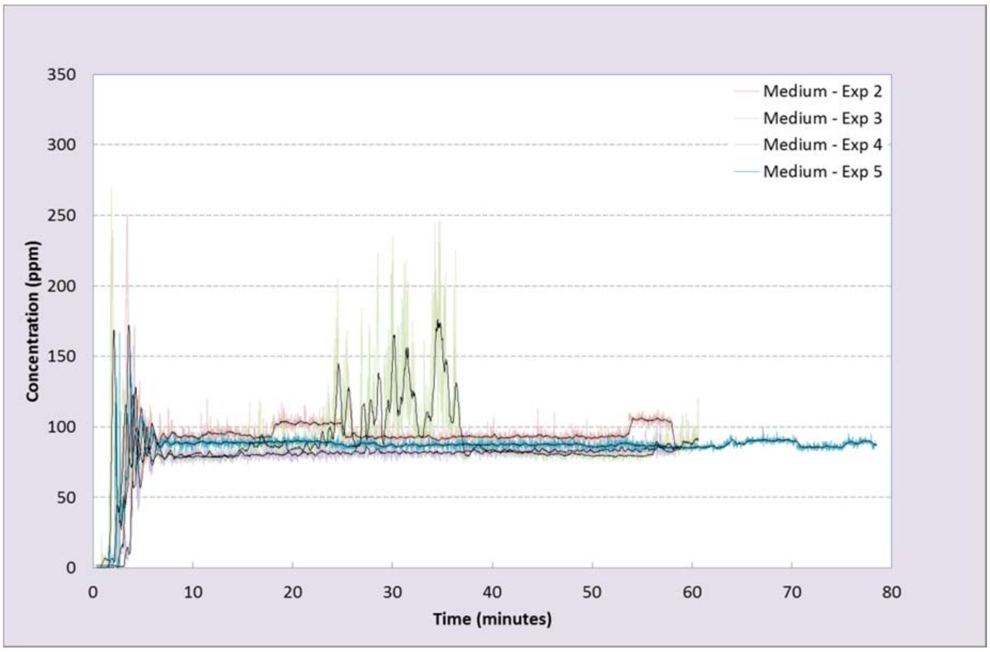


Figure 4. Medium energy: Oil concentration (in ppm) from the in situ LISST measurements. Oil on LISST window in exp 3 from approximately 25 to 35 minutes.

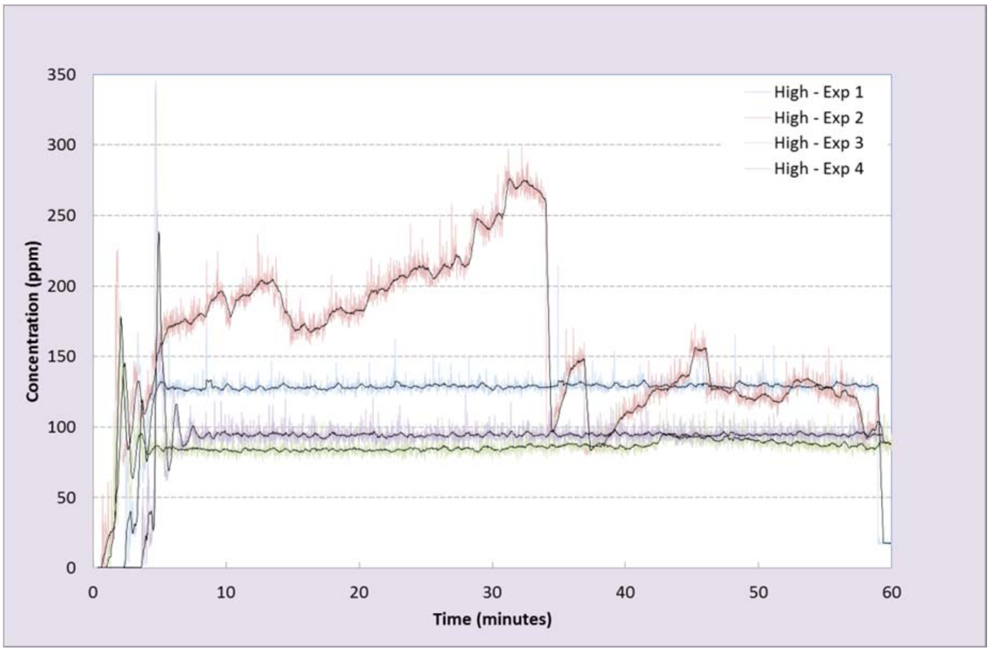


Figure 5. High energy: Oil concentration (in ppm) from the LISST measurements. Oil on LISST window in exp 2.

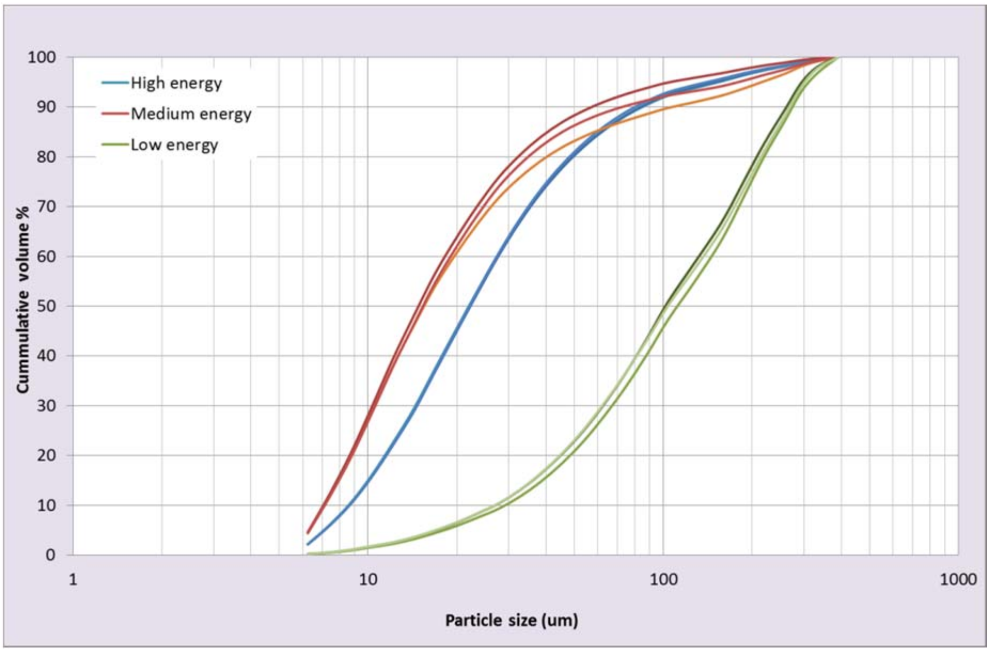


Figure 6. Cumulative droplet size distribution. All energy levels, after 20, 40, and 60 minutes.

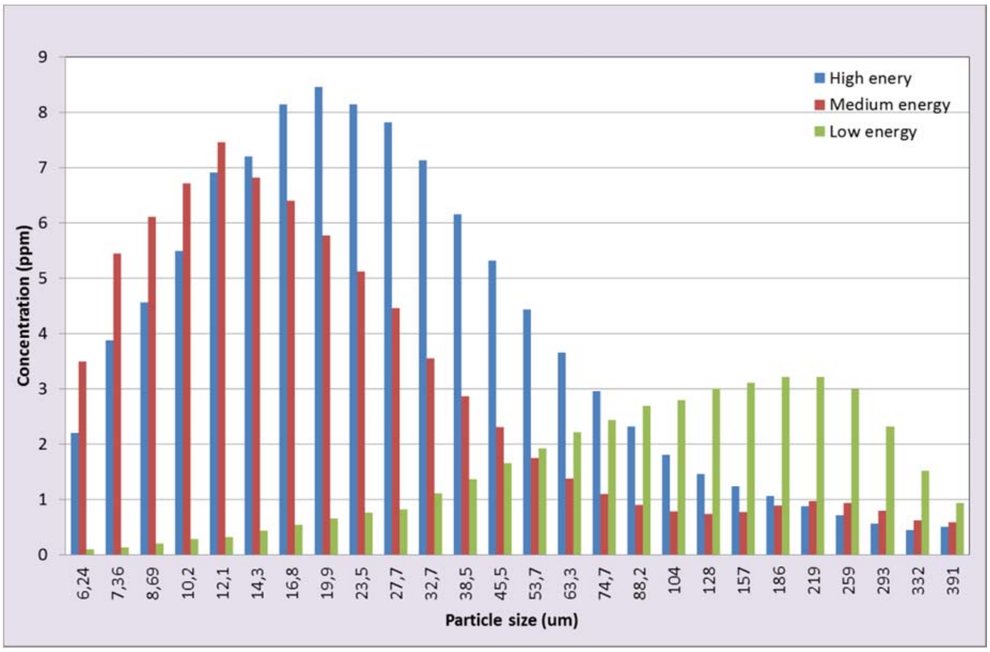


Figure 7. Average droplet size distribution for three energy levels, after 60 minutes.

A summary of the oil concentrations measured by the LISST (approximately from 10 to 60 minutes) and from the extracted water samples (average for the samples collected at 20, 40 and 60 minutes) and estimated dispersant efficiencies for all experiments are given in Table 5.

The comparison of the oil concentration measured by the LISST and the extracted water samples show that the LISST data are approximately two times lower than the water samples. In order to investigate this further, an additional experiment were performed (Medium level 5). In this experiment the LISST-100x both with a 50% and 90 % PRM were tested. The LISST was moved around in the flume to check if there could be inhomogeneity between the location for water sampling and LISST measurements, or concentration shifts in the flume. The concentration differences in the flume were insignificant, both from the LISST and the water samples. Oil concentration measurements from water samples and LISST have previously been compared at SINTEF using the factory-provided calibration, and it has always been very close (although this was with droplets that were >~70 microns and at lower concentrations). More investigation regarding this will be performed, and SINTEF has also contacted the manufacturer Sequoia Scientific. In the comparison between the flumes and the further discussions, it is therefore suggested that the water sample concentrations from SINTEF are used.

The dispersant efficiencies for the low energy tests were 44 ± 2 %, and 92 ± 2% for both medium and high energy tests. There was not a significant difference between the medium and the high energy tests, and as the oil droplet measurements indicated, the droplet size in the medium energy test is smaller than in the high energy tests. This could be explained by the frequency of the high energy waves leading to collision between the existing and the newly generated wave resulting in a wave dampening effect, possibly by changing the wave direction making it bounce between the flume walls. The energy levels will be slightly adjusted prior to further testing in Task 2.

Table 5. Oil concentrations and dispersant efficiency (Conc: concentration, disp: dispersant, wt, weight, DOR: dispersant to oil ratio, ave: average, stdev: standard deviation). Grey values not include in average.

