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TESTING SUB-SEA DISPERSANT INJECTION AT LABORATORY SCALE

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June 2014 (reviewed August 2014)

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TESTING SUB-SEA DISPERSANT INJECTION AT LABORATORY SCALE

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TESTING SUB- SEA DISPERSANT INJECTION AT LABORATORY SCALE

Executive summary

OGP-IPIECA requested a laboratory screening study to understand more precisely the range conditions under which dispersant is effective and efficient for different crude oils in the context of subsea dispersant application and to check any possible evolution of the plume and droplets size in time.

A dedicated test and protocol have been designed and set up by Cedre for the specific purpose of this study.

This laboratory study was conducted on 4 different oils / 3 concentrated dispersants / 2 different Dispersant-Oil Ratios (DOR) / 2 energy levels, low and high (LE, HE) / 2 dispersant injection methods: PreMix, upstream the oil injection nozzle (PM) and Direct Injection of dispersant, downstream the oil injection nozzle, in the oil plume (DI).

The experiments were conducted in a 120 L cylindrical tank. The dispersion was assessed by measuring the dispersed oil droplet size with a Malvern particle size analyzer, as well as the oil concentration in the water column along vertical profiles using a Turner spectrofluorometer. These parameters were monitored for 20 minutes in order to check for possible short term evolution of the plume of dispersed oil. Therefore 3 data were considered, the median droplet sizes at the beginning (oil injection) and at the end of the test (20 minutes.) and the amount of oil still in the water column at the end of the test, called "efficiency" (integration of the oil concentration along a vertical at 20 minutes). In addition, when oil resurfaced at the end of the test, its re-dispersibility was assessed using the LaboFina-WSL rotating flask method.

85 tests were completed corresponding to 72 tests from the original request and 13 additional ones.

Furthermore, complementary tests were carried out to study the behavior of oil which would rise to the surface from high depth (simulation of a virtual ascent of 2h30). An experimental simulation of oil droplets ascent has been completed in a water column in which a downward stream was adjusted just to balance the rising speed of the oil droplets.

The subsea dispersant effectiveness bench scale test protocol developed by *Cedre* for the study allowed the following observations:

1. The oil type and characteristics were obviously the most influent parameters ; Grane oil remained poorly dispersible on the contrary of the the 3 others crude oils.
2. Averaging the different measurements, Corexit 9500 seemed to give a slightly better efficiency than Finasol OSR52, Dasic Slickgone and Finasol OSR 62. However, observed differences remained not significant. Comparison between Finasol OSR52 and Finasol OSR 62 showed that these 2 dispersants are very similar as they give very close results for similar conditions.
3. The level of energy was too high for the 3 lightest oils, and the tests gave results close to the maximum efficiency (≈90%). Therefore, in order to better highlight the difference between the dispersants, it would have been more appropriate to adopt more restrictive conditions for each oil, particularly a specific mixing energy level for each oil, (such a protocol was not planned in the design of the original study).
4. There is no special benefit to monitor the oil dispersion for a long period of time (20 minutes) as the observed variations remained low and did not change the ranking of dispersants in terms of efficiency.
5. The results showed that oil droplets obtained after dispersant injection fell mainly into 2 distinct areas: less than 35 µm and more than 100 µm. Good efficiency matched with droplets diameter <35 µm, while poor efficiency matched with > 100 µm. There was very few data in the range of 35 to 100 µm which appeared to be a threshold area.
6. The DOR at 5% gave little coalescence of the dispersed plume and no oil resurfacing even with Direct Injection mode. On the contrary more coalescence and sometimes dispersed oil resurfacing were observed at DOR of 2 and 1%. At DOR 0% (oil alone), dispersion was poor as large recoalescence occurred.
7. The Premix injection mode gave better results than the Direct Injection mode in terms of efficiency and dispersed oil stability (absence of resurfaced oil at the end of the test). The injection in the plume did not lead to an optimum oil-dispersant mixing. With a better mixing it should even be possible to use a reduced DOR. Obviously, the quality of the mixing between the oil and the dispersant is a key parameter; therefore, in the field, the design of the dispersant injection device should be carefully designed (e.g. the dispersant injection assembly mounted at the end of the injection wand).
8. According to the different testing conditions, on a few tests, discrepancies appeared between the droplet size distributions and the efficiency measurements: the ranking of the dispersant from the particle size analyzer was not always in agreement with the one from the spectrofluorometer. In order to better understand the confidence level of the test results it seems important to indicate the residual of the droplet size measurement (assessment of the measurement uncertainty). In this respect, the use of both droplet size measurements and oil concentration measurements increases the reliability of the results.
9. When oil resurfaced at the end of the test (20 min), it was easily re-dispersed when vigorously mixed (i.e. LaboFina WSL test method), except for Grane oil at low dosage and Direct Injection mode (injection into the plume).
10. A simple experimental simulation of a long ascent (2h30) in a long virtual water column has been completed in order to observe the oil droplets behavior during their ascent. It has been

observed that dispersion process occurred during the ascent: under the friction of the water against the oil droplets, a large part of the oil (between 40 to 60%), split into thinner droplets which were flushed out of the flask by the stream of water. In a real situation, this part of the oil would not likely reach the sea surface. Moreover, the oil which reached the surface lost about half of its dispersibility. These processes depend obviously on the type of oil and on the dispersant effect (reduction of the interfacial tension). In other terms, these observations showed that, after the dispersant addition, the dispersion process may be produced just by energy produced by the rising ascent of the oil from a high depth, independently from the conditions of the oil leakage at the bottom (pressure, velocity, turbulence which lead to the atomization of the oil).

Understanding these new questions is of tremendous importance for modeling the behavior of dispersed oil plume along the water column and for optimizing subsea dispersant applications. A specifically designed controlled experimental simulation could be undertaken, in a virtual water column at larger scale, in order to define the real droplet size evolution as a function of time, the final droplet size which could be achieved under the water friction as well as the final dispersibility of the oil which would reach the surface according to the oil type and to the dispersant used. Such an experiment could be undertaken in *Cedre*'s testing facilities, especially in its 5 meter high experimental water column which has already been used to study the behavior of chemical and oil and gas underwater releases.

TESTING SUB- SEA DISPERSANT INJECTION AT LABORATORY SCALE

Table of content

TESTING SUB- SEA DISPERSANT INJECTION AT LABORATORY SCALE

1) Objectives & Context

The experimental study aimed at determining the optimum operating conditions for dispersant subsea injection in a sub-sea blowout, at laboratory scale and checking any possible evolution of the dispersed oil plume over time.

Macondo incident demonstrated the possibility to use dispersant injected at the sea bed level to respond to a subsea oil blow out. However, since this incident, a lot of uncertainties have remained concerning the understanding of the dispersion mechanism and its efficiency and stability.

Deep sub-sea dispersion differs from regular surface slick dispersion as the oil, which is often mixed with gas, is more or less violently released in the very quiet deep sea environment. In this respect, the energy needed for the dispersion process is brought very briefly before the dispersed plume arrives in the deep quiet environment. Therefore, in deep sub-sea, the dispersion must occur very rapidly and must produce a very stable dispersed oil plume to avoid coalescence and/or rising up of oil towards the sea surface.

OGP-IPIECA requested a laboratory screening study to understand more precisely the range conditions under which dispersant is effective in terms of efficacy against different crude oils in the context of subsea dispersion, in other words to assess the optimum operating conditions for dispersant subsea injection.

The result of this study should be used to set the testing conditions of the validation a second set of experiments which will be conducted at larger scale in SINTEF's testing facilities.

Additionally it was requested to assess the possibility that the wave action (at the sea surface) could re-disperse the treated oil which would have resurfaced.

The request covers a large number of testing conditions:

- 4 different oils, / 3 concentrated dispersants / 2 different Dispersant-Oil Ratios (DOR)/ 2 energy levels, low and high (LE, HE) / 2 dispersant injection methods: premix (PM) and direct injection of dispersant (DI).

Cedre's view

According to a previous study conducted in a large hydraulic canal in *Cedre*'s testing facilities, it had been observed that coalescence of dispersed oil droplets could occur during the first 10 minutes after the initial dispersion. Therefore it seemed useful to design a test method which enables to monitor the dispersed plume during such a period of time.

2) Description of the technical approach: principle

Each test consisted in dispersing a known quantity of oil with a known quantity of dispersant in a tank and in monitoring the quality of the dispersed oil plume for 20 minutes while the plume was progressively rising up towards the water surface. Then, the oil which resurfaced was collected and tested for its re-dispersibility. This second step aimed at assessing if this treated oil which would have reached the surface of the sea could be re-dispersed under the mixing energy of the waves.
The test was conducted in a transparent cylindrical vertical tank in which the oil was injected from a nozzle while the dispersant was added either at the outlet of the oil nozzle (Direct Injection) or upstream the oil nozzle (PreMixed injection) in order to simulate a dispersant treatment straight in the oil plume or in the pipe or wellhead, upstream the leakage point.

By locating the oil nozzle in the upper section of the water column and orientating the oil flow downwards, the oil plume was sent down in order to leave more time for observation and measurement during its travel back up to the water surface. This could also favor re-coalescence of poorly dispersed oil and/or give information on the ability of the dispersant to prevent the oil re-coalescence.

The dispersed plume was monitored in terms of oil concentration in the water column and oil droplet size: a suction head which could be moved vertically along the water column sampled water which was sent to a spectrofluorometer and a laser particle analyzer for measuring both oil concentration and oil droplet size.

To avoid the plume to stick to the tank walls during its ascension and to allow collecting the resurfaced oil, the water column is kept very slowly rotating with a magnetic stirrer at its bottom.

Then, secondly, the oil which had possibly resurfaced was gently collected and put into a separatory flask in which its re-dispersibility could be measured according to the LaboFina-WSL method.

Lastly, additional tests were carried out in order to simulate oil droplets ascent from great depth to surface. These tests aimed at checking the possible effect of a prolonged contact time (several hours) between the dispersed oil droplets and the water during the droplets ascension toward the water surface. This long upward migration of the oil droplets was simulated by keeping the droplets in the vertical section of a separatory flask set upside down, in slow stream of sea water, running downward at the same speed than the upward velocity of the rising oil droplets. After few hours, the oil droplets were let to recoalesce at the water surface and then their re-dispersibility was checked. These additional tests have been carried out with and without dispersant in order to check if dispersant keeps with the oil during this rising up movement.

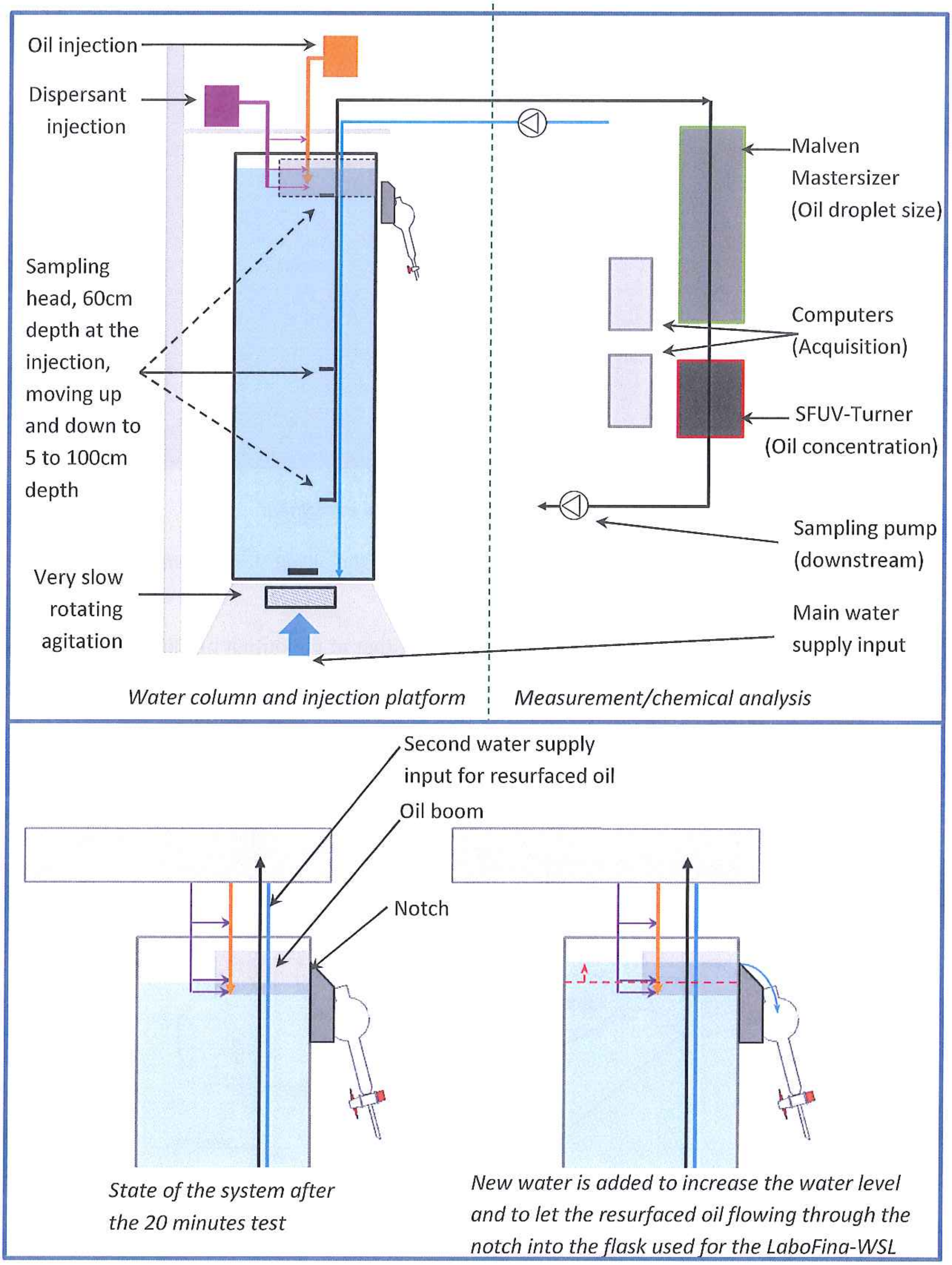
3) Testing equipment description

3.1) The test tank

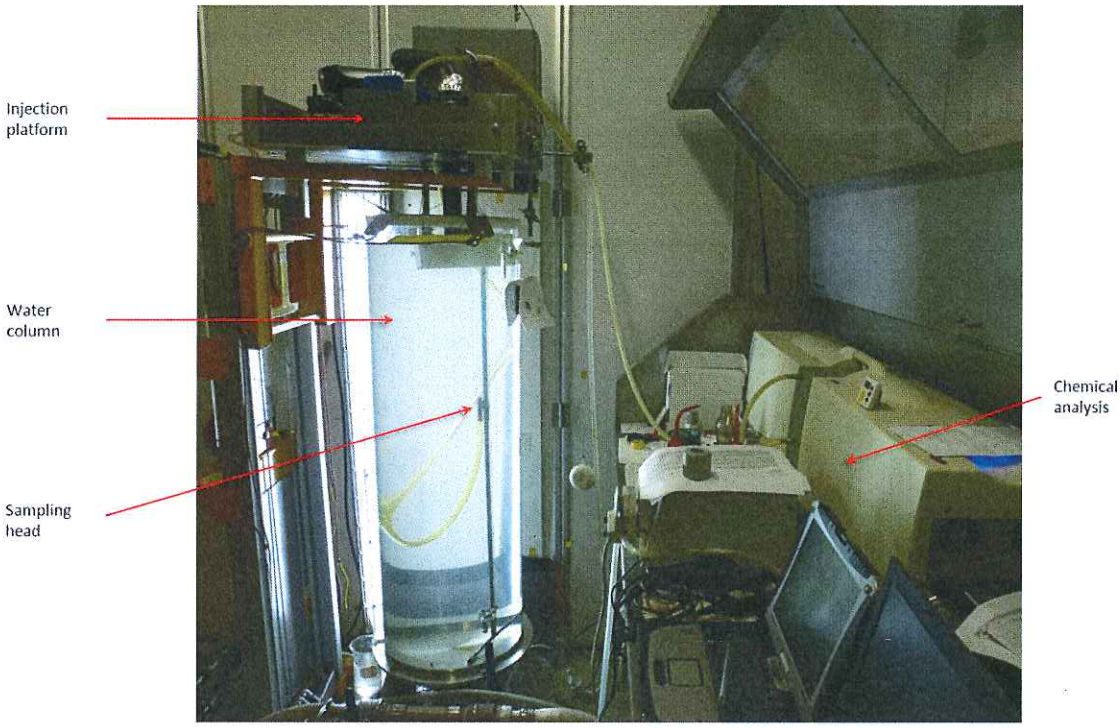
See pictures 1 & 2

The tests were conducted in a Plexiglas column (120cm height & 40cm diameter). A magnetic stirrer at the bottom of the tank maintains the water column rotating very slowly.

The oil was injected downward at 5 cm under the surface water level, through a nozzle 0.8 mm diameter. The oil was driven to the nozzle using a piston pump operating at 50 strokes/second which strokes could be adjusted according to the desired flowrate.



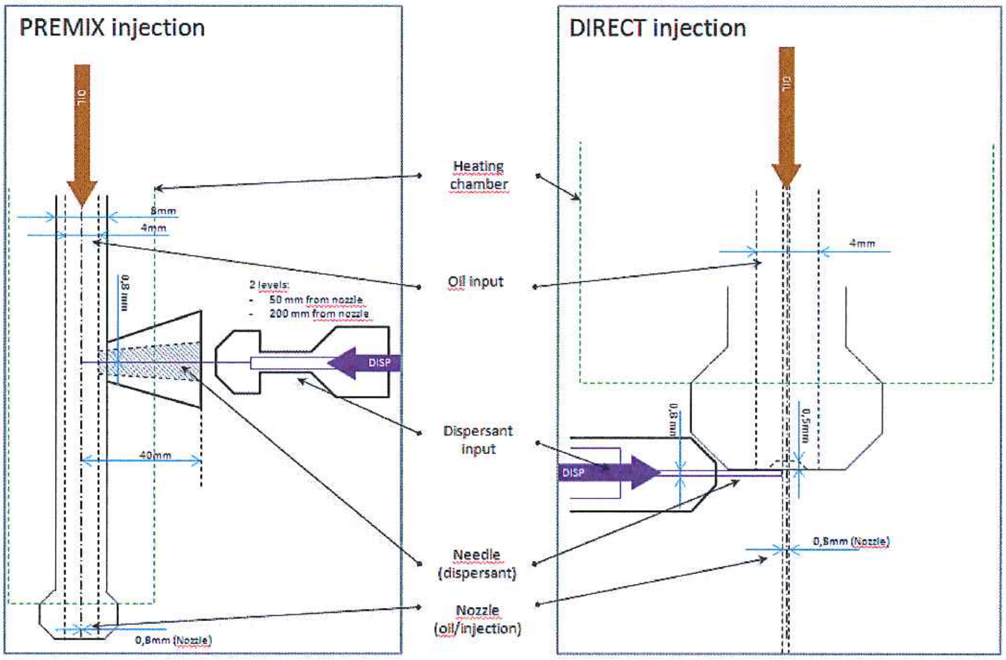
Picture 1: Description of the testing equipment



Picture 2: View of the experimental equipment

In order to heat (when necessary) the oil prior to its injection, the oil injection assembly was enclosed in a chamber which can be heated with a flow of warm air.

The dispersant was added using an automatized syringe either at the output of the whole oil nozzle (<u>D</u>irect <u>I</u>njection) or in the oil pipe 5 cm ahead of the nozzle (<u>P</u>reMix <u>I</u>njection) [cf picture 3]



Picture 3: The two injection modes design

A sampling head slided along a vertical bar in the tank, in order to collect water at different heights in the water column between 10 cm above the bottom and 5 cm under the water surface. The collected water was sent to a *Malvern particle size analyzer,* then to a *spectrofluorometer Turner Design,* in order to assess the dispersed oil droplet size and the dispersed oil concentration.

Slightly above the water level there was a notch in the tank wall. An oblique plate extending down to the water level was set from the notch to the center of the tank. Under the slow rotating movement of the water the oil which resurfaced was gathered along the plate (which acted as an oil boom) and accumulated toward the notch. Filling the tank with some additional water, it was possible to raise the water level up to the notch in order to let the resurfaced oil confined by the plate to flow out through the notch into a flask.

3.2) The laboFina-WSL equipment

The flask which received the resurfaced oil was a regular conical separatory flask used for the LaboFina-WSL dispersant efficiency test.

3.3) Complementary test equipment

The separatory flask, in which the droplet ascension was simulated, was a spherical flask.

4) Products used for the tests

4.1) The oils

The testing oils were Grane, Norne, Oseberg and Kobbe oils supplied by SINTEF; their properties are listed in the table 1.

Table 1: Properties of the testing oil

	Grane 2006-1060	**Norne blend** 2007-0260	**Oseberg blend** 2012-0347	**Kobbe** 2006-1061
Specific gravity (kg/l)	0.941	0.860	0.832	0.797
Pour Point (°C)	-24	21	-6	-36
Viscosity (mPa.s @ 13°C)	640	89	44	22
Asphaltene (wt%)	1.4	0.3	0.3	0.03
Waxes (wt%)	3.2	4.2	3.2	3.4
150°C – Evap loss (vol%)	3	9	22	34
200°C – Evap loss (vol%)	5	18	34	43
250°C – Evap loss (vol%)	13	28	45	54

4.2) The dispersants

The dispersants tested were the Corexit 9500, the Dasic Slickgone NS, the Finasol OSR 62 and the Finasol OSR 52.

On the occasion of the mid-completion meeting, on the 8th of April, when OGP representatives visited *Cedre*, discussions showed that confusion occurred on the choice of the FINASOL dispersant: while SINTEF completed its tests with Finasol OSR 52, *Cedre* used mistakenly Finasol OSR 62 which is a dispersant very close to Finasol OSR 52 in terms of formulation.

As it was no longer possible in terms of deadline and in terms of quantities of testing oils left to reperform the full test matrix with Finasol OSR 52, it has been agreed to complete the test matrix with Finasol OSR 52 and performing few additional tests with Finasol OSR 52 in order to check if results from the 2 dispersants are correlated (in other words, checking if the two dispersants lead to similar results).

5) Protocol

5.1) Testing temperature

As originally agreed at the kickoff meeting in Trondheim (June 2013) the tests were completed at low temperature, 5.2 +/- 0.7 °C.
However, at such a temperature it was not possible to inject the waxy Norne Blend oil (which pour point is 21°C). Therefore, this oil was tested after being heated up to 17.6 +/- 1.2 °C.
In addition, in order to see the possible influence of the injection temperature, an additional test was carried out with the Grane heated at 18 °C to be compared with the a similar test carried out with Grane injected at 5°C.

5.2) Oil injection conditions

The energy level was adjusted by tuning the oil pump flowrate:

- The low oil injection energy level (LE) has been chosen in order to obtain a poor dispersion with the most viscous oil (Grane).
- The high energy level (HE) has been chosen as the highest one which the injection system could achieve.

The viscosity of the oil affects the pump injection flow rate; therefore the duration of the injection has been adjusted for each oil, in order to inject the same oil quantity in each test (between 15 and 20 sec).

