

## Experimental Studies on the Weathering of Chemical Products in Open Cells to Predict Their Behaviour in Case of a Spill

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### Abstract

Recent data highlights the growing trend in the transport of dangerous substances and the consequent evolution in legislation concerning such substances, whether on a European level (evolution of the Standard European Behaviour Classification, or SEBC code) or on a worldwide scale (new MARPOL-Annex II classification which entered into force on 1<sup>st</sup> January 2007, the OPRC-HNS Protocol, as well as the HNS Convention). This evolution is however based exclusively on data from the literature which all too often cannot be applied to spills, as it does not take into account the influence of factors in the marine environment on the physico-chemical characteristics of the product spilled. For instance, the salinity and temperature of seawater can significantly affect chemicals' hydrosolubility.

This study aimed to obtain experimental data on the behaviour of 3 chemical products when released at sea and to characterise them through *in situ* experiments. For this purpose, experimental open cells were set up in Brest Bay, each of them made of a 9-square-metre surface surrounded with a 3-metre-high nylon skirt set on a metal structure. As the cell sides were flexible, the contained water was subject to the influence of swell and waves. As the cells were not covered, the sea surface was subject to atmospheric influences (wind, sunshine and rain). After releasing the products at the surface, weathering processes were studied by measuring the viscosity of the slick, solubility in the water column and gas cloud formation. These experiments were carried out in two stages: *in situ* analyses first to characterise solubility in the water column (SF-UV turner design), then the vapour in the atmospheric compartment (PhotoIonization detector). In addition, slick samples were taken to monitor the emulsification process and/or to characterise its extension.

### 1. Introduction

Over the past years, chemical shipping has been relentlessly increasing, leading to changes in regulations governing this mode of transport. While many HNS<sup>1</sup> classifications exist according to requirements, such as danger for man or their chemical nature, three categorisations are particularly important in terms of marine pollution: the International Maritime Organization (IMO) MARPOL<sup>2</sup> classification, the hazard profiles produced by the IMO group of experts (Gesamp, 2002) and the Standard European Behaviour Classification system of chemicals spilled into the sea (SEBC code). A chemical substance therefore cannot be transported in bulk, i.e. in one or more tanks of a chemical tanker, without being referenced in one of these three classifications. The aim of this referencing effort, under the

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<sup>1</sup> Hazardous and Noxious Substances

<sup>2</sup> MARPOL: International Convention for the Prevention of Pollution from Ships.

responsibility of IMO, is to cover all types of pollution liable to be caused by ships and their cargo. Liquid substances are divided into four categories (IMO, 2006):

Category X: Noxious Liquid Substances which, if discharged into the sea from tank cleaning or deballasting operations, are deemed to present a major hazard to either marine resources or human health and, therefore, justify the prohibition of the discharge into the marine environment;

Category Y: Noxious Liquid Substances which, if discharged into the sea from tank cleaning or deballasting operations, are deemed to present a hazard to either marine resources or human health or cause harm to amenities or other legitimate uses of the sea and therefore justify a limitation on the quality and quantity of the discharge into the marine environment;

Category Z: Noxious Liquid Substances which, if discharged into the sea from tank cleaning or deballasting operations, are deemed to present a minor hazard to either marine resources or human health and therefore justify less stringent restrictions on the quality and quantity of the discharge into the marine environment; and

And, Other Substances: Substances which have been evaluated and found to fall outside Categories X, Y or Z because they are considered to present no harm to marine resources, human health, amenities or other legitimate uses of the sea when discharged into the sea from tank cleaning or deballasting operations. The discharge of bilge or ballast water or other residues or mixtures containing these substances are not subject to any discharge requirements of MARPOL Annex II.

The MARPOL classification, which draws upon the SEBC code (Bonn Agreement 1994), therefore takes into account the behaviour of products in the event of a spill at sea, i.e. the way in which they will evolve over the first hours once in contact with water. Predicting this behaviour is one of the key phases in establishing a spill response strategy. The SEBC code is used to determine the theoretical behaviour of a substance according to its intrinsic physico-chemical properties (density, solubility, vapour pressure and viscosity), and then to place it within one of the 12 families identified (Bonn Agreement 1994). It is therefore possible to identify the environmental compartment (atmosphere, surface, water column or sediment) towards which a given substance will tend to migrate, based on its material safety data sheet. This information is crucial for operators in charge of spill response.

However, this classification draws upon the physico-chemical properties obtained in laboratory conditions, i.e. in experimental conditions that are far removed from the reality of the field. For instance, solubility is determined in freshwater at a temperature of 20°C. However, in the event of a spill, the environmental conditions will be quite different, and the substance's actual behaviour may in reality differ from that predicted in the SEBC code. This gap between theory and field reality will be all the more perceptible when there is a long delay between the spill and the detection of the product in the environment. Immediately after the spill, the physico-chemical properties of the slick will evolve under the influence of environmental factors such as wind and sunshine, which promote evaporation, as well as temperature and salinity, which promote dissolution (Xie, 1997).

The aim of this paper is to present an experimental system developed by *Cedre* which can be used to monitor the fate in the medium and long term, in conditions as close as possible to field reality, of a substance spilled at sea. This study focuses on the behaviour of three chemicals, whose persistence at the water surface, evaporation and dissolution were monitored.

