

**Natural water
fluorescence
characteristics based on
lidar investigations of
a surface water layer
polluted by an oil film;
the Baltic cruise
– May 2000**

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Abstract

Several sea and laboratory experiments have been carried out to detect and determine the fluorescence characteristics of seawater and its natural fluorescing components and to analyse the influence of oil in the surface water layer on the lidar-induced return signal. The aim of the laboratory investigations was to create a database of different natural water types and of water polluted with Petrobaltic oil. During the r/v 'Oceania' cruise in the southern Baltic Sea two lidar methods were applied simultaneously to detect and analyse the influence of the fluorescence emission of oil on the fluorescence spectrum of seawater. Detection of oil pollution can be used to correct the fluorescent factors of fluorescing components on the basis of natural seawater fluorescence spectra.

1. Introduction

The presence of contaminants such as oil and its products in the surface seawater layer affects the energy exchange balance. The most typical seawater components (referred to as natural water components)

– phytoplankton and yellow substances (coloured dissolved organic matter – DOM) – and oil pollutants of seawater have been studied for many years. Displaying strong absorption and fluorescence emission in the UV and VIS spectra, these pollutants significantly affect the photosynthesis active radiation. As a result, the satellite methods for estimating the chlorophyll *a* concentration on the basis of solar-simulated ocean colour reflectance and airborne fluorescence spectra are prone to error (Hoge & Swift 1986, Hengsterman & Reuter 1990).

Lidars are used in numerous situations for studying oceanographic and optical parameters. Laser systems constitute a highly reliable means of investigating the marine environment and of detecting and determining the spatial and quantitative distributions of several optical and physical parameters of seawater with good resolution and accuracy at long distances. Lidar methods applied to remote sensing techniques or at sea can supply instantaneous information on environmental parameters.

One of the many lidar applications involves detecting an oil-polluted seawater surface through the recording of the attenuated Raman scattering signal in the water versus the reference signal (Hengsterman & Reuter 1990, Piskozub et al. 1997, Piskozub et al. 1998). Lidars are also applied in bathymetric research, and to study the transmission of the optical properties of seawater by the application of signals scattered at different depths and recorded with different time resolutions (Kung & Itzkan 1976, Hoge & Swift 1981, Poole & Esaias 1982). The registration of the induced backscattered spectra at several spectral bands is employed to detect and create maps of below-surface plankton blooms, and to produce maps of hydrographic parameters, especially in areas where such parameters change relatively quickly (Hoge et al. 1988).

The fluorescence lidars recording the continuous spectrum of lidar-induced backscattering and fluorescence emission, known as the return signal, are used to create databases of the fluorescence spectra for particular regions. With the aid of such databases, the marine environment can be monitored rapidly and pollution soon detected (Koblentz-Mishke 1987, Dudelzak et al. 1991, Poryvkina et al. 1994, Drozdowska & Kowalczyk 1999).

Dittman (1988) pointed out the effects of the attenuation of the lidar backscattered signal from seawater, mainly by phytoplankton cells, and found that the degree of this attenuation depended not only on excitation and emission wavelengths, but also on the pigment concentration. Bristow et al. (1981) presented a method for correcting lidar data through the normalisation of the recorded pigment fluorescence signal to the water Raman scattered signal. However, as the results of those studies are

inapplicable to the Baltic Sea, having been obtained elsewhere; moreover, they refer to a different spectral region of the fluorescence emission of seawater obtained by another lidar system using an excitation wavelength of 532 nm (Koblentz-Mishke 1987).

The aim of the present paper is to analyse the spatial distribution of the natural fluorescing components of seawater in the surface layer of the Baltic Sea, and to investigate the influence of oil pollution on seawater fluorescence spectra. Several lidar methods were applied to carry out the above tasks.

The investigations of the natural water emission spectrum were conducted during experiments in the laboratory and at sea. The KLS-10 lidar (Dudelzak et al. 1991) was used during the former, and two lidar systems – FLS-12 (Król et al. 1994, Piskozub et al. 1996) and FLS-UV (Piskozub et al. 1997) – during the latter.

Equipped with a CCD-camera, the KLS-10 lidar system enabled the fluorescence characteristics of natural water types to be investigated in order to generate a natural water fluorescence spectra database and to examine the changes in the fluorescence spectra of water polluted by small quantities of Petrobaltic oil.

