

**RESEARCH ARTICLE**

**EXPERIMENTAL STUDY OF USING FLUORESCENT METHOD IN  
DETECTING HYDROCARBON LEAKAGES FROM SUBSEA  
STRUCTURES AND COMMERCIAL SHIPS**

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**Abstract**

A large quantity of hydrocarbon is leaked along the Libyan coast every year. The leakage of hydrocarbon comes from commercial ships and/ or hydrocarbon transporting pipelines and structures. In order to detect oil leakages in subsea environment as early as possible, there should first be a method that can distinguish between oil leakages from offshore oil and gas structures from those that have been leaked from commercial ships (either at the ship yard or during waiting times at sea). The leakage if not detected and stopped will lead to a serious of environmental and economical losses. This paper represents a small-scale experiment to study the effectiveness of fluorescent method in detecting hydrocarbon spills which can then be employed on large-scale by the Libyan authorities to minimise hydrocarbon leakages to the environment. This fluorescent method relies on adding a fluorescent material, such as a dye, into the fluid. Also, a light source such as laser or LED is required to excite the fluorescence present in the leaking fluid. The result of the fluorescence experiment confirmed our expectations that naturally present or added fluorescence to seawater can be detected with high sensitivity.

**Key Words:** Subsea, hydrocarbon, leakages, detection, environment, fluorescent.

## دراسة تجريبية لاستخدام طريقة الفلور في الكشف عن تسرب الهيدروكربون من الهياكل البحرية والسفن التجارية

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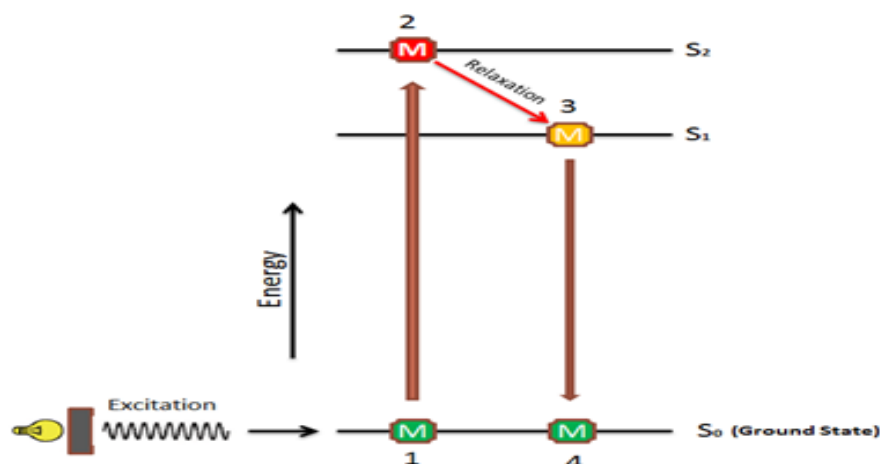
### المستخلص

يتم تسريب كمية كبيرة من الهيدروكربون على طول الساحل الليبي كل عام. تسرب الهيدروكربون يأتي من السفن التجارية و / أو خطوط نقل الهيدروكربون. من أجل الكشف عن تسرب النفط تحت سطح البحر في أقرب وقت ممكن ، يجب أن يكون هناك أولاً طريقة يمكن أن تميز بين تسرب النفط من هياكل النفط والغاز البحرية من تلك التي تم تسريبها من السفن التجارية (إما في ساحة السفينة أو أثناء أوقات الانتظار في البحر). إن التسرب إذا لم يتم اكتشافه وتوقعه سيؤدي إلى خسائر بيئية واقتصادية خطيرة. تمثل هذه الورقة تجربة صغيرة لدراسة فعالية طريقة الفلورسنت في الكشف عن تسربات الهيدروكربونات التي يمكن بعد ذلك استخدامها على نطاق واسع من قبل السلطات الليبية لتقليل تسرب الهيدروكربونات إلى البيئة. تعتمد طريقة الفلورسنت هذه على إضافة مادة الفلورسنت ، مثل الصبغة ، إلى السائل. أيضا ، مطلوب مصدر لإثارة الفلورسنت الموجود في السائل المتسرب. أكدت نتيجة هذه التجربة توقعاتنا أنه يمكن الكشف عن التسربات الهيدروكربون بشكل دقيق من خلال الفلورسنت الموجود أو المضافة بشكل طبيعي لمياه البحر .