## **2. Materials and methods**

The aim of this experimental investigation was to characterise the fate of liquid chemicals released at the sea surface.

## 2.1. Floating cell enclosures

Three floating cells were constructed by *Cedre* and moored in Brest harbour, France (48°22'32N and 4°29'32W) to study the influence of natural conditions (i.e., prevailing air and sea temperatures, wind, radiant energy) on the weathering of products spilled onto the sea surface (Figure 1). Each cell consisted of a 3 x 3 m rigid aluminium framework surrounded by a nylon skirt which extended approximately 2 m below and 0.6 m above the sea surface. As cells were not covered and were open to the sea at the base, evaporation and dissolution processes were unimpaired but lateral dispersion at the sea surface was restricted. Observations have shown that while wind-induced capillary waves were reduced by the presence of the skirt, the majority of the mixing action of the sea passed into the cell almost unimpeded. The volume of seawater inside the skirt of each floating cell was approximately 18 m<sup>3</sup>. As the floating cell enclosures were independent of each other, replica experiments could be conducted simultaneously.



Figure 1: Floating cell enclosure

## 2.2. Chemical products tested

Following an investigation into the chemicals transported most frequently and in the greatest quantities, three products were selected: xylene, methyl methacrylate and methyl ethyl ketone.

Xylene is a volatile aromatic hydrocarbon produced through petrochemistry. It is in liquid form at room temperature and is often a mixture of isomers (ortho, para and meta – often the most dominant) containing variable proportions of ethylbenzene, although always below <15%. In France, xylene is the most commonly used hydrocarbonated solvent, with 34,000 tonnes in 2004, in particular in the paint, varnish, glue and printing ink industries. The main physico-chemical characteristics of this substance indicate that it will, in theory, behave as a floater/evaporator with very low solubility ( $V_p = 8.9\text{hPa}$  at 20°C, density of  $0.88\text{g}\cdot\text{cm}^{-3}$ , and solubility between 175 and 200 mg per litre of seawater; Cedre, 2007 and Lyman et al., 1996).

Methyl methacrylate (MMA) is described as a transparent volatile liquid with a characteristic smell that can be detected at very low concentrations (< 1 ppm in air). It is mainly used in the polymer and copolymer industry (plastic sheets such as Plexiglas®, Perspex® and Lucite...). Its vapour pressure is 3.9 kPa at 20°C, its solubility  $15\text{ g}\cdot\text{L}^{-1}$  and its density  $0.944\text{ g}\cdot\text{cm}^{-3}$  (Cedre, 2008).

Methyl ethyl ketone (MEK) is a sweet-smelling colourless liquid. It is a volatile organic compound, that is readily flammable and is of little harm to the environment. MEK is

an eye and respiratory irritant as well as a central nervous system depressant in humans. It is mainly used as a solvent in various coatings including vinyl, nitrocellulose and acrylic coatings. It is also used as an extraction agent in certain oils as well as in the processing of products and food ingredients. Its vapour pressure is 10.5 kPa at 20°C, its solubility 158 g.L<sup>-1</sup> and its density 0.805 g.cm<sup>-3</sup>. It is therefore a highly evaporative and soluble product, with particularly low persistence at the water surface (Cedre, 2009).

### **2.3. Experimental protocol**

The test, replicated for each product, required 25 litre releases at the sea surface of each floating cell. To monitor physical oceanographic conditions that may influence the behavioural processes of products, weather conditions, prevailing light, wind speed and temperature conditions were recorded, for both air and sea.

#### **2.3.1. Slick sampling**

Slicks were sampled daily, with a funnel equipped with a tap, in order to characterise the emulsification processes. Samples were transferred into 0.5 L amber bottles and brought back to the laboratory for viscosity measurement and water content determination.

Before the release of xylene, the internal standard pentacosane, a non-evaporating and insoluble substance, was introduced. By monitoring the pentacosane/xylene relation throughout the trial, it was possible to monitor the disappearance kinetics of xylene at the water surface.

#### **2.3.2. Water column sampling**

The natural dispersion of the products in the water column was monitored by performing *in situ* fluorescence measurements at 2 positions in each floating cell at 3 depths (0.5, 1 and 1.50 m). In addition, for each *in situ* fluorescence measurement (at one position and one depth), a sample of seawater (1 L) was taken. All samples were placed in an amber bottle and were stored in a refrigerator before analysis, performed the following day. These samples, which were analysed by GC-MS after liquid-liquid extraction, were used to correct data obtained by fluorimetry measurements (Katz, 1987).

#### **2.3.3. Air monitoring**

During the tests, evaporation was monitored by measuring the Volatile Organic Carbon using two PhotoIonization Detectors (PID MiniRAE 2000, RAE systems). A stationary PID was placed in a corner of the floating cell, while the second was placed on a wind vane so as to monitor evaporation independently of the wind direction.