Injection conditions were assessed according to the rationale developed by SINTEF in [Oistein Johansen et al. 2013 [1]]. These data are presented for information, in the table 2. The calculations of the Ohnesorge and Reynolds numbers were completed using an Excel sheet supplied by M Nicolas Passade-Boupat from TOTAL. (picture 4)

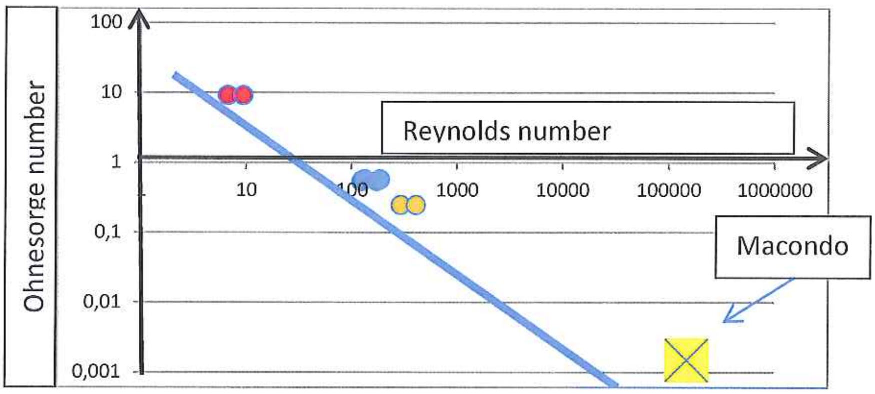
[1] Oistein J., Branddvick P.J., Farooq U.; Droplet breakup in subsea oil releases – Part 2: Predictions of droplet size distributions with and without injection of chemical dispersant- Marine Pollution Bulletin -2013

In this respect:

- The flowrate was considered as the double of the pump flowrate (because the single piston pump can be supposed to pump half time),
- The specific gravity and viscosity used were supplied by Sintef (data@13°C), and corrected to the actual injection temperature (correction made using regular chart viscosity vs temperature).
- The interfacial tension σ was considered as 20 mN/m (which is an order of magnitude which can be encountered in the literature).

Table 2: Oil injection specifications for each oil for Low and High energy level, Diam: nozzle diameter, D: pump flow rate; D_{inst} instantaneous flowrate; viscosity at the injection temperature (calculated) , Sp Grav oil specific gravity, IFT oil (alone) surface tension extrapolated from the literature. Reynold number and Ohnesorge numbers calculated using excel sheet from M Passade Boupat.

Oil	Energy	D Pump L/h	D_{inst} L/h	Diam noz (mm	Sp Grav Kg/m^3	Visco @13°C (cP)	Visco @ inj temp (cP@C°)	IFT (mN/m)	Re	Oh
GRANE	LE	9.0	18.0	0.8	941	640	1130 @ 5	20	6.63	9.21
	HE	12.6	25.2	0.8	941	640	1130 @ 5	20	9.28	9.21
NORNE	LE	10.6	21.2	0.8	860	89	65 @ 18	20	124	0.55
	HE	14.8	29.6	0.8	860	89	65 @ 18	20	173	0.55
OSEBERG	LE	12.0	24.0	0.8	832	44	67 @ 5	20	132	0.58
	HE	16.8	33.6	0.8	832	44	67 @ 5	20	185	0.58
KOBBE	LE	12.0	24.0	0.8	797	22	29 @ 5	20	292	0.25
	HE	16.8	33.6	0.8	797	22	29 @ 5	20	408	0.25



Picture 4: Representation of the injection condition for oil only in the Ohnessorge / Reynold numbers (Norne and Oseberg dot superpose).Dots represent the operating conditions for the 4 oils; red : Grane, green Norne blend; blue: Oseberg blend; Orange Kobbe.

6) Testing procedure

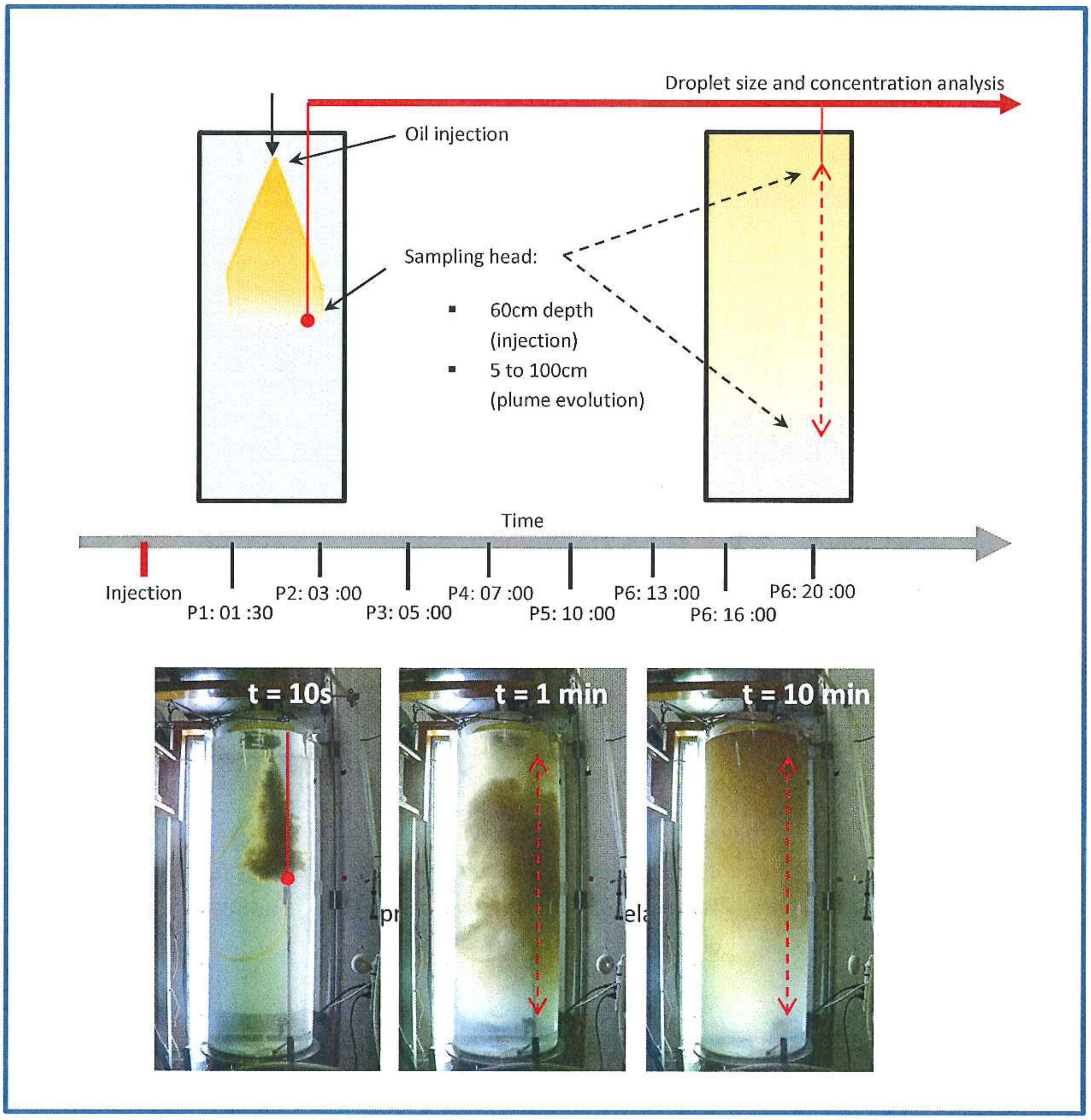
6.1) Preparation

In a first step the background levels of the spectrofluorometer and the particle size analyzer were recorded.

6.2) Dispersed plume monitoring

The test started with the oil and dispersant injection (T0). While the oil and dispersant were injected at appropriate flow rate and during the first minute the sampling head was kept at 60 cm depth in order to monitor the plume characteristics at the injection.

After one minute the sampling head was moved periodically from 5cm to 100 cm deep (20 cm from the bottom of the tank) in order to acquire vertical profiles of the dispersed oil plume (oil concentration and droplet size). For 20 minutes, 8 profiles were done for each test respectively at 1:30, 3:00, 5:00, 7:00, 10:00, 13:00, 16:00 & 20:00 minutes, (duration of a profile ~15s) (picture 5).

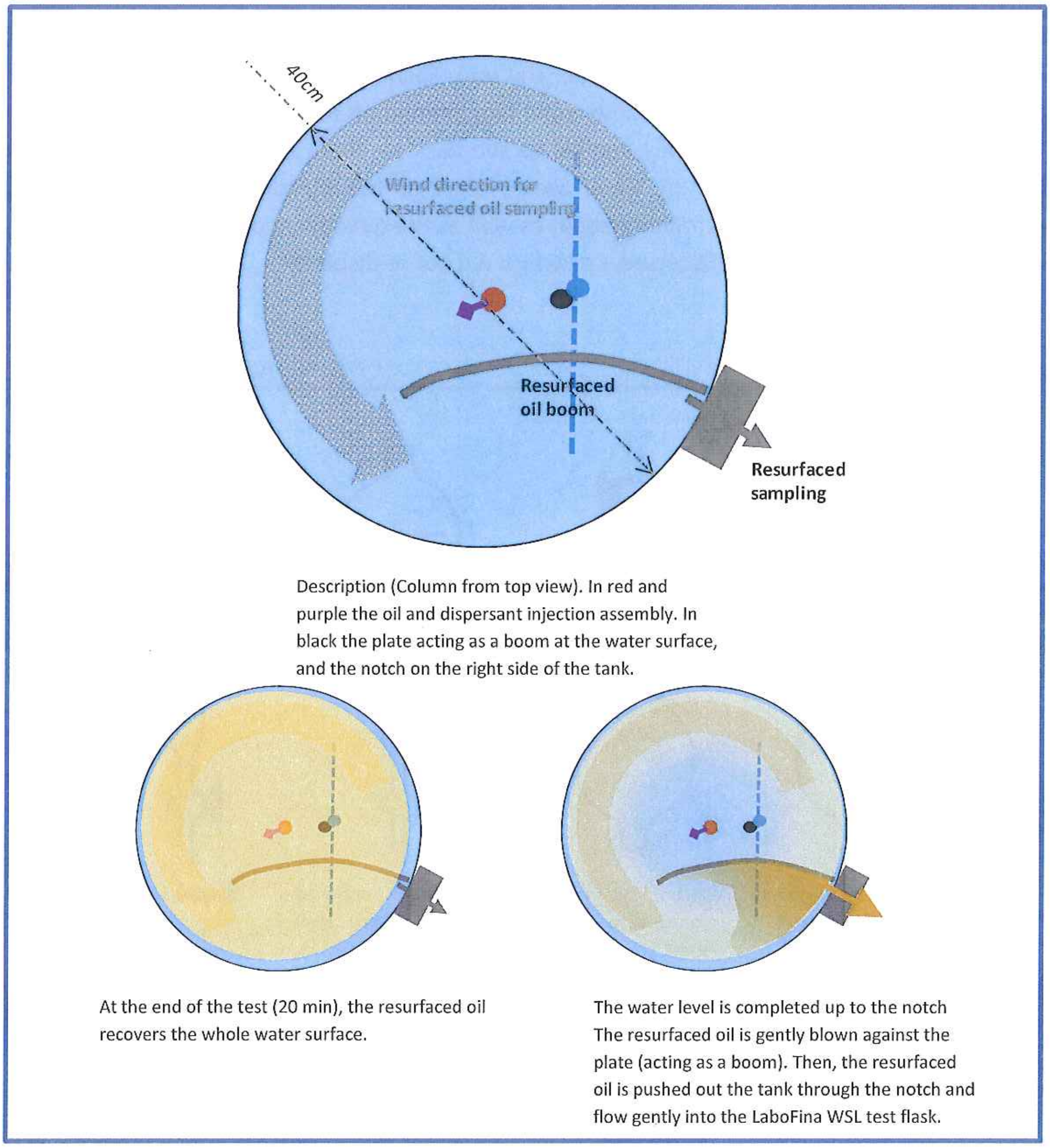


Picture 5: Sampling the plume in the tank; during the first minutes the sampling head monitors the plume at 60 cm deep; then, from 1 to 20 minutes, the sampling head is moved vertically from 5 to 100 cm deep to acquire vertical profiles of the plume

6.3) Resurfaced oil re-dispersiblity assessment

After 20 minutes, when the quantity of resurfaced oil was significant, sea water was added in the tank to raise the water surface level up to the notch. The resurfaced oil which had been gathered against the plate (boom) by the slow rotating stream was gently pushed out of the tank through the notch using an air blow. After passing the notch the oil flew gently into the separatory flask which has been used to complete the LaboFina-WSL test.

The LaboFina WSL test was performed according to the regular procedure giving the bottom sample [sample 1], and, in addition, the rest of the separatory funnel was sampled too [sample 2]. The oil was quantified in the 2 samples in order to assess the whole quantity of oil which had been collected [sample 1 and sample 2] and the part which had been re-dispersed [sample 1].



Description (Column from top view). In red and purple the oil and dispersant injection assembly. In black the plate acting as a boom at the water surface, and the notch on the right side of the tank.

At the end of the test (20 min), the resurfaced oil recovers the whole water surface.

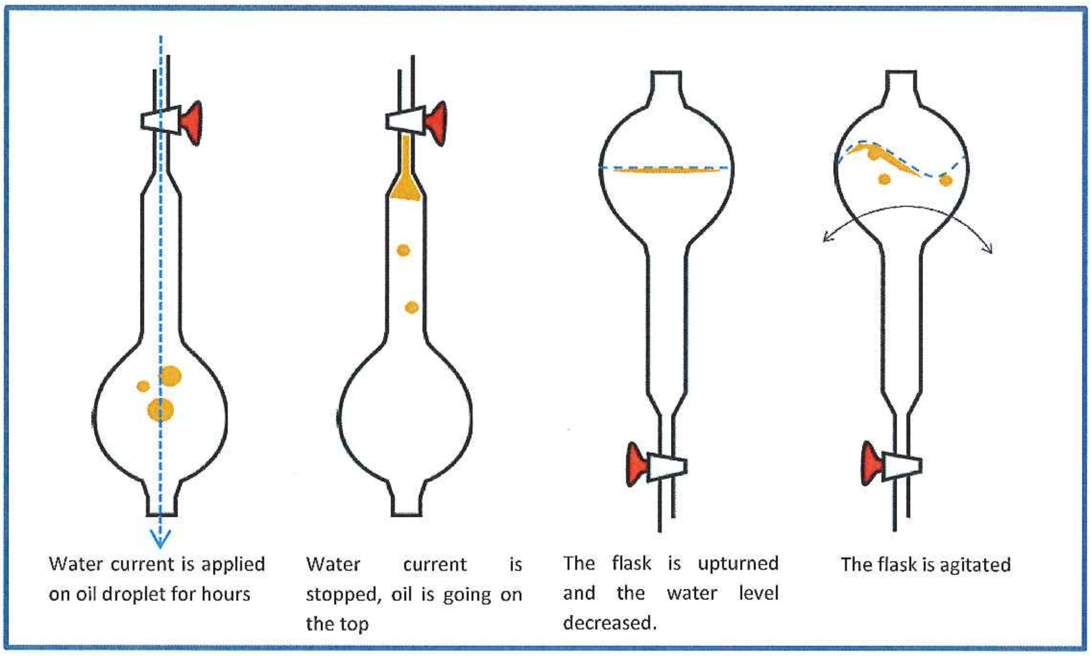
The water level is completed up to the notch The resurfaced oil is gently blown against the plate (acting as a boom). Then, the resurfaced oil is pushed out the tank through the notch and flow gently into the LaboFina WSL test flask.

Picture 6: Collection of the resurfaced oil at the end of the test (20 min)

6.4) Simulation of oil droplets ascent in a long virtual water column

This additional test aimed at simulating the long (several hours) ascent of oil droplets coming back from deep environment to the sea surface.

The simulation was completed in a separatory flask (see picture 7). At the beginning, the flask was set upside down (tap at the top, plug at the bottom). The bottom of the flask was dived in a water basin and a known quantity of oil with or without dispersant (pre-mixed) was gently introduced into the flask full of water. Then water was slowly injected from the top of the flask in order to create a downward stream. This stream was increased progressively to reach the point at which the rising speed of the oil droplet was balanced by the downward stream of water. The oil droplets could be kept in the middle of the flask for several hours, which simulated the ascension of oil droplets from high depth.
At the end of the test, the water flow was stopped and the oil droplets were left free to rise and to accumulate at the top of the flask (just under the tap). Then the flask was gently turned back to its regular position (tap at the bottom). Some water was drained off in order to lower the water level in the flask. Then a slow agitation was applied to the flask to check the oil behavior, especially its possible re-dispersion. Samples of water were taken at the bottom of the flask for further quantification of the dispersion (oil dispersed) as well as the quantification of the total oil left in the flask at the end of the test (oil dispersed + oil which did not re-disperse).



Picture 7: Experimental diagram for the simulation of a long ascent for dispersed oil droplets.

7) Test matrix

The initial test matrix considering, the 3 dispersants, 2 levels of energy and 2 dispersant injection modes (72 tests) is given in the table 3

Table 3: Initial test matrix

OIL	DISP	ENERGY	DOR	INJECTION	n°	OIL	DISP	ENERGY	DOR	INJECTION	n°
GRANE	C 9500	high	1/100	premix	1	NORNE BLEND	C 9500	high	1/100	premix	37
		high	1/100	direct	2			high	1/100	direct	38
		high	1/50	premix	3			high	1/50	premix	39
		high	1/50	direct	4			high	1/50	direct	40
		low	1/20	premix	5			low	1/20	premix	41
		low	1/20	direct	6			low	1/20	direct	42
	OSR62	high	1/100	premix	7		OSR62	high	1/100	premix	43
		high	1/100	direct	8			high	1/100	direct	44
		high	1/50	premix	9			high	1/50	premix	45
		high	1/50	direct	10			high	1/50	direct	46
		low	1/20	premix	11			low	1/20	premix	47
		low	1/20	direct	12			low	1/20	direct	48
	DASIC NS	high	1/100	premix	13		DASIC NS	high	1/100	premix	49
		high	1/100	direct	14			high	1/100	direct	50
		high	1/50	premix	15			high	1/50	premix	51
		high	1/50	direct	16			high	1/50	direct	52
		low	1/20	premix	17			low	1/20	premix	53
		low	1/20	direct	18			low	1/20	direct	54
OSEBERG BLEND	C 9500	high	1/100	premix	19	KOBBE	C 9500	high	1/100	premix	55
		high	1/100	direct	20			high	1/100	direct	56
		high	1/50	premix	21			high	1/50	premix	57
		high	1/50	direct	22			high	1/50	direct	58
		low	1/20	premix	23			low	1/20	premix	59
		low	1/20	direct	24			low	1/20	direct	60
	OSR62	high	1/100	premix	25		OSR62	high	1/100	premix	61
		high	1/100	direct	26			high	1/100	direct	62
		high	1/50	premix	27			high	1/50	premix	63
		high	1/50	direct	28			high	1/50	direct	64
		low	1/20	premix	29			low	1/20	premix	65
		low	1/20	direct	30			low	1/20	direct	66
	DASIC NS	high	1/100	premix	31		DASIC NS	high	1/100	premix	67
		high	1/100	direct	32			high	1/100	direct	68
		high	1/50	premix	33			high	1/50	premix	69
		high	1/50	direct	34			high	1/50	direct	70
		low	1/20	premix	35			low	1/20	premix	71
		low	1/20	direct	36			low	1/20	direct	72

To answer few technical questions raised by the tests completion and taking into account the discussions with OGP representatives from the mid-completion meeting, additional tests have been carried out (table 4):

1) Tests on the 4 oils without any dispersant.
2) A set of tests with the Finasol OSR 52 with the 4 oils at one condition (high energy, DOR 1/50, and premixed injection mode).
3) A test with the Grane preheated at 18°C (in order to check the possible effect of the injection temperature by comparison with the same test completed at 5°C)
4) Tests at extra low energy and extra low DOR (0.7%) to bring out difference of efficiency between the dispersants; these tests were carried out with the Oserberg oil at DOR 1/150 in premix injection mode. In order to reduce the energy level, the oil injection flowrate was reduced to 5 L/h.

Table 4: Additional tests matrix

OIL	DISP	ENERGY	DOR	INJECTION	n°
Grane		high			73
Norne B		high			74
Oseberg B		high			75
Kobbe		high			76
Grane	OSR52	high	1/50	premix	77
Norne B	OSR52	high	1/50	premix	78
Oseberg B	OSR52	high	1/50	premix	79
Kobbe	OSR52	high	1/50	premix	80
Water instead of oil	C95	high	1/50	premix	X
Grane at 18°C	C95	high	1/50	premix	81
Oseberg B	C95	extra low 5 L/h	1/150	*MIXTURE*	82
Oseberg B	OSR62		1/150	*MIXTURE*	83
Oseberg B	DASIC		1/150	*MIXTURE*	84
Oseberg B	OSR52		1/150	*MIXTURE*	85

10 tests were completed to simulate the ascent of oil droplets from high depth (table 5)

Table 5: Test matrix of the tests of simulation of long ascent of oil droplets

Oil	dispersant	Ascent
Grane	Corexit 9500	2h30 ascent
Grane	Corexit 9500	no ascent
Grane	-	2h30 ascent
Oseberg	Corexit 9500	no ascent
Oseberg	Corexit 9500	2h30 ascent
Oseberg	Dasic	no ascent
Oseberg	Dasic	2h30 ascent
Oseberg	Finasol OSR 52	no ascent
Oseberg	Finasol OSR 52	2h30 ascent
Oseberg	Finasol OSR 62	no ascent
Oseberg	Finasol OSR 62	2h30

8) Results

The results are presented in the table 6 (original test matrix) and in table 7 (additional tests).