				By LISST			By water analysis		
	Oil	Disp	DOR	Conc	Conc.	Efficiency	Conc	Conc.	Efficiency
	g	g	by wt	ppm	StDev	%	ppm	StDev	%
Low energy 1	901	37,6	24	33,7	7,0	18,0	78,6	14,2	41,9
Low energy 2	922	36,0	26	45,1	7,8	23,5	82,4	2,3	42,9
Low energy 3	915	38,6	24	37,9	9,0	19,9	88,8	18,6	46,6
Ave low				38,9		20,4	83,3		43,8
Medium	*879*	*38,1*	*23*	*104**	*4,3*	*56,1*	*173*	*1,5*	*94,2*
Medium	950	40,2	24	95,2	5,6	48,1	180	2,8	91,2
Medium	*917*	*41,9*	*22*	*82,4*	*5,1*	*43,1*	*177*	*1,6*	*92,6*
Medium	929	38,8	24	81,9	3,5	42,4	183	5,6	94,3
Medium	890	36,4	24	87,8	2,4	47,3	167	2,5	90,0
Ave mid				88,3		45,9	177		91,8
High energy 1	930	33,5	28	129*	4,1	66,6	178	1,9	91,7
High energy 2	*891*	*36,5*	*24*	*127*	*11*	*68,3*	*165*	*6,3*	*89,1*
High energy 3	870	42,6	20	86,4	5,3	47,7	167	0,2	92,1
High energy 4	964	38,6	25	94,6	4,4	47,1	187	3,5	93,0
Ave high				103		53,8	177		92,3

**LISST-measurements with LISST-Deep and no path reduction module.*

5 Summary

Dispersant effectiveness of one oil-dispersant combination has been performed using the pre-weathered Norwegian crude oil Grane and Corexit 9500 as the dispersant (dispersant to oil ratio of 1 to 25). Three different energy conditions (low, medium, high) were established. Triplicate dispersant effectiveness tests have been conducted at each energy condition.

The dispersant efficiencies for the low energy tests were 44 ± 2 %, and 92 ± 2% for both medium and high energy tests. There was not a significant difference between the medium and the high energy tests, and as the oil droplet measurements indicated, the droplet size in the medium energy test is smaller than in the high energy tests, (14 and 22 µm, respectively). It is assumed that this could be explained by a too high frequency of the high energy waves which is leading to collision between the existing and the newly generated wave resulting in a wave dampening effect.   The droplet size in the low energy experiments was larger than the medium and high energy experiments as the volume median oil droplet diameter (d50) was 104 µm.

APPENDIX C. CEDRE'S RESULTS

INTER-BASIN CALIBRATION OF MESO-SCALE FLUMES

Between SINTEF, SL Ross and Cedre - Cedre's results

Loïc MERLIN

1 Context

This research program conducted for the International Association of Oil and Gas Producers (OGP) is seeking scientific expertise for supporting the Arctic Oil Spill Response Technology – Joint Industry Program (JIP).

This program aims at conducting research investigations on the use of dispersant in Arctic conditions.

Among the different tasks of this program dealing with chemical dispersants, experimental work plans to carry out in meso-scale basin tests on the efficacy of chemical dispersant and mineral fines in Arctic marine waters conditions in order to define the operational limits of each with respect to oil type, oil viscosity, ice cover (type and concentration), and mixing energy (natural, water jet, and propeller wash).

Taking into account the large number of tests to be completed, the work has been split between the 3 laboratories, SL ROSS, SINTEF and Cedre which own similar testing facilities, flume test or hydraulic canal in shape of a loop equipped to reproduce the natural sea conditions wave, wind and temperature.

In order to compare the 3 equipment it has been requested by OGP to start the research program by an inter-calibration of the 3 flume tests, especially to look how the dispersion process can be promoted in each flume according to different level of mixing energy

This report describes the work done on Cedre's flume test equipment, (called the *Polludrome*), concerning this inter-calibration.

2 Objective

This study aimed to assess the dispersion which can be promoted in the *Polludrome* according 3 levels of energy (Low, Medium and High)

3 Principle

Fixed amounts of oil were spilt into the *Polludrome* and treated with dispersant. After addition of mixing energy using a wave generator, the level of dispersion was monitored for one hour looking to the oil concentration in the water column and to the oil droplet size distribution.

4 Materials

4.1 Products

- Oil: 200°C+ residue of the asphaltenic Norwegian Grane crude oil (Supply by SINTEF)
- Dispersant: Corexit 9500A (Supply by SL Ross)

4.2 Equipment

- Flume with wave & wind generator. (water volume 7.2m³)
- MALVERN MASTERSIZER 2000 – Drop size distribution of oil particles.
- 10-AU FLUOROMETER TUNER design (SFUV) – Concentration of oil.
- Sampling circuit (pipes, valves & one pump downstream the measurement equipment)
- A floating containment to lock the oil. (circular – 0.25m² – Ø = 0.56m)
- WAGNER 450 – Sprayer.
- Spectrophotometer EVOLUTION 600 UV-VIS. (for SFUV calibration)

4.3 Testing arrangement

- Water volume: 7.2 m³
- Water depth: 0.9 m
- Sampling point depth: 0.4 m from the surface

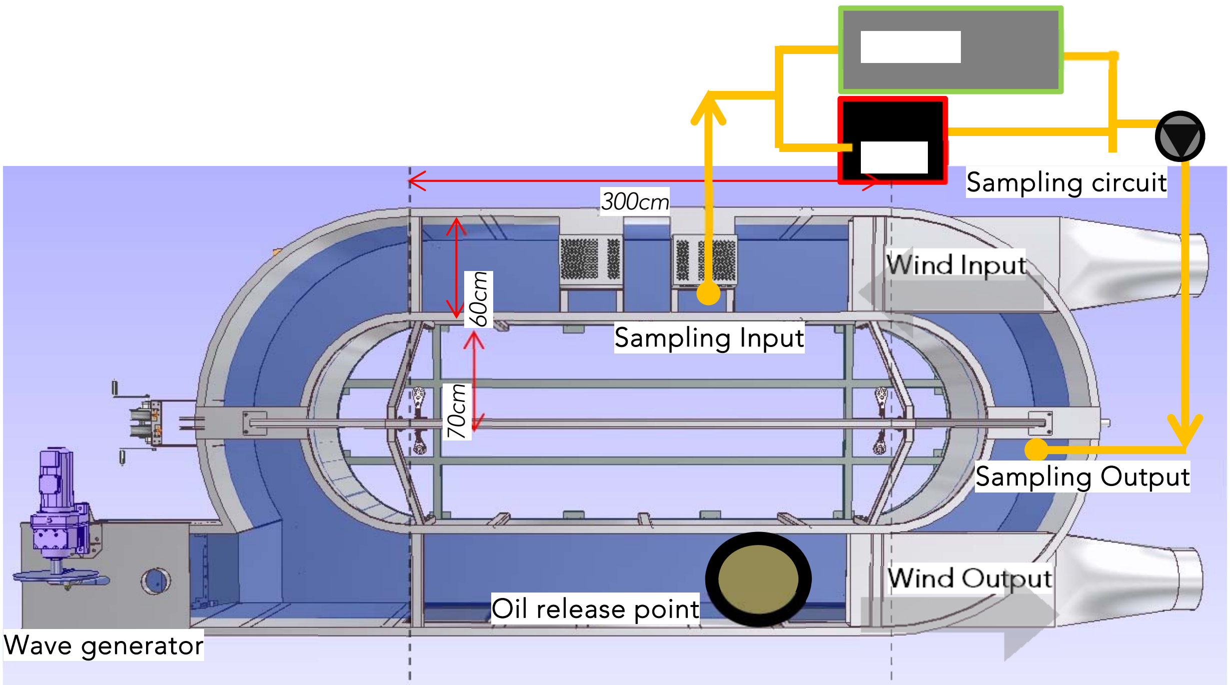


Figure 2 Experimental design

Note: Cedre flume is a larger than SL Ross & SINTEF ones (7.2 m³ > 5 m³).

4.4 Wave generator

The waves are generated by a board oscillating around a horizontal axis. This board is moved by electric motor through an acentric rod. The level of energy can be adjusted by changing the speed of the electric engine (i.e. changing the number of oscillation of the board per minute). In the report the level of energy is expressed in rotation speed of the engine (RPMm).

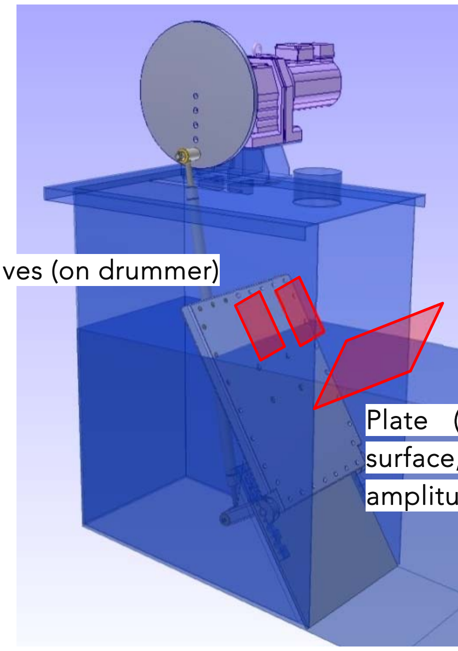


Figure 3 Wave generator design

5 Method

5.1 Parameters

- Room temperature: 10°C
- Water temperature: 13°C – Volume 7.2m³
- Oil temperature: 13°C – Volume 1L
- Dispersant: 10°C – Volume 40ml
- Wind velocity 3m/s.

5.2 Protocol

A summary of the major steps followed for each test is provided below.

- First of all, the baseline of SFUV & Malvern is measured.
- 1L of oil is poured into the containment ring (figure 3).
- The oil is let for 5minutes to let the oil to spread and to let temperature to equalize between the oil and the water.

Note: the exact quantity of oil introduced in the flume is measured by weighting the oil beaker before and after.

Figure 3 Oil applied on the circular containment

- SFUV measurements start;
- After 1 minute, the dispersant is sprayed with the Wagner sprayer, over the oil, for 20seconds (40ml at 2ml/s), (*figure 4*) which shows the spraying procedure.

Figure 4 Dispersant sprayed on the oil layer

- Starting on the center of the oil layer *(1)* then turning all around *(2)* (3 rounds) then ended at the center *(3)*.
- The containment ring is lifted after 2 minutes; the time origin for the data collection;
- 25 seconds after, the wind is started, then 10 seconds after, the wave generator is started
- The test lasted for one hour.
- The droplet size is periodically measured with the Malvern.
- At t=3000 secondes, a sample is taken to be used for afterwards calibration of the SFUV response.

-

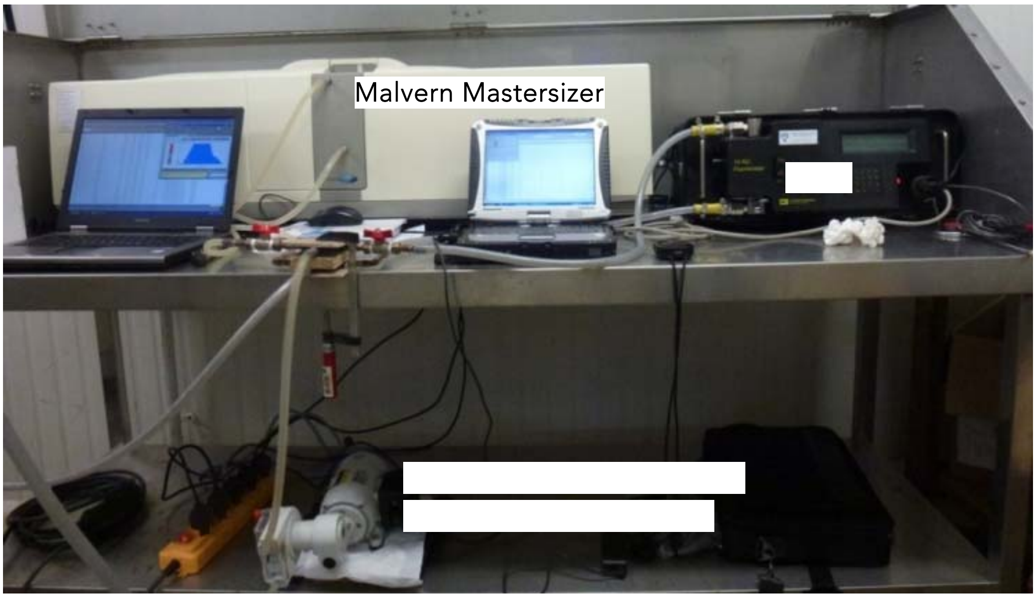


Figure 5 measurement system

6 Experimental conditions

6.1 Water current

Table 1 presents the water speeds at the surface and at the bottom for a wind set at 3 m/s.