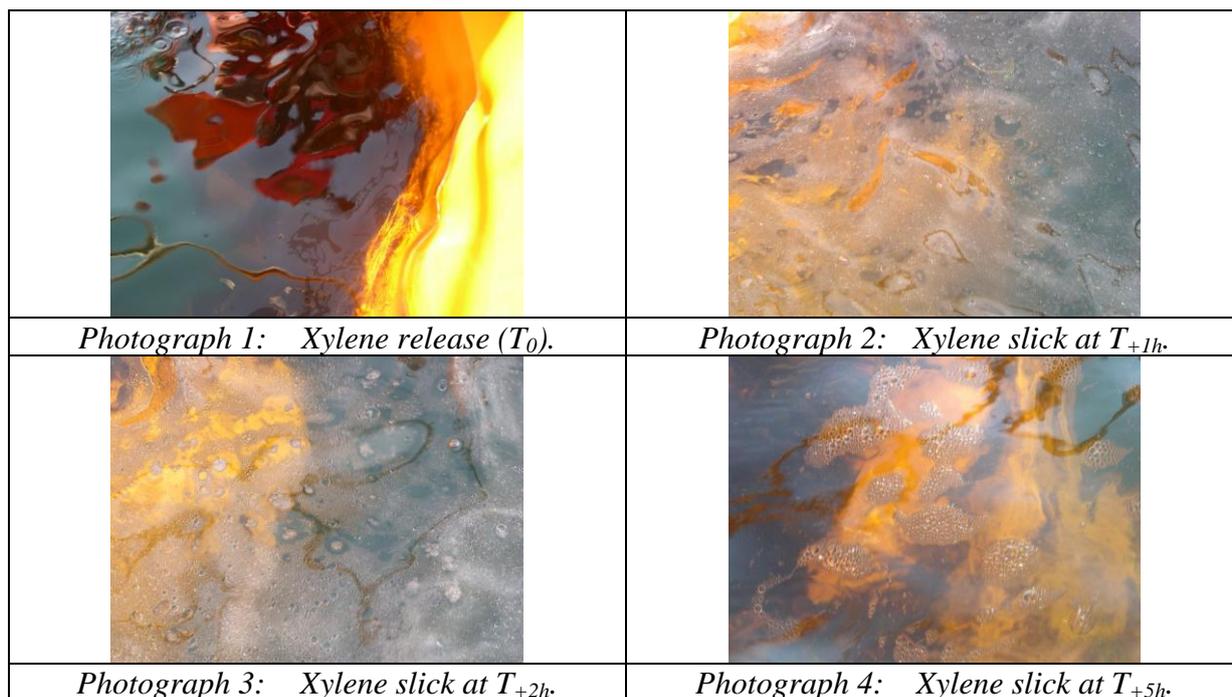
## **3. Results and discussion**

### **3.1. Xylene**

To study the fate of xylene, two 25 litre releases were performed.

#### **3.1.1. Observations**

Photograph 1, taken at T<sub>0</sub>, illustrates the chemical release. One hour after the release (Photograph 2), the slick began to emulsify and, two hours later, the emulsion covered the entire surface of the floating cell (Photograph 3). After 5 hours, the slick began to fragment and several small slicks scattered across the entire cell appeared (Photograph 4).



### 3.1.2. Prevailing sea and weather conditions

The week of the trials was marked by relatively harsh weather conditions, with a slight sea according to the Douglas sea scale (sea 3), a north-easterly wind at force 3 on the Beaufort scale ( $12$  to  $19 \text{ km.h}^{-1}$ ) and low temperatures (between  $3$  and  $5^\circ\text{C}$ ). Several snow showers occurred during this week.

### 3.1.3. Slick persistence

Figure 2 shows the persistence of the xylene slick at the water surface. The results are expressed as a percentage of the quantity spilt at  $T_0$ . After 2 hours, over 50% of the slick had disappeared, and after 5 hours, less than 30% of the initial quantity remained. The quantities lost were either transferred from the surface to the air (evaporation) or from the surface to the water column (natural dispersion). After day 2, no traces of the slick remained visible at the surface. The time during which the slick was present was estimated at 34 hours.

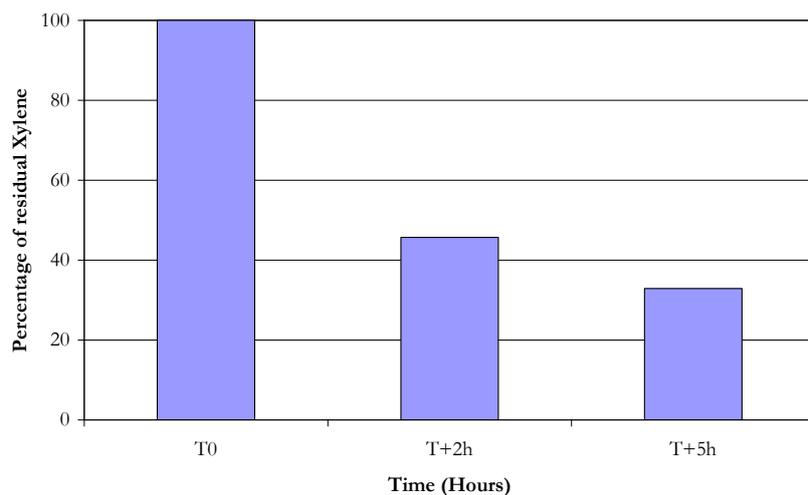


Figure 2: Evolution over time of the slick remaining at the surface (average of results obtained using samples taken from cells 1 and 2).