Table 6: Results of the original tests matrix

n°	OIL	injection temperature	INJECT METH	DISPERSANT	ENERGY	DOR (%)	Efficiency		Median droplet size	
							Subsea dispersion	Resurfaced dispersion	At the injection	After 20 minutes
1	GRANE	5	DI	C95	HE	1	7%	27%	176	131
3	GRANE	5	DI	C95	HE	2	6%	27%	177	145
5	GRANE	5	DI	C95	LE	5	8%	64%	242	88
7	GRANE	5	DI	OSR62	HE	1	5%	19%	183	231
9	GRANE	5	DI	OSR62	HE	2	5%	11%	182	210
11	GRANE	5	DI	OSR62	LE	5	7%	55%	155	50
13	GRANE	5	DI	DASIC	HE	1	29%	34%	47	91
15	GRANE	5	DI	DASIC	HE	2	33%	31%	108	170
17	GRANE	5	DI	DASIC	LE	5	5%	48%	273	192
2	GRANE	5	PM	C95	HE	1	38%	68%	119	99
4	GRANE	5	PM	C95	HE	2	35%		121	106
6	GRANE	5	PM	C95	LE	5	29%		129	100
8	GRANE	5	PM	OSR62	HE	1	27%	53%	135	116
10	GRANE	5	PM	OSR62	HE	2	27%		132	110
12	GRANE	5	PM	OSR62	LE	5	30%		129	107
14	GRANE	5	PM	DASIC	HE	1	22%	55%	123	103
16	GRANE	5	PM	DASIC	HE	2	25%		117	92
18	GRANE	5	PM	DASIC	LE	5	20%		126	104
19	*NORNE BLEND**	*18*	DI	C95	HE	1	74%	62%	26	66
21	*NORNE BLEND**	*18*	DI	C95	HE	2	89%		14	29
23	*NORNE BLEND**	*18*	DI	C95	LE	5	91%		31	39
25	*NORNE BLEND**	*18*	DI	OSR62	HE	1	65%	59%	13	85
27	*NORNE BLEND**	*18*	DI	OSR62	HE	2	80%		13	82
29	*NORNE BLEND**	*18*	DI	OSR62	LE	5	81%		22	50
31	*NORNE BLEND**	*18*	DI	DASIC	HE	1	73%	74%	15	40
33	*NORNE BLEND**	*18*	DI	DASIC	HE	2	83%		15	30
35	*NORNE BLEND**	*18*	DI	DASIC	LE	5	88%		11	27
20	*NORNE BLEND**	*18*	PM	C95	HE	1	91%		11	27
22	*NORNE BLEND**	*18*	PM	C95	HE	2	95%		15	31
24	*NORNE BLEND**	*18*	PM	C95	LE	5	91%		17	31
26	*NORNE BLEND**	*18*	PM	OSR62	HE	1	86%		16	28
28	*NORNE BLEND**	*18*	PM	OSR62	HE	2	88%		19	32
30	*NORNE BLEND**	*18*	PM	OSR62	LE	5	89%		15	36
32	*NORNE BLEND**	*18*	PM	DASIC	HE	1	89%		18	35
34	*NORNE BLEND**	*18*	PM	DASIC	HE	2	88%		22	31
36	*NORNE BLEND**	*18*	PM	DASIC	LE	5	90%		18	29
37	OSEBERG BLEND	5	DI	C95	HE	1	89%		7	14
39	OSEBERG BLEND	5	DI	C95	HE	2	92%		9	13
41	OSEBERG BLEND	5	DI	C95	LE	5	92%		14	15
43	OSEBERG BLEND	5	DI	OSR62	HE	1	89%		13	34
45	OSEBERG BLEND	5	DI	OSR62	HE	2	89%		15	20
47	OSEBERG BLEND	5	DI	OSR62	LE	5	87%		6	13
49	OSEBERG BLEND	5	DI	DASIC	HE	1	81%		10	21
51	OSEBERG BLEND	5	DI	DASIC	HE	2	85%		9	8
53	OSEBERG BLEND	5	DI	DASIC	LE	5	92%		9	13
38	OSEBERG BLEND	5	PM	C95	HE	1	87%		13	17
40	OSEBERG BLEND	5	PM	C95	HE	2	83%		12	16
42	OSEBERG BLEND	5	PM	C95	LE	5	97%		16	16
44	OSEBERG BLEND	5	PM	OSR62	HE	1	92%		13	16
46	OSEBERG BLEND	5	PM	OSR62	HE	2	83%		13	17
48	OSEBERG BLEND	5	PM	OSR62	LE	5	75%		15	15
50	OSEBERG BLEND	5	PM	DASIC	HE	1	95%		13	17
52	OSEBERG BLEND	5	PM	DASIC	HE	2	89%		12	16
54	OSEBERG BLEND	5	PM	DASIC	LE	5	92%		16	16
55	KOBBE	5	DI	C95	HE	1	90%		14	26
57	KOBBE	5	DI	C95	HE	2	91%		6	27
59	KOBBE	5	DI	C95	LE	5	90%		11	16
61	KOBBE	5	DI	OSR62	HE	1	91%		17	34
63	KOBBE	5	DI	OSR62	HE	2	91%		13	32
65	KOBBE	5	DI	OSR62	LE	5	91%		10	12
67	KOBBE	5	DI	DASIC	HE	1	91%		14	29
69	KOBBE	5	DI	DASIC	HE	2	90%		12	17
71	KOBBE	5	DI	DASIC	LE	5	91%		8	23
56	KOBBE	5	PM	C95	HE	1	93%		10	9
58	KOBBE	5	PM	C95	HE	2	95%		9	9
60	KOBBE	5	PM	C95	LE	5	93%		8	8
62	KOBBE	5	PM	OSR62	HE	1	93%		9	10
64	KOBBE	5	PM	OSR62	HE	2	92%		9	9
66	KOBBE	5	PM	OSR62	LE	5	90%		8	7
68	KOBBE	5	PM	DASIC	HE	1	89%		10	9
70	KOBBE	5	PM	DASIC	HE	2	93%		9	9
72	KOBBE	5	PM	DASIC	LE	5	90%		8	8

Table 7: Results of the additional tests

n°	OIL	Injection temperature	DISPERSANT	INJECT METH	ENERGY	DOR (%)	Efficiency: Subsea dispersion	Efficiency: Resurfaced dispersion	Median droplet size: At the injection	Median droplet size: After 20 minutes
73	GRANE	5			HE	0	3%		267	100
74	NORNE BLEND	5			HE	0	33%		39	30
75	OSEBERG BLEND	5			HE	0	35%		45	37
76	KOBBE	5			HE	0	29%		31	27
77	GRANE	5	OSR52	PM	HE	2	19%		146	121
78	NORNE BLEND	5	OSR52	PM	HE	2	88%		14	25
79	OSEBERG BLEND	5	OSR52	PM	HE	2	90%		8	10
80	KOBBE	5	OSR52	PM	HE	2	91%		10	9
81	GRANE	18	C95	PM	HE	2	78%		49	
82	OSEBERG BLEND	5	C95	PM	HE	0,7	66%		61	79
83	OSEBERG BLEND	5	DASIC	PM	HE	0,7	38%		75	103
84	OSEBERG BLEND	5	OSR62	PM	HE	0,7	35%		83	111
85	OSEBERG BLEND	5	OSR52	PM	HE	0,7	44%		70	105

9) Legends

All the pictures of the report use the same color code according to the oil:

Tests on GRANE oil are figured in **RED**

Tests on NORNE BLEND oil are figured in **GREEN**

Tests on OSERBERG oil are figured in **BLUE**

Tests on KOBBE oil are figured in **YELLOW**

Tests of the regular matrix (Corexit 9500, Dasic Slickgone, Finasol OSR62) are figured by a dot ●, ●, ●, ●,

Tests completed with Finasol OSR52 are figured as triangle, Δ, Δ, Δ, Δ

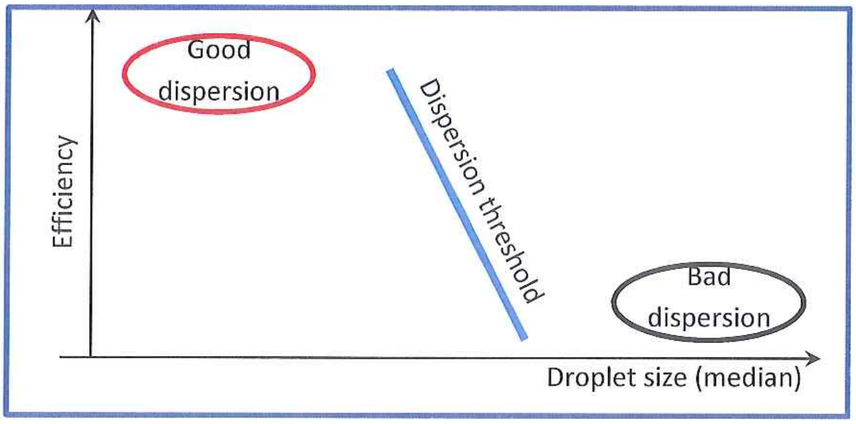
Tests without dispersants are figured as open diamond ◊, ◊, ◊, ◊

10) Efficiency definition

Efficiency refers to the ratio between the quantity of oil initially injected in the tank and the quantity of oil which is still present in the water column after 20 minutes, as assessed by integrating the last vertical profile of oil concentration carried out at the end of the test.

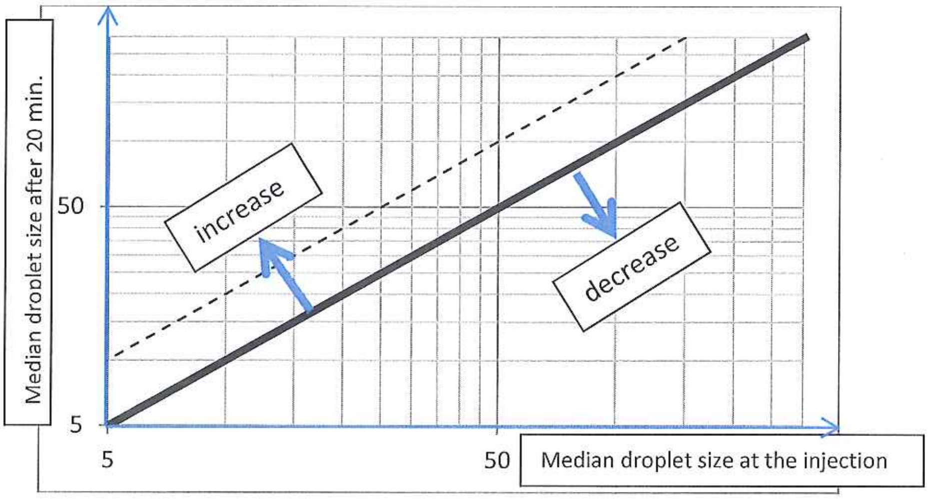
11) Representation of the results

Most of the results are presented on charts showing the efficiency versus the oil droplet size (picture 8). In these charts, a good dispersion which can be characterized as having high efficiency and a small droplet size will be located in the left upper part of the chart while a poor dispersion which can be characterized as having a low efficiency and large droplet size will be located at the right lower part of the chart.



Picture 8: Principle of the chart efficiency versus median oil droplet diameter

Another parameter assessed in this study is the stability of the oil droplet size along the test (20 min) which can be illustrated using diagrams droplet size at 20 min versus initial droplet size. The position of the dots with regards to the line 1:1 (in black bolt) indicates if the droplet size increases or decreases during the 20 min test (picture 9).



Picture 9: Principle of the chart showing the evolution of the droplet size between the injection and the end of the test (20 min)

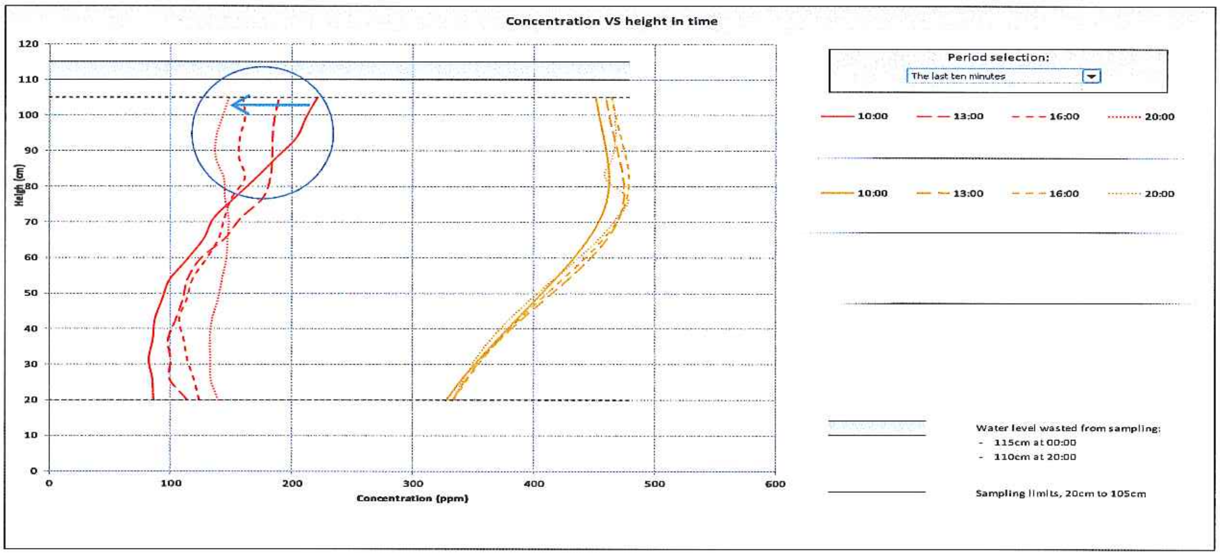
Lastly, the presentation of the results at the meeting held in June 2014, at IPIECA facilities in London, showed that Cedre and Sintef presented their results in different ways. In order to help the reader to compare these two similar laboratory studies, Cedre reworked afterwards its results to present them in the same format as Sintef. Cedre's results in Sintef's format are presented in the Annex 2.

12) Results exploitation & interpretation

12.1) Observation of the tests: evolution of the oil concentration in the water column and of the oil droplet size distribution.

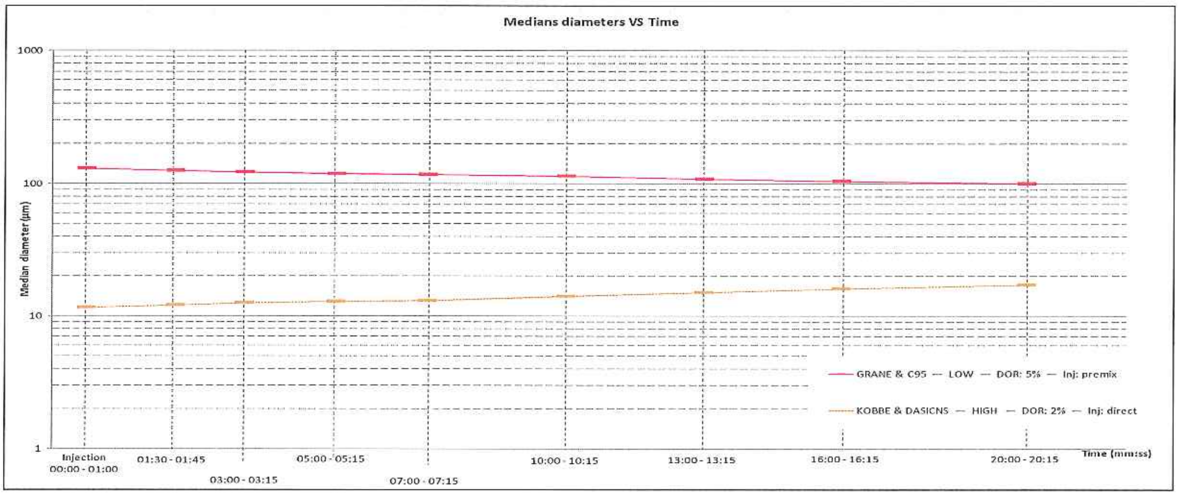
Picture 10 shows the evolution of the oil concentration in the water column through the successive vertical profiles from the beginning to the end of the test (20 min after injection), for the Grane, in red (poor dispersion), and for the Kobbe, in yellow (good dispersion).

In the case of a good dispersion, as expected, the dispersed oil plume stays in the whole water column (no evolution of the profile). For a poor dispersion, the oil concentration in the water column kept much lower from the beginning and the evolution of the successive profiles shows that a large part of the oil resurfaces (decrease of the oil concentration of the upper part of the profile).



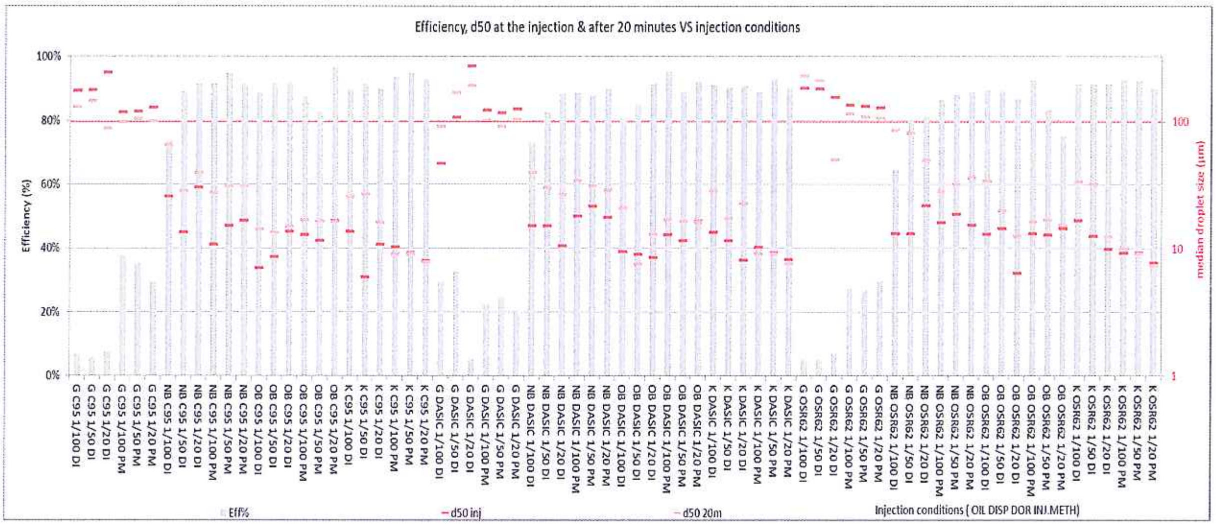
Picture10: Example of a good and a poor dispersion: vertical profiles of dispersed oil concentration in the tank. In red test on Grane & C95 at 5% with PREMIX injection mode, in orange/yellow test on Kobbe & DASIC at 2% with DIRECT injection mode.

When considering the evolution of the oil droplet size distribution, in case of a good dispersion despite a small increase of the droplet size along the 20 minutes of the test, the median droplet size kept much smaller than in a poor dispersion. It should be noted that in a poor dispersion the droplet size tended to reduce progressively during the test due to the loss of the large droplets which resurfaced quickly (only the smallest ones remained in the water column) (Picture 11).



Picture 11: Median droplet size vs time (by profile). In red test on Grane & C95 at 5% with PREMIX injection mode, in orange test on Kobbe & DASIC at 2% with DIRECT injection mode.

Lastly, the droplet size was globally well correlated with the efficiency as shown by picture 12. The largest droplets (around 100 µm) matched with the lowest efficiency (<40%), especially for the tests with Grane oil, while the other tests for which the efficiency is high (80% and more) presented much smaller droplets (for initial droplets at the injection time, from 6 to 31 µm).



Picture 12: Global view of the test results corresponding to the matrix. Grey bars: efficiency, bold red dash, median droplet size at the injection, pale red dash median droplet size at the end of the test (20 min).

12.2) Injection temperature

In order to assess the effect of the oil temperature at the injection, a test with Grane was repeated at 18°C. As expected, the dispersibilty was greater at 18°C (droplet size: 50µm, efficiency: 78%) than at 5°C (droplet size: 120µm, efficiency: 35%), due to the reduction of oil viscosity while the temperature increases (Grane viscosity: 4°C; 1443 cP, 18°C; 641 cP); see table 8.

Table 8: Effect of the temperature

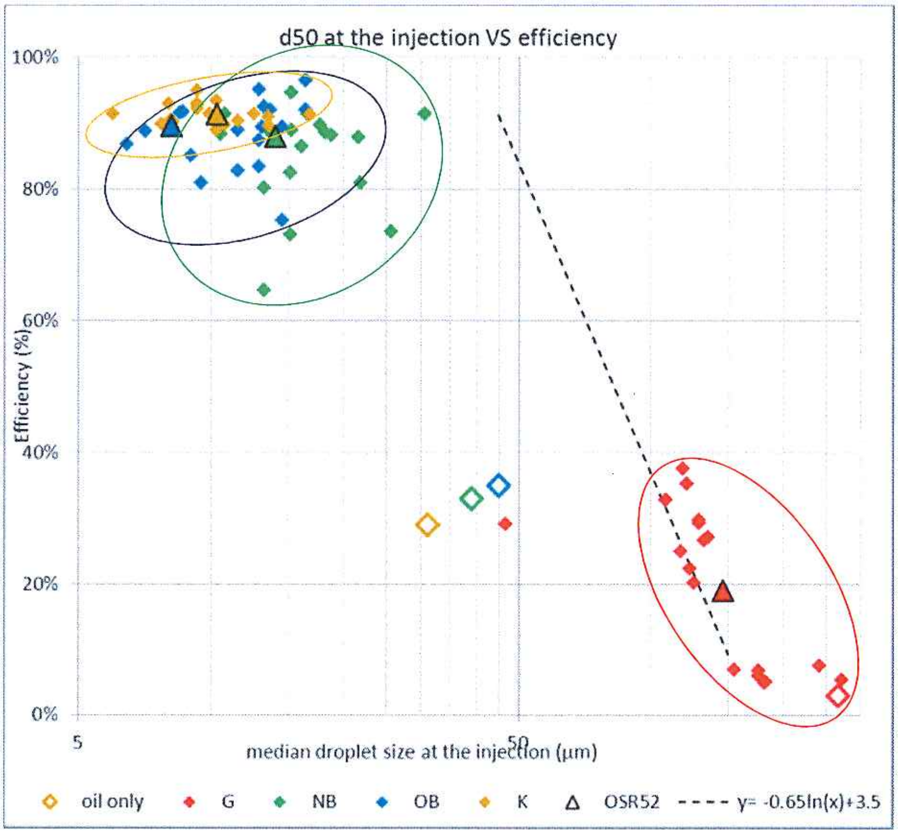
Oil	dispersant	temperature °C	DOR %	Energy	injection mode	Efficiency %	Initial droplet diameter µm
GRANE	Corexit 9500	5	2	HE	Premixed	35	121
		18				78	49

The following chapters consider the effect of the different parameters on the quality of the dispersion.

12.3) Oil type

Regarding the quality of the dispersion, the oil type and characteristics were obviously the most influent parameter.

There were clear differences between the oils: the Oseberg blend, Kobbe (at 4°C) and Norne blend (at 18°C) gave a good dispersion in the different testing conditions (Energy, DOR, injection mode), whereas the Grane remained difficult to disperse. With the Grane, the efficiency was low and the oil droplet size was larger than for the 3 other oils. This could be mainly attributed to the viscosity which was much higher for the Grane (picture 13).



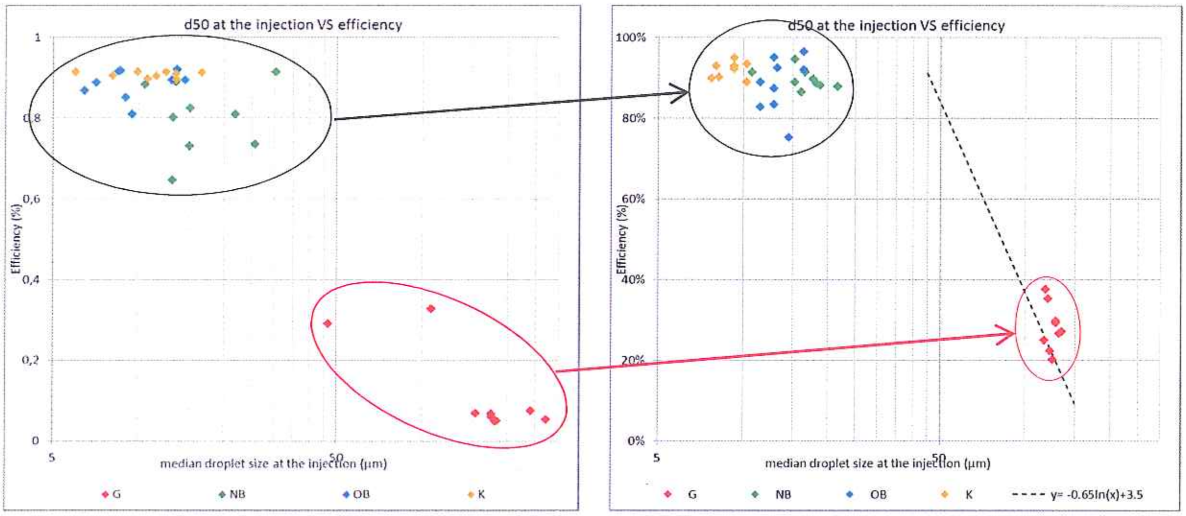
Picture 13: For all the tests, the efficiency vs the median droplet size at the injection

12.4) Injection method

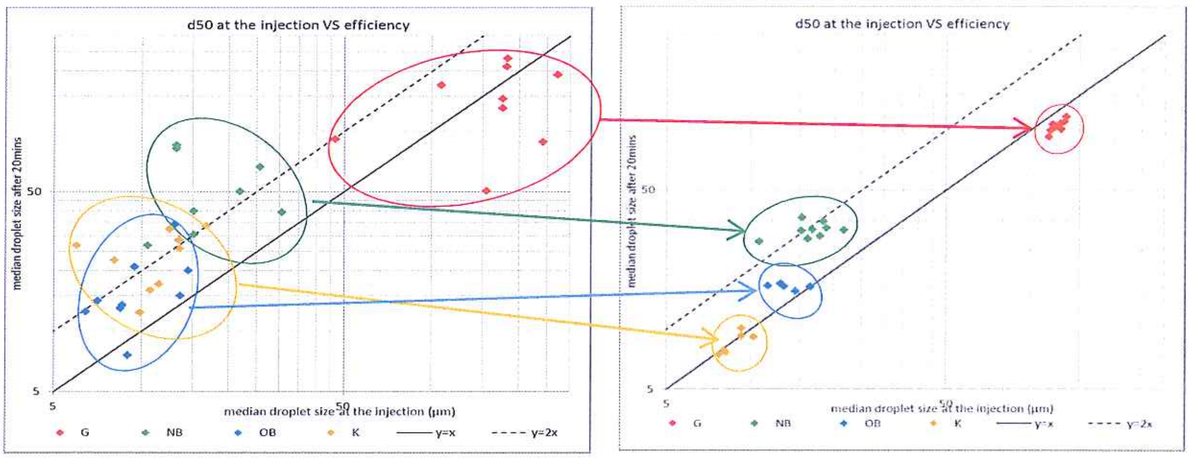
Tests completed with the PREMIX injection mode (dispersant added upstream the oil injection nozzle) showed a better reproducibility and proved to be more stable (along the 20 minutes of the test) than those completed with the DIRECT injection mode (picture 14 & 15):

- With the Direct Injection mode, the dispersion was less stable, and droplet size could increase enabling the final droplet median size to be between x1 to x5 times the original median size (Test with Oseberg, Norne blend and Kobbe),
- For the Grane, as already stated, the diminution of the median droplet size could be attributed to the loss of the largest droplets which rose to the surface and so, disappeared from the droplet size distribution.