Table 1 Water speed

ENERGY	Water speed close to the surface (cm/s)	Water speed close to the bottom (cm/s)
300	4	2
400	5	2
500	~9 (unstable)	4
600	unstable	8

6.2 Correlation between RPM motor & RPM wave maker

The energy can be adjusted according to the electric engine speed value. The correlation between the frequency of the movements of the oscillating board (RPM) and the electric engine speed (RPMm) is given in the *figure 6*.

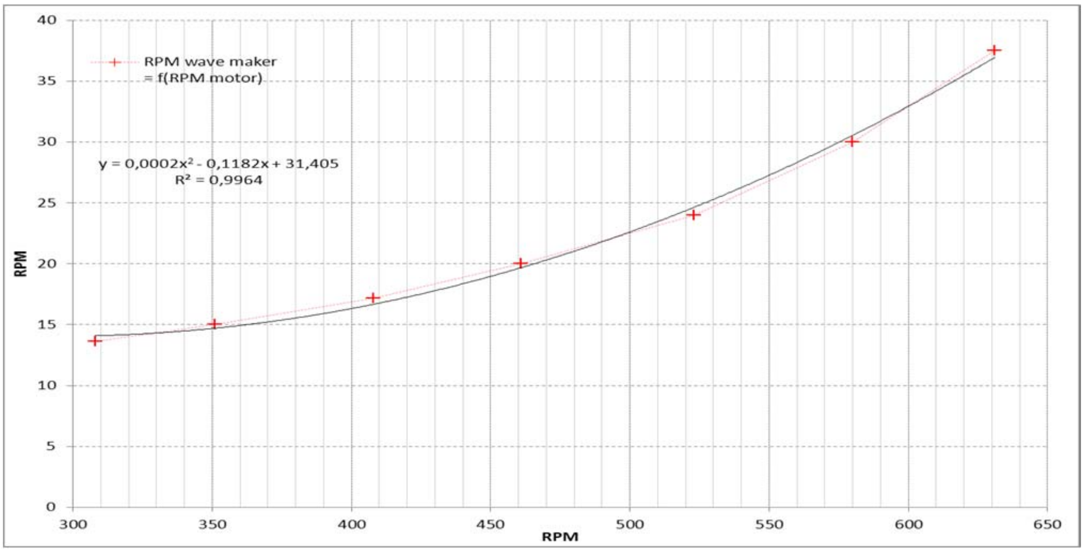


Figure 6 RPM wave generator relative to RPMm

6.3 *Waves amplitude assessment*

The measure of the wave's amplitude shows 3 regimes (*picture 7*):

- Phase 1: Below 400 RPMm, no significant difference, the waves remain weak.
- Phase 2: After 400 RPMm up to 500 RPMm the wave's amplitude increases keeping non breaking regime.
- Phase 3: Upper than 500 RPMm, the waves regime change into breaking regime.

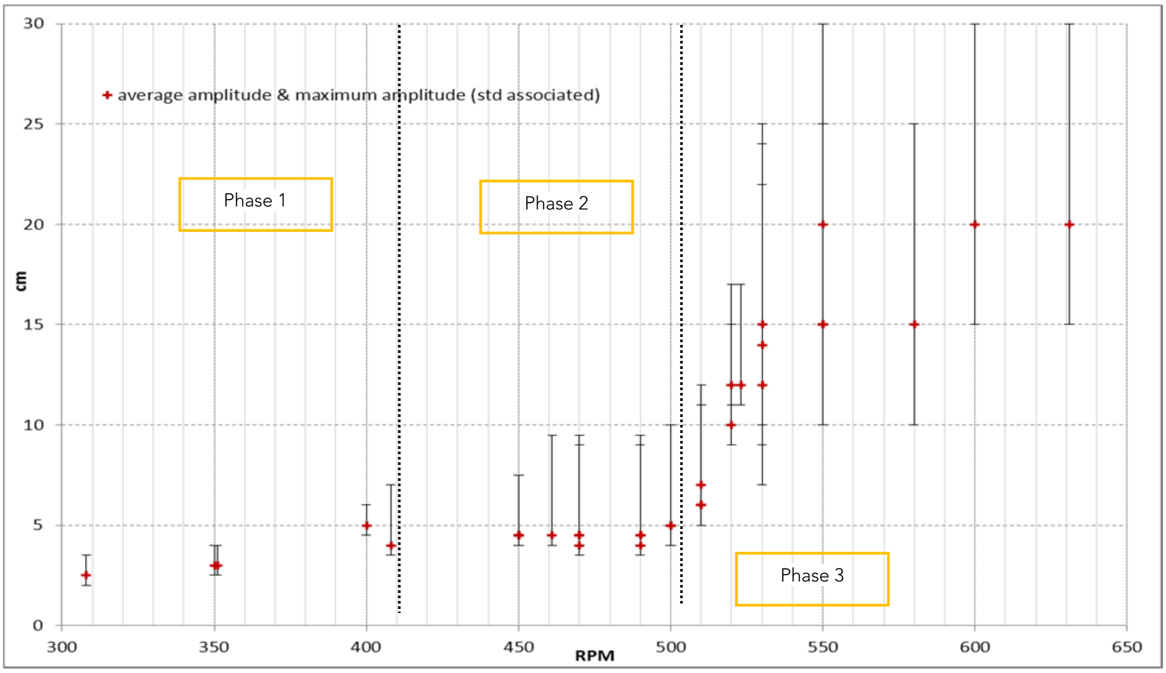


Figure 7 Waves amplitude relative to energy (RPMm)

6.4 Calibration of the SFUV

The SFUV used to measure the concentration during the test needs to be calibrated.

This calibration was completed by taking a water sample at the end of each test (at 3000sec after the oil release) which was analyzed afterwards in the laboratory (extraction with DCM and measurement with spectrophotometer at 390 nm wavelength). The analysis results were compared to the average concentration from SFUV at the end of the tests (2700 to 3300sec).

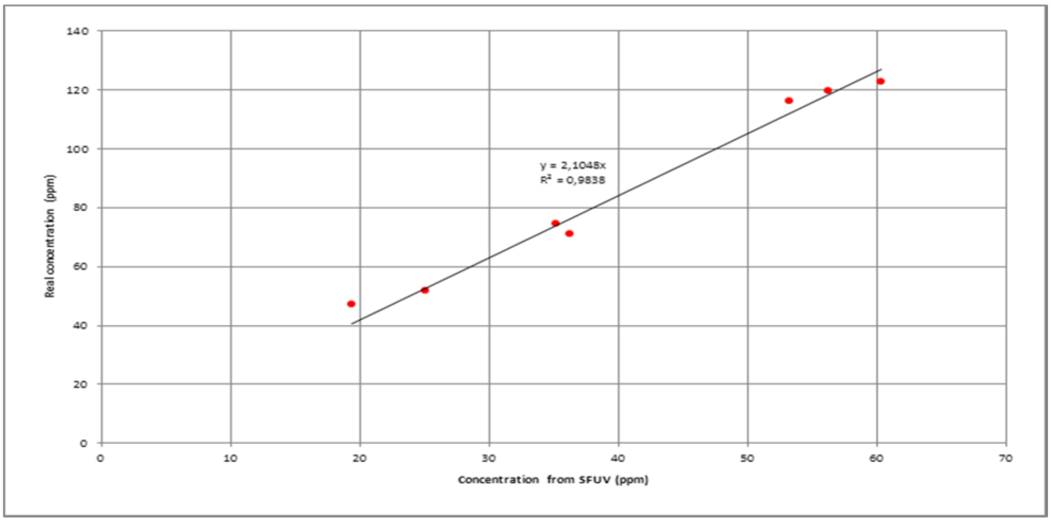


Figure 8 Calibration of the SFUV with the analysis results completed in the laboratory;

The calibration showed that the indication from SFUV was the half of the real concentrations. All the results presented further have been corrected according to this correlation: (see *Figure 8*)

- 'Real concentration' = 'SFUV concentration' x 2,1 (r^2 = 0,98)

6.5 Checking the quality of the dispersant application

4 tests were conducted to assess the efficiency of the method used to apply uniformly the dispersion over the oil slick (spraying quality).

The containment ring was put over a square of sorbent (1.2m x 1.2m); then dispersant is sprayed according to the same procedure than for a real test (cf. figure 5). The quantity of dispersant applied was measured by weight differences of the sorbent before and after the spraying application. The effective quantity of dispersant reaching the oil layer measured was:

- 28.3 ± 1.7 ml.

7 Experimental matrix

The tests have been completed in 3 phases:

1) Exploratory tests over a large energy range (from 350 to 550 RPMm)to select the 3 levels of energy required (low, medium and high).
2) The required inter-calibration tests at the 3 levels of energy (Low, Medium and High)
3) Complementary tests to explore possible improvement to the operating protocol.

The *table 2* lists the tests which have been completed. The levels of energy adopted for the inter-calibration were 350 RPMm for low energy, 425 RPMm for medium energy, 460 RPMm for high energy. In red are the tests which have been considered for the inter-calibration (3 energy levels – triplicate).

Table 2 Experimental matrix

N°	DATE	ENERGY (RPMm - RPM)	INFORMATIONS
1	01-10-2013	350 – 14.5	First test (determination of energy level) Recorded as Low energy test – A
2	02-10-2013	450 – 18.7	Free test (determination of energy level)
3	03-10-2013	550 – 26.9	Free test (determination of energy level)
4	04-10-2013	300 – 13.9	Free test (determination of energy level & exploration)
5	07-10-2013	400 – 16.1	Free test (determination energy level & exploration)
6	08-10-2013	425 – 17.3	Medium energy test – A
7	09-10-2013	425 – 17.3	Medium energy test – B
8	10-10-2013	460 – 19.5	High energy test – A
9	11-10-2013	460 – 19.5	High energy test – B
10	14-10-2013	350 – 14.5	Low energy test – B
11	15-10-2013	350 – 14.5	Low energy test – C
12	16-10-2013	425 – 17.3	Medium energy test – C
13	18-10-2013	460 – 19.5	High energy test – C
14	21-10-2013	500 – 22.3	Free test (exploration of an intermediate energy)
15	22-10-2013	350 to 450 to 550 – 14.5 to 18.7 to 26.9	Free test (Efficiency improvement, energy increasing slowly)
16	23-10-2013	550 – 26.9	Free test (efficiency improvement for >460RPM) Oil layer just before the Wave maker
17	24-10-2013	550 – 26.9	Free test (efficiency improvement for >460RPM) Oil layer just before the Wave maker & alginate film on flume side
18	25-10-2013	460 – 19.5	Free test (Rapprochement to SINTEF & SL Ross magnitude & improvement of Malvern response) Oil quantity: 3L

8 Results

8.1 Comparison of the 3 energy levels

- LOW energy level: 350 RPMm = 14.5 RPM (wave amplitude: 3 to 4cm)
- MEDIUM energy level: 425 RPMm = 17.3 RPM (wave amplitude: 4 to 7cm)
- HIGH energy level: 460 RPMm = 19.5 RPM (wave amplitude: 5 to 10cm)

The table 3 summarizes the result for the 9 tests kept for the inter-calibration.