With the FLS-12 and FLS-UV lidar systems, seawater emission spectra were recorded in several regions of the Baltic Sea, as were oil films on the sea surface. The experiments enabled the fluorescence characteristics of water according to its chlorophyll *a* and organic matter content to be examined.

2. Experimental arrangement

The laboratory experiment was intended to assess the impact of the origin of the water – port, Gulf of Gdańsk, clean distilled water, river mouth, coastal area, open sea – on its fluorescence spectra, and was focused on the variations in the distribution and forms as well as the intensities of the different spectral bands (different contents of natural components) in the 250–600 nm range excited by the lidar (308 nm). The two main objectives of the experiment were to create a database of natural water types and to assess the effect of oil in the surface water on the latter's fluorescence spectrum.

The experiment at sea involved measuring the lidar-induced fluorescence emission of yellow substances and chlorophyll *a* in the 400–800 nm range, and detecting oil films on the water surface using UV light. The spatial distribution of seawater component concentrations and oil film thickness according to water-types were subsequently analysed.

2.1. The laboratory equipment

2.1.1. The KLS–10 lidar system

The source of the UV radiation of the KLS–10 lidar used in the laboratory experiment was a XeCl (308 nm) excimer laser with a beam divergence of 5 mrad, a laser pulse power of 10 mJ, and a laser pulse repetition rate of 1 or 10 Hz. The receiving system contained a hermetically sealed Cassegrain-Maksutow telescope (diameter of the receiver mirror – 180 mm) and a grating polichromator appropriate to the 250–800 nm spectral range. A receiving telescope of this type can be operated over distances of 8 m to infinity without having to be refocused. The photoreceiver used a CCD-line with a time-gated light intensifier (an electron-light converter with a microchannel plate). By means of a CCD camera, the software controller governed the operating mode of the emitter and receiver systems and the processing of the spectral signals.

2.2. Instrumentation during the sea experiment

2.2.1. The FLS-12 lidar system

The FLS–12 lidar measured the spectral characteristics of seawater during the experiment at sea, and consisted of an excimer laser (308 nm) used as a pumping source, a tuneable dye-laser – the source of the lidar light, and a receiving block containing a telescope, polichromator and electronic set-up. Its emission tunability lay in the 320–670 nm range. The dye could be changed by quickly replacing the cell without disturbing the resonator adjustment. The beam divergence was < 1 mrad and the pulse repetition rate was 1 or 10 Hz. The receiving system was the same as in the FLS–10. A gated linear CCD-detector coupled to the polichromator recorded the lidar-induced fluorescence signal of seawater, the time-space distribution being defined by an impulse delay of 14–99 ns and a strobe of 0.2–999.9 μ s in the 400–850 nm range.

Coumarin 120 was the dye used in the sea experiment described here.

2.2.2. FLS-UV lidar system

The oil film measurements were carried out at sea with a FLS–UV lidar. This consisted of a solid state laser (Nd:YAG) with frequency transformers and a receiver system comprising a polichromator and PMs, and a control unit controlled by a computer. The UV light (299 nm) excites the water and any oil and organic substances in the surface layer of the sea. Before every sea experiment, a laboratory test is carried out to record the reference line

of the Raman signal for clean water, I_0 . During the sea experiment, the water Raman signal came from the oil-polluted water, I , and the changes in the water Raman intensities supplied information about the surface pollution causing the attenuation of the Raman line. The oil film thickness $d = 1/a * \ln(I_0/I)$ was calculated from the ratio of the intensity of the water Raman backscattering signal of pure water I_0 to that of oil-coated water I , where a is the extinction coefficient of the oil (Hengsterman & Reuter 1990).

The oil film thickness calculations refer to Petrobaltic oil, ($a = 0.5 \text{ m}^{-1}$) extracted from the Baltic Platform. The fluorescence spectra are given in Fig. 2, while the other optical characteristics are given by Otremba (2000) and Stelmaszewski (1998).

3. The laboratory experiment

The laboratory measurements were carried out in two stages. In the first step the fluorescence of ‘pure water’ from various sources – tap water, distilled water, sea, Gulf (bay), port, river mouth – was measured (Fig. 1). In the next step small but equal quantities of oil were added to each water sample. The changes in the shape of the water-oil fluorescence emission spectra noted for an oil film and a water-oil emulsion.