These observations resulted from a lesser quality of the mixing between the oil and the dispersant when the dispersant was added downstream the nozzle.



Picture 14: Droplet size at the injection vs efficiency for Direct Injection (left) and Premix injection modes (right).

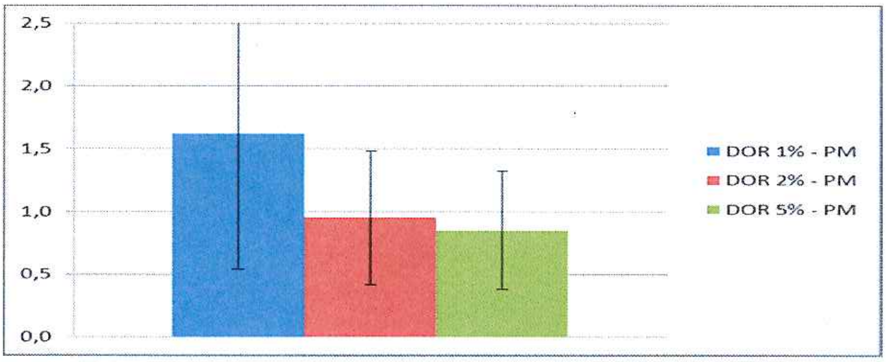


Picture 15: Droplet size evolution between injection and 20 min. for Direct Injection (left) and Premix injection modes (right).

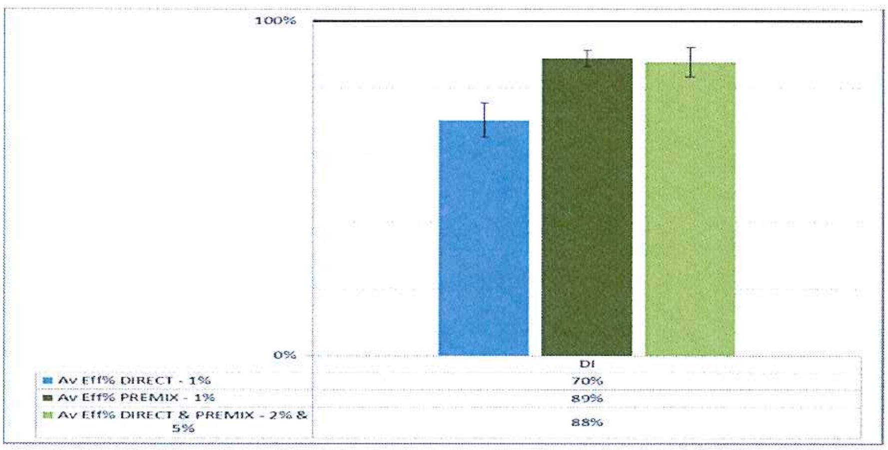
12.5) DOR & associated energy level

When looking at the whole set of tests results, the variation which could be attributed to the DOR and to the energy levels were hidden by the large variations due to the oil type and the injection mode which are the main parameter): no real difference on the efficiency or on the droplet size, attributable to the DOR / energy level could be highlighted.

In order to find differences between the 3 DORs, it has been necessary to consider only the tests with Direct Injection mode (as there are more discrepancies in the results within this group) and the differences observed within each oil subgroup. This has been done on the droplet stability (size evolution between the injection and 20 min) on the tests of the initial matrix: the absolute difference between the beginning and the end of each test has been divided by the average of the difference between initial and 20 min of its subgroup "oil" (12 tests/group). Finally, the average of this last value has been calculated for each DOR (picture 16).



Picture 16: For Direct Injection mode, average of the droplet size evolution by DOR (divided by the average by oil)



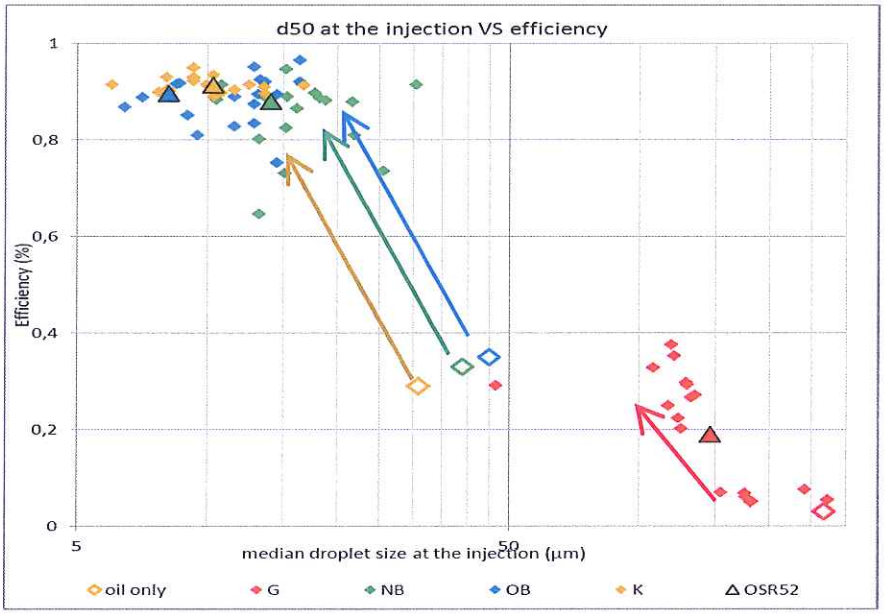
Picture 17: For the Norne blend, efficiency according to the DOR: in blue the average efficiency for DOR 1%/ HE in DIRECT injection mode, in dark green the average efficiency for DOR 1% / HE in PREMIX injection mode, and in green the average for all other test on Norne blend (PREMIX and DIRECT, DOR 2 / HE & 5%/ LE).

The droplet variation at DOR 2% / HE and 5% / LE were not really different, whereas they are much more different from the droplet variation at DOR 1% / HE, although remaining inside the confidence interval. This could be considered as an indication that there was probably a higher ability for coalescence at DOR 1%.

When considering only the tests on Norne blend, we can observe differences in efficiency between the DOR 1% / HE in Direct Injection mode and the DOR 1% / HE in Premix injection modes and also the other DOR at 2% / HE and 5 % / LE for any injection mode: on the picture 17, the average efficiency for DOR 1% / HE in Direct Injection mode (blue) is significantly lower than the average efficiency of the same DOR in Premix injection mode (dark green), or the average efficiency of all other tests conducted at DOR 2 / HE and 5% / LE (soft green)

12.6) Oil only vs oil and dispersant

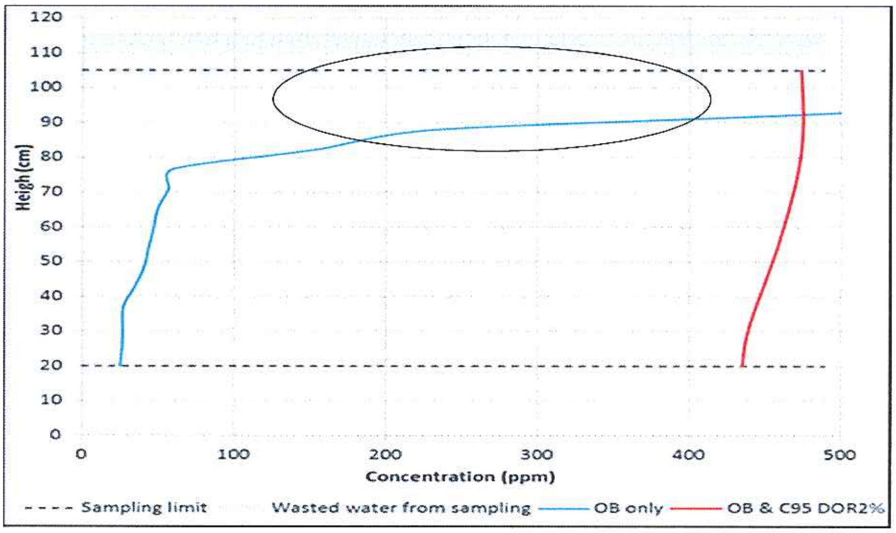
The picture 18 shows the effect of adding dispersant: for each oil, the tests without dispersant or oil alone (open diamond), clearly differentiated from the tests with dispersant (bold triangle): the use of dispersant moved the dots towards the good efficiency area.



Picture 18: Median droplet size at the injection vs the efficiency for all tests (open marker is oil only)

Without dispersant (oil only), even when the dispersion looked fine enough at the beginning of the test (d50 < 50µm for Norne, Oseberg blend and Kobbe), re-coalescence occurred and led finally to a low efficiency (< 40%). Application of dispersant increased the efficiency (increase to ~90%) which confirmed the ability of dispersants to prevent (or to reduce) the coalescence process (see an example on picture 19).

Remark: a previous study carried out at CEDRE showed that coalescence is observed during the very first minutes, while the plume is still under turbulent regime. It also showed that under approximately 100 ppm re-coalescence is not significant anymore.



Picture 19: Comparison of dispersed oil profiles at the end of the test for Oseberg blend alone (blue) and with Corexit 9500 at 2% (red). Without dispersant the dispersed oil concentration remained much lower and this oil kept close to the surface.

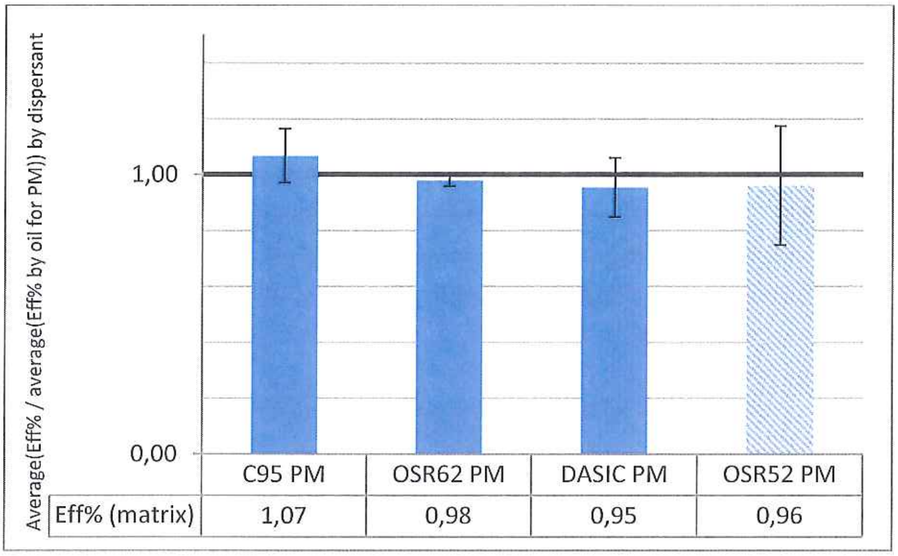
12.7) Dispersant brand

As previously seen, the effect of the parameters -oil type- and -injection mode- hid the effect of the other parameters.

In order to highlight differences between the dispersants, it was necessary to consider only the tests performed with the Premix Injection mode, for which the standard deviation was much lower than for the Direct Injection mode and to normalize the efficiency on each oil: to get comparable values, we considered the efficiency of each test related to the average of the efficiency of all the tests completed on the same oil with the 3 dispersants [9tests]. Then it was possible to pool these relative efficiencies to get an average relative efficiency for each dispersant (see Picture 20, blue columns).

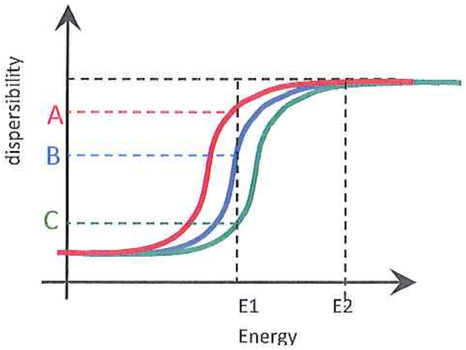
In addition, considering Finasol ORS 52 dispersant, tests were performed with the 4 oils at one testing condition -Premix, HE, DOR 2%- (4 tests). The values of efficiency of these tests have been divided by the average efficiency of all tests performed on each oil (see Picture 20, pale blue columns).

There was no significant difference between the 4 dispersants. However the Corexit 9500 seems to be slightly more efficient than the 3 others. The 3 other dispersants kept close to each other. All results are in the uncertainty interval.



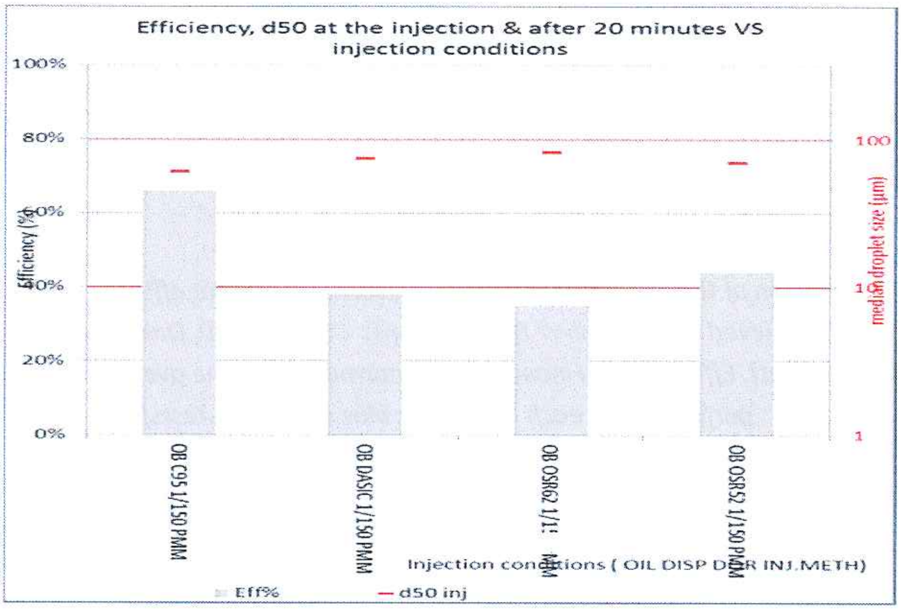
Picture 20: Comparison of the efficiency of each dispersant: Dispersant efficiency normalized to the average efficiency achieved on each oil => Blue columns: Corexit 9500, Dasic Slickgone, Finasol OSR 62 [12 tests / dispersant]. Efficiency of Finasol OSR 52 normalized to the average efficiency of all tests performed on each oil => Pale blue column [4 tests].

In terms of energy level, dispersion process was a "threshold process": the dispersibility was either low under the threshold value or high over this value with a sharp change from low to high (picture 21). This threshold energy level is dependent on the oil type. In this experimental program the lower energy level has been chosen high enough to disperse, even coarsely, the less dispersible oil (Grane). Therefore this level was too high for the 3 other oils which were much lighter.



Picture 21: General diagram; principle of the relation between energy and dispersibility : Considering different dispersants(A,B,C), red, blue, green. The level of energy should be adjusted at E1 to see differences between 3 dispersants for which results are close.

In order to rank clearly the dispersants it would have been better to choose specific testing conditions for each oil, especially, an appropriate energy level, which would be close to the threshold energy level of the oil.
Therefore, in order to reveal differences between dispersants, additional tests have been completed on one of the easily dispersible oils, Oseberg, but using an extra low energy and an extra low DOR (0.7%). In these last tests, in terms of efficiency, the ranking between dispersants was more observable: Corexit kept higher than the others which kept close together in the following order, Finasol OSR52, then Dasic, and at last Finasol OSR 62. Picture 22 gives the efficiency and the initial median droplet size for these tests.



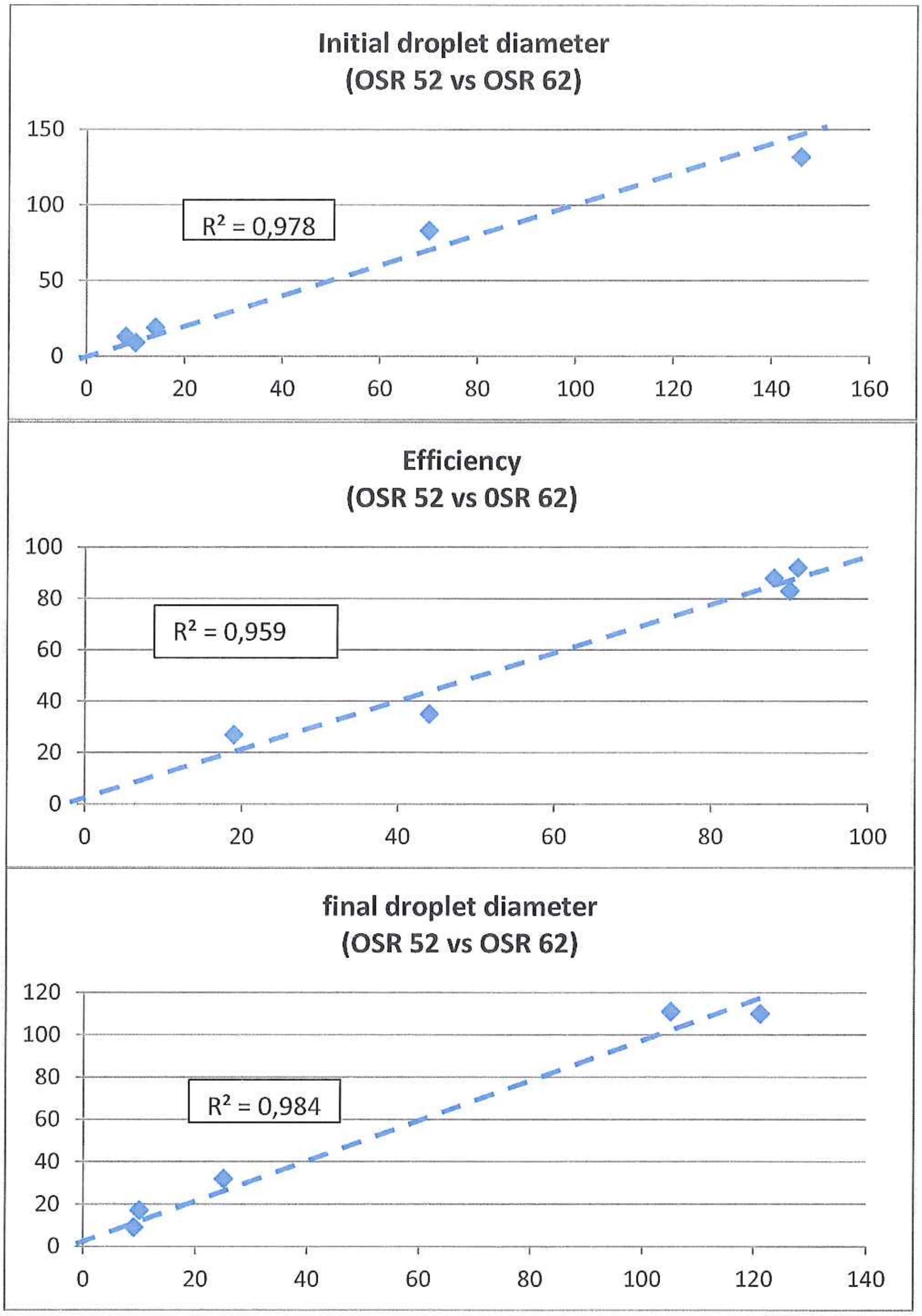
Picture 22: Efficiency and median droplet size at the injection according to the injection conditions for the 4 dispersants (C95, DASIC, OSR 62 & 52) on the Oseberg blend at DOR 0,7% and extra low energy.

12.8) Comparison between Finasol OSR62 and Finasol OSR52

As the study has been completed using mainly Finasol OSR62 instead of Finasol OSR 52, it was necessary to compare the tests results obtained from these two dispersants.

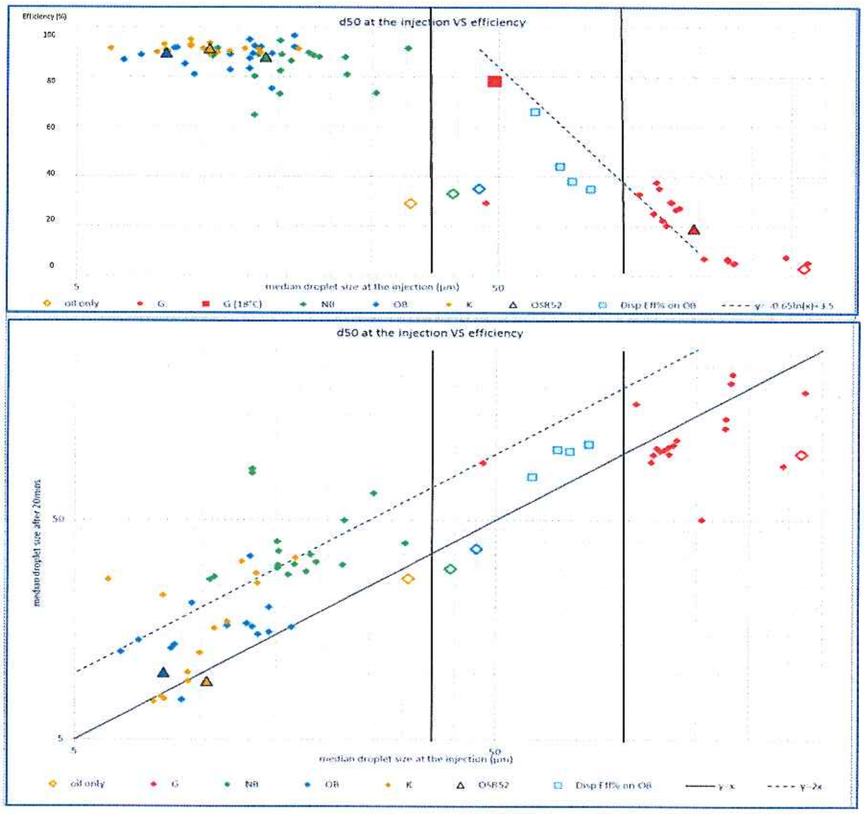
The 5 series of tests completed with these two dispersants were compared for the 3 main parameters: efficiency, initial droplet diameter and final droplet diameter.

This comparison which is illustrated in the picture 23, shows the two products give very similar results and are very well corralled (r > 0.95). This observation confirms that both products are quite similar and, in these testing conditions, show very similar performance.



Picture 23: Comparison of the results obtained on Finasol OSR 62 and Finasol OSR 5, (dotted line y=x).

12.9) Relation between the efficiency and the initial droplet size, and between the initial and final droplet size



Picture 24: Relations between the efficiency and the initial droplet size, and between the droplet sizes initial and final (20 min).

Considering the initial droplet size (at the injection), we could see that, under 35 µm, the efficiency of chemical dispersion was good. On the other hand, low efficiency matched with droplet size higher than 100. (picture 24). There was almost no data in between 35 and 100.