### 3.1.4. Monitoring of emulsification

Emulsification kinetics could only be monitored during the first day of the experiment and are presented in Figure 3.

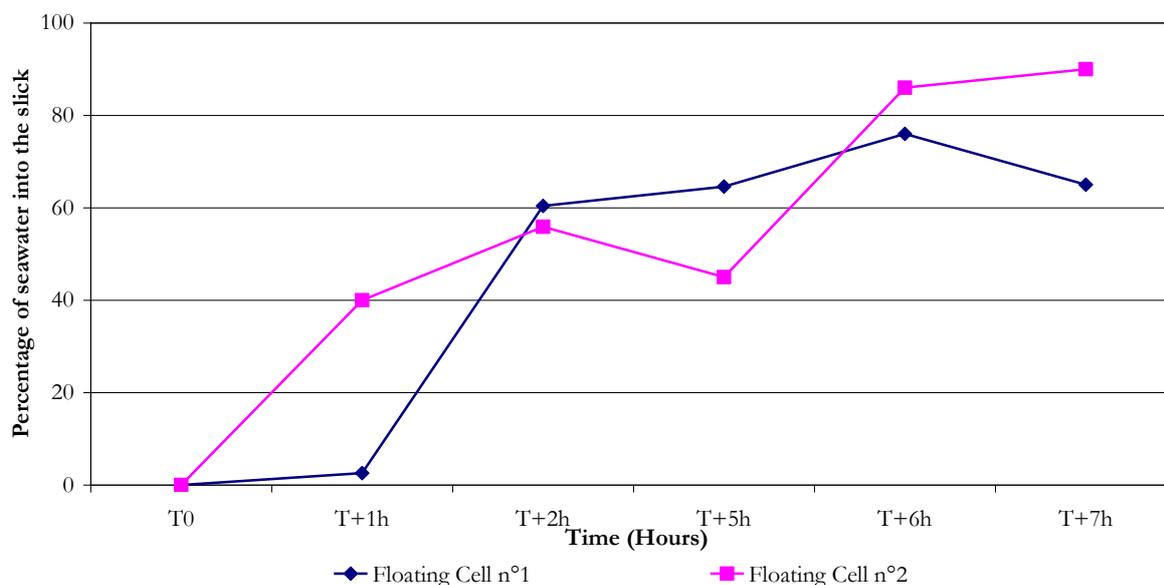


Figure 3: Emulsification kinetics of xylene slicks within cells 1 and 2.

Xylenes have a high water sorption capacity and rapidly form emulsions. At  $T_{+7h}$  the water content of slicks in cells 1 and 2 were respectively 65% and 90%. However, these emulsions are unstable as they are non-persistent: from day 2, it was no longer possible to sample the surface slick due to insufficient quantities, and on day 3, no pollution remained visible.

**3.1.5. Monitoring of dispersion in the water column**

Figures 4 and 5 show the xylene concentrations in the water column, respectively in cells 1 and 2, at the three sampled depths (0.5, 1 and 1.5 m) over time. After 29 hours, concentrations of the substance were no longer detected.