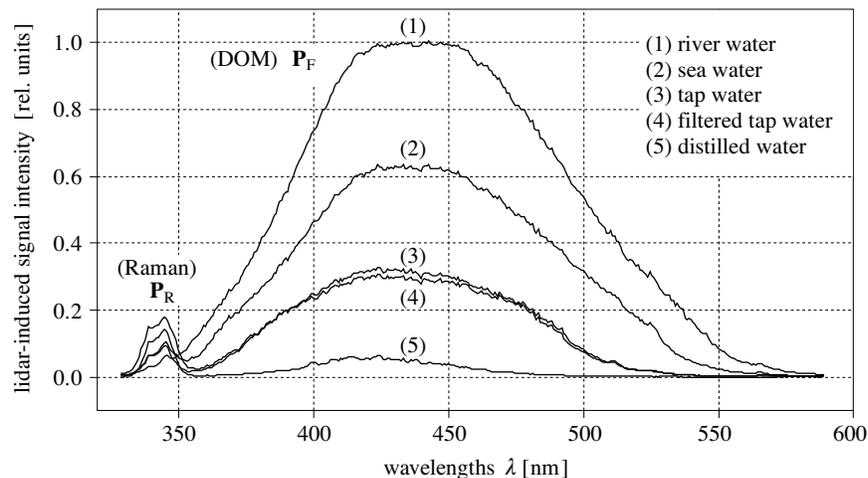


Fig. 1. Lidar-induced fluorescence spectra of various natural water types; the excitation wavelength at 308 nm, maximum of the water Raman scattering at 344 nm and the maximum of the organic matter fluorescence at 430 nm

The lidar-induced spectra of each type of water were characterised by a distinctive band of the water Raman scattering emission (344 nm) and

a broad band of the fluorescent emission due to DOM suspended in water (370–500 nm). The spectra of the various types of water differed in the intensities of these two bands.

The intensity of the organic matter fluorescence band (i.e. the relative concentration of organic matter) was highest in the river water (at the river mouth) and the bay water. The distilled water was the cleanest: its spectrum is characterised by a hardly visible fluorescent organic matter band. Moreover, the maximum of the fluorescence band of organic substances for the bay and river water is shifted slightly to the longer wavelength side in relation to tap and distilled water, which indicates that the DOM consists of different combinations of substances.

Tap water was chosen as the typical natural water for the following measurements to investigate the influence of oil on the fluorescence spectra of natural water.

The fluorescence properties of Petrobaltic oil, the most common oil type for the southern Baltic Sea, were examined (Fig. 2).