Table 3 Table data resumed for the 3 energy levels

	TEST CONDITIONS	TIME RESULTS				CONCENTRATION (Calibrated)		EFFICIENCY		PARITICLES SIZES					
		Δt		Period						d10%		d50%		d90%	
LOW ENERGY AVERAGE ± STANDARD DEVIATION	*Temperature (oil & water) : 13,2 ± 1,0°C* *Oil Quantity : 956,4 ± 13,2g* *Wave amplitude : 3 to 4cm* *Wave maker : 14,6 ± 0,0 rpm*	459	± 93 sec	428	± 82 sec	46,2	± 6,0 ppm	34,8%	± 4,0%	14	± 2 µm	41	± 3 µm	90	± 8 µm
MEDIUM ENERGY AVERAGE ± STANDARD DEVIATION	*Temperature (oil & water) : 13,2 ± 1,0°C* *Oil Quantity : 956,6 ± 10,6g* *Wave amplitude : 4 to 7cm* *Wave maker : 17,4 ± 0,1 rpm*	339	± 25 sec	254	± 4 sec	72,5	± 4,3 ppm	54,6%	± 3,7%	12	± 4 µm	33	± 10 µm	76	± 6 µm
HIGH ENERGY AVERAGE ± STANDARD DEVIATION	*Temperature (oil & water) : 12,2 ± 0,3°C* *Oil Quantity : 955,5 ± 31,4g* *Wave amplitude : 5 to 10cm* *Wave maker : 19,6 ± 0,1 rpm*	195	± 28 sec	223	± 16 sec	118,8	± 7,4 ppm	89,4%	± 3,0%	6	± 1 µm	20	± 5 µm	59	± 21 µm

Data descriptions: appendix I

Data details for all inter-calibration tests: Appendix II, III, IV

Figure 9 presents the evolution of concentration of dispersed oil in the water column according to the time for the 3 energies tested. We can see that, according to the energy tested, the concentration of oil change significantly.

It can be observed that, for all tests, the dispersed oil concentration follow periodical variations which weaken progressively until being stable. This represents the oil plume moving in the flume, which homogenizes progressively in the whole tank.

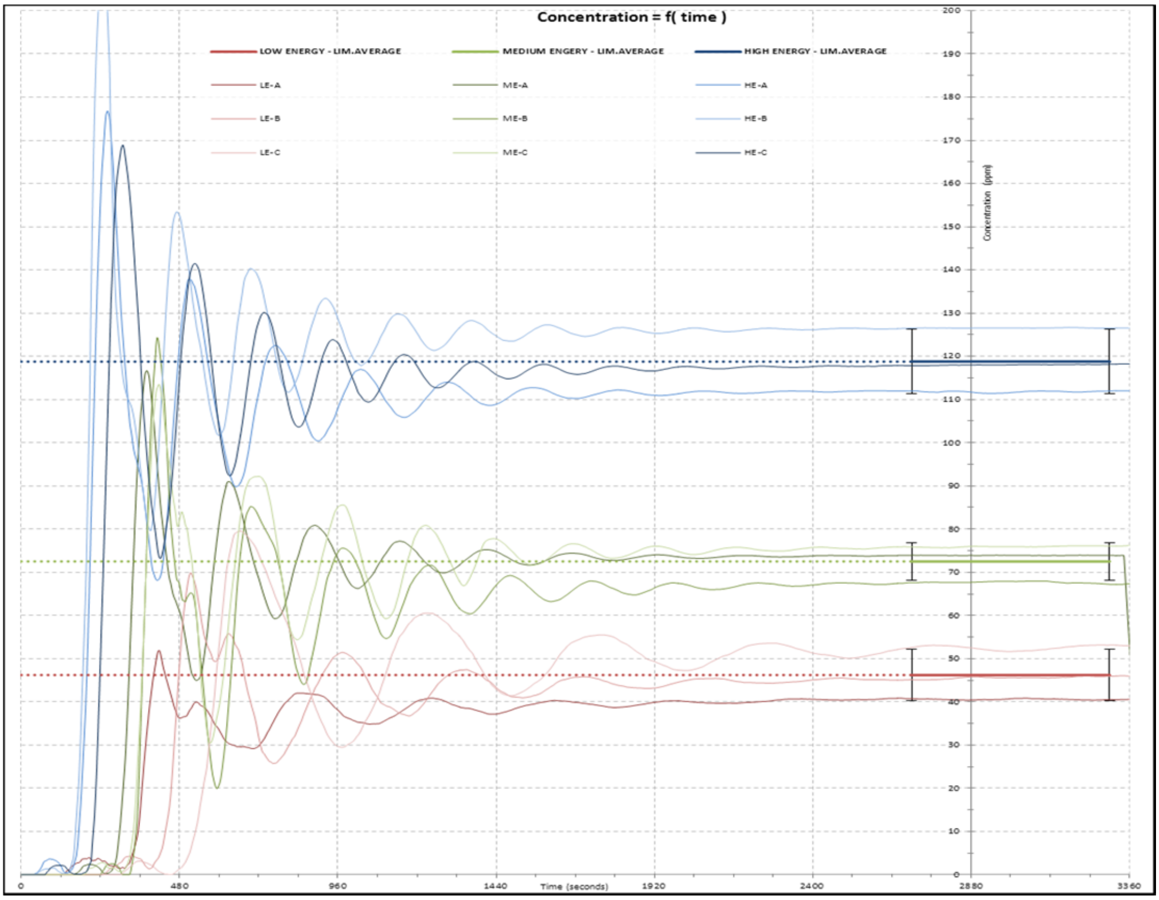


Figure 9 The average distribution of the oil droplet size by energy level (low, medium, high).

The whole droplet size ranges from 2 - 3 µm to 200 µm whatever the energy level. However, the median of the population changed according to the energy. This evolution of medians diameters let think there is a correlation between the energy and the droplet size, but the standard deviation, keeps too high to validate this statement. (*figures 10, 11 & 12*)

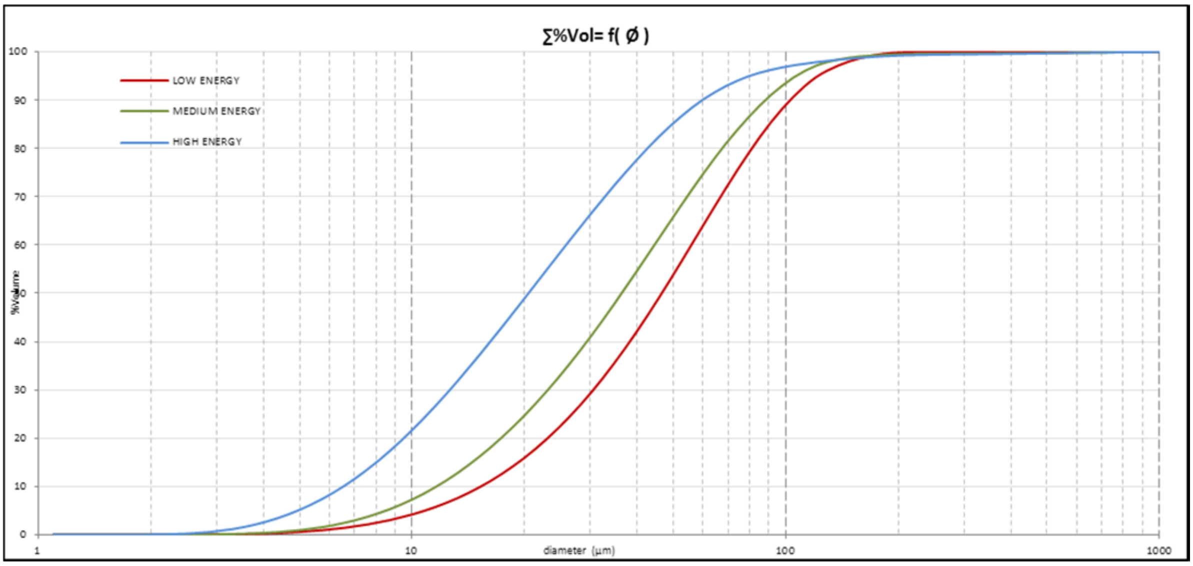


Figure 10 Cumulative droplet size distribution for the 3 energy levels

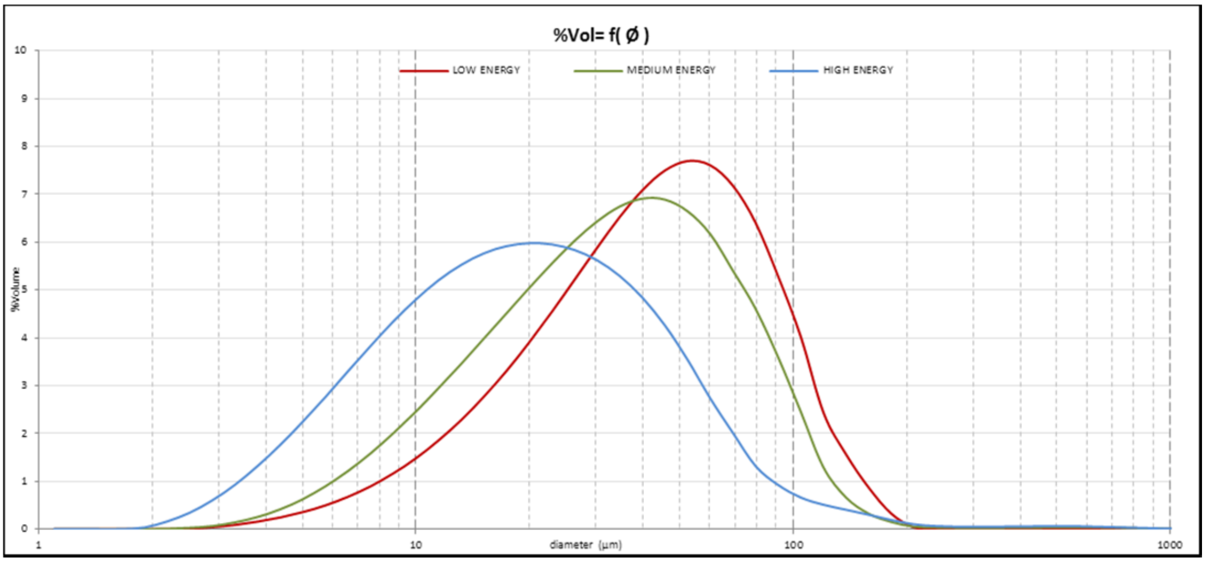


Figure 11 Droplet size distributions for the 3 energy levels

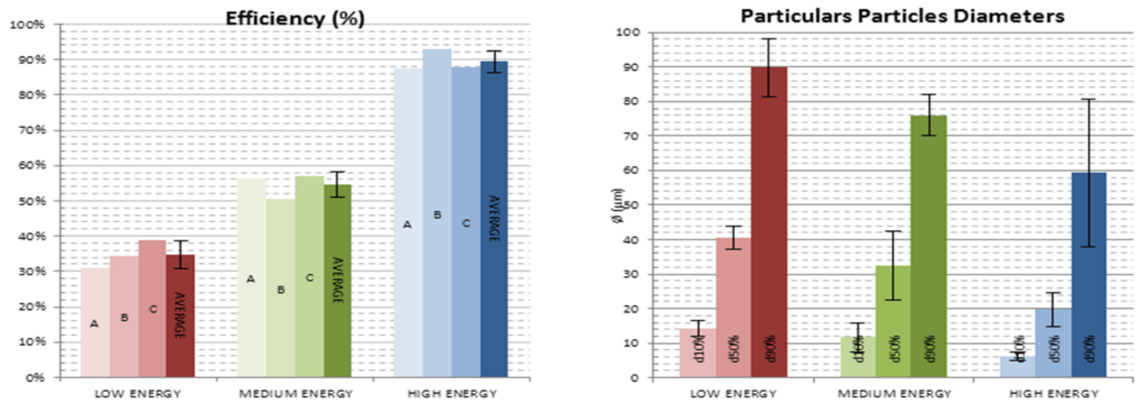


Figure 12 Dispersion efficiency & particular particles diameters by energy level

8.2 Additional issues

The additional tests which were completed in these experiments raise several issues

8.2.1 Energy level versus dispersion efficiency & waves amplitude

The level of dispersion is not linear with the energy level: we see 3 phases corresponding to the 3 phases of waves regimes as described in the wave's assessment.