## 1. Introduction

The fluorescence method (FM) is defined as a method that uses different light wavelengths to detect leakage through fluorescent material that have been added to fluids in subsea structures. [1]

This technology relies on adding a fluorescent material, such as a dye, into the fluid. [2] Also, a light source such as laser or LED is required to excite the fluorescence present in the leaking fluid. Figure 1.1 shows the process on which the fluorescent method is based. First, the light source is pointed at the material (M) to be monitored which is initially at ground state ( $S_0$ ). The material then gets excited after absorbing the light and, as a result, its energy rises to its highest level at state ( $S_2$ ). After that, the material relaxes and moves to a lower energy level at state ( $S_1$ ) before it returns again to ground state after releasing a photon fluorescent light. [1] The whole process from point 1 to 4 happens very quickly.[3]

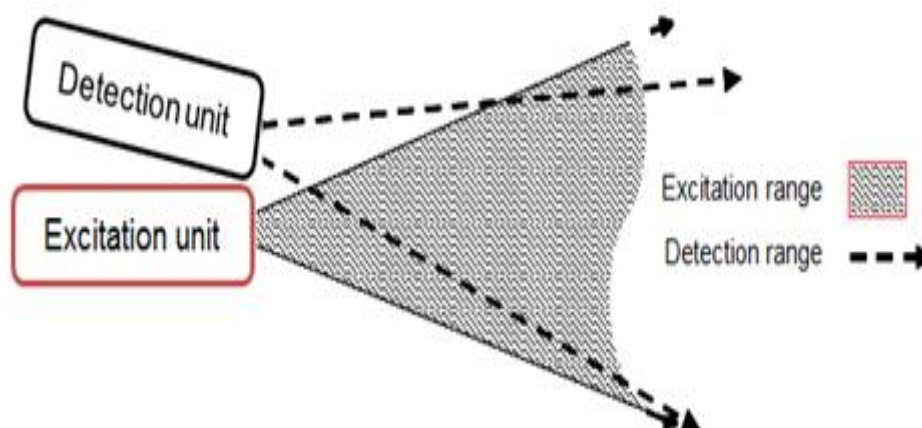


**Figure 1.1:** Principle of the fluorescent method

The fluorescent method is very sensitive in detecting fluorescence in crude oil with ppm detection levels.[3][4][5] This means detecting small leaks is more effective and can be faster than other detection methods. Moreover, the presence of other sources of fluorescence material in sea water, such as dyes added to hydraulic fluids, will not affect the performance of FM, as this technology has the potential to distinguish between fluorescent material added to hydrocarbon fluids and those added to hydraulic fluids, as the two have different fluorescent spectroscopy ranges when exposed to a light source. [3][6]

The fluorescent method is commercially available and has been successfully used with ROVs in non-continuous monitoring of subsea structures. However, permanent monitoring of subsea structures using FM is still immature. [1][7]

Figure 1.2 shows a schematic of the alignment of a fluorescent detection unit and an excitation source unit. It can be seen that both units are pointing in the direction of material to be monitored. The FM is used as point sensor and needs to be close to the subsea structure to achieve an effective leak detection range. [3][8]