The droplet size evolution along the test duration (difference between the median diameter at the injection and the median diameter at 20 min) remained low and did not provide much additional information.

In the Annex 2, the tests results are presented under the format used by Sintef; the performances reached by the different tested dispersants are displayed on the same chart:

a) The relative distributions of the initial oil droplet size for each dispersant in volume.
b) The efficiency measured for each dispersant.
c) The residual for the droplet size distribution of each dispersant. (This value which exists for any droplet size analyzer, gives an idea of the quality of the distribution).

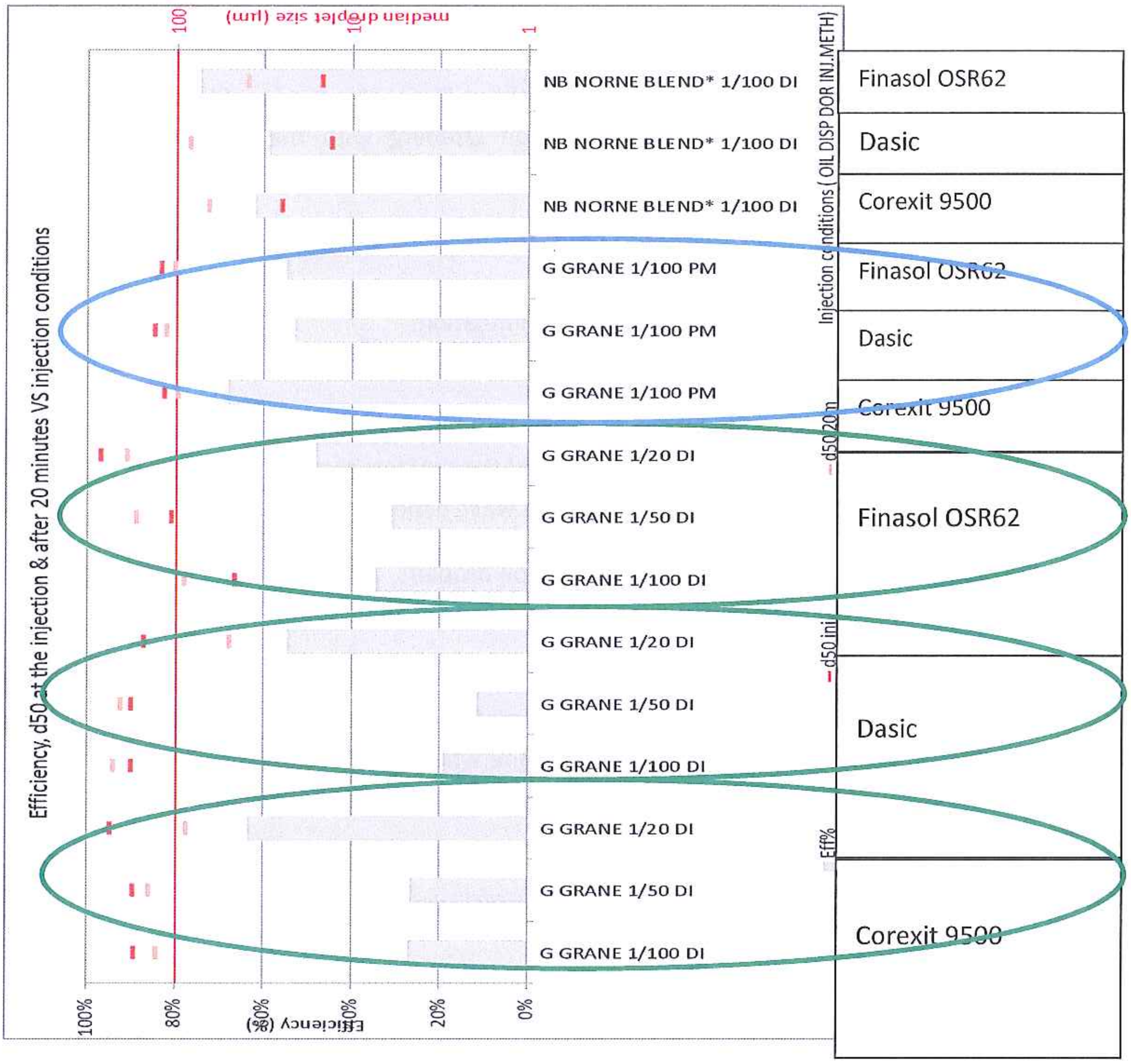
Globally, these results confirm those presented in the report. However, it can be seen on few charts some discrepancies between the ranking of the dispersants according to the oil droplet size distribution and according to the measurement of the efficiency.

The residual values show that, on few oil droplet distributions, the confidence level can be weak. Therefore, in order to better understand the confidence level of the test results it seems important to indicate the residual of the droplet size measurement. More, in this respect, the use of both droplet size measurements and oil concentration measurements leads to increase the reliability of the results.

12.10) Resurfaced oil dispersion results (LaboFinaWSL)

For all the tests with Kobbe, Oseberg and for most of the tests with Norne, there was no oil resurfacing during the test as the dispersed oil plume was stable enough to remain in the water column during the 20 minutes of the test. Only the tests ran with the Grane and few tests ran with the Norne Blend in Direct Injection led to significant quantities of oil which resurfaced during the test duration.

For these last tests, this resurfaced oil was collected and its re-dispersability was measured using the Labo-Fina test method (rotating flask). The picture 25 shows the result of these last tests.



Picture 25: Results of LaboFina-WSL for the dispersion of the resurfaced oil

The oil which resurfaced remained dispersible with efficiency results higher or close to 60% except for the Grane at low dosage (DOR = 1 and 2%) in Direct Injection mode (green circles). In these cases, these low re-dispersibility confirmed that with Direct Injection mode, the amount of dispersant which succeeded to mix actually with the oil was too low. In the same tests carried out in Premix Injection mode, the dispersant appeared to be properly incorporated into the oil, and the oil appeared easily re-dispersible (blue circle).

For Norne, which dispersion was much easier than the one of the Grane, there was significant quantity of resurfaced oil at the end of the test only for the Direct Injection mode and at the lowest DOR; however the resurfaced oil was easily re-dispersible.

Considering the good re-dispersibility observed in these last tests, it should be kept in mind that the LaboFina-WSL test method is a high energy test. Such results may not be achieved if a low energy test such as the IFP flow-through test. However, globally, in this experimental model, the oil which resurfaces seemed to be easily dispersible if the dispersant had been well mixed with it.

12.11) Simulation of the oil droplet ascent in a long virtual water column

These tests were carried out on Grane and Oseberg oils. The first tests which were used to set the testing protocol were carried out on Grane with and without dispersant (Corexit 9500) and consisted in visual observations. The other tests completed on Oseberg with the 4 dispersants led to quantification of:

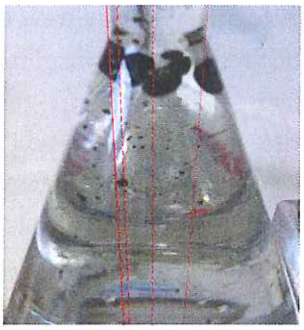
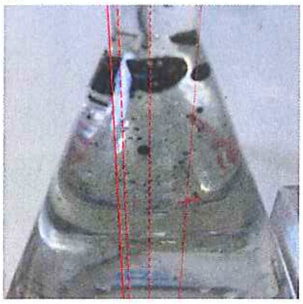
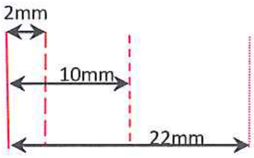
1. the oil which escaped from the flask during the simulation, -droplets too small which has been flushed out of the flask-,
2. the oil which remained in the flask during the simulation,
3. the oil which remained in the flask at the end of the test and could be re-dispersed.

As a simple analogy we could consider the first category as the oil which could stay in the water column, the second as the oil which would have reached the surface, the last one, the part of the oil reaching the surface which was re-dispersible under the wave action.

In order to highlight the effect of a long ascent on the oil droplets, 2 types of tests were completed: with and without a long simulated ascent (2h30 = 150 min).

The picture 26 shows the tests completed with the Grane with and without dispersant. It can be seen that without dispersant (DOR=0) very few small oil droplets were produced; most of the oil was as very large droplets which remained in the flask during the whole test. With dispersant at DOR 1%, a lot of small oil droplets were produced and part of them (the thinnest) could not stay in the flask during the test.

The table 9 gives the results of the quantitative tests completed on the Oseberg oil.

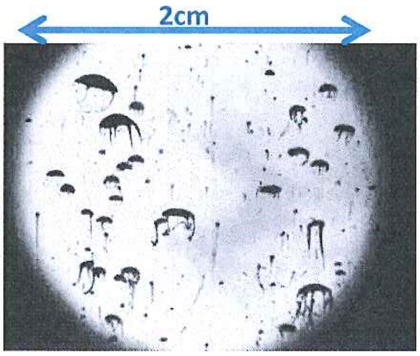
Grane & C95 ; DOR 1% (150 minutes)	Grane only (150 minutes)
5 minutes 	5 minutes 
60 minutes 	60 minutes 
120 minutes 	120 minutes 
Scale: An approximated scale is indicated in red in the different pictures 	

Picture 26: Observation of the Grane oil behavior, with and without dispersant during the 2h30 ascent simulation.

Table 9: Simulation of oil droplets ascent in a long virtual water column: results of the quantitative tests completed with Oseberg oil.

Dispersant	Ascent duration (minutes)	Initially introduced (g)	Flush out the flask (g)	Remaining in the flask (g)	Oil redispersed (%)
C-95	150	3.6	1.5	2.1	36
C-95	0	3.6	0	3.6	58
OSR 52	150	3.6	2.1	1.5	25
OSR 52	0	3.6	0	3.6	54
DASIC	150	3.6	1.8	1.8	34
DASIC	0	3.6	0	3.6	57
OSR 62	150	3.6	1.7	1.9	34
OSR 62	0	3.6	0	3.6	61

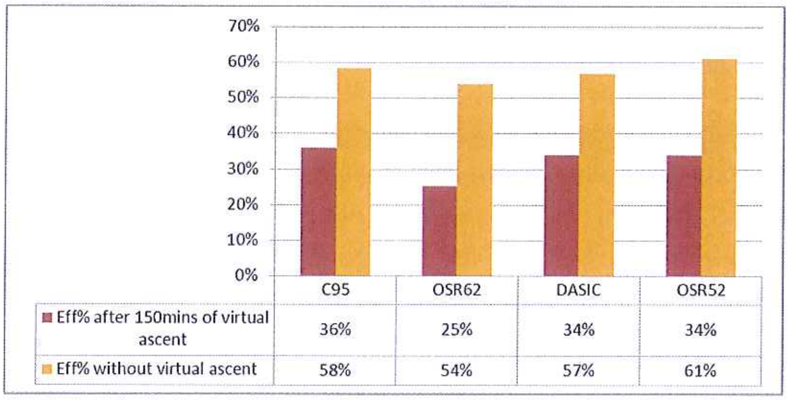
These figures show that, just by travelling in the water column for a long time, an important amount of the oil did not reach the surface: between 58 and 41 % of the oil initially introduced were lost. In fact, during their ascent, just by the friction with the surrounding water, the oil droplets are fractionated into smaller dropplets. Picture 27 coming from another recent experimental program conducted at Cedre illustrates this process in which large droplets form thin strand which break into very small droplets. These thin droplets can be small enough to remain into the water column as a stable plume of dispersed oil.



Picture 27: Large droplets of dispersed oil splitting into thinner droplets during their ascent through the water column. Picture taken with an high speed camera -250 pic/sec- in macroscopic mode – picture diameter 2 cm-.

This process is dependent on the characteristics of the oil mixed with the dispersant (density viscosity but also the interfacial tension). In other terms, in some circumstances, possibly if the surface tension is low enough by the effect of dispersant addition, the dispersion process can be produced just by the rising of the oil from a great depth, independently from the conditions of the oil leakage at the bottom (pressure, velocity, turbulences which lead to the atomization of the oil). This process is of key importance for understanding and modeling the actual behavior of dispersed plume in the water column.

Considering the re-dispersibility of the oil which was collected in the flask at the end of the test (the oil supposed to reach the sea surface in real situation), the oil which traveled for a long time (2h30) in the virtual water column was about half less re-dispersible than the oil which did not travel. See picture 28. In other terms, after 2h30 of friction with the surrounding water, the oil and dispersant mixture lost a significant part of its ability to be re-dispersed by waves. However, the ranking of the different dispersants in terms of efficiency remained quite similar to what has been observed previously.



Picture 28: Comparison of the re-dispersibility of the oil which would reach the surface according to the virtual ascent time (2h30 or no ascent).

13) Conclusions

OGP-IPIECA requested a laboratory screening study to understand more precisely the range conditions under which dispersant is effective and efficient for different crude oils in the context of subsea dispersant application and to check any possible evolution of the plume and larger droplets in time.

A dedicated test and protocol have been designed and set up by Cedre for the specific purpose of this study.

This laboratory study was conducted on 4 different oils / 3 concentrated dispersants / 2 different Dispersant-Oil Ratios (DOR) / 2 energy levels, low and high (LE, HE) / 2 dispersant injection methods: PreMix, upstream the oil injection nozzle (PM) and Direct Injection of dispersant, downstream the oil injection nozzle, in the oil plume (DI).

The experiments were conducted in a 120 L. cylindrical tank. The dispersion was assessed by measuring the dispersed oil droplet size with a Malvern particle size analyzer, as well as the oil concentration in the water column along vertical profiles using a Turner spectrofluorometer. These parameters were monitored for 20 minutes in order to check for possible short term evolution of the plume of dispersed oil. Therefore 3 data were considered, the median droplet sizes at the beginning (oil injection) and at the end of the test (20 min.) and the amount of oil still in the water column at the end of the test, called "efficiency" (integration of the oil concentration along a vertical at 20 minutes). In addition, when oil resurfaced at the end of the test, its re-dispersibility was assessed using the LaboFina-WSL rotating flask method.

85 tests were completed corresponding to 72 tests from the original request and 13 additional ones.

Furthermore, complementary tests were carried out to study the behavior of oil which would rise to the surface from high depth (simulation of a virtual ascent of 2h30). An experimental simulation of oil droplets ascent has been completed in a water column in which a downward stream was adjusted just to balance the rising speed of the oil droplets.

The subsea dispersant effectiveness bench scale test protocol developed by Cedre for the study allowed the following observations:

1. The oil type and characteristics were obviously the most influent parameters ; Grane oil remained poorly dispersible on the contrary of the the 3 others crude oils.
2. Averaging the different measurements, Corexit 9500 seemed to give a slightly better efficiency than Finasol OSR52, Dasic Slickgone and Finasol OSR 62. However, observed differences remained not significant. Comparison between Finasol OSR52 and Finasol OSR 62 showed that these 2 dispersants are very similar as they give very close results for similar conditions.
3. The level of energy was too high for the 3 lightest oils, and the tests gave results close to the maximum efficiency (≈90%). Therefore, in order to better highlight the difference between the dispersants, it would have been more appropriate to adopt more restrictive conditions for each oil, particularly a specific mixing energy level for each oil, (such a protocol was not planned in the design of the original study).
4. There is no special benefit to monitor the oil dispersion for a long period of time (20 minutes) as the observed variations remained low and did not change the ranking of dispersants in terms of efficiency.
5. The results showed that oil droplets obtained after dispersant injection fell mainly into 2 distinct areas: less than 35 μm and more than 100 μm. Good efficiency matched with droplets diameter <35 μm, while poor efficiency matched with > 100 μm. There was very few data in the range of 35 to 100 μm which appeared to be a threshold area.
6. The DOR at 5% gave little coalescence of the dispersed plume and no oil resurfacing even with Direct Injection mode. On the contrary more coalescence and sometimes dispersed oil resurfacing were observed at DOR of 2 and 1%. At DOR 0% (oil alone), dispersion was poor as large recoalescence occurred.
7. The Premix injection mode gave better results than the Direct Injection mode in terms of efficiency and dispersed oil stability (absence of resurfaced oil at the end of the test). The injection in the plume did not lead to an optimum oil-dispersant mixing. With a better mixing it should even be possible to use a reduced DOR. Obviously, the quality of the mixing between the oil and the dispersant is a key parameter; therefore, in the field, the design of the dispersant injection device should be carefully designed (e.g. the dispersant injection assembly mounted at the end of the injection wand).
8. According to the different testing conditions, on a few tests, discrepancies appeared between the droplet size distributions and the efficiency measurements: the ranking of the dispersant from the particle size analyzer was not always in agreement with the one from the spectrofluorometer. In order to better understand the confidence level of the test results it seems important to indicate the residual of the droplet size measurement (assessment of the measurement uncertainty). In this respect, the use of both droplet size measurements and oil concentration measurements increases the reliability of the results.

9. When oil resurfaced at the end of the test (20 min), it was easily re-dispersed when vigorously mixed (i.e. LaboFina WSL test method), except for Grane oil at low dosage and Direct Injection mode (injection into the plume).
10. A simple experimental simulation of a long ascent (2h30) in a long virtual water column has been completed in order to observe the oil droplets behavior during their ascent. It has been observed that dispersion process occurred during the ascent: under the friction of the water against the oil droplets, a large part of the oil (between 40 to 60%), split into thinner droplets which were flushed out of the flask by the stream of water. In a real situation, this part of the oil would not likely reach the sea surface. Moreover, the oil which reached the surface lost about half of its dispersibility. These processes depend obviously on the type of oil and on the dispersant effect (reduction of the interfacial tension). In other terms, these observations showed that, after the dispersant addition, the dispersion process may be produced just by energy produced by the rising ascent of the oil from a high depth, independently from the conditions of the oil leakage at the bottom (pressure, velocity, turbulence which lead to the atomization of the oil).

Understanding these new questions is of tremendous importance for modeling the behavior of dispersed oil plume along the water column and for optimizing subsea dispersant applications. A specifically designed controlled experimental simulation could be undertaken, in a virtual water column at larger scale, in order to define the real droplet size evolution as a function of time, the final droplet size which could be achieved under the water friction as well as the final dispersibility of the oil which would reach the surface according to the oil type and to the dispersant used. Such an experiment could be undertaken in *Cedre*'s testing facilities, especially in its 5 meter high experimental water column (picture 29) which has already been used to study the behavior of chemical and oil and gas underwater releases. (see Annex2).

Picture 29: Cedre's experimental water column (5m high)

Annex 1

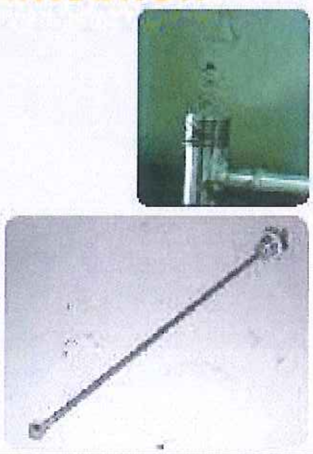
Description of *Cedre* Experimentation column

Cedre experimentations Column (CEC)

The Cedre Experimentation Column is designed to study the behaviour of bubbles, droplets or an object rising to the surface or settling to the bottom of a water column (Le Floch et al 2009).

It is a hexagonal column, 5 m high by 0.8 m in diameter, with a total volume of 2770 L. Four of its sides are made of glass and two of stainless steel. It can be supplied with fresh water or salt water.
The substance studied can be injected either from the top or the bottom of the column by a gear pump to ensure a regular flow at a controlled rate, in order to obtain isolated droplets rather than a plume.
A 40 cm long injection rod enables the substance to be released in the centre of the column and can be fitted with nozzles of different diameters and/or shapes. Several cameras can be placed up the side of the column to monitor the evolution of the droplets according to the level.

INJECTION



Column characteristics		
Shape		Hexagonal column
[illegible]		[illegible]
Observation distance		3.05 m
[illegible]		[illegible]
[illegible]	Shape	Circulaire

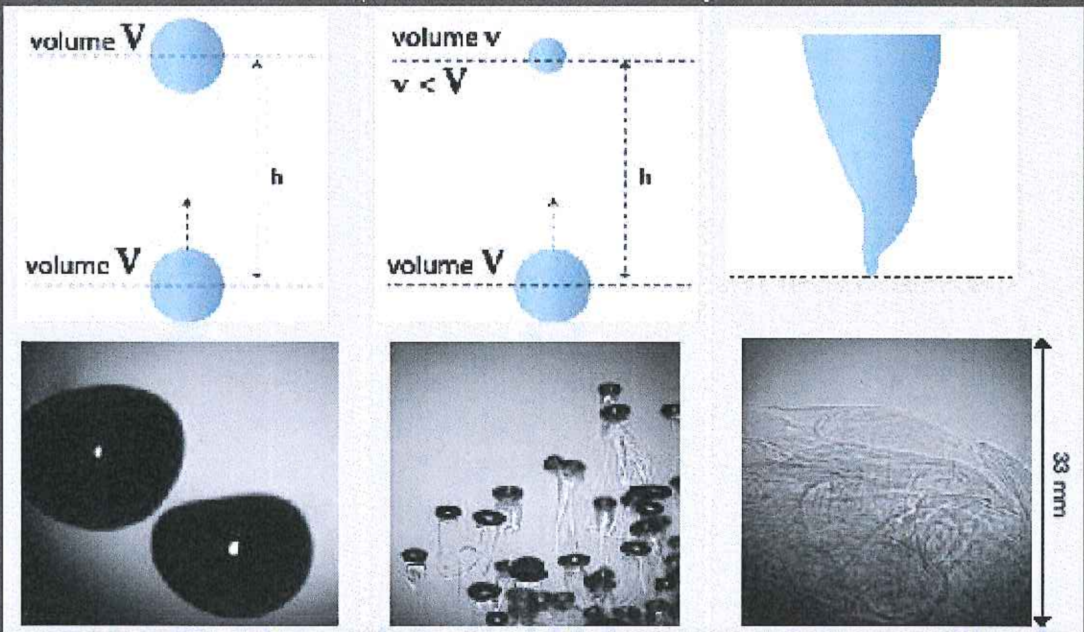
Example

In the case of a floating substance released from the bottom, 3 main types of behaviour can be observed (similarly to a sinking substance released from the surface):

1. No dissolution: insoluble or poorly soluble substances rise up through the water column in the form of droplets and their volume remains unchanged.
2. Dissolution: soluble substances rise up through the water column in the form of droplets and their volume decreases as they rise.
3. Immediate dissolution: highly soluble substances dissolve into a plume.

The table below presents these different types of behaviour and the operational information obtained from the analysis of experiments conducted in the Cedre Experimentation Column.

1 - No dissolution | 2 - Dissolution | 3 - Immediate dissolution



A - Droplet of Di(2-ethylhexyl)phthalate

Determination of the speed at which droplets rise
- Time taken for a surface slick to appear

B - Droplets of n-butanol

Determination of the speed at which droplets rise and of the decrease in volume
- Time taken for a surface slick to appear
- Volume at the surface

C - Plume of ethanol

Determination of the speed at which the plume rises and of dissolution rates
- Appearance of a slick or total dissolution
- Volume at the surface

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Annex 2

Cedre's results presented according to Sintef's format

At the presentation of the results (meeting held in June 2014, in IPIECA facilities in London), it appeared that *Cedre* and Sintef presented their results in different ways. In order to make comparison of results easier, *Cedre* reworked afterwards its results to present them in the same format as Sintef.
These are presented in this annex.

For each testing condition, (Oil, Energy/DOR, Injection mode) the performances reached by the different tested dispersants are displayed on the same chart according to Sintef's color code: black: oil alone; **red**: Corexit 9500; **green:** Dasic Slickgone; **blue**: Finasol OSR 52; **violet**: Finasol OSR 62.

The reader will find:
As for Sintef:

a) The relative distributions of the initial oil droplet size for each dispersant in volume given by the Malvern particle size analyzer.

