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Experimental Findings on the Dispersion Effectiveness of Magna 1100 and Magna 1101 Oil Spill Dispersants Using the Modified United Kingdom (UK) Warren Spring Laboratory (WSL) LR448 test and Australia Semi-Quantitative Test (SQT)

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Abstract

The aim of this experiment is to determine the dispersion effectiveness of Magna 1100 and Magna 1101 dispersants using the modified United Kingdom (UK) Warren Spring Laboratory (WSL) LR448 test (i.e. rolling flask test) [1]. The dispersant-to-oil ratio used in this experiment is 1:20 (i.e. 1 part of dispersant to 20 parts of oil).

Key Words

Modified (UK) Warren Spring Laboratory (WSL) LR448 Test, Australia Semi-quantitative test (SQT) Magna 1100, Magna 1101, Oil Spill Dispersant

Introduction

An oil dispersant consists of a mixture of surfactants and solvents [2], [3], [4]. A surfactant has a hydrophilic and hydrophobic group [5], [6]. It can break down oil slick into small oil patches or droplets [6]. The solvents transmit the surfactants to the oil slick and improve the penetration ability of the surfactants into the oil slick [2], [7, p. 32]. These small droplets of oil can easily disperse into the water column and may be more readily biodegraded by microorganisms that live in the seawater [8], [9], [10], [11], [12].

1.1 Type of dispersant

There are 3 types of dispersant [1], [13], [14], [15, p. 22], [16, pp. 3-4], [17]:

Type I: Traditional hydrocarbon-based dispersant. Generally, the number of aromatic hydrocarbons in type I dispersants is low or none. The dispersant usually consists of about 15% to 25% surfactants. Type I dispersants are applied undiluted on seas or beaches.

Type II: Water-dilutable concentrates. Type II dispersants are normally made up of non-aromatic hydrocarbons and oxygenated compounds such as glycol. It normally contains about 25% to 65% of surfactants. Type II dispersants are required to be diluted with seawater (dilution ratio of 1:10, 1part dispersant to 10 parts seawater) before its application to the sea.

Type III: Concentrate. Type III dispersants are similar to type II dispersants except that type III dispersants can be used without diluting with seawater.

Magna 1100 is a type I dispersant while Magna 1101 is a type II dispersant.

WSL LR448 test

In this experiment, a modified Warren Spring Laboratory (WSL) LR448 test is used to determine the dispersion effectiveness of the oil dispersants of interest (i.e. Magna 1100 and Magna 1101). The WSL LR448 test, also known as the rotating flask test, is a comparatively energetic test [7, p. 48], [15, p. 5], [16, p. 22] that is simple and efficient to carry out [15, p. 16]. It makes use of the rotation of the flask to simulate the mixing action of the waves on the dispersant and test oil [15, p. 19], [16, p. 22]. The purpose of the WSL LR448 test is to determine the percentage of dispersed test oil that has diffused into the column of seawater [15, p. 21]. This measurement indicates the dispersion effectiveness of the oil dispersant in an ideal test condition (i.e. the oil droplets are fully and uniformly dispersed into the seawater) [1], [18] .

To accurately establish the dispersion effectiveness of the dispersant, colourimetry is applied. A spectrophotometer is used to measure the amount of light that passes through the test solution (i.e. mixture of dispersed oil and clean solvent) and standard solutions (i.e. known proportion of oil dissolved in clean solvent) [1], [18]. The light absorbance result obtained from the test solution is then compared to that of the standard solutions to identify the dispersion effectiveness of the dispersant [1], [18].

However, in this experiment, due to unavailability of spectrophotometer, visual colour analysis is used to deduce the dispersion effectiveness of the dispersant. This method of analysis is adapted from the Australia Semi-quantitative test (SQT) (refer to section 2.4 for more details). It involves the comparison of the colour of the dispersed oil layer in the test solution to that of standard solutions that have known percentages of oil dissolved in clean solvent [19], [20], [21]. The darker the layer of dispersed oil, the higher the content of dispersed oil droplets present in the water column, the more effective the dispersant.