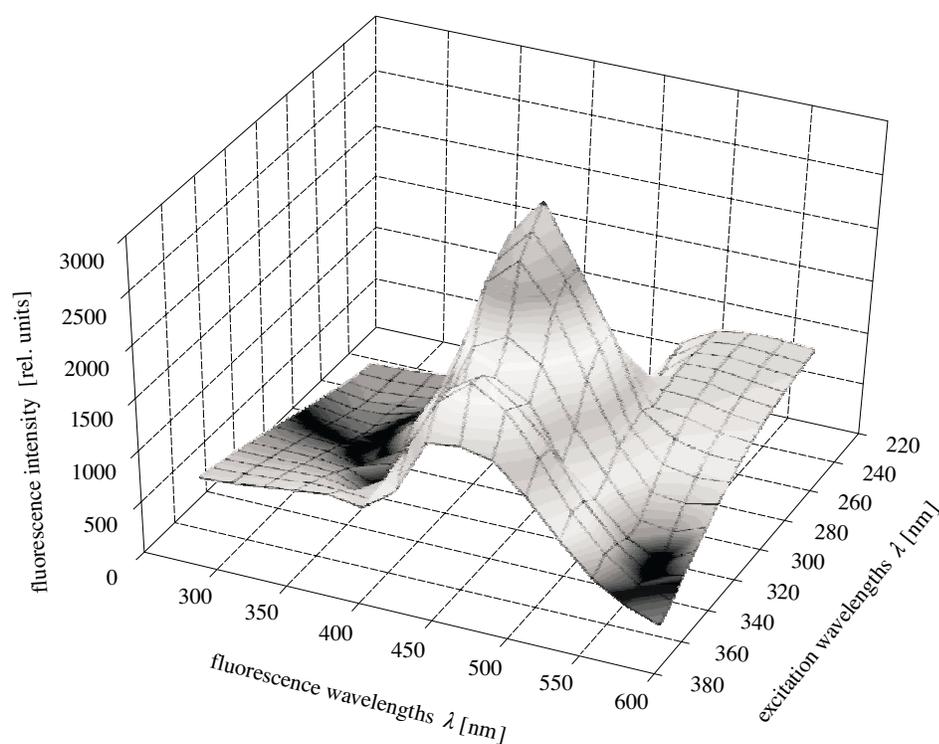


Fig. 2. 3D Petrobaltic oil fluorescence spectrum; concentration of oil in water 25 mg l^{-1}

At an excitation wavelength of 308 nm, this oil fluoresced in the 350–500 nm region (Fig. 2), i.e. in the same spectral range as organic matter (Fig. 1). The presence of oil on the water surface thus affected the intensity and shape of the spectral band produced by both organic matter and oil.

Small amounts (0.5 ml) of oil dissolved in acetone were added in order to create an oil film on the water surface. The fluorescence emission was then measured, after which the oil film was mixed with water and the fluorescence spectrum measured once more.

There were dissimilarities between the water Raman line and the fluorescence of organic-oil spectral bands obtained for an oil film and an oil-water emulsion for a constant angle of refraction (Fig. 3).

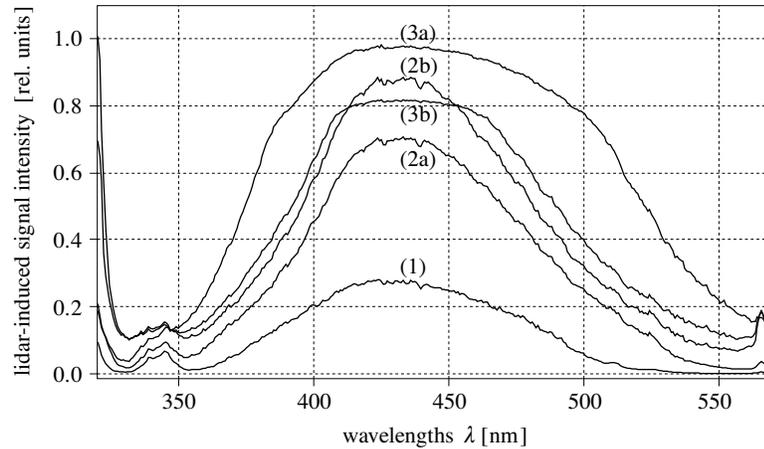


Fig. 3. Lidar-induced fluorescence spectrum of clean tap-water (1) and water polluted with a Petrobaltic oil film: 5 μm (2a) and 9 μm (3a), and water-oil emulsion (2b) and (3b), respectively; excitation wavelength: 308 nm

The water Raman scattering band overlapped the wide spectral band of organic-oil fluorescence in the oil film and oil-water emulsion alike. Moreover, in the case of a thick oil film (9 μm) and its emulsion, the water Raman line partly overlapped the lidar backscattered light. The water Raman line of the oil-water emulsions was intense, while that of the oil films was weaker, the suppression of the water Raman line being caused by the attenuation of the return signal by the oil film covering the water surface.

The fluorescence signal of the organic matter was also attenuated to different degrees by the oil film and oil-water emulsion. Both thick (9 μm) and thin (5 μm) oil films on the water surface caused a distinct increase

in the intensity of the oil fluorescence emission, but strong absorption of the fluorescence of organic substances. In case of the oil-water emulsion, the fluorescence emissions of the oil and yellow substances were additive. Superposition of the oil and organic matter fluorescence spectra yielded a higher intensity than the spectrum obtained for a thin oil film ($5 \mu\text{m}$) and was slightly weaker than the intensity of a thick oil film ($9 \mu\text{m}$) – curves (2a) and (3a) in Fig. 3.

In the subsequent experiments oil-water emulsions were used to investigate the presence of oil and its influence on the fluorescence spectrum of water (Fig. 4).