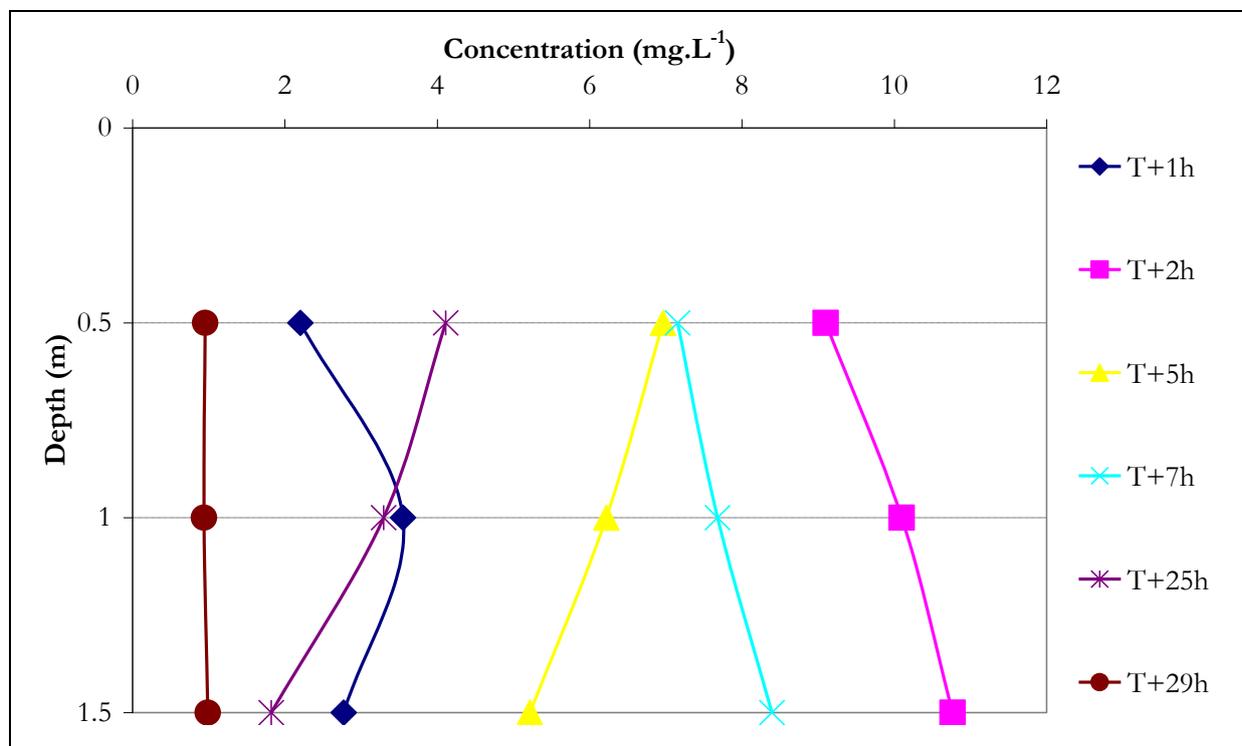


Figure 4: Evolution over time of xylene concentrations in the water column in cell 1.

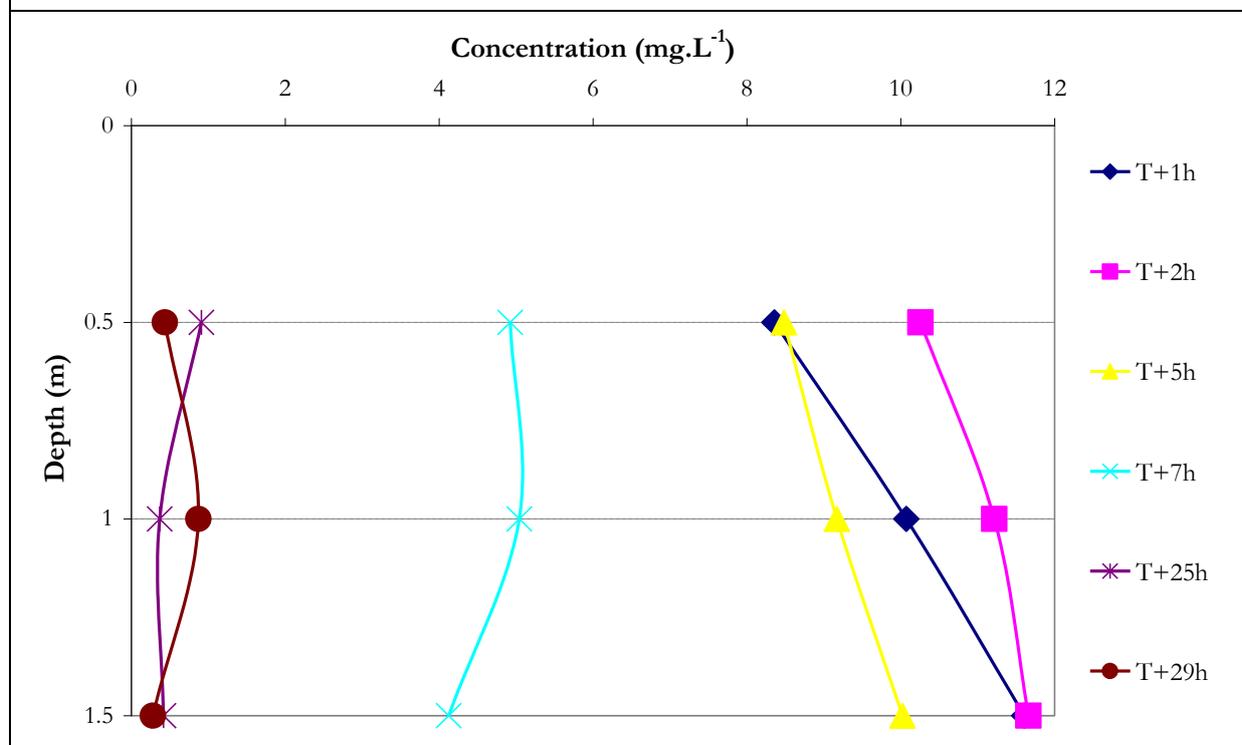


Figure 5: Evolution over time of xylene concentrations in the water column in cell 2.

In the two cells, the maximum concentrations were obtained at  $T_{+2h}$  (10.8 and 11.7 mg.L<sup>-1</sup>, respectively in cells 1 and 2), and represented a quantity of around 200 g of dispersed xylenes. These concentrations then gradually decreased over time, and dropped below 1 mg.L<sup>-1</sup> at  $T_{+29h}$ . Beyond this sample time, traces of xylene were no longer detected in the water column.

During these *in situ* trials, the natural dispersion of xylenes in the water column (dissolution + emulsification) showed rapid kinetics, and the concentrations measured were higher than expected: 100 times higher than the theoretical solubility found in the literature.