In the UK, the WSL test is used to evaluate the dispersion effectiveness of dispersants before they are approved for application in the sea [22]. According to the UK's requirement for dispersion effectiveness, type I and II dispersants should be at least 30% effective while type III dispersants should have a dispersion effectiveness of 60% or higher [1], [15, p. 22], [23].

1.2 Australia Semi-quantitative test (SQT)

The SQT is an oil dispersant dispersion effectiveness test that is relatively simple and quick to carry out, making it a practical on-site dispersion effectiveness evaluation test for oil dispersants [19]. The test procedure of the SQT is quite similar to that of the WSL LR448 test as both tests make use of the rotation of the flask to imitate the mixing effect of the waves on the dispersant and oil. However, there are some differences in the SQT, mainly in terms of the sequence of the addition of the test oil and dispersant to the seawater, rotation technique as well as the result analysis.

The SQT procedure requires the oil and dispersant to be mixed prior to the addition of a specified amount of the mixture to the seawater for further mixing [19], [20], [21]. In the case of the WSL test, the oil, dispersant and seawater are mixed in a single flask [1].

The rotation technique used in the SQT is slightly different from that of the WSL test. According to the SQT procedure, the flask containing the mixture of oil, dispersant and seawater is rotated end-over-end, compared to the rotation along the horizonal axis for the WSL test [1], at a rate of about 4 seconds per turn for 2 minutes [19], [20], [21].

In the SQT, the dispersion effectiveness of an oil dispersant is established by visual comparison of the colour of the dispersed oil layer in the test solution to standard solutions with known percentages of dissolved oil in the clean solvent [19], [20], [21]. A darker colour indicates a higher dispersion effectiveness.

The SQT is part of the Australia 2012 National Plan Operational Guide, which aims to provide oil spill response teams with an efficient and effective method of evaluating the dispersion effectiveness of oil dispersants on-site [16, p. 23], [19]. It is associated to the WSL test and adapted from the United States Environmental Protection Agency (US EPA) Field Dispersant Effective Test (FDET) [19].

Experiment

- Solutions

- Magna 1100
- Magna 1101
- Seawater
- Oil to be dispersed (test oil) – crude oil, bitumen based

- Clean solvent (used for extraction of dispersed oil) – white spirit

Equipment

- 500ml beaker
- 250ml beaker
- Syringe/measuring cup/dropper
- 100ml and 250ml of separating funnels (substitute: 110ml and 500ml squeeze bottles respectively)
- 50ml measuring cylinder
- 250ml conical flask
- Measuring flask
- Test tubes/small vials
- Stopwatch

Experimental procedures

Part A – Preparing Test Solution

1. Using a 500ml beaker, measure 250ml of seawater and pour it into a 500ml squeeze bottle.
2. Using a measuring cup, transfer 20ml of test oil to the surface of the seawater. Start the stopwatch.
3. After 1min, transfer 1.0ml of dispersant to the test oil. The dispersant should be added dropwise, starting from the middle of the oil lens and moving radially towards the outside of the lens. This is to ensure uniform distribution of the dispersant on the surface of the oil.
4. Secure the squeeze bottle.
5. When the stopwatch reads 2.5mins, start the rotation of the squeeze bottle for 2mins. The bottle should be rotated along its horizontal axis, at right angles to its longitudinal axis, at about 33rpm.

6. After 2mins (stopwatch should read 4.5min), stop the rotation. Leave the mixture (seawater + oil + dispersant) to stand for 1 min.
7. After 1 min (stopwatch should read 5.5min), stop the stopwatch. Using a measuring cylinder, transfer 50ml of the mixture into a 110ml squeeze bottle. This should be done within 10s.
8. Rinse the measuring cylinder with white spirit. Then use it to transfer 10ml of white spirit into the 110ml squeeze bottle.
9. Seal the squeeze bottle and shake for 1min.
10. After 1min, pour the solution into a test tube/beaker labelled Mixture M. Let the dispersed oil and seawater settle into its layers before colour comparison with standards solutions.