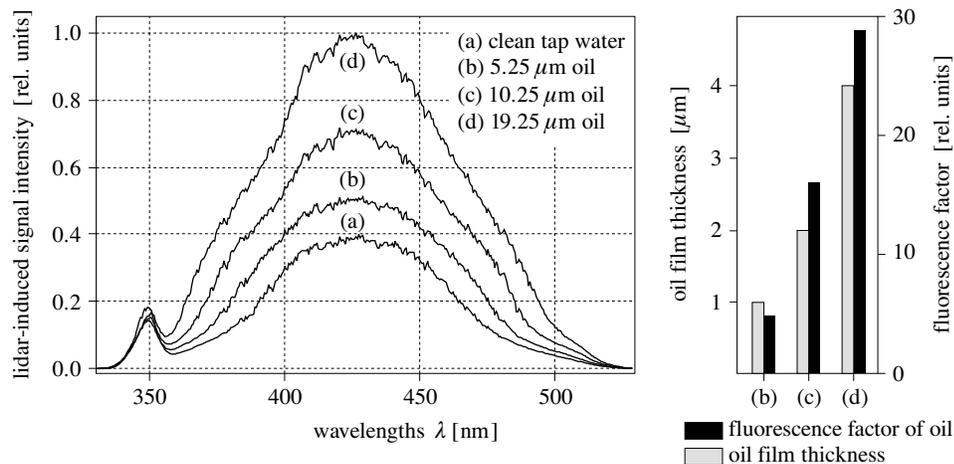


Fig. 4. Lidar-induced fluorescence spectra of tap water polluted with different amounts of Petrobaltic oil; excitation wavelength – 308 nm

Adding equal quantities of Petrobaltic oil increased both the intensity of the organic-oil fluorescence spectral band and the attenuation of the water Raman line (Fig. 4) due to absorption by the oil film. Since the water Raman line overlapped the organic-oil fluorescence band, the increasing oil fluorescence band suppressed the water Raman signal. Changes in the oil film thickness and the oil concentration in water altered the oil's fluorescence factor.

4. Marine water measurements *in situ*

The idea of the next step was to create a database of the spectral characteristics for various types of water from lidar data. The measurements were carried out on board r/v 'Oceania' in different parts of the Baltic Sea

(coastal waters, bay, open sea, river mouths), and the DOM and chlorophyll *a* concentrations in the surface water layer obtained. Additionally, the oil content in various areas of the southern Baltic Sea was assessed (Fig. 5).

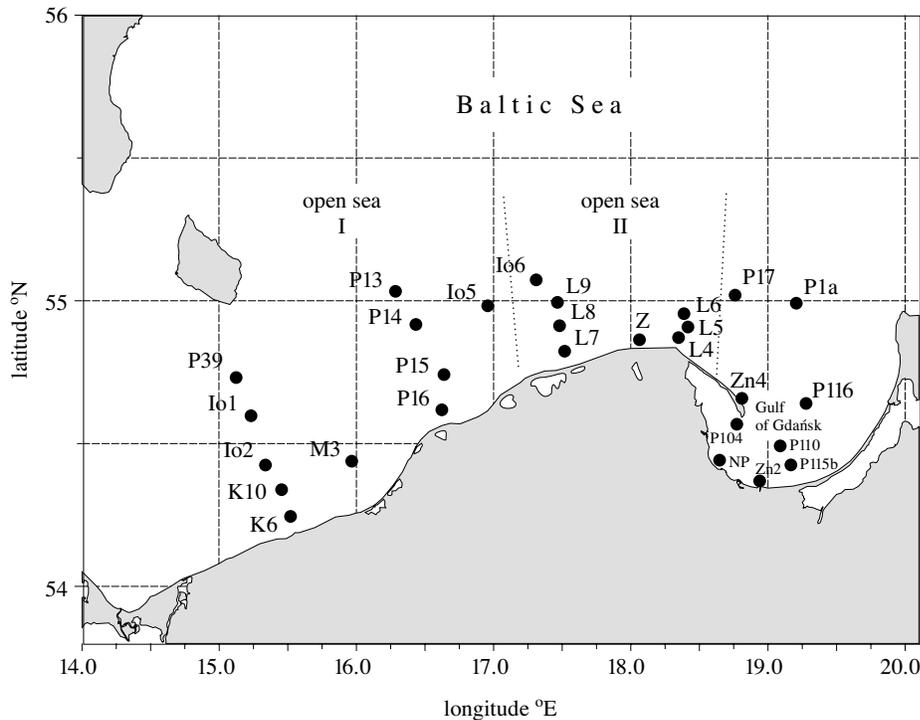


Fig. 5. A map of the stations in the Polish zone of the Baltic Sea studied during the Baltic cruise on 7–14 May 2000, divided into 3 areas (open sea I and II, and Gulf of Gdańsk) according to the amounts of oil pollution detected by the lidar measurements (Fig. 7)