In addition:

b) The efficiency measured for each dispersant with the spectrofluorometer. This is the integration of the oil concentration measured in the water column along the vertical profile at the end of the test (20 minutes after the oil injection); its represents the oil still dispersed in the water column after 20 minutes.

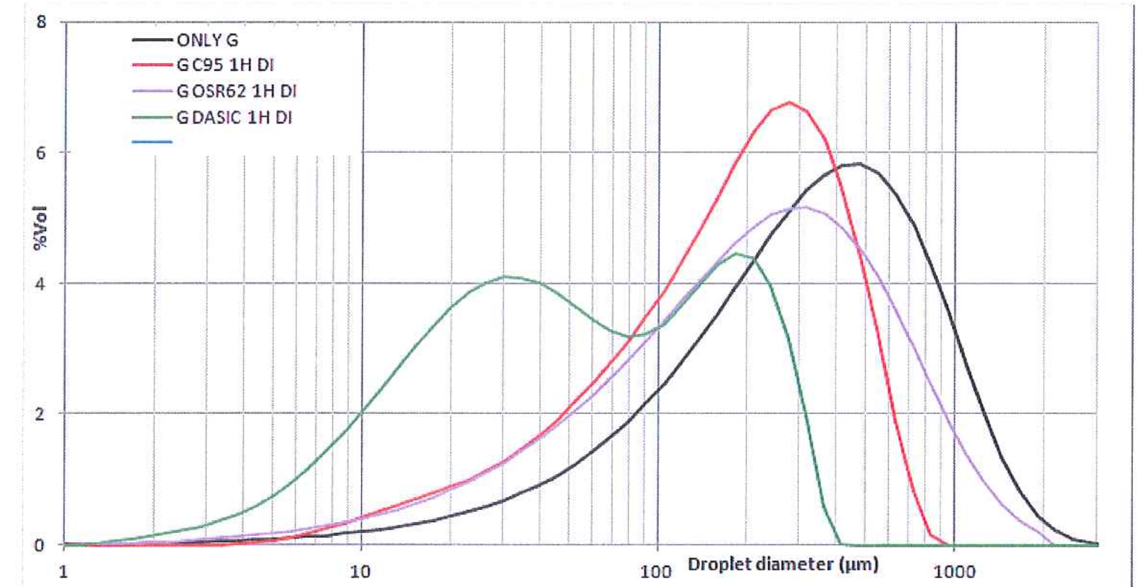
c) The residual for the droplet size distribution of each dispersant. This value which exists for any droplet size analyzer, gives an idea of the quality of the distribution. As a general rule when the residual is less than 1 the quality is good, higher than 1 the quality becomes mediocre and further measurement loses its reliance (e.g. over 5). Different conditions can alter the quality of the measurement such as the oil concentration which must remain in a range acceptable by the apparatus.

Each chart is labeled with the oil name, the injection mode and the energy and DOR. (i.e. "GRANE – PM 2H" for, test completed on **GRANE** oil in **P**re**M**ix injection mode and at DOR **2**% and **H**igh energy)

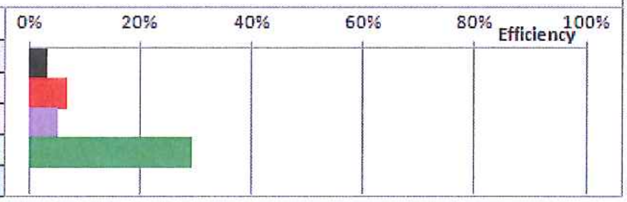
Globally, these results confirm those presented in the report.
However, some discrepancies appeared on a few charts between the ranking of the dispersants according to the oil droplet size distribution and according to the measurement of the efficiency.

The residual values show that, on few oil droplet distributions, the confidence level can be weak. Therefore, in order to better understand the confidence level of the test results it seems important to indicate the residual of the droplet size measurement.
In this respect, using simultaneously droplet size and oil concentration measurement leads to a better reliability of the test results.

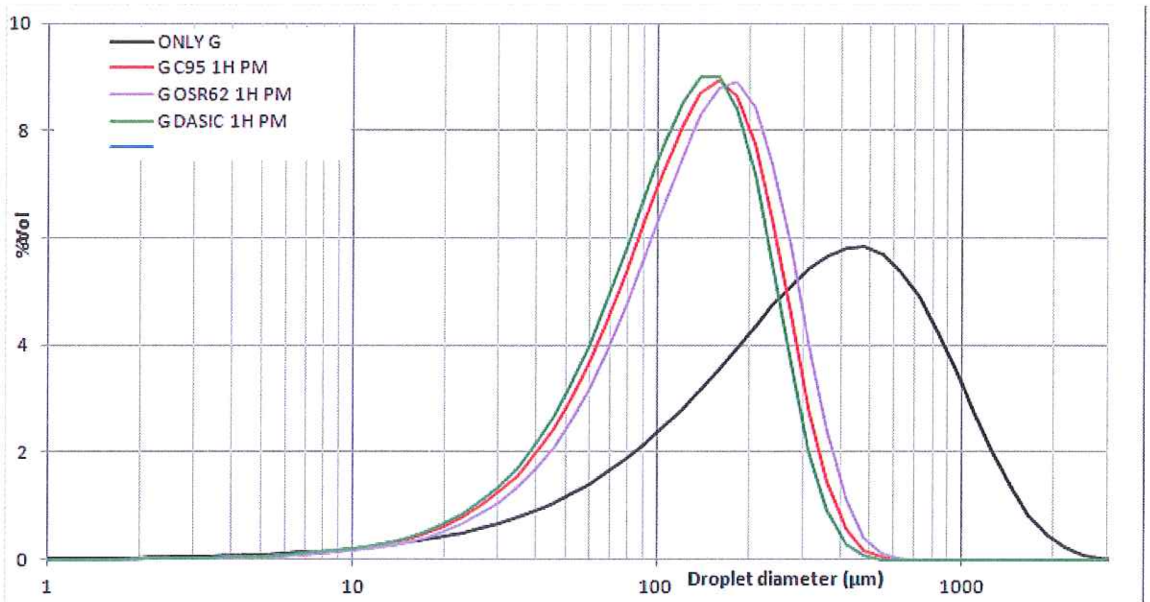
GRANE DI 1H



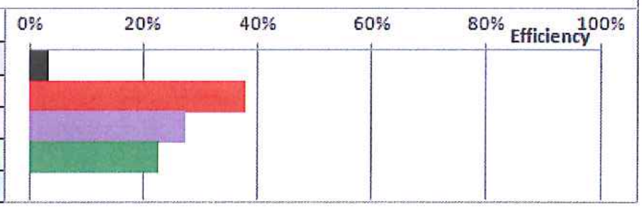
	Residual
ONLY G	
G C95 1H DI	0,680
G OSR62 1H DI	1,735
G DASIC 1H DI	2,988



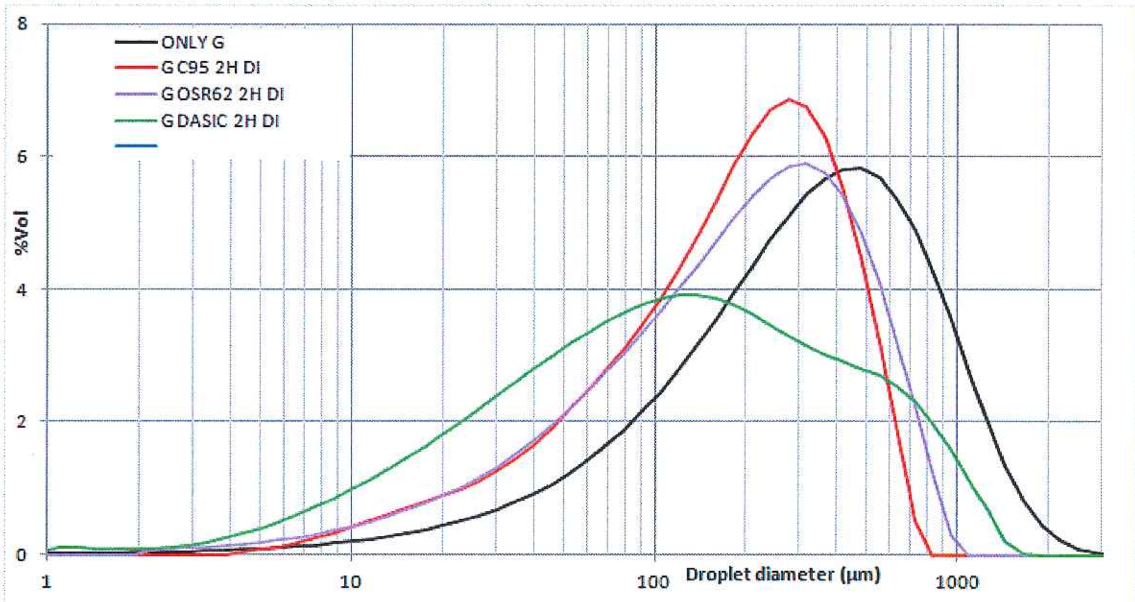
GRANE PM 1H



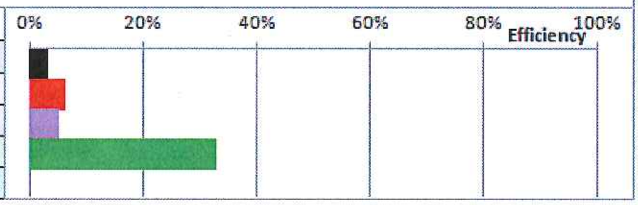
	Residual
ONLY G	
G C95 1H PM	1,850
G OSR62 1H PM	1,742
G DASIC 1H PM	0,669



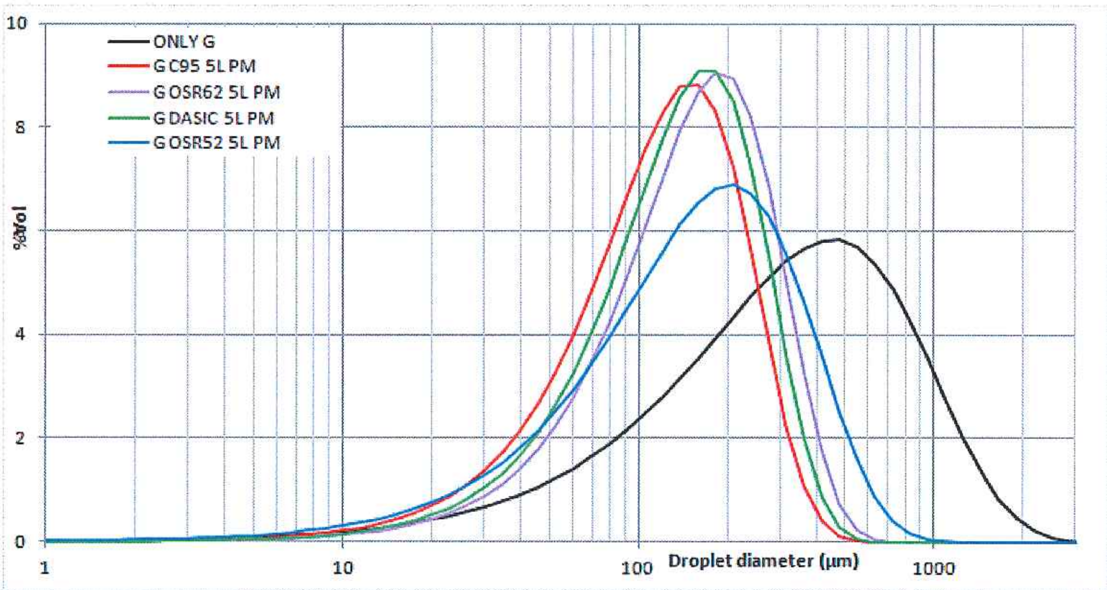
GRANE DI 2H



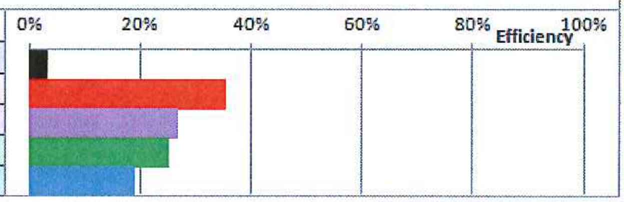
	Residual
ONLY G	
G C95 2H DI	1,360
G OSR62 2H DI	1,524
G DASIC 2H DI	0,369



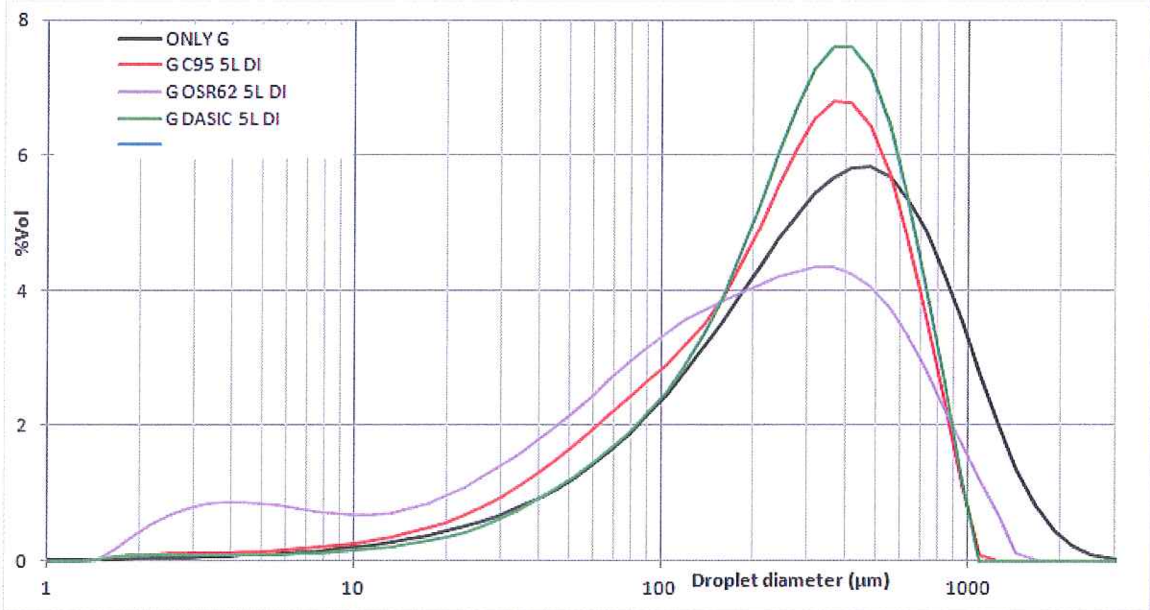
GRANE PM 2H



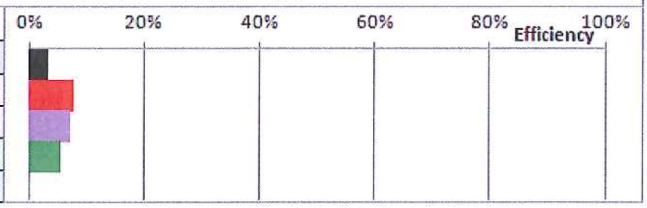
	Residual
ONLY G	
G C95 5L PM	1,799
G OSR62 5L PM	1,677
G DASIC 5L PM	1,277
G OSR52 5L PM	1,566



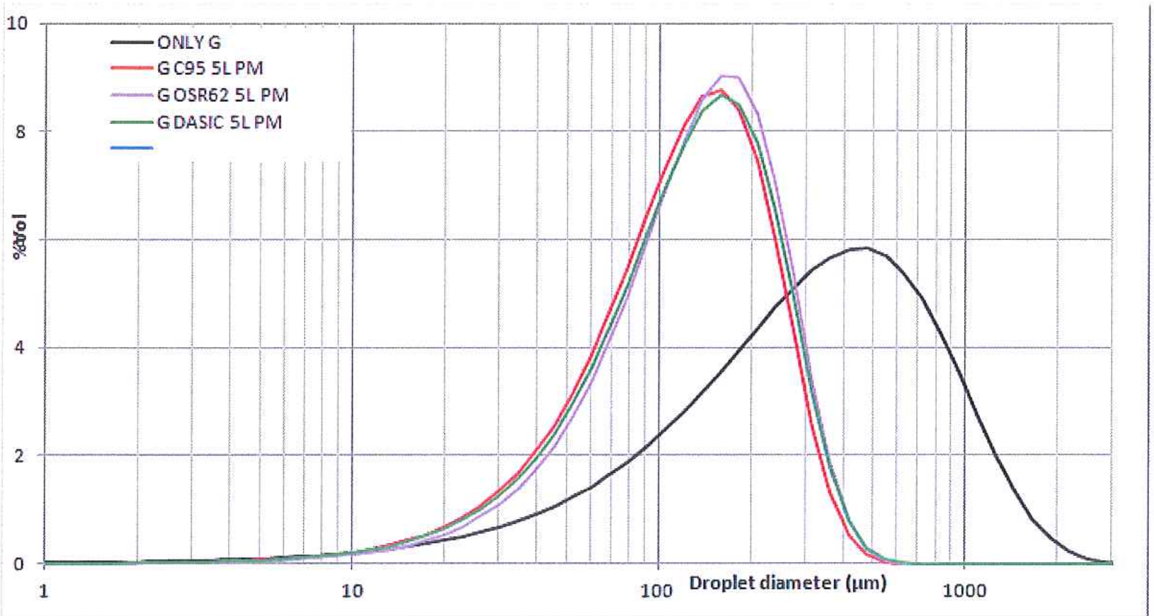
GRANE DI 5L



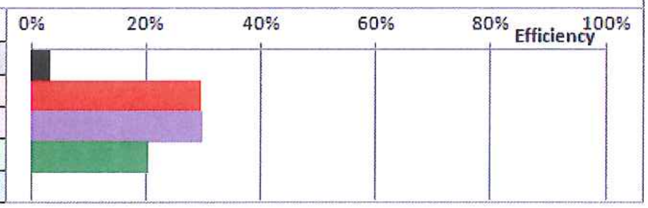
	Residual
ONLY G	
G C95 5L DI	0,679
G OSR62 5L DI	0,411
G DASIC 5L DI	0,998



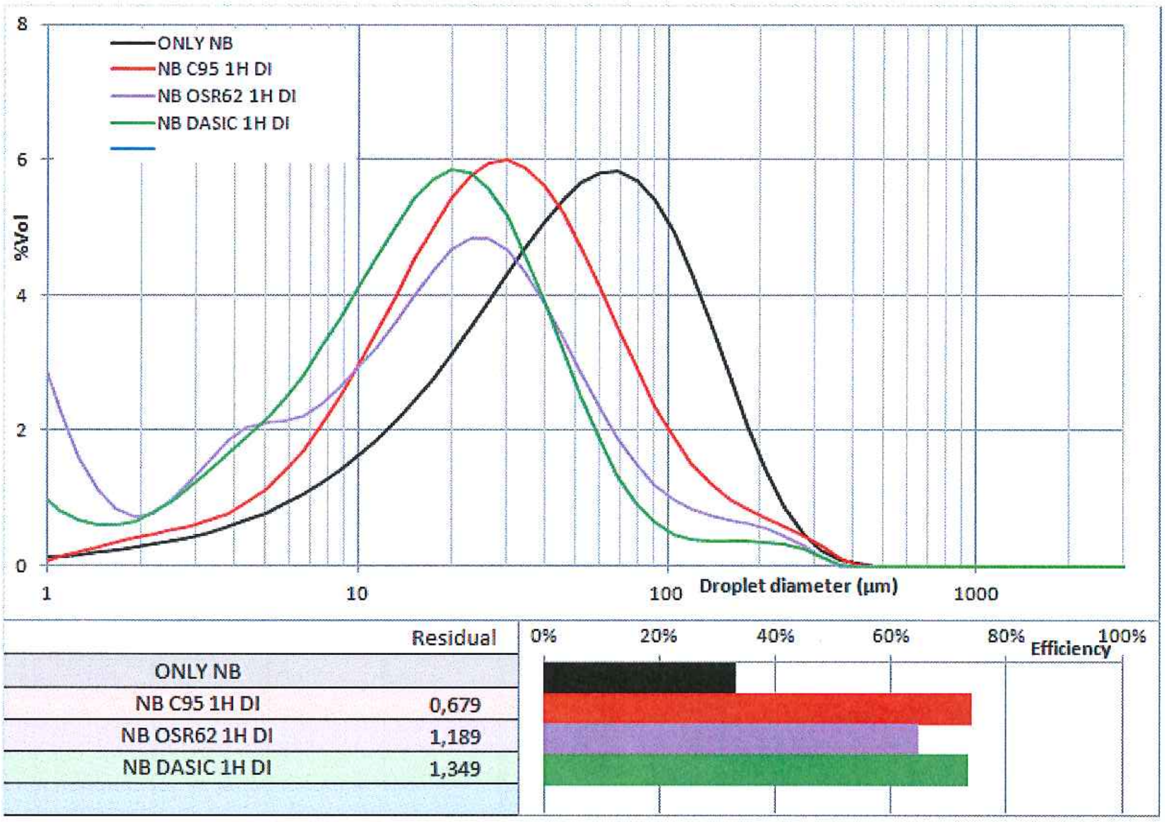
GRANE PM 5L



	Residual
ONLY G	
G C95 5L PM	1,730
G OSR62 5L PM	1,327
G DASIC 5L PM	0,777

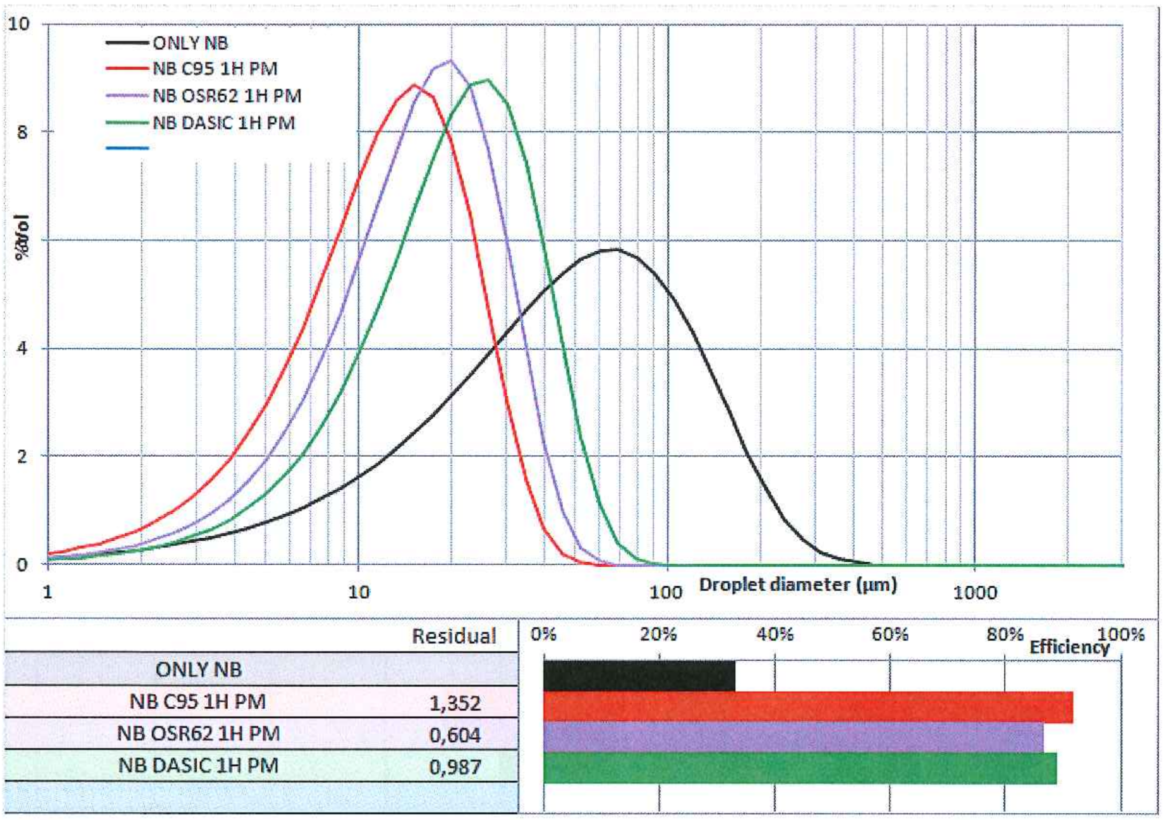


NORNE BLEND DI 1H



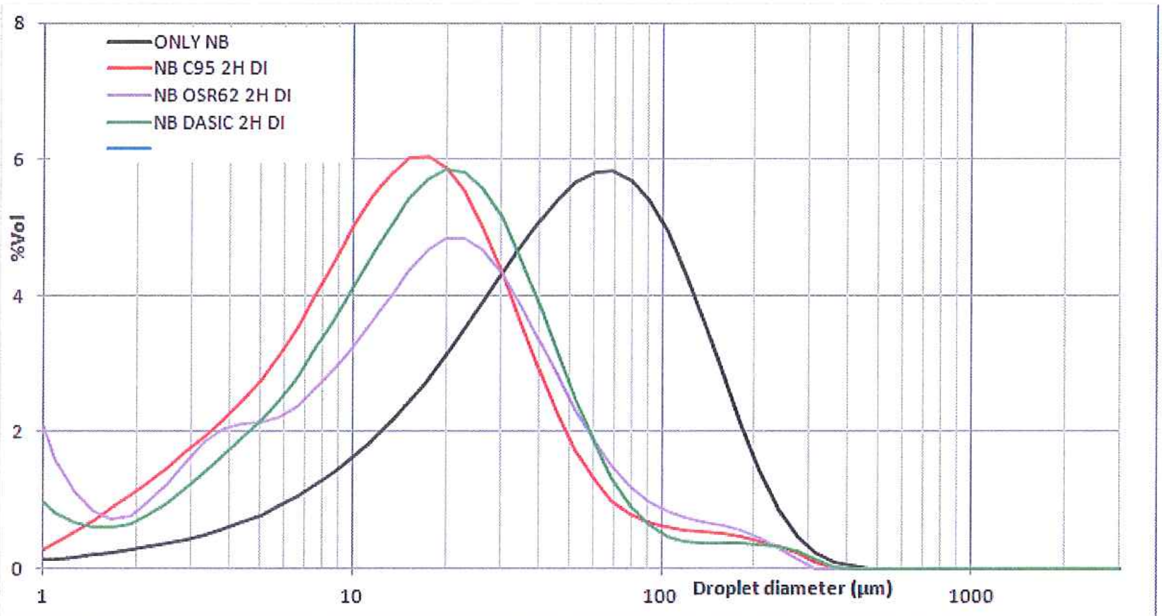
	Residual
ONLY NB	
NB C95 1H DI	0,679
NB OSR62 1H DI	1,189
NB DASIC 1H DI	1,349