### 3.1.6. Behaviour of xylenes

These trials demonstrated that the persistence of a slick of xylenes at the water surface is relatively short-lived (around 2 days in these trial conditions, not very conducive to evaporation) and that this disappearance could be explained by dissolution processes which were higher than expected (in the literature this chemical is identified as having low solubility). In terms of the transfer of the chemical into the atmosphere, evaporation kinetics could not be measured due to the particularly harsh weather conditions (the snow interfered with the photoionisator). However, by comparing it to styrene (these two products have an equivalent vapour pressure and are classified as FE according to the SEBC code), it can be supposed that xylenes evaporate less quickly than theory predicts: after 5 hours, the entire styrene slick had disappeared, while this took 34 hours with xylenes. These results can be explained by emulsification processes: the xylene slicks were rapidly emulsified to a great extent during the first day (90% at  $T_{+7h}$ ), a process which does not take place with styrene. This raises the question of surface tension which is not taken into account in the SEBC classification; yet it is this factor that defines a product's capacity to emulsify, and emulsification promotes natural dispersion in the water column.

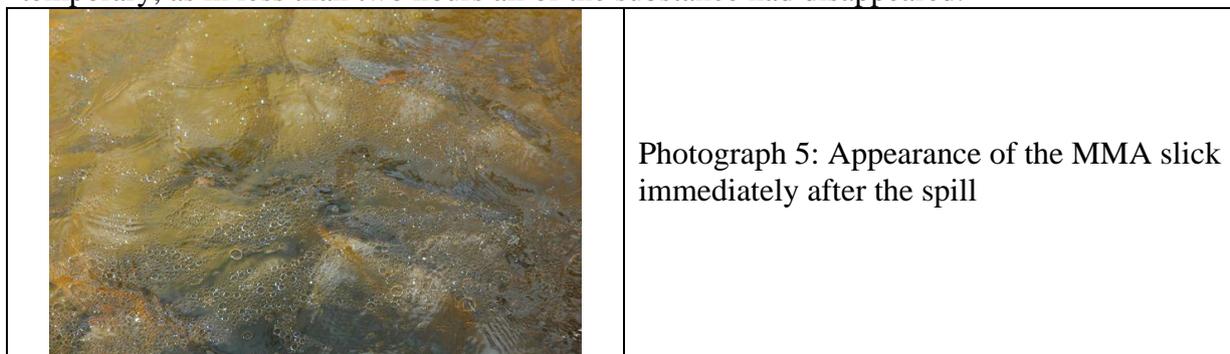
The results obtained *in situ* therefore directly contradict the literature which mainly describes the behaviour of a xylene slick in terms of transfer into the atmosphere. It is clear that the slick will initially emulsify, causing evaporation kinetics to slow down and promoting transfer into the water column by natural dispersion processes (dissolution + emulsification). It is important, however, to remember that the sea and weather conditions on site should be taken into account when considering these results, as, in these trials, the low temperatures curbed evaporation processes (we note that in December 2005, following a spill of benzene in the Chinese Songhua River, high concentrations were measured in the water over a long period of time, although this product is classed E and insoluble, however due to low winter temperatures, dissolution processes became dominant over evaporation), and surface agitation promoted emulsification. In terms of response, this means that all the issues related to pollution by soluble products (contamination of the water column and problem of water intakes, impact on benthic flora and fauna...) must be taken into account. Nevertheless, the gas cloud must not be overlooked, as it presents a risk for responders both in terms of intoxication and as an explosion hazard. In the case of xylene, vapours are heavier than air and will move around just above the water surface, pushed by the wind.

## 3.2. Methyl methacrylate

Methyl methacrylate (or MMA) is classed ED according to the SEBC classification, and its main behaviour is evaporation according to its Henry's law constant.

### 3.2.1. Observations

The MMA spread out across the water surface forming a heterogeneous slick: various sized clusters of floating droplets were observed (Figure 28). This phenomenon was only temporary, as in less than two hours all of the substance had disappeared.



### 3.2.2. Prevailing sea and weather conditions

The prevailing weather conditions onsite at the time of the trial are presented in Table 1. The key point is the presence of wind.

Table 1: Evolution of weather conditions during the release of MMA.

| Date                                  | 25/06       | 25/06       | 25/06       |
|---------------------------------------|-------------|-------------|-------------|
| Time                                  | 11:00-12:00 | 12:00-13:00 | 13:00-14:00 |
| Air T (°C)                            | 16.4        | 16.7        | 17.1        |
| Wind speed (m/s)                      | 5.5         | 5.6         | 5.6         |
| Solar intensity (mW/cm <sup>2</sup> ) | 61.5        | 71.2        | 83.4        |

### 3.2.3. Evaporation

The MMA evaporated very rapidly: it was released at 11:18 and the PIDs began to detect significant values from 11:25 (1 ppm at 11:24, then 53 ppm at 11:25). A peak in concentration was reached at 11:27 with 130 ppm, the time at which a local maximum was recorded for light intensity (130 mW/cm<sup>2</sup>), while the wind varied little throughout the duration of measurements (5 to 6 m/s on average with peaks at 7 m/s and minimums at 4 m/s). These results, illustrated in Figure 6, show the importance in evaporation processes of sunshine, which had a greater influence than the wind: positive correlation between solar peaks and evaporation peaks (T<sub>+9</sub>, T<sub>+14</sub>, T<sub>+35</sub>, T<sub>+49</sub>, T<sub>+56</sub>, T<sub>+74</sub> min).