An FLS-12 lidar was employed to measure the spectral characteristics of the fluorescence properties of seawater.

The lidar light excites the fluorescence of chlorophyll *a* (685 nm) and yellow substances (DOM) (500–650 nm) as well as the water Raman scattering emission (517 nm) (Babichenko et al. 1993, Vodacek et al. 1994).

Fig. 6a illustrates the typical return (1) and background (2) signals averaged from about one hundred spectra recorded by the lidar. The lowest curve (known as the ‘real signal’) (3), which is amazingly smooth, is the real seawater emission spectrum induced by the lidar light and is obtained as a result of subtracting the background signal from the return signal. This third curve can be decomposed into a number of spectral bands owing to the

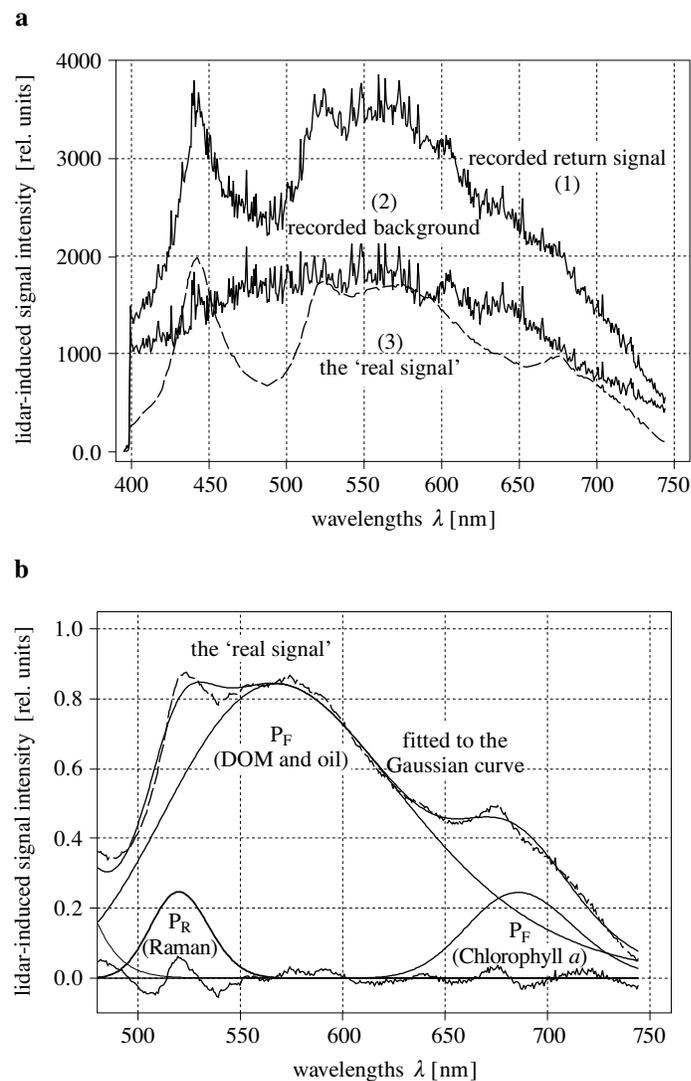


Fig. 6. A typical lidar-induced marine water fluorescence spectrum, excitation wavelength – 440 nm: recorded return signal (1), recorded background (2), and the 'real signal' (3) emitted by seawater (a); the 'real signal' curve and its best fit to the Gaussian curve (by the least squares method), and separate scattering and fluorescence emission spectral bands (b)

elastic (P_L) and non-elastic (water Raman) (P_R) scattering of the lidar light and the fluorescence of DOM (P_{DOM}) and chlorophyll a ($P_{Chl\ a}$). By means of the least squares method an overall Gaussian curve consisting of separate Gaussian curves each reflecting one of the above processes was obtained. The best fit of the 'real signal' enabled the integrals of the intensity of all