NORNE BLEND PM 1H

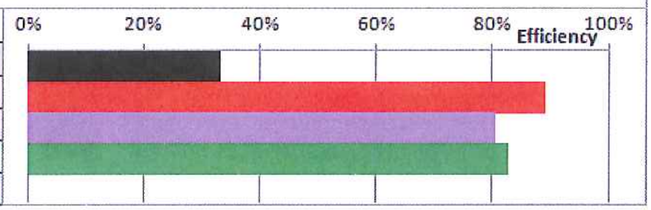


	Residual
ONLY NB	
NB C95 1H PM	1,352
NB OSR62 1H PM	0,604
NB DASIC 1H PM	0,987

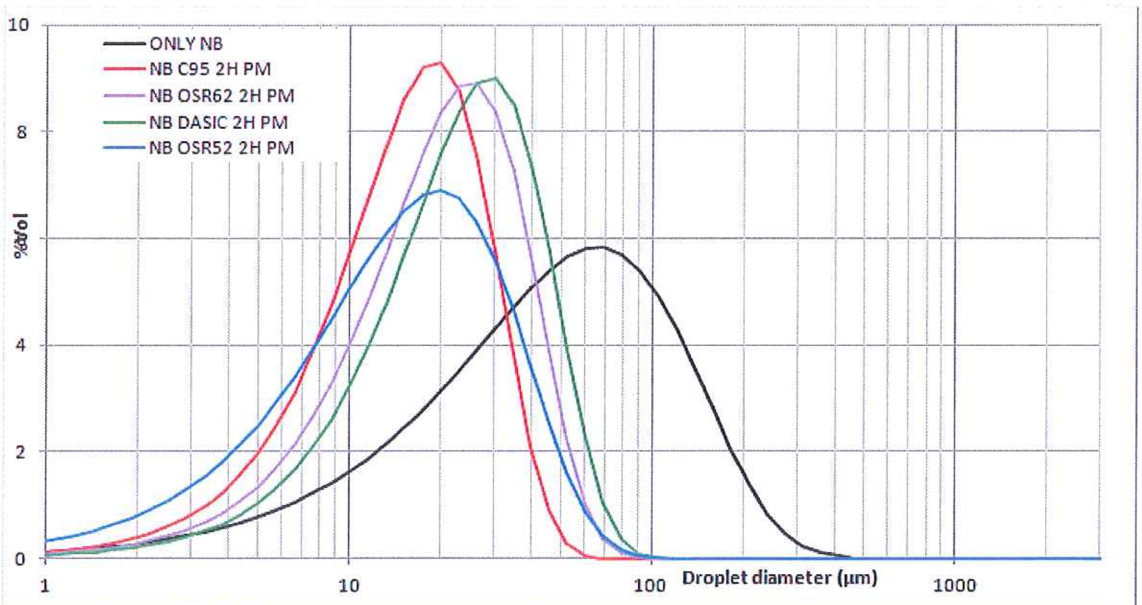
NORNE BLEND DI 2H



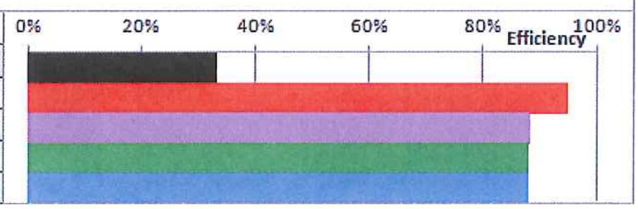
	Residual
ONLY NB	
NB C95 2H DI	0,470
NB OSR62 2H DI	2,315
NB DASIC 2H DI	0,605



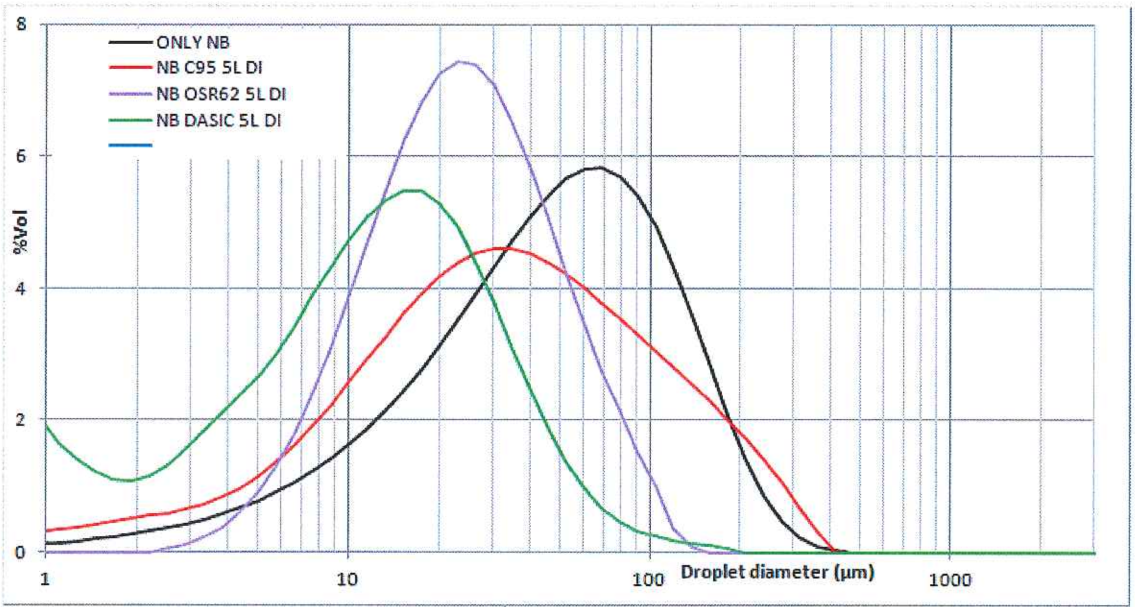
NORNE BLEND PM 2H



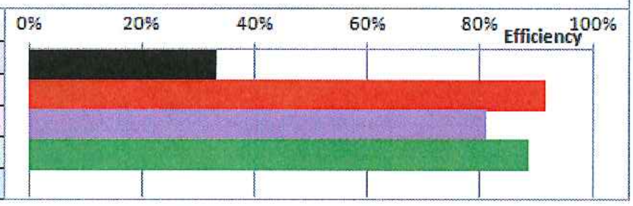
	Residual
ONLY NB	
NB C95 2H PM	1,402
NB OSR62 2H PM	0,697
NB DASIC 2H PM	0,659
NB OSR52 2H PM	0,633



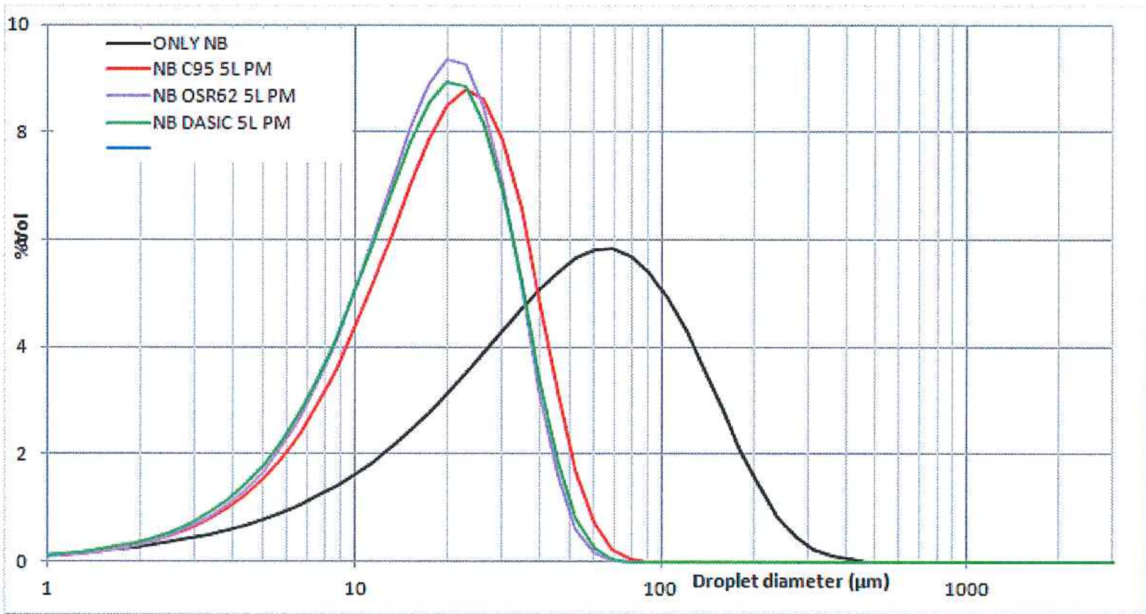
NORNE BLEND DI 5L



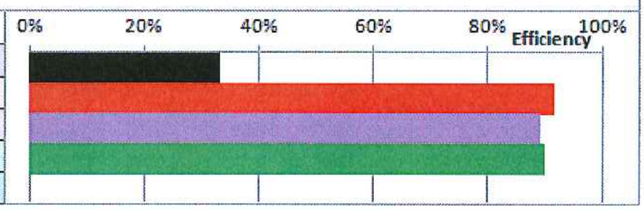
	Residual
ONLY NB	
NB C95 5L DI	1,012
NB OSR62 5L DI	2,775
NB DASIC 5L DI	1,044



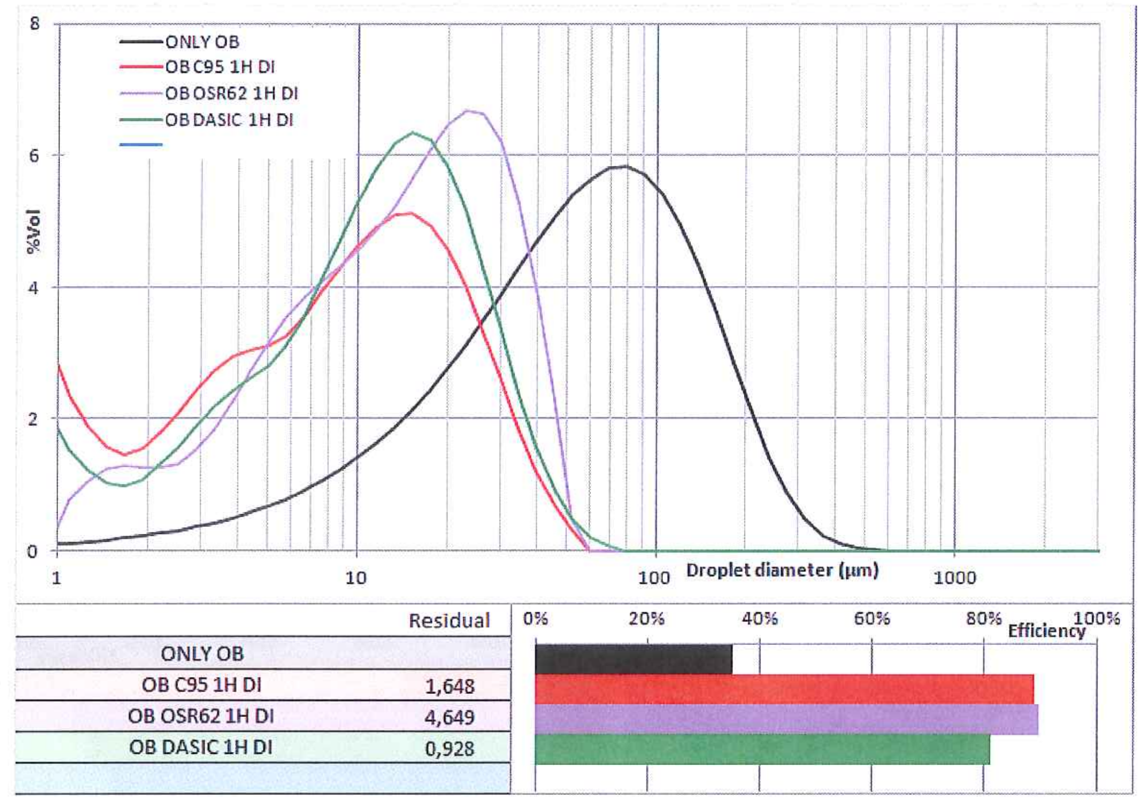
NORNE BLEND PM 5L



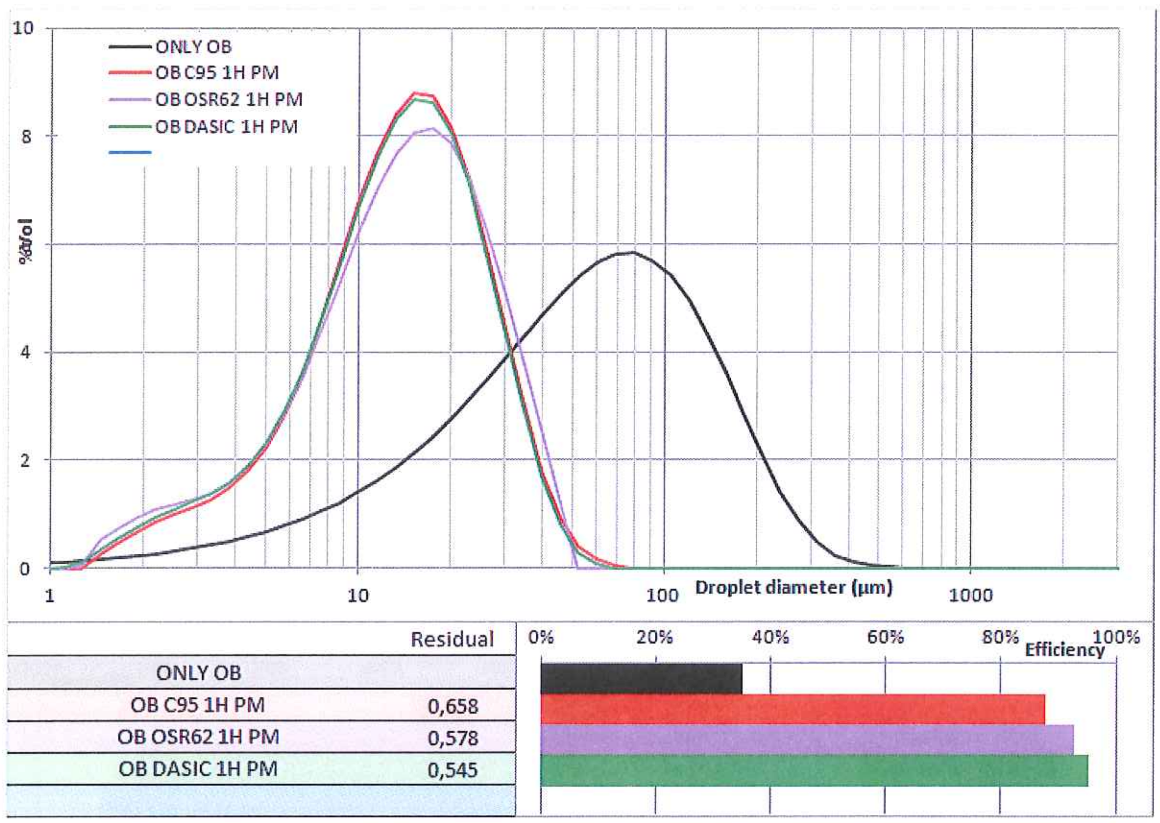
	Residual
ONLY NB	
NB C95 5L PM	0,921
NB OSR62 5L PM	0,775
NB DASIC 5L PM	0,517



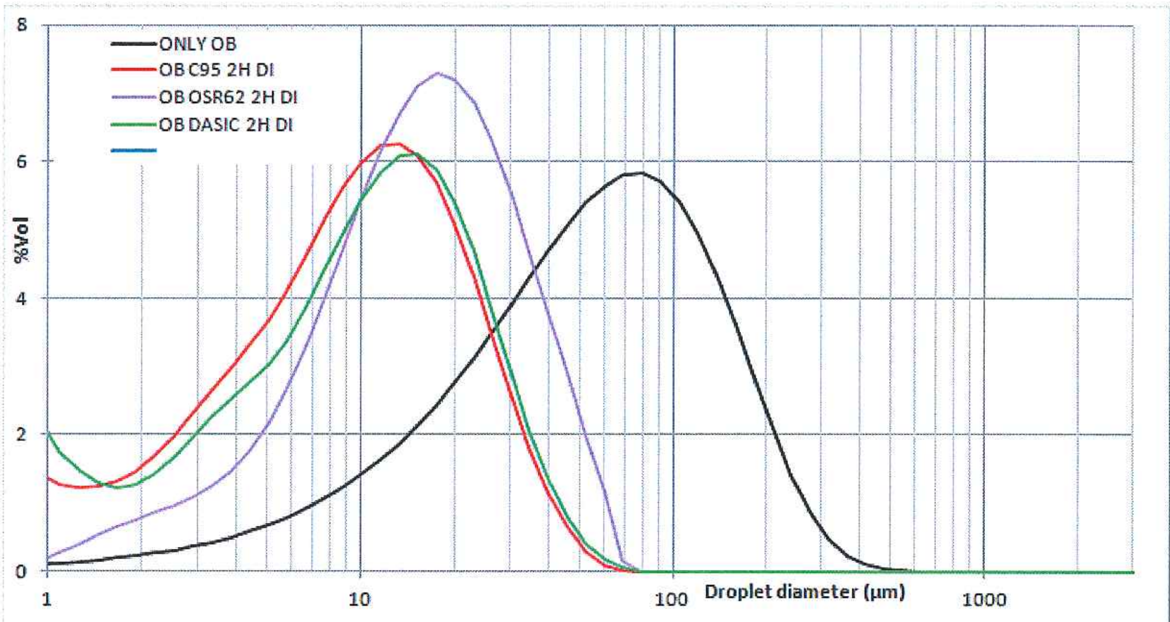
OSEBERG BLEND DI 1H



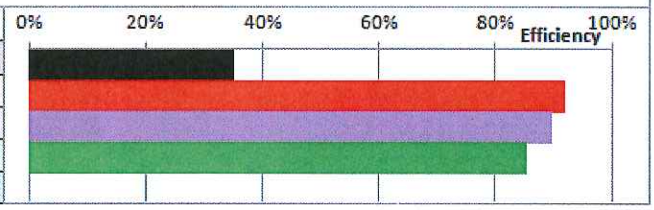
OSEBERG BLEND PM 1H



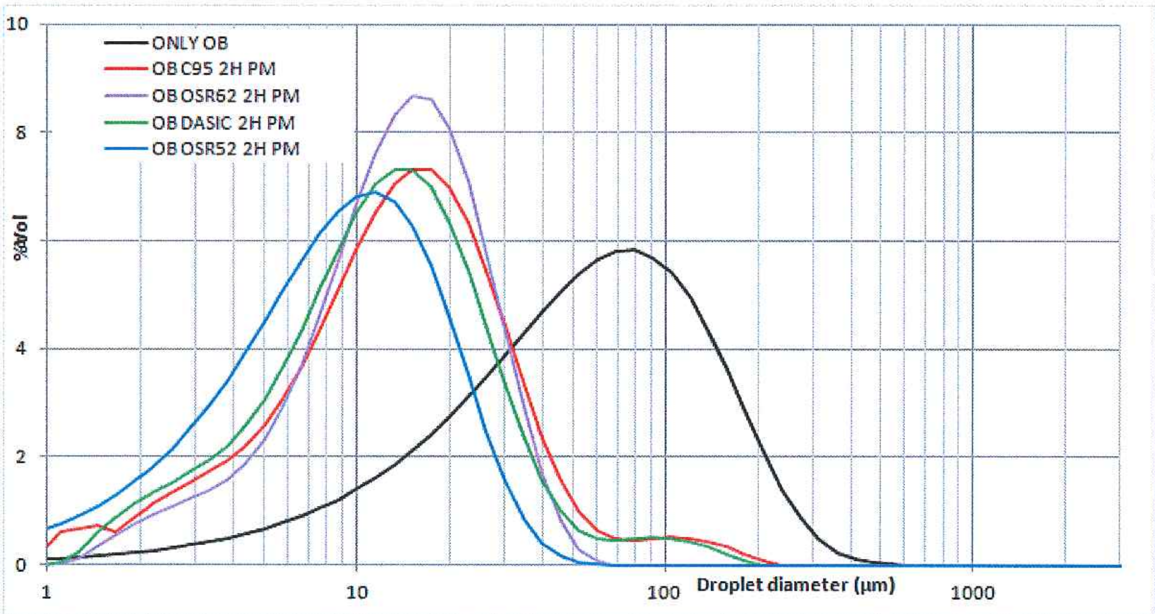
OSEBERG BLEND DI 2H



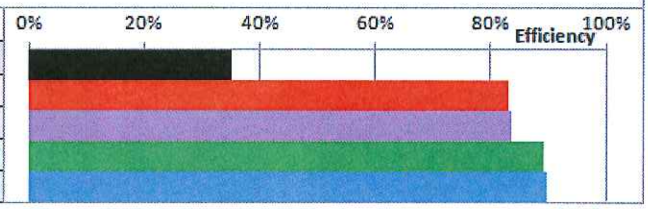
	Residual
ONLY OB	
OB C95 2H DI	0,719
OB OSR62 2H DI	0,909
OB DASIC 2H DI	0,920