The decrease of dispersing efficiency over 460 RPMm can be explained by the oil behavior during the first round: Splashed by the breaking waves and stick before be really dispersed when passing in front of the wave generator.

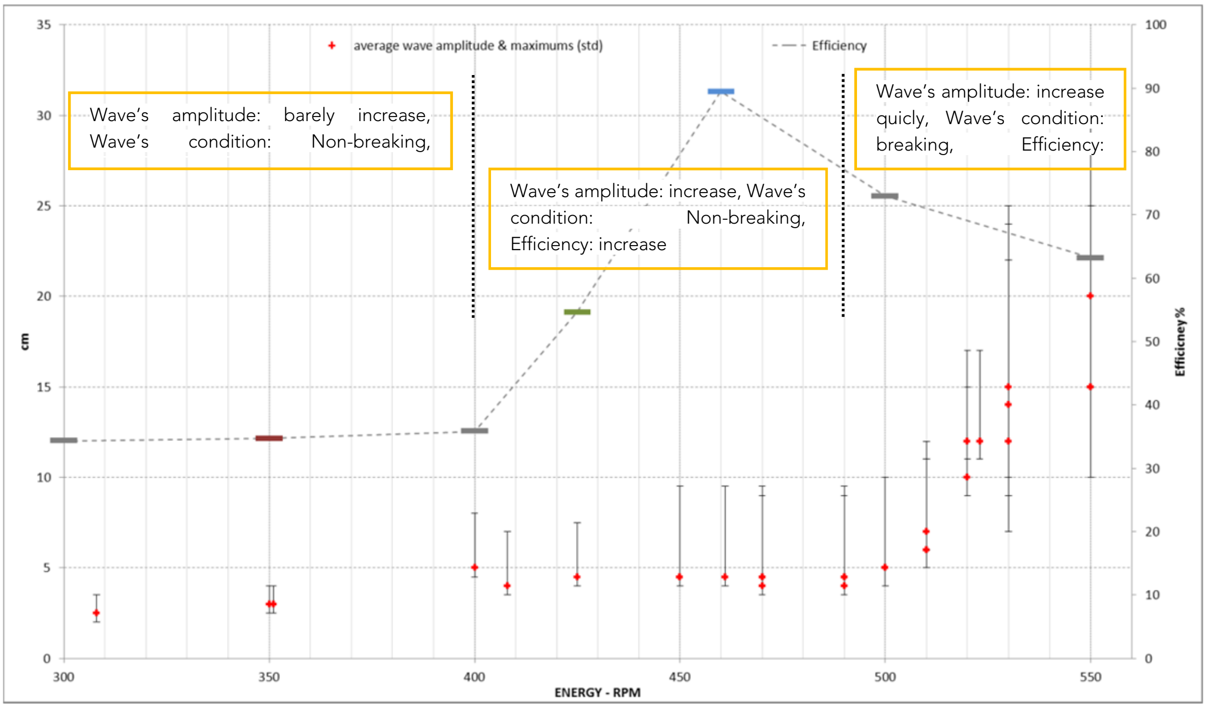


Figure 13 Waves amplitude & efficiency relative to the energy (RPMm)

8.2.2 *Period versus the wave generator frequency*

The concentration curves show that the oil concentration oscillate around the final concentration (#weaken sinusoid). The oscillations characterized by the period, which directly depends to the water speed, represent the oil dispersed plume in rotation in the flume. The figure 14 shows that the wave generator is the main parameters affecting the water speed. It can be approximately calculated (in considering the speed is constant in the whole water column).

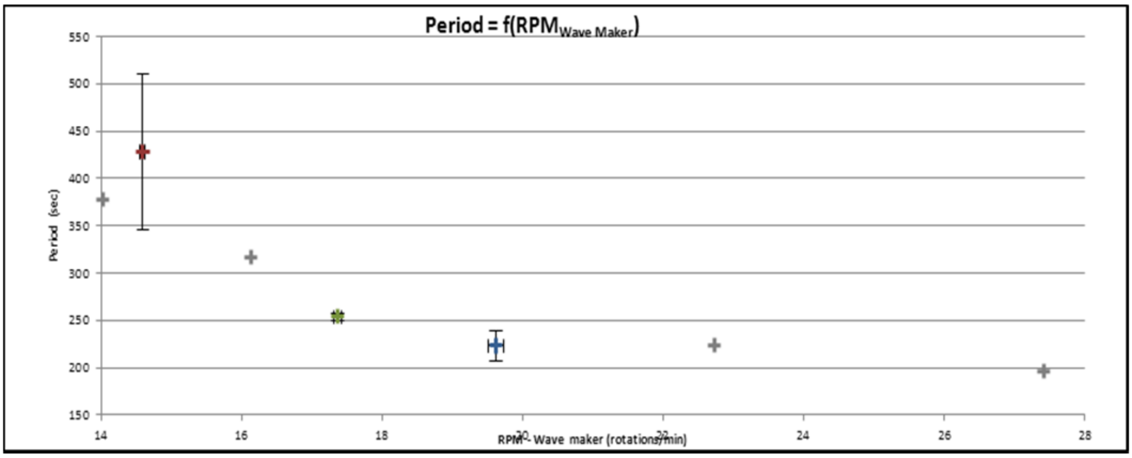


Figure 14 Period (Peak to peak) relative to the energy levels

The following table (*table 4*) compares the water speed measured (*table 1*) to the water speed calculated with the period from oscillations curves.

Table 4 Water speed comparison

ENERGY	Water speed close to the surface (cm/s)	Water speed close to the bottom (cm/s)	Calculated using the period (cm/s)
300	4	2	3
350			3
400	5	2	4
425			5
460			6
500	~9 (inconstant)	4	5
550			6
600	Inconstant	8	

8.2.3 Protocol optimization

To reduce the oil sticking on the walls at higher energy 3 tests were completed.

A. Increasing progressively the energy, after oil slick & plume associated made one lap (starting at 350 RPMm then 450, then 550)

B. The oil release point located before the wave generator to ensure dispersion during the first meter. With an "earliest" dispersion, (550 RPMm)

C. Operating as previously (B) but with the application of a film forming agent.

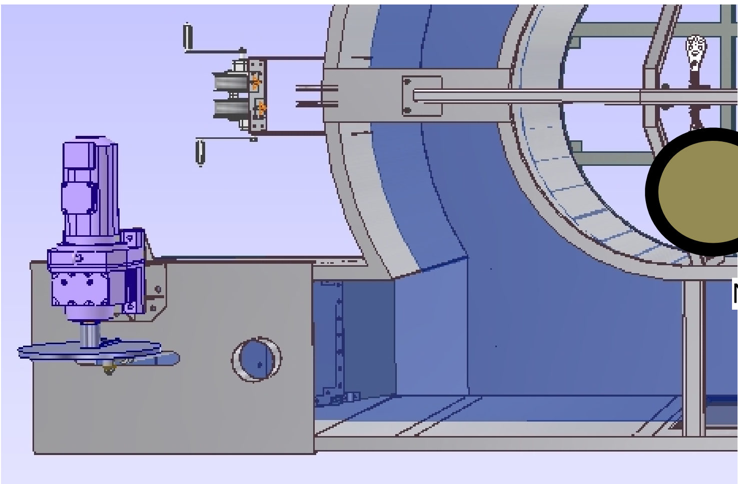


Figure 15 New oil release point position

Result: The efficiencies of the 3 tests have to be compared to the equivalent energy level made in the previous (regular) conditions: 550 RPMm; 63.2% of efficiency.

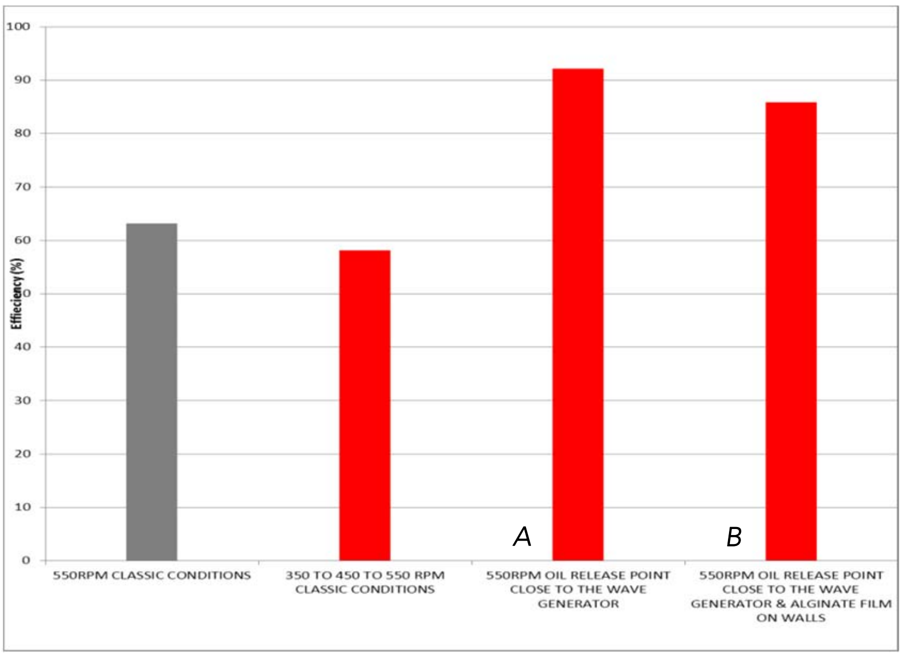


Figure 16 Efficiency relative to the conditions

A. Efficiency = 58% *(≈63.2%)*
   - Increasing the energy progressively does not affect the efficiency.

B. Efficiency = 92% *(>>63.2%)*
   - Release of oil just before the wave generator increases the efficiency for highest energies.

C. Efficiency = 86% *(≈92% from second test, same conditions without film)*
   - The presence of a film protecting agent has no effect on the efficiency.

8.2.4 Improvement for droplet size measurement

The Malvern results show an uncertainty due to the fact the dispersed oil concentration kept too low for optimal measurement.

In order to reduce this uncertainty it could be suitable to increase the quantity of oil.

A test has been performed at 460 RPMm, using a larger quantity of oil: 3L Oil with 120ml of dispersant.

The high concentration allowed to make an auto-acquisition of 100 measurements during the one hour test (compare to 5 to 10 measurements during the intercalibration tests). The residual was quite better, (as well as the obscuration).