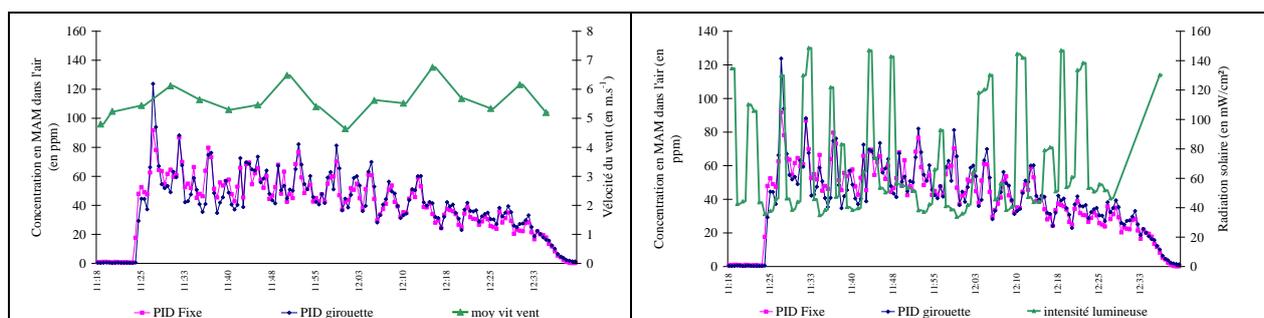


Figure 6: MMA concentrations in the air measured using PIDs (stationary and on the wind vane) and variations in wind and sun intensity throughout the experiment.

The advantage of using the wind vane to monitor the concentration of the product in the atmosphere can be observed in Figure 6: while the shapes of the two curves (stationary PID and PID on wind vane) match, the concentration peaks are more intense for the PID on the wind vane and, in general, its curve shows slightly higher values, indicating better quantification of the vapours emitted by the slick. This result was obtained despite a wind that retained a relatively constant direction throughout the experiment ( $250 - 270^\circ$ ), therefore suggesting that the gain would be even greater in the case of quantification with a swirling wind.

### 3.2.4. Dissolution

Despite its solubility ( $10 \text{ g.L}^{-1}$ ), the MMA did not dissolve at all in these experimental conditions: the chemical was not detected at any of the three depths sampled (0.5, 1 and 1.5 m).

### 3.2.5. Behaviour of methyl methacrylate

MMA is listed as ED according to the SEBC code, i.e. it is first and foremost an evaporator, but also dissolves. In the prevailing weather conditions during the trial, this product only evaporated, and it did so with rapid kinetics: in less than 90 minutes, no traces of MMA were visible at the water surface or detected by the PIDs. Given its negligible dissolution, an atmospheric transfer coefficient can be estimated at around 4.1 litres per square metre per hour ( $4.1 \text{ L.m}^{-2}.\text{h}^{-1}$ ), in these experimental conditions.

In the event of a spill, response should therefore focus on the gas cloud as it will be explosive (MMA can form flammable vapours with air) and toxic (TEEL<sup>3</sup> of 400 ppm). It is therefore essential to have operational tools to visualise the formation and movement of a gas cloud as well as a computer forecast model.

## 3.3. Methyl ethyl ketone

Methyl ethyl ketone (MEK), like MMA, is classed ED.

### 3.3.1. Observations

Immediately after the spill, the MEK spread across the entire water surface within the floating cell to form a homogeneous, colourless slick that was difficult to detect. This observation is consistent with the results obtained in the laboratory on MEK-water interfacial tension which predict complete spreading, or even infinite spreading in the absence of containment. This high degree of spreading promoted the transfer of the product into the atmosphere and the water column, resulting in complete disappearance of the slick in less than 1 hour and the end of sampling and PID measurements at 12:00 (no signal from PID).

### 3.3.2. Prevailing sea and weather conditions

The prevailing weather conditions on site at the time of the experiment are presented in Table 3, and can be summarised as alternating sunny spells and threatening clouds due to the constant wind.

<sup>3</sup> TEEL = TEmporary Exposure Limit

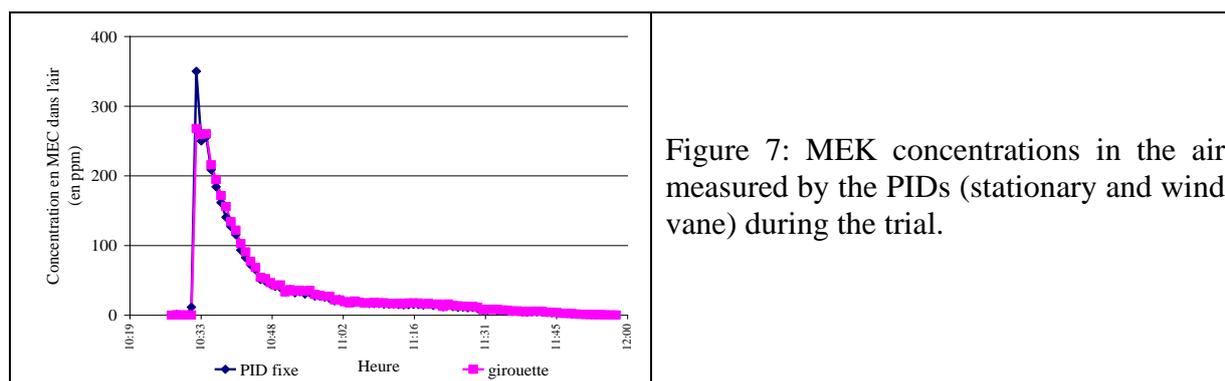
Table 2: Evolution of weather conditions during the MEK release.