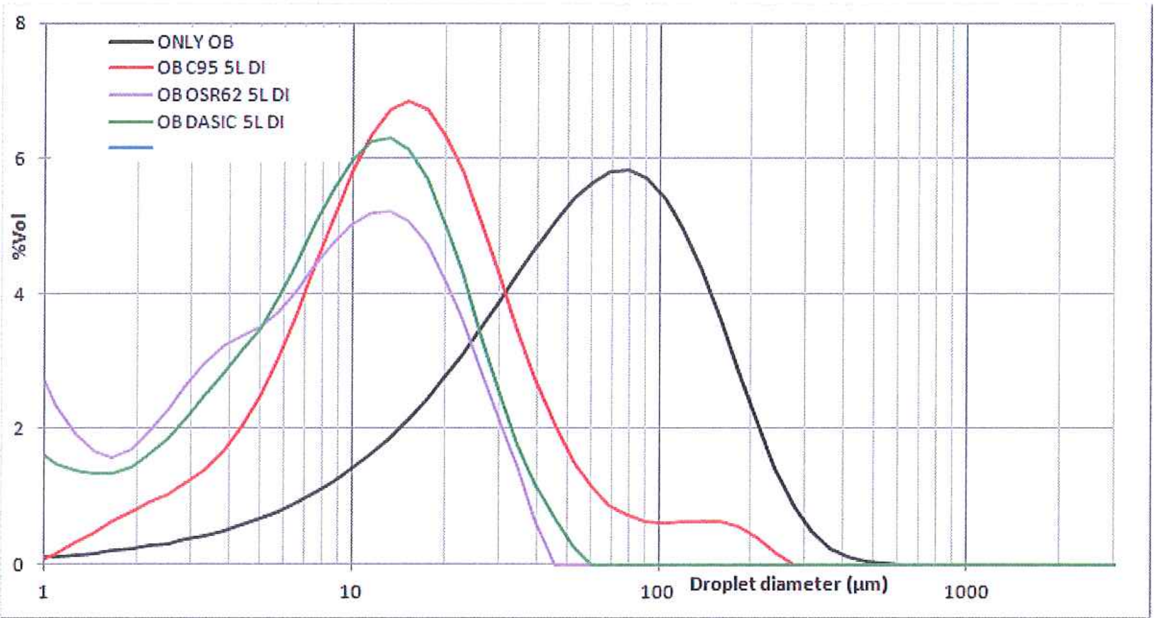
OSEBERG BLEND PM 2H



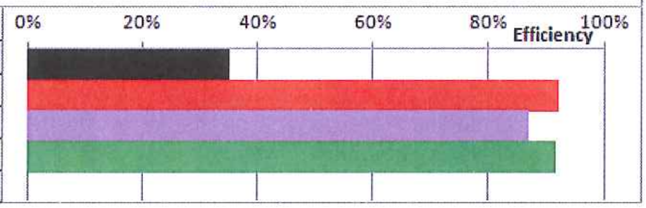
	Residual
ONLY OB	
OB C95 2H PM	0,626
OB OSR62 2H PM	0,468
OB DASIC 2H PM	0,727
OB OSR52 2H PM	0,323



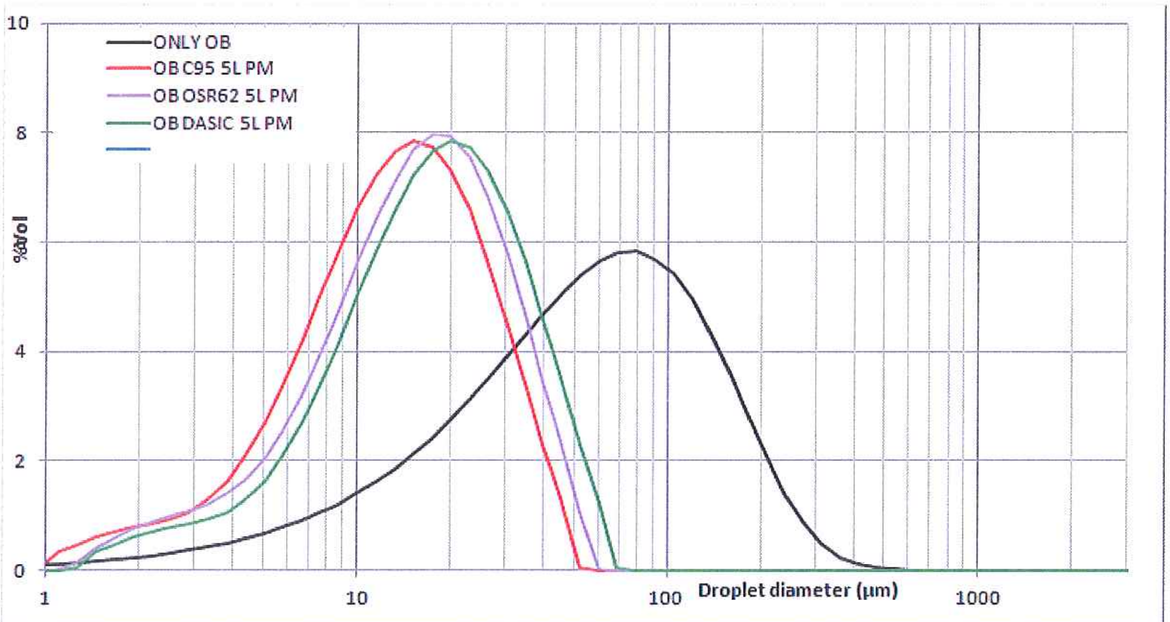
OSEBERG BLEND DI 5L



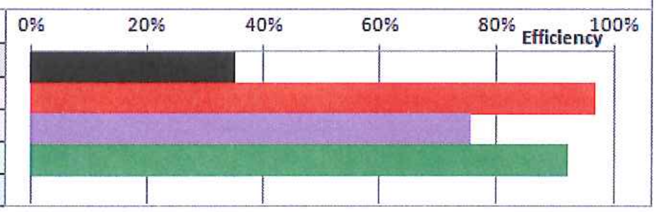
	Residual
ONLY OB	
OB C95 5L DI	1,248
OB OSR62 5L DI	1,437
OB DASIC 5L DI	0,725



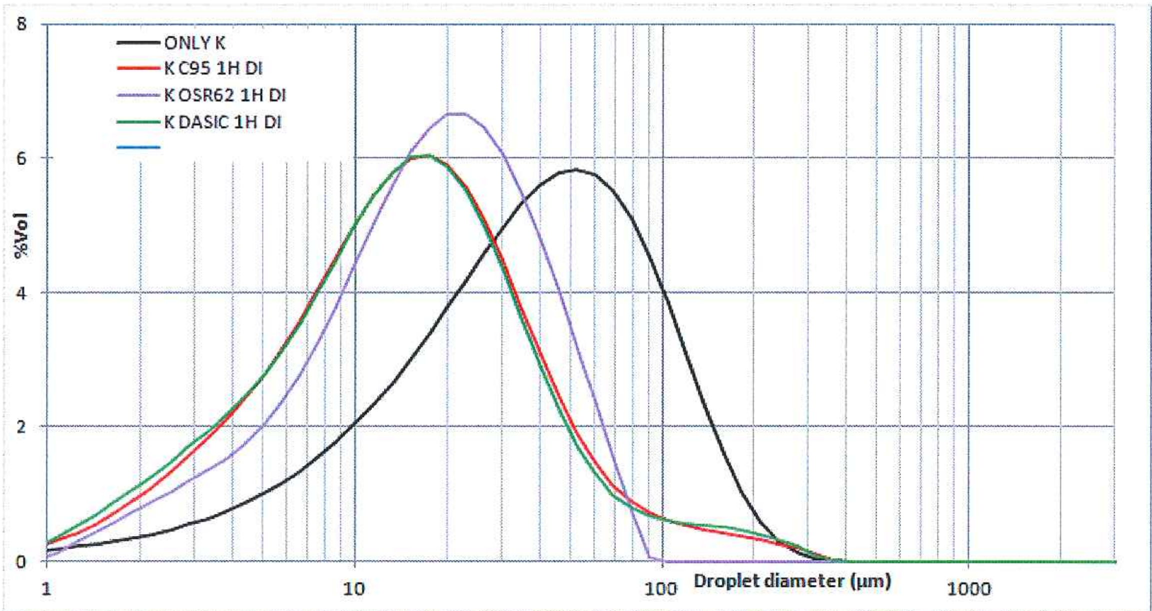
OSEBERG BLEND PM 5L



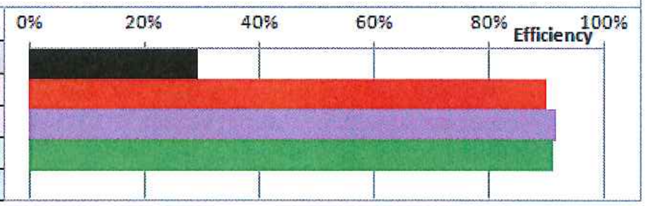
	Residual
ONLY OB	
OB C95 5L PM	0,575
OB OSR62 5L PM	0,474
OB DASIC 5L PM	0,175



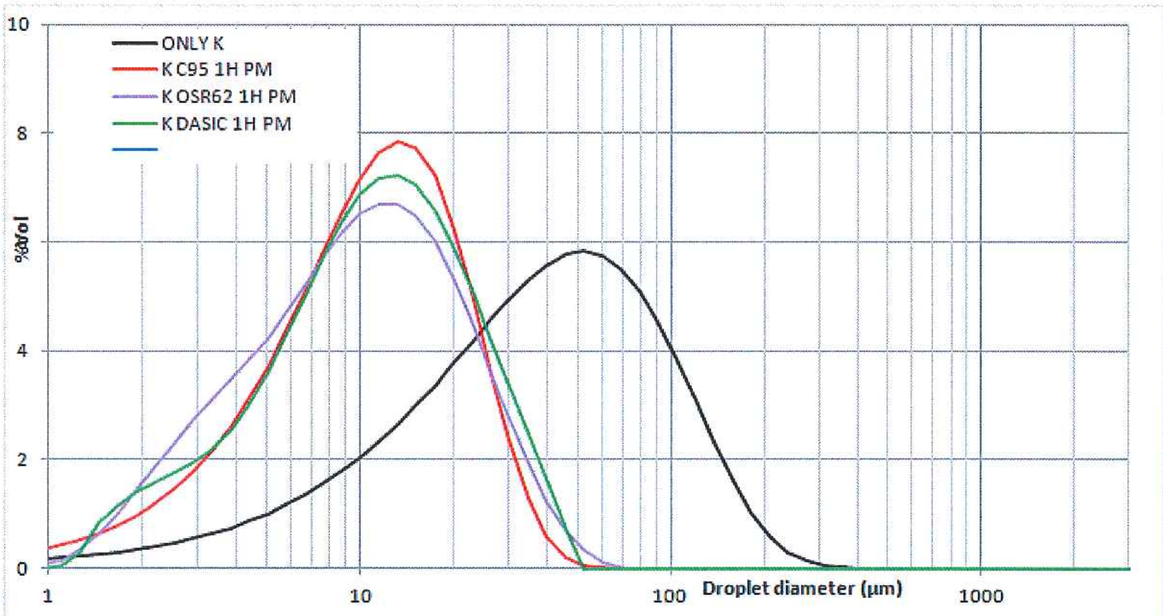
KOBBE DI 1H



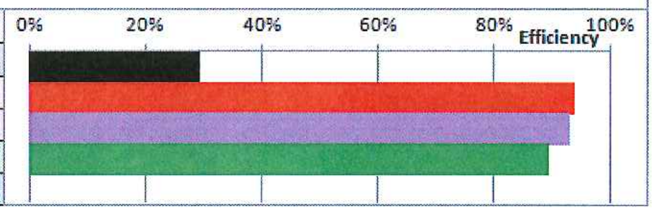
	Residual
ONLY K	
K C95 1H DI	0,473
K OSR62 1H DI	0,923
K DASIC 1H DI	0,470



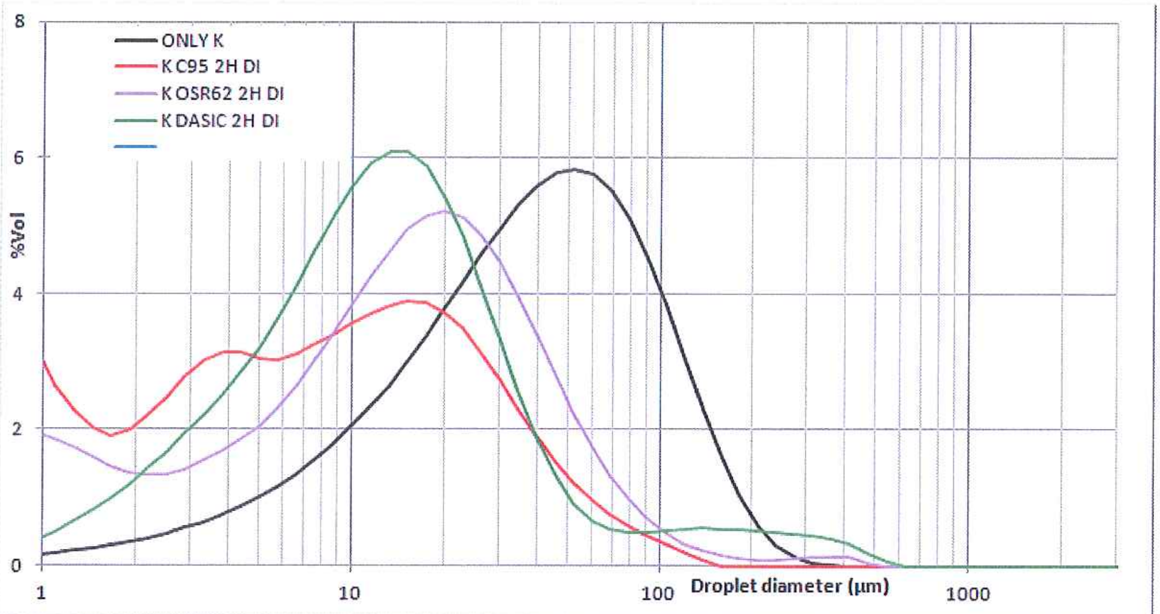
KOBBE PM 1H



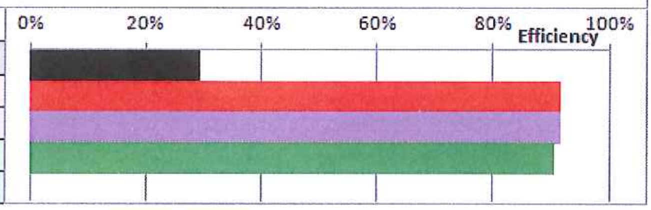
	Residual
ONLY K	
K C95 1H PM	0,128
K OSR62 1H PM	0,361
K DASIC 1H PM	0,810



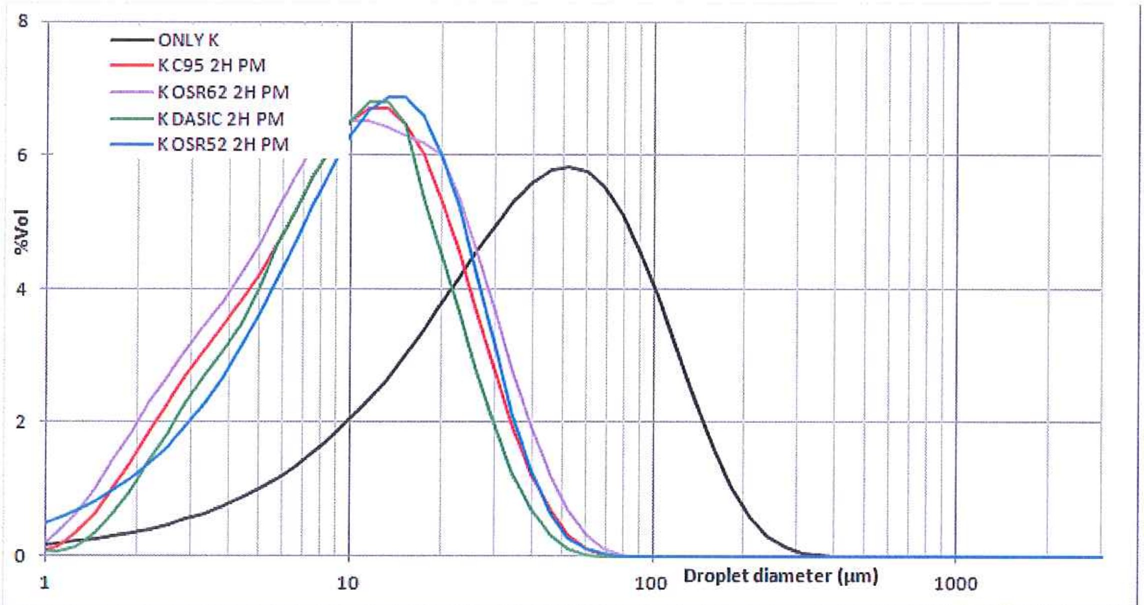
KOBBE DI 2H



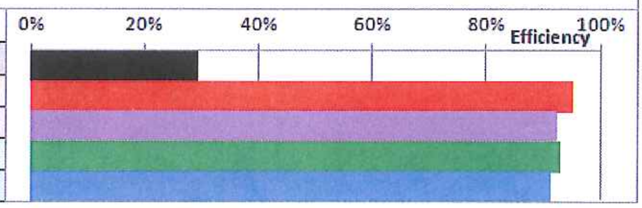
	Residual
ONLY K	
K C95 2H DI	1,751
K OSR62 2H DI	1,180
K DASIC 2H DI	0,554



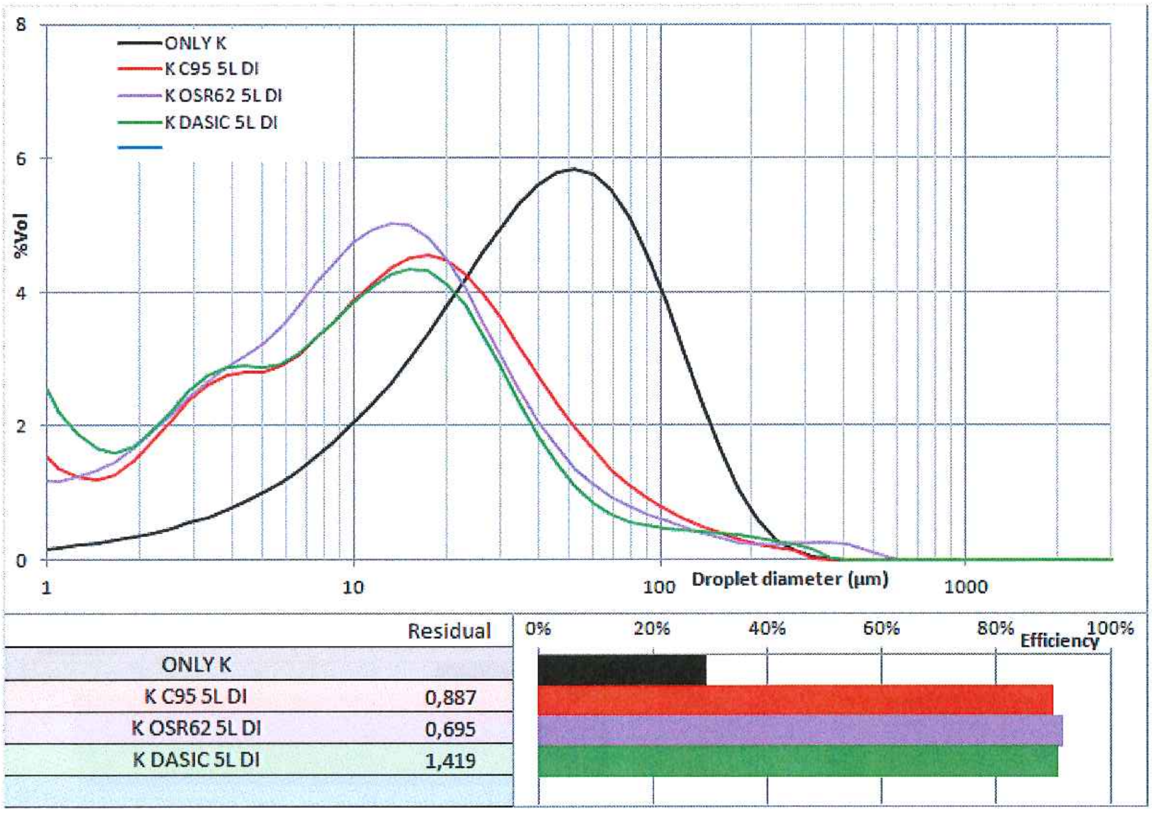
KOBBE PM 2H



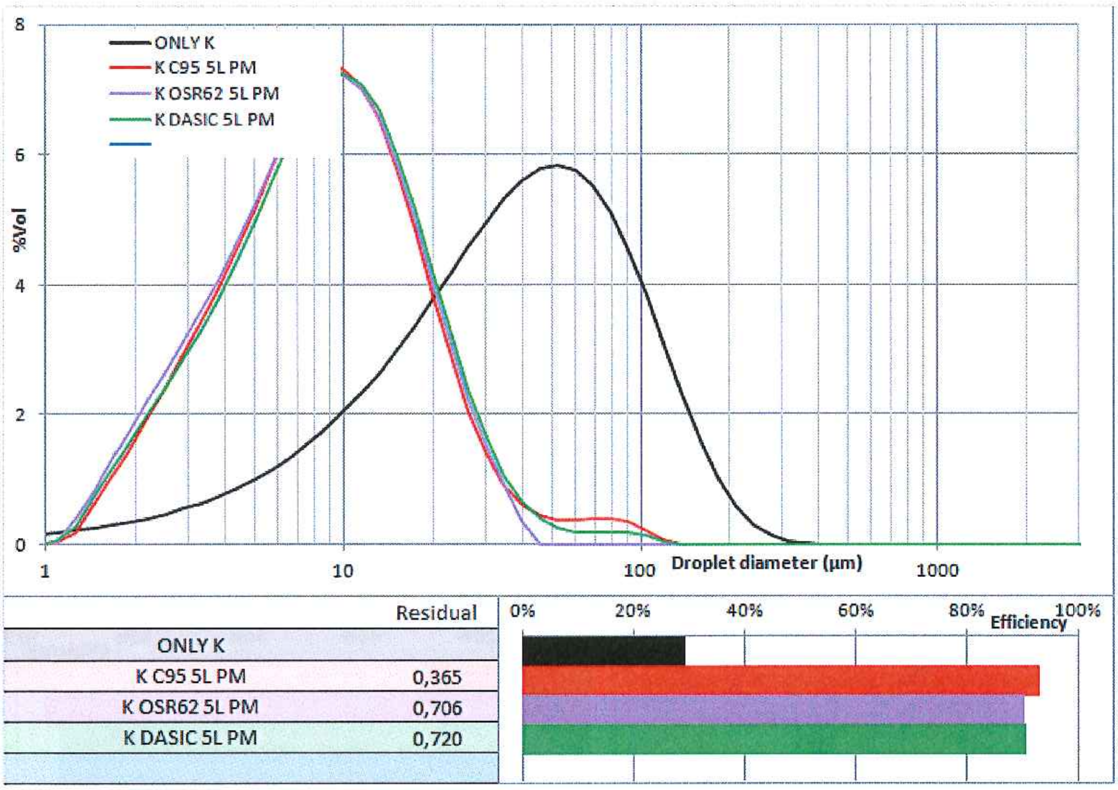
	Residual
ONLY K	
K C95 2H PM	0,985
K OSR62 2H PM	0,585
K DASIC 2H PM	0,593
K OSR52 2H PM	0,496



KOBBE DI 5L



KOBBE PM 5L



OSEBERG BLEND – VL – 0.7