Table 5 Interval time relative to the equation

	Parameter description	Value / Range
RESIDUAL	Average for the test	1.33 ± 0.10
	Average for all inter-calibration tests	5.77 ± 3.18
	Optimal range	1 - 0
OBSCURATION	Average obscuration for the test	16.3%
	Obscuration range for all inter-calibration tests	3% - 15%
	Optimal range	15% - 25%

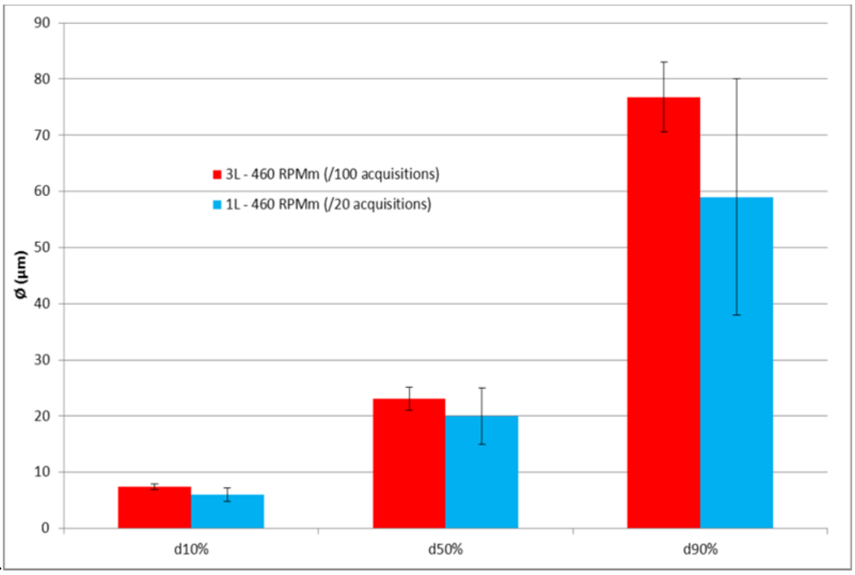


Figure 17 Comparison of the particulars particles diameters for 3L & 1L at 460 RPMm with the standard deviation associated

8.3 *Modeling the concentration*

An attempt to model the evolution of the dispersed oil concentration during a test.

Considering 3 ranges in the time: (*see Table 6*)

RANGE 1 – From 0 to A

RANGE 2 – From B to C

RANGE 3 – From C to the end

The evolution has been modeled using ∑*Sygmaplot™*, an example is given *Appendix V*.

Table 6 Interval time relative to the equation - Software use for correlation: ∑Sygmaplot™

Interval	RANGE 1	RANGE 2	RANGE 3

time (sec)	0 to A	B to C	C to the end
Equation or value	0: 0 A (Δt(20ppm)-1): 0	B (Δt(20ppm)): 20 C (Δt(C0ppm)): C0	$C = C_0 + Ae^{-\left(\frac{t}{d}\right)} \sin\left(\frac{2\pi t}{B} + c\right)$

The following table presents the average parameters to modeled the concentration evolution by energy level:

Table 7 Average parameters for equations - Software use for correlation: ∑Sygmaplot™

Parameter	Representation	LOW ENERGY	std	MEDIUM ENERGY	std	HIGH ENERGY	std
A	Magnitude of amortization (ppm)	27,3	7,1	44,5	5,4	64,8	8,5
B	Period of the amortization (sec)	471 *(≈ 428*)*	88	268 *(≈ 254*)*	12	233 *(≈ 223*)*	22
c	Phase (#0)	0,3	0,0	0,3	0,1	0,3	0,1
d	Amortization (sec)	567	108	408	54	340	38
C0	Constant concentration at the end (ppm)	47,7 *(≈ 46,2**)*	4,7	71,2 *(≈ 72,5**)*	4,5	117,8 *(≈ 118,8**)*	7,3
Δt(20ppm)	Average first time at 20 ppm	459	93	339	25	195	28
Δt(C0ppm)	Average first time at C0 ppm	489	104	367	22	233	30
** Comparison with the period from the tests (experimental result), ** Comparison with the average concentration at the end of the tests (experimental result)*							

Result: The oil plume evolution in the flume model, according to the time and the energy level.

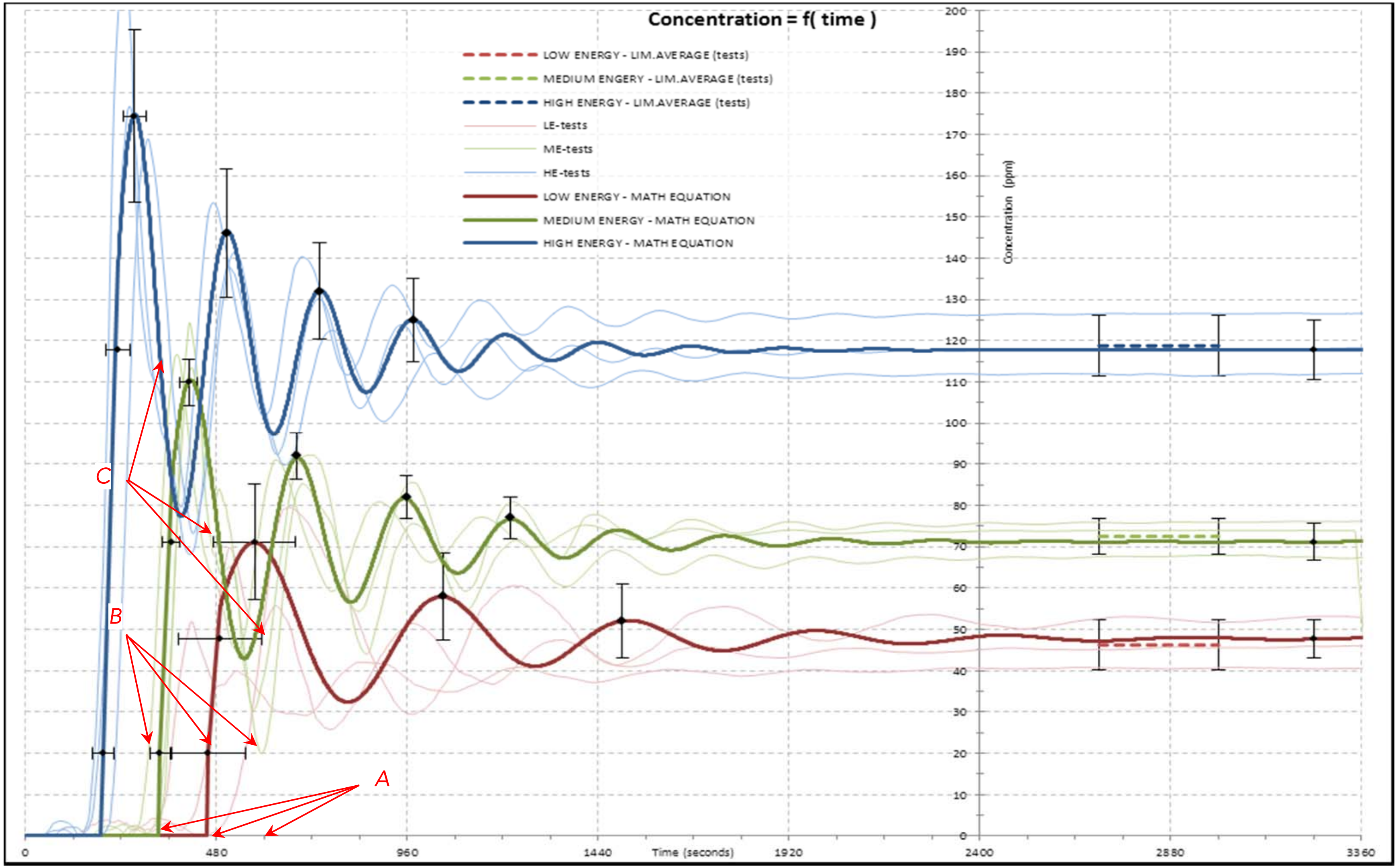


Figure 18 Model of plume evolution (Concentration VS time for Low, Medium and High energy)

9 Conclusion

This study aimed at inter-calibrating the 3 flume test canals of Sintef, SL Ross and Cedre.

This report presents the results related to Cedre flume test canal (the *Polludrome*).

Dispersibility tests were conducted on dedicated oil at different mixing energy levels quantified with the rotating speed of the wave generator. The quality of the oil dispersion was assessed by measuring the dispersed oil concentration in the flume with a SFUV Tuner calibrated in the laboratory and the dispersed oil droplet diameter with a Malvern Master Sizer 2000.

First, different energy levels were tested on a wide range of rotating speed (350 – 550 RPMm).

These tests showed that the operating range of Cedre flume is from 350 to 460 RPMm. Accordingly the 3 levels of energy required for conducting the inter-calibration tests were defined as follows: Low 350, Medium 425, High 460 RPMm.

The *table 8* summarizes the results from these intercalibration tests.

Table 8 Resumed data result for inter-calibration tests

	Conditions: Air: 10°C – Water: 13°C – Oil: 1L, 13°C – Dispersant: 40ml (28.3 eff.), 10°C – Wind speed: 3m/s			
N°	INFO – ENERGY – RPMm	Efficiency (%)	Droplet size range (µm)	Median droplet size (µm)
1, 10, 11	LOW ENERGY (350)	34.8 ± 4.0	3 - 200	41 ± 3
6, 7, 12	MEDIUM ENERGY (425)	54.6 ± 3.7	3 - 200	33 ± 10
8, 9, 13	HIGH ENERGY (460)	89.4 ± 3.0	2 - 200	20 ± 5

We can observe that,

A. For the concentration, the reproducibility is good, with a small standard deviation.

B. For the oil droplet size measurement, there is a correlation between the median droplet size and the energy, despite the whole size range distribution keeps the same global range for all energy levels. However the reliability remains poor (high residual) due to the fact the oil concentration in the flume keeps under the optimal operating range of the Malvern..

Additional tests were completed in view to improve the operating testing protocol (see *table 9*)

Table 9 Resumed data result for additional tests

	Conditions: Air: 10°C – Water: 13°C – Oil: 1L, 13°C – Dispersant: 40ml (28.3 eff.), 10°C – Wind speed: 3m/s			
N°	INFO – ENERGY – RPMm	Efficiency (%)	Droplet size range (µm)	Median droplet size (µm)
4	300	34	3 - 200	35 ± 4
5	400	36	3 - 200	28 ± 4
14	500	73	2 - 200	19 ± 3
3	550	63	2 - 200	17 ± 3
16	Oil release downstream the wave maker (550)	92		
18	3L of oil (460)		2 - 200	23 ± 2

It can observe that:

A. At higher energy (> 460 RPMm) the efficiency is decreasing due to the loss of oil which sticks on the walls of the flume (splash over due to breaking waves).

B. As a way to improve the accuracy of the Malvern measurements will be to increase the oil quantity used for each test. It is proposed to double this quantity. More, this would make the testing conditions more similar between the 3 flume tests as Cedre flume test has a higher water volume than the two others.

C. Higher dispersion concentration (i.e. higher dispersion efficiency) and therefore a better accuracy (for Malvern) could be obtained by moving the location for the oil release just upstream the wave generator.

10 APPENDIX

10.1 DATA DEFINITIONS

Dispersed oil concentration:

- *Uncalibrated data*: Average concentration at the end of the test – Data from SFUV.
- *Calibrated data*: Average concentration at the end of the test – Corrected data after laboratory calibration.

Droplet size:

- *d10% / 90%*: value for which 10 or 90% of the population is under the diameter
- *d50%*: Median diameter.

(*d10%, d50%, d90%* are called particulars particles diameters)

Measurement's relevance:

- *Deviation relative to the average*: Deviation in percentage of the value to the average value (average of the 3 tests); the deviation relative to the average is different from the standard deviation.
- *Average relative deviation*: Average of the relative deviation for the 3 tests.
- *Residual*: This coefficient shows the measurement's relevance of the Malvern.