Part B – Preparing Standard Solution (refer to Figure 1)

1. Measure 250 ml of clean solvent into a measuring flask.
2. According to the dispersant-to-oil (DOR) ratio of 1:20, first measure 20ml of test oil using a measuring cup labelled Mixture D/O. Next, add 1ml of dispersant dropwise into the same measuring cup using a 1ml syringe. Ensure that the dispersant is evenly spread over the surface of the oil.
3. Stir the solution thoroughly to ensure homogeneity.
4. Using a 1ml syringe, add 0.5ml of Mixture D/O into the measuring flask to obtain S1. This represents 100% dispersion. The ratio is calculated based on the same rationale as the dispersion test i.e. 2ml dispersant/oil in 100ml seawater diluted 10 times.
5. Add 10ml of S1 to vial labelled S1.
6. Using a measuring cylinder, add 60ml of S1 from the measuring flask to 20ml of clean solvent in a separate measuring flask. This is S2 with 75% dispersion.
7. Transfer 10ml of S2 into a vial labelled S2.
8. Using a measuring cylinder, add 40ml of S1 from the measuring flask to 40ml of clean solvent in a separate measuring flask. This is S3 with 50% dispersion.

9. Transfer 10ml of S3 into a vial labelled S3.
10. Using a measuring cylinder, add 20ml of S1 from the measuring flask to 60ml of clean solvent in a separate measuring flask. This is S4 with 25% dispersion.
11. Transfer 10ml of S4 into a vial labelled S4.
12. Add 10ml of Mixture M from part A of the experiment into the vial labelled Test.
13. Compare the colour of the solution in the vial labelled Test to the other standard solutions in vials S1 to S4.

Note: For Type II dispersant such as Magna 1101, dilution with seawater has to be carried out before its application to seawater. The dilution ratio is 1:10 (1 part of Magna 1101 dispersant to 10 parts of seawater).

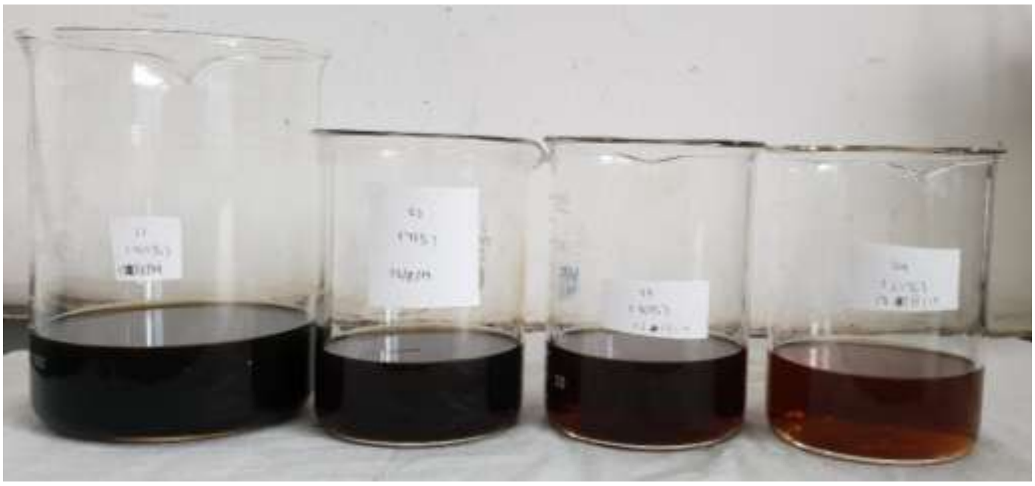

Figure 1. Standard solutions. From left: S1(100%), followed by S2(75%), then S3(50%) and S4(25%)

Results and Discussion

Magna 1100 (Type I dispersant)

TABLE I.
Observation Table of Magna 1100

OBSERVATION			REMARK
DAY	WEEK	MONTH	
15/8/19	-	-	2 layers of liquid is observed. The top layer has a dark brown colour while the bottom layer is chalky.
16/8/19	-	-	2 layers of liquid is still observed. The top layer remains dark brown while the bottom layer has become clear.

Figure 2. Mixture M (DOR 1:20)

As seen from Figure 2, there are 2 layers of liquid. The darker top layer represents the layer of dispersed oil suspended in clean solvent while the chalky bottom layer is seawater with some dispersed oil that is not extracted by the clean solvent.