**Figure 1.2:** Schematic alignment of a fluorescent detection unit [9]

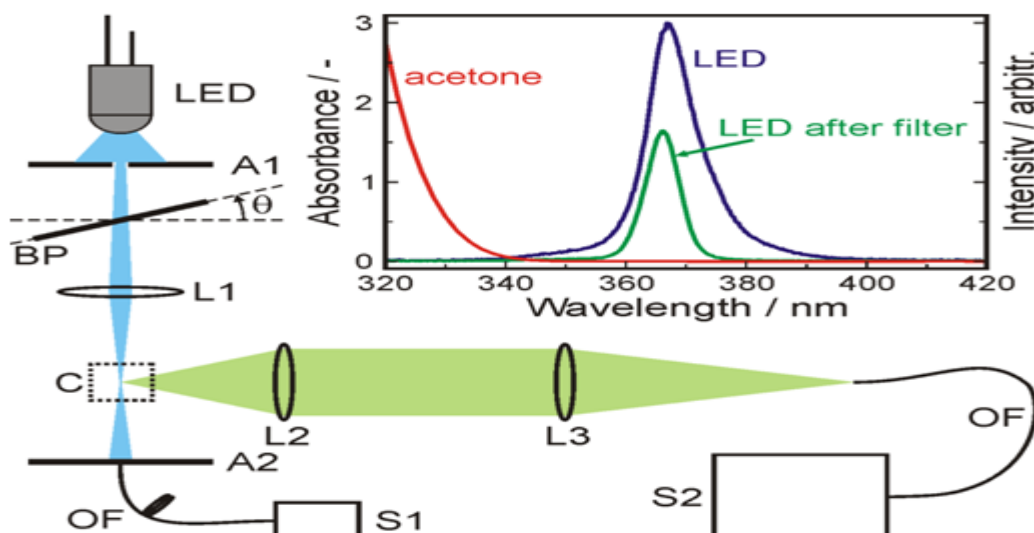
## 2. Fluorescent Method Experiment

This experiment involved testing five samples of water, the aim being to understand the principle of the fluorescent leak detection method when detecting fluorescence present in water. The experiment was carried out in a dark laboratory environment in an attempt to replicate actual subsea conditions.

### 2.1 Equipment

- Ultraviolet Light Emitting Diode (UV-LED) is a light source that was used for exciting the material to be detected. The LED has a wavelength of 365 nanometres.
- Monochromator: This was used to focus incoming light wavelengths from wide to narrow.
- Bandpass (BP) filter: A 10nm nominal BP was used to filter incoming LED light at FWHM bandwidth.
- Imaging spectrograph: This is a detector used to register the emitted photons and create electrical signals.
- Ocean optics (Fiber-coupled USB-spectrometer) were used for monitoring the wavelength and spectral shape of the emitted light.
- Cuvette: This device was used for handling samples.
- Optical Lenses were employed to reflect light wavelengths.
- Computer software was used to analyse received data.

Figure 2.1 shows the equipment set-up and the spectrum of the LED before and after the BP filter.



**Figure 2.1:** Illustration of the fluorescent experiment equipment and the spectrum of the LED before and after the bandpass filter [10]

## 2.2 Preparation of samples

The five samples tested in this experiment were:

1. Distilled water
2. Tap water
3. Sea water
4. Artificial leakage. This was prepared by simply adding crude oil to seawater
5. Dye in seawater. This was prepared by adding a very small amount of dye to sea water.[11][12]

## 2.3 Experiment procedure

After considering all safety aspects, the five samples were tested as follows:

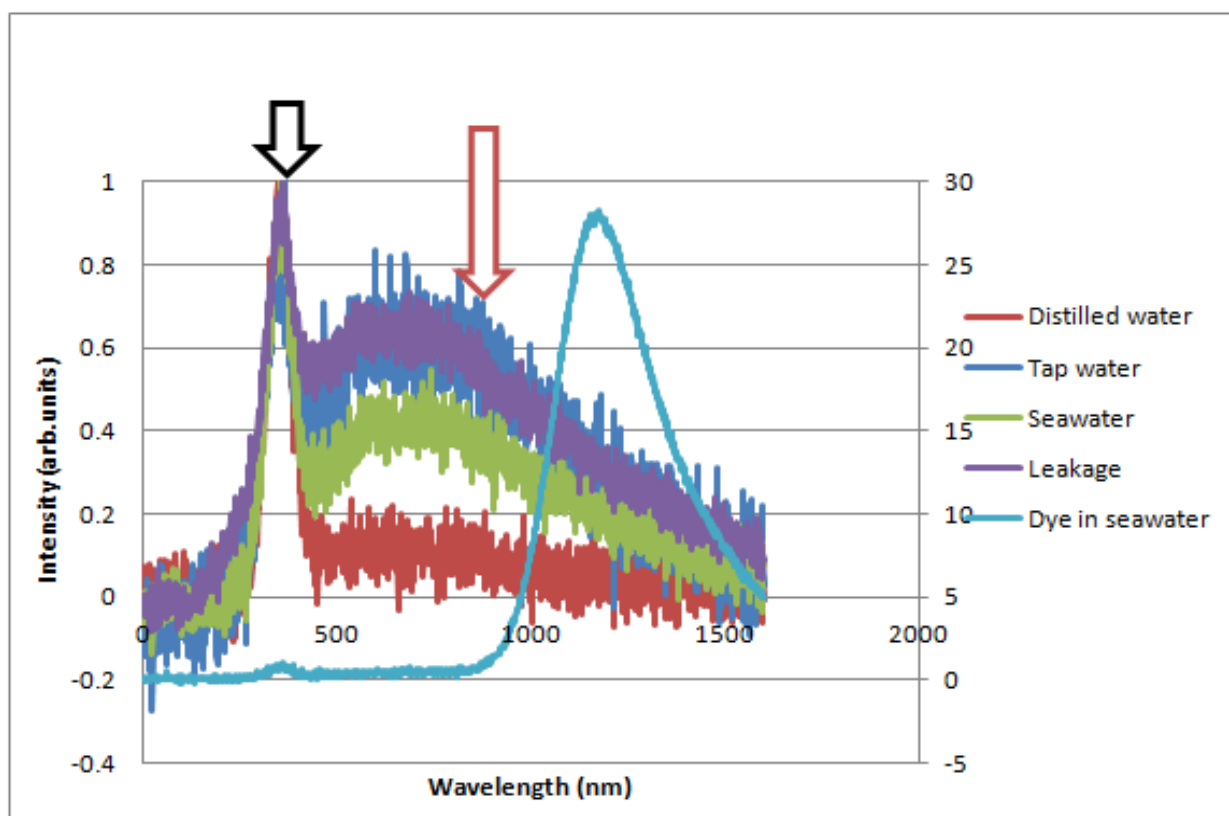
- The LED source was employed together with a 370nm Bandpass filter.[6]
- The light source was then loosely focused into the cuvette holding the sample. The focus diameter was one millimetre.
- Ocean optics were then used for monitoring the light wavelength and spectral shape of the light emission.[6]
- The emitted light signal was then collected at 90° from the incident beam.
- Next, the imaging spectrograph was used to analyse the collected signal. Imaging spectrographs are equipped with an electron-multiplying charged coupled device (CCD) camera detector with an exposure time of 10 seconds.[1][11]
- The computer software was then used to plot the results obtained in a graph of intensity versus wavelength. [13]
- The above steps were undertaken in a dark laboratory environment in order to reflect actual subsea environments. The results of the five samples are reported below.