Energy units (wave generator):

- *RPMm*: Rotation per minute of the wave generator motor (~300-650 range)
- *RPM*: Frequency of the oscillating board (~10-40 range)

Times: specific points considered in the diagrams)

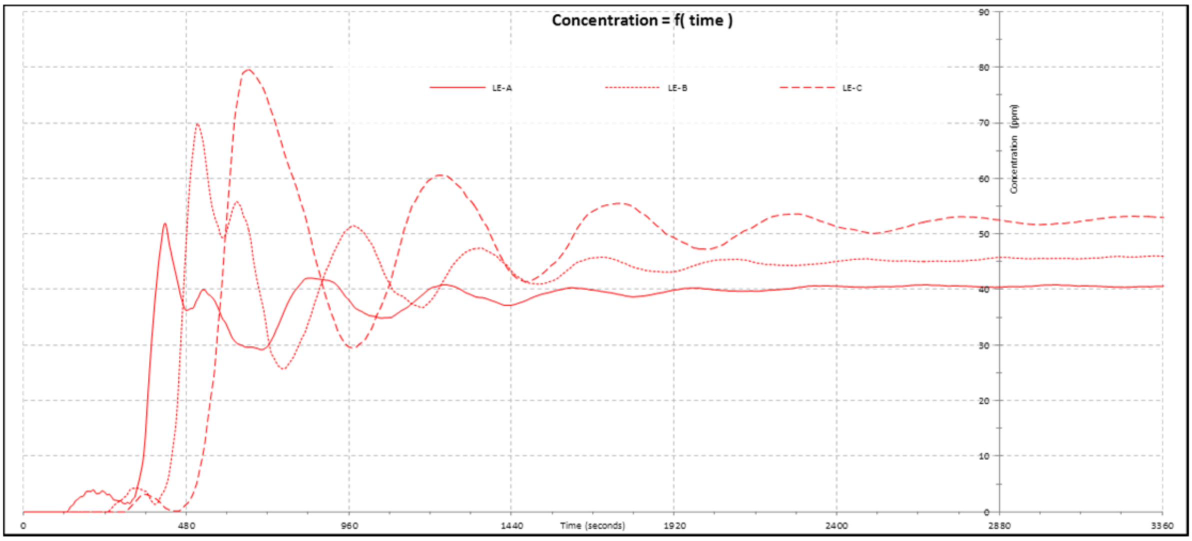
- ***Δ****t*: First time (sec) when the dispersed oil concentration reaches 20ppm

Period: Average time (sec) from peak to peak

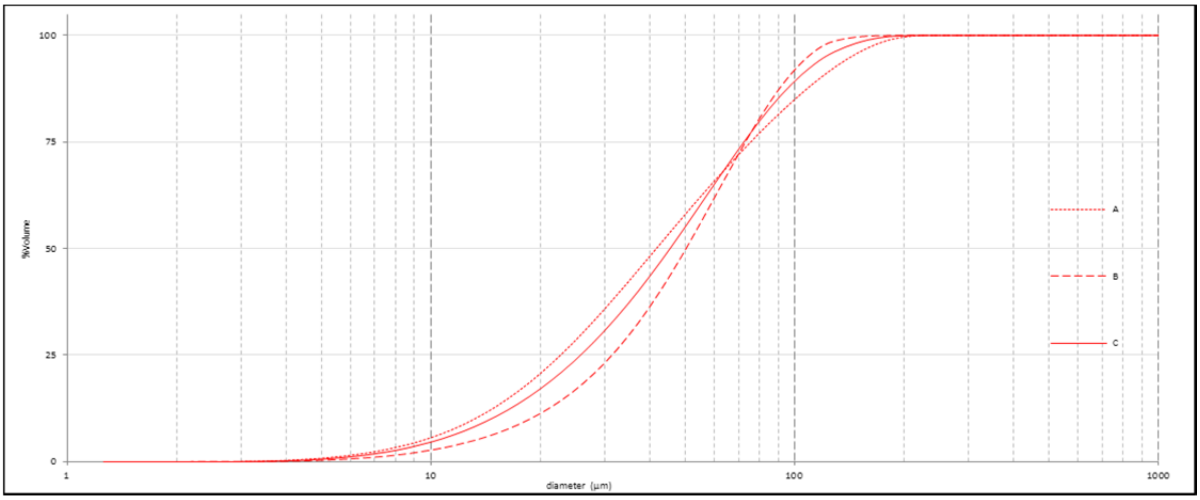
10.2 LOW ENERGY LEVEL DETAILS

Table data for Low energy level

		TEST CONDITIONS					TIME RESULTS						CONCENTRATION RESULTS							PARTICLES SIZES RESULTS									
					RPM (rotation/min)								Concentration (ppm)								Particulars Particles Diameters (µm)								
		Oil & Water temperature (°C)	Oil quantity (g)	Average waves amplitudes To maximums amplitudes (cm)	Engine (RPMm)	Wave Maker	Δt (sec) (1st time at 20ppm)	Deviation relative to the average	Average deviation	Period (peak to peak) (sec)	Deviation relative to the average	Average deviation	Uncalibrated datas (SFUV)	Calibrated datas	Deviation relative to the average	Average relative deviation	Efficiency (%)	Deviation relative to the average	Average relative deviation	Residual (optimal range : 0 - 1)	d10%	Deviation relative to the average	Average relative deviation	d50%	Deviation relative to the average	Average relative deviation	d90%	Deviation relative to the average	Average relative deviation
LOW ENERGY	A	12,0	943,4		353	14,6	368	20%		384	10%		19,3	40,6	12,2%		31,0%	10,9%		*3,98*	12	15%		38	7%		99	10%	
	B	13,5	956,1	3 to 4	354	14,6	455	1%	14%	378	12%	15%	21,7	45,6	1,4%	9%	34,3%	1,3%	8%	*10,70*	17	16%	11%	44	9%	6%	83	8%	7%
	C	14,0	969,8		351	14,6	553	21%		523	22%		25,0	52,5	13,6%		39,0%	12,2%		*6,93*	14	1%		40	2%		88	2%	
AVERAGE ± STANDARD DEVIATION		*Temperature (oil & water) : 13,2 ± 1,0°C - Oil Quantity : 956,4 ± 13,2g - Wave amplitude : 3 to 4cm - Wave maker : 14,6 ± 0,0 rpm*					**459 ± 93 sec**			**428 ± 82 sec**				**46,2 ± 6,0 ppm**			**34,8% ± 4,0%**				**14 ± 2 µm**			**41 ± 3 µm**			**90 ± 8 µm**		



Evolution of concentration according to time (averaged on 60 sec & calibrated – Low energy)

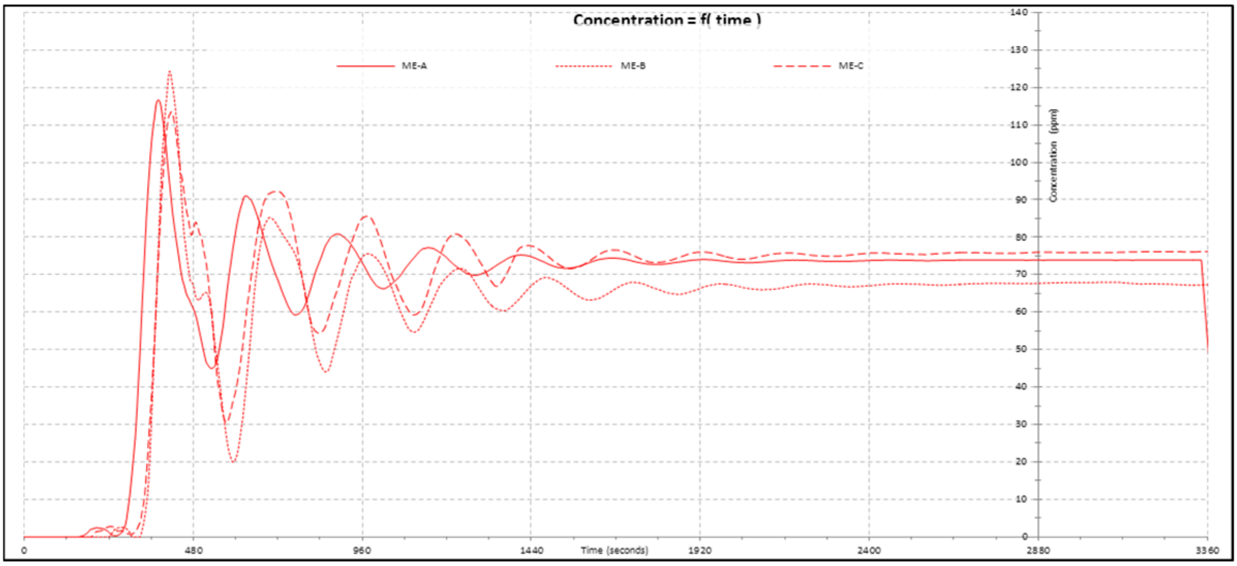


Cumulative droplet size distribution (Low energy)

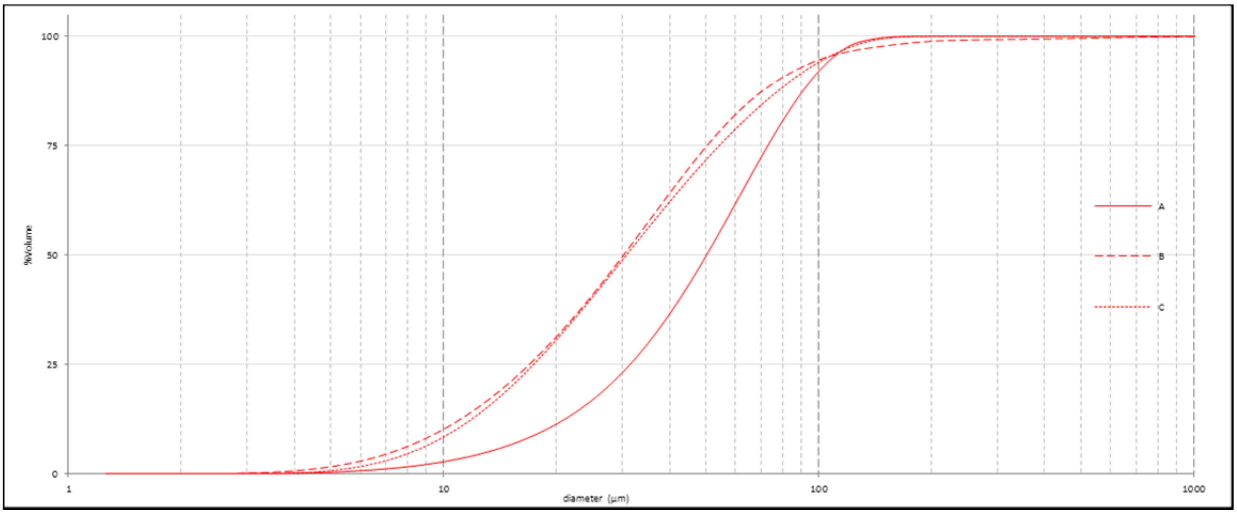
10.3 MEDIUM ENERGY LEVEL DETAILS

Table data for Medium energy level

		TEST CONDITIONS					TIME RESULTS						CONCENTRATION RESULTS							PARITICLES SIZES RESULTS									
		Oil & Water temperature (°C)	Oil quantity (g)	Average waves amplitudes To maximums amplitudes (cm)	RPM (rotation/min) Engine (RPMm)	RPM (rotation/min) Wave Maker	Δt (sec) (1st time at 20ppm)	Deviation relative to the average	Average deviation	Period (peak to peak) (sec)	Deviation relative to the average	Average deviation	Concentration (ppm) Uncalibrated datas (SFUV)	Concentration (ppm) Calibrated datas	Deviation relative to the average	Average relative deviation	Efficiency (%)	Deviation relative to the average	Average relative deviation	Residual (optimal range : 0 - 1)	Particulars Particles Diameters (µm) d10%	Deviation relative to the average	Average relative deviation	d50%	Deviation relative to the average	Average relative deviation	d90%	Deviation relative to the average	Average relative deviation
MEDIUM ENERGY	A	14,0	946,1		427	17,4	310	8%		259	2%		35,2	73,9	1,9%		56,2%	3,0%		9,86	17	42%		44	36%		83	9%	
	B	12,0	967,2	4 to 7	425	17,3	356	5%	6%	254	0%	1%	32,2	67,7	6,6%	4%	50,4%	7,7%	5%	6,71	9	22%	28%	27	18%	24%	71	6%	6%
	C	13,5	956,6		427	17,4	350	3%		250	2%		36,2	76,0	4,7%		57,2%	4,7%		3,93	9	20%		27	18%		74	3%	
AVERAGE ± STANDARD DEVIATION		*Temperature (oil & water) : 13,2 ± 1,0°C - Oil Quantity : 956,6 ± 10,6g - Wave amplitude : 4 to 7cm - Wave maker : 17,4 ± 0,1 rpm*					**339 ± 25 sec**			**254 ± 4 sec**				**72,5 ± 4,3 ppm**			**54,6% ± 3,7%**				**12 ± 4 µm**			**33 ± 10 µm**			**76 ± 6 µm**		