Figure 3. Colour Comparison of Mixture M (DOR 1:20) with standard solutions

From Figure 3, it is observed that the colour of the dispersed oil layer of Mixture M (DOR: 1:20) is similar to standard solution S2, which represents 75% dispersion effectiveness. This means that the dispersion effectiveness of Magna 1100 DOR of 1:20 is approximately 75%.

According to the UK WSL test standard, a dispersant is only approved for use if its dispersion effectiveness is at least 30%. Since Magna 1100 has a dispersion effectiveness of about 75%, it can be approved to be used as a dispersant as its dispersion effectiveness has exceeded the requirement of 30% (i.e. passed the WSL test).

Magna 1101 (Type II dispersant)

TABLE II.
Observation Table of Magna 1101

OBSERVATION			REMARK
DAY	WEEK	MONTH	
20/8/19	-	-	2 layers of liquid is observed. The top layer has a brown colour while the bottom layer is chalky.
21/8/19	-	-	2 layers of liquid is still observed. The top layer remains brown while the bottom layer has become clear.

Figure 4. Mixture M (DOR 1:20)

As illustrated in Figure 4, 2 layers of liquid is observed. The darker top layer shows the layer of dispersed oil suspended in clean solvent while the chalky bottom layer represents seawater with some dispersed oil that is not extracted by the clean solvent.

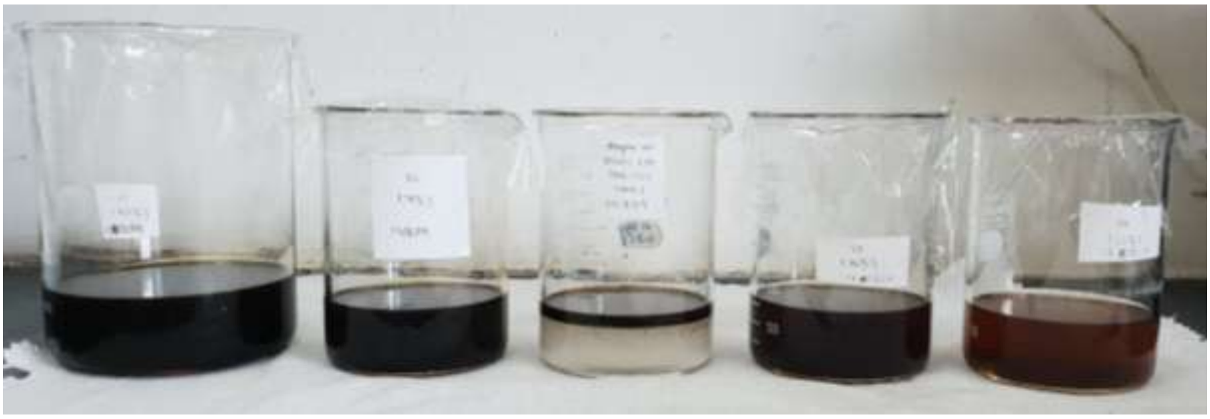

Figure 5. Colour comparison of Mixture M (DOR 1:20) with standard solutions

As shown in Figure 5, the colour of the dispersed oil layer of Mixture M (DOR: 1:20) is similar to standard solution S2, which represents 75% dispersion effectiveness. This means that the effectiveness of Magna 1101 dispersant in oil dispersion with DOR of 1:20 is around 75%.

According to the UK WSL test, a dispersant is approved for use only if its dispersion effectiveness is 30% or higher. Since the dispersion effectiveness of Magna 1101 (DOR 1:20) is more than 30%, it can be approved for use (i.e. passed the WSL test).

Discussion

As demonstrated by the results, both Magna 1100 and Magna 1101 are highly effective oil dispersants with a dispersion effectiveness of approximately 75%. However, it is important to note the limitations in this experiment.

The first limitation is the nature of standard solutions used for colour comparison. In this experiment, the standard solutions are made according to the SQT procedure (reference to [19]). These standard solutions contain about 0.2% of test oil dissolved in clean solvent (refer to Figure A). Conversely, the test solution produced from the WSL test consist of about 6% test oil (refer to Figure B). This difference in the test oil content implies that the colour of the standard solutions may appear lighter compared to that of the test solution, leading to misleading conclusions about the dispersion effectiveness of the dispersants. As a result, the actual dispersion effectiveness of the dispersants may be lower than the test result obtained in this experiment. Hence, further investigations should be carried out to verify the dispersion effectiveness of the dispersants.