| Date                                  | 25/06       | 25/06       | 25/06        |
|---------------------------------------|-------------|-------------|--------------|
| Time                                  | 10:25-11:00 | 11:00-11:30 | 11:30 -12:00 |
| Air T (°C)                            | 15.6        | 15.7        | 15.7         |
| Wind speed (m/s)                      | 5.1         | 5.1         | 5.2          |
| Solar intensity (mW/cm <sup>2</sup> ) | 93.1        | 106.1       | 104.6        |

\* Note that the mean solar intensity on this day was higher than that measured during the MMA trial.

### 3.3.3. Evaporation

The MEK evaporated very swiftly and to a great extent: less than 5 minutes after the spill, a peak in concentration was measured both by the stationary PID and the PID on the wind vane (350 ppm). Concentrations then gradually decreased and levelled out at around 20 ppm at  $T_{+30 \text{ min}}$  before disappearing completely at  $T_{+90 \text{ min}}$  (Figure 7). The evaporation of MEK was so intense that it was unaffected by the wind or sun; no peaks could be correlated with a gust of wind or spell of sunshine.



### 3.3.4. Dissolution

Despite its high solubility ( $\approx 300 \text{ g.L}^{-1}$  i.e. 30 times higher than MMA), MEK did not dissolve at all in the conditions of this experiment: the chemical was not detected at any of the three depths sampled (0.5, 1 and 1.5 m).

### 3.3.5. Behaviour of methyl ethyl ketone

MEK is listed as ED according to the SEBC code, i.e. it is first and foremost an evaporator, but also dissolves. In the weather conditions during this trial, the product only evaporated, and did so with intense, rapid kinetics: in less than 5 minutes, an evaporation peak was observed and thereafter concentrations in the air continually decreased. This behaviour was similar to MMA with, however, even faster evaporation kinetics, unaffected by the wind or sun. This trend can be explained by MEK's capacity to spread widely at the water surface and to form a homogeneous slick. The coefficient is the same as for MMA, i.e.  $4.1 \text{ L.m}^{-2}.\text{h}^{-1}$ , bearing in mind that for MEK it is not constant throughout evaporation. It is estimated that 80% of the slick evaporated in 15 minutes, giving a coefficient of  $8.9 \text{ L.m}^{-2}.\text{h}^{-1}$ ,

while the remaining 20% took around 75 minutes, giving a considerable lower transfer coefficient of around  $0.45 \text{ L.m}^{-2}.\text{h}^{-1}$ .

In the event of a spill, response should therefore focus on the gas cloud which will form rapidly and will be flammable and toxic.

#### 4. Conclusion

In the event of a chemical spill at sea, it is crucial to be aware of the behaviour of the substances involved to determine the most appropriate response strategy. With this in mind, *Cedre* has developed an experimental tool, floating cells, which can be used to characterise the main processes governing a substance's fate.

This paper presents the results obtained during a series of trials in these floating cells, which aimed to characterise the behaviour of three chemicals: xylene, methyl methacrylate and methyl ethyl ketone.

These trials provided two vital pieces of information on these chemicals, to add to the information provided by the SEBC classification. The first relates to xylene which is listed as FE. It became apparent that, in certain weather conditions, xylene will indeed behave as a Floater/Evaporator, however if the temperature drops and especially if the surface agitation and wind are sufficient (wind speed greater than  $3 \text{ m.s}^{-1}$ ), it will form an emulsion and will mainly dissolve, thus exhibiting FDE behaviour. The two other products (MMA and MEK) are classed ED while, in these experimental conditions, they behaved only as Evaporators.

These results highlight the need to study *in situ* the behaviour of chemicals to add to the theoretical classification based only on laboratory data, especially as it is clear that factors such as surface agitation, wind, sunshine and outside temperature significantly affect the transfer of products from the surface to another environmental compartment. The floating cells, developed by *Cedre*, adequately fulfil this purpose, and, what's more, are an innovative tool which, in addition to helping to define a response strategy in the event of a spill, can be used routinely as realistic input for forecast software.

Concretely, such experiments on the behaviour of HNS releases in sea water allow:

(i) adding further informations to the chemical emergency response guides. For example, on the one hand xylene which is considered as a Floater/Evaporator can behave as FDE product; in a case of emergency response in the same weather conditions of the trials, the attention of the intervention team has to be also focalized onto the water column. On the other hand for both other substances (MMA and MEK), the atmospheric compartment has to be especially considered by the intervention team which has, firstly, to protect itself versus the gaseous cloud.

(ii) providing experimental data for the implementation of existing software. Forecast software are an useful tool but few are really validated by field experiments. Here, results are used to validate the Clara software which is fully dedicated to predict the behaviour of HNS at sea.

(iii) to provide the French Navy with operational information. The French navy is in charge of the emergency response at sea in case of incident and, they need accurate data in order to organize the more appropriate response.

### Acknowledgments

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