Please note that the amount and concentrations of dye to sea water was small. (It was not measured). Future work may consider studying the effect of dye concentration to sea water. However, this was not within the scope of this experiment as florescent method can detect very small amounts of dye and/or hydrocarbon in sea environment.

### 3. Results and Discussion

The results generated from the five samples are shown in figure 3.1. It can be observed that there are two spectra peaks: short and wide. The short peak indicated by the black arrow is from the LED emission; the wider spectra indicated by the red arrow are the result of hydrocarbon and/or added fluorescence. For the purpose of this experiment, our interest is focused on the wider spectra. This is because the wider spectra tell us about the intensity of crude oil fluorescence and/or added fluorescence in the tested samples.

It can be clearly noted that the distilled water sample showed the lowest fluorescence spectra emission intensity. This is because the distilling process removes impurities, including fluoride which can be fluorescent in water. This is confirmed by comparing the fluorescence spectra emission intensity from the second sample, (undistilled) tap water. Interestingly, the fluorescence spectra emission intensity from the tap water sample is almost the same as that of the artificial leakage sample. Meanwhile, the fluorescence spectra emission intensity from seawater is more intense than distilled water and less intense than both tap water and artificial leakage. The most obvious trend in figure 3.1 is the added dye in seawater sample.



**Figure 3.1:** Fluorescence spectra for the five tested samples

As can be seen, there is a large difference in the light emission intensity between LED emission spectra and light spectra from added fluorescence (dye). Moreover, the difference in the fluorescence spectra emission intensity between the first four samples and the dye in seawater sample is noticeably large. This large difference in spectra is the result of adding a very small drop of dye to the seawater sample.



## 4. Conclusion

The fluorescence experiment confirmed our expectations that naturally present or added fluorescence to seawater can be detected with high sensitivity. However, applying the fluorescent method to the actual subsea environment can give different results, depending on subsea conditions. One of the challenges of this experiment was keeping the glass cuvettes clean. For instance, when handling cells, some fingerprints were left on the surface, which seemed to cause spectrophotometric measurement errors.

Overall, the success of this experiment indicates that such a leak detection method has the potential to successfully detect subsea hydrocarbon leakages along the Libyan coastline. Furthermore, current research aims to develop this technology further so that it can detect natural fluorescence in hydrocarbon fluids rather than relying on added fluorescence material.

## 5. Acknowledgment

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