Concentration relative to the time (averaged on 60 sec & calibrated – Medium energy)



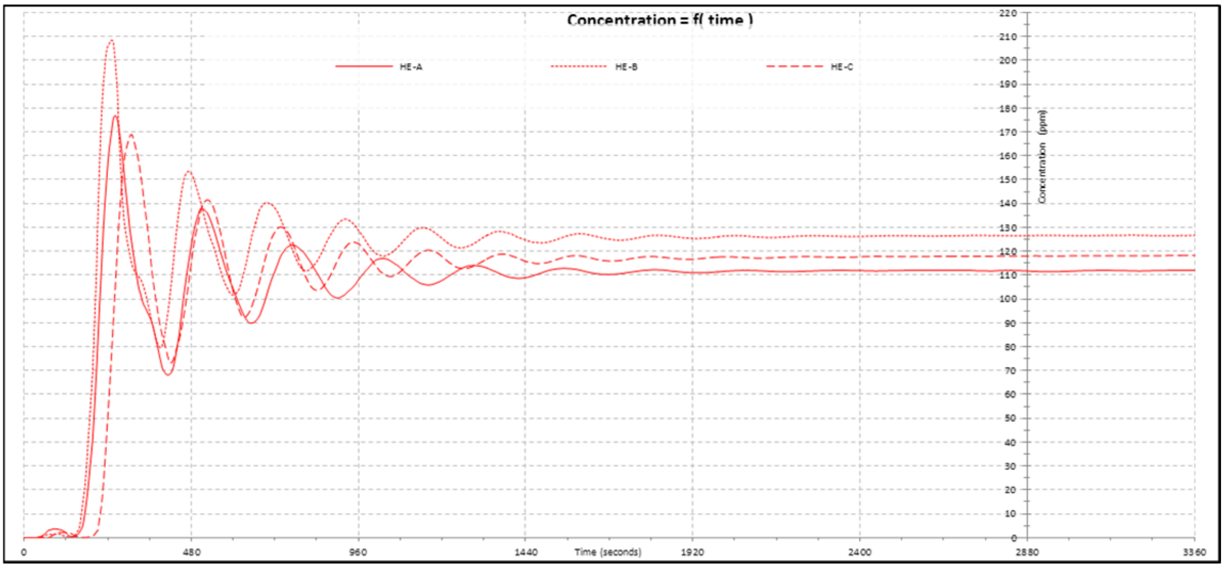
Cumulative droplet size distribution (Medium energy)

10.4 HIGH ENERGY LEVEL DETAILS

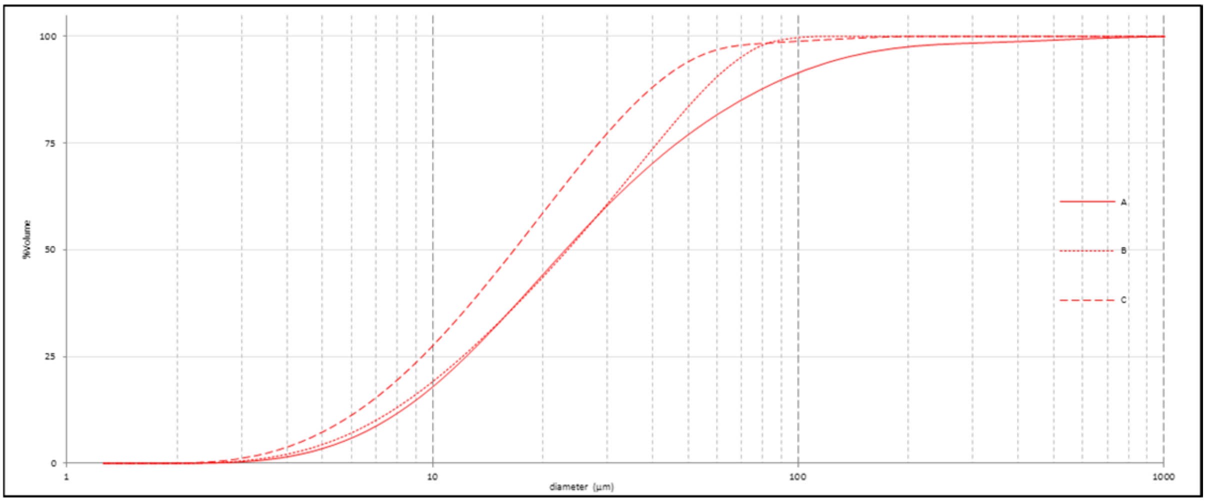
Table data for High energy level

	TEST CONDITIONS					TIME RESULTS					
	Oil & Water temperature (°C)	Oil quantity (g)	Average waves amplitudes To maximums amplitudes (cm)	RPM (rotation/min) Engine (RPM/m)	RPM (rotation/min) Wave Maker	Δt (sec) (1st time at 20ppm)	Deviation relative to the average	Average deviation	Period (peak to peak) (sec)	Deviation relative to the average	Average deviation
HIGH ENERGY A	12,0	920,3	5 to 10	462	19,5	184	5%	11%	241	8%	5%
HIGH ENERGY B	12,5	980,4		465	19,7	174	11%		222	1%	
HIGH ENERGY C	12,0	965,9		465	19,7	226	16%		208	7%	
AVERAGE ± STANDARD DEVIATION	*Temperature (oil & water) : 12,2 ± 0,3°C - Oil Quantity : 955,5 ± 31,4g - Wave amplitude : 5 to 10cm - Wave maker : 19,6 ± 0,1 rpm*					**195 ± 28 sec**			**223 ± 16 sec**		

	CONCENTRATION RESULTS							PARITICLES SIZES RESULTS									
	Concentration (ppm) Uncalibrated datas (SFUV)	Concentration (ppm) Calibrated datas	Deviation relative to the average	Average relative deviation	Efficiency (%)	Deviation relative to the average	Average relative deviation	Residual (optimal range : 0 - 1)	Particulars Particles Diameters (µm) d10%	Deviation relative to the average	Average relative deviation	d50%	Deviation relative to the average	Average relative deviation	d90%	Deviation relative to the average	Average relative deviation
HIGH ENERGY A	53,2	111,8	5,9%	4%	87,5%	2,2%	3%	*0,45*	7	3%	14%	20	2%	17%	81	37%	24%
HIGH ENERGY B	60,3	126,6	6,6%		93,0%	3,9%		*5,35*	7	17%		24	24%		59	2%	
HIGH ENERGY C	56,2	118,0	0,7%		87,9%	1,7%		*4,04*	5	21%		15	26%		38	35%	
AVERAGE ± STANDARD DEVIATION		**118,8 ± 7,4 ppm**			**89,4% ± 3,0%**				**6 ± 1 µm**			**20 ± 5 µm**			**59 ± 21 µm**		

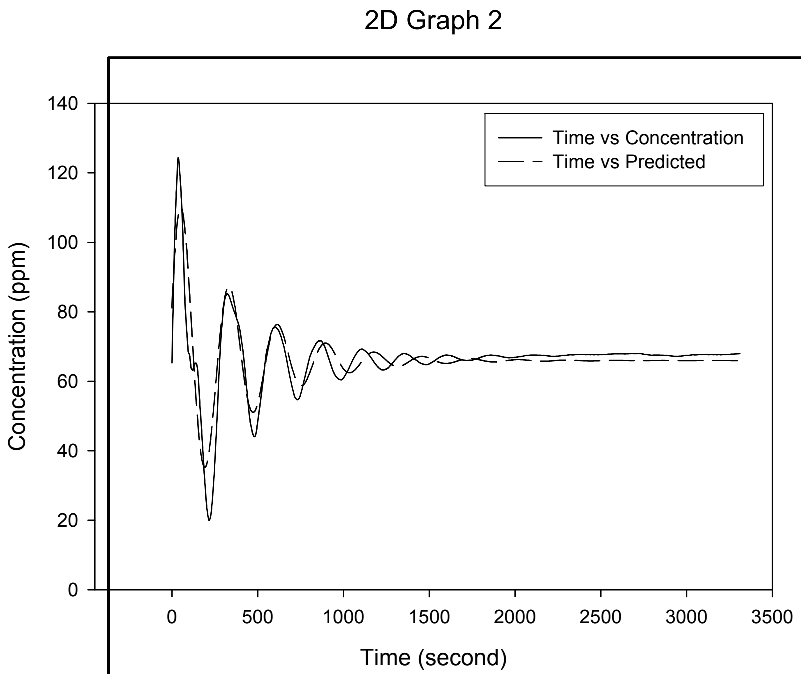


Concentration relative to the time (averaged on 60 sec & calibrated – High energy)



Cumulative droplet size distribution (High energy)

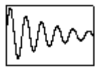
10.5 EXAMPLE OF SYGMAPLOT REGRESSION



Nonlinear Regression

$$y = y_0 + a e^{-\left(\frac{x}{d}\right)} \sin\left(\frac{2\pi x}{b} + c\right)$$

Data Source: Data 1 in Notebook1
Equation: Damped Sine, 5 Parameter



R	Rsqr	Adj Rsqr	Standard Error of Estimate
0,9975	0,9950	0,9950	4,7575

	Coefficient	Std. Error	t	P
a	50,7102	0,6749	75,1420	<0,0001
b	281,0872	0,6317	444,9532	<0,0001
c	0,3037	0,0143	21,2041	<0,0001
d	388,6691	7,4066	52,4764	<0,0001
y0	65,9584	0,0832	793,0901	<0,0001

Analysis of Variance:

	DF	SS	MS
Regression	5	14969696,8461	2993939,3692
Residual	3308	74871,2244	22,6334
Total	3313	15044568,0705	4541,0710

Statistical Tests:

PRESS 75781,7166

Durbin-Watson Statistic 0,0019 Failed

Normality Test Passed (P = <0,0001)

K-S Statistic = 0,2118 Significance Level = <0,0001

Constant Variance Test Passed (P = <0,0001)

Power of performed test with alpha = <0,0001: 0,0000

(**Time:** time – t_{y0})

∑Sygmaplot™ Regression wizard example (Medium energy – B)