Figure A

Percentage of Oil in Standard Solutions from SQT

The following is calculated with reference to the SQT from the National Plan Oil Spill Dispersant Effectiveness Field Test Kit (Nat-DET) Operational Guide by AMSA (or reference to section 3.3 part B) .

Table III.
Percentage of crude oil in SQT standard solution

Standard Solutions	Volume of S1/ml	Volume of solvent/ml	Effective dispersion/%	Amount of crude oil/% (2s.f)
S1	-	250	100	0.19
S2	60	20	75	0.14
S3	40	40	50	0.095
S4	20	60	25	0.048

For S1: gb

0.5ml of dispersant/oil mixture was mixed with 250ml of solvent

The original dispersant/oil mixture has 1ml dispersant and 20ml oil.

Total volume: 21ml

% of oil in original mixture = $\frac{20}{21}$ x100 = 95.23809524 ≈ 95%

Volume of oil in S1 = 0.5 x $\frac{95.23809524}{100}$ = 0.476190476

% of oil in S1 = $\frac{0.476190476}{250.5}$ x 100 = 0.190095998 ≈ 0.19

For S2:

Volume of oil in S2 = 60 x $\frac{0.190095998}{100}$ = 0.114057599

% of oil in S2 = $\frac{0.114057599}{80}$ x 100 = 0.142571999 ≈ 0.14

For S3:

Volume of oil in S3 = 40 x $\frac{0.190095998}{100}$ = 0.076038399

% of oil in S3 = $\frac{0.076038399}{80}$ x 100 = 0.095047999 ≈ 0. 095

For S4:

Volume of oil in S4 = 20 x $\frac{0.190095998}{100}$ = 0.0380192

% of oil in S4 = $\frac{0.0380192}{80}$ x 100 = 0.047524 ≈ 0. 048

Figure B

Percentage of Oil in test solution obtained from WSL

The following is calculated with respect to the WSL test from [1] (or reference to section 3.3 part A).

With reference to experimental procedure part A,

Procedure 1: 1ml of dispersant to 20ml of crude oil to 250ml of seawater (oily water).

Total volume: 271ml

% of oil in mixture D/O = $\frac{20}{271}$ x 100 = 7.380074%

Procedure 2: 50ml of oily water to 10ml of clean solvent (dispersed oil solution)

Volume of oil in 50ml of oily water = $\frac{7.380074}{100}$ x 50

= 3.690037ml

% of oil in dispersed oil solution = $\frac{3.690037}{60}$ x 100

= 6.150062%

≈ 6%

The second limitation is the use of visual colour analysis instead of a spectrophotometer. Determination of dispersion effectiveness via visual colour analysis is not very accurate as it does not provide an exact value [19]. Furthermore, visual colour analysis may be relatively subjective as one's perception of colour may differ from others [24], [25]. Nonetheless, the visual colour analysis method is still useful as it can provide an estimate of the dispersion effectiveness of the dispersant, especially when swift evaluations are desired.

The third limitation is the type of equipment used in certain parts of the test procedure. For instance, the mixing of the seawater, oil and dispersant (reference to Part A steps 1-5) is carried out using a squeeze bottle instead of a separating funnel.

Another example is the use of manual rotation in Part A step 5 of the experimental procedure instead of a machine-powered rotation such as a motor driven rack. The deviation from the specified apparatus in the reference test could result in systematic errors, leading to inaccuracies occurring in the test results [26], [27].

Conclusion

In a nutshell, through the modified WSL LR448 test, both Magna 1100 and Magna 1101 were shown to have a high dispersion effectiveness of 75%. This means that both oil dispersants have passed the WSL test requirement of minimum 30% dispersion effectiveness for type I and type II dispersants.

To improve the accuracy and reliability of this experiment, a spectrophotometer can be used in place of visual colour analysis for dispersion effectiveness evaluation. A spectrophotometer is more precise than visual colour comparison; thus, it can better assess the dispersion effectiveness of the dispersants. Additionally, proper equipment should be utilized to reduce the systematic errors that may arise due to the use of unsuitable equipment.

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