spectral bands to be calculated, i.e. the power of every process, which is proportional to the concentration of molecules relating to the respective process (Exton et al. 1983). The interpretation of the lidar-induced marine water fluorescence spectra was based on the calculation of the fluorescence factor, P_F/P_R , of the fluorescing substance (Babichenko et al. 1993, 1995). This factor was obtained from the ratio of the integrals of the fluorescence spectral band intensity of pigment, P_F , to the Raman scattering band on water molecules, P_R (Fig. 6b) (Bristow et al. 1981, Hoge & Swift 1981, Exton et al. 1983).

The recorded spectra supply information about the concentration of the fluorescent components in the water column. The length of the water column, i.e. the surface layer of seawater examined by the lidar, is defined by the penetration depth of the lidar light.

The fluorescence factors are proportional to the relative concentration of the substance investigated. The values in Fig. 7 present the distribution of DOM and chlorophyll *a* as well as the oil film thickness distribution in the Gulf of Gdańsk and in the Southern Baltic Sea, which were investigated during the May cruise.

The fluorescence factors of DOM and chlorophyll *a* (Fig. 7a) were the highest near the mouths of the Vistula and Łeba rivers (Zn2, P115b, L7, L8, Z), in the Gulf of Gdańsk and in the coastal zones (P104, Zn4, P116, P1a). This means that the seawater in these regions contains much riverine organic matter that is subsequently distributed along shipping routes and in ports.

a

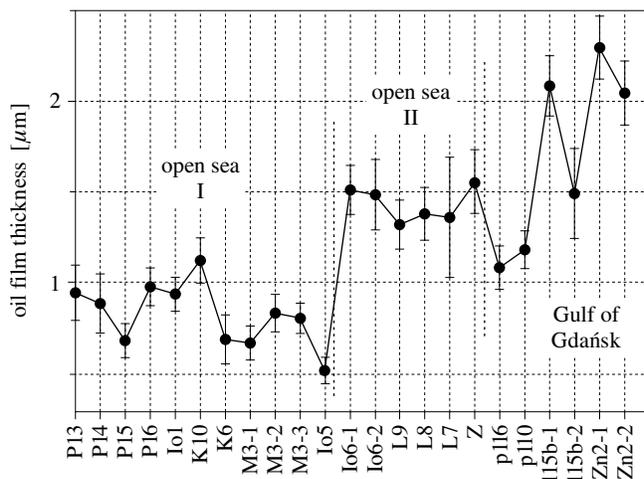


Fig. 7.

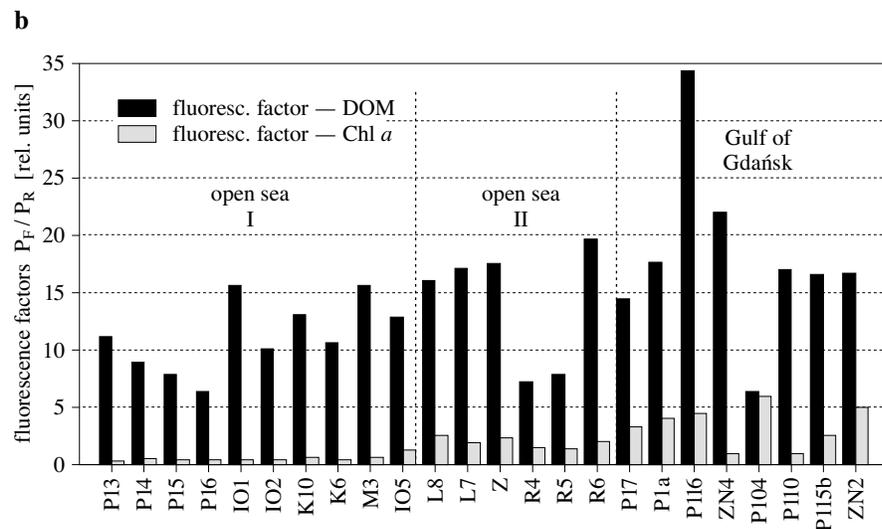


Fig. 7. Lidar measurements in the southern Baltic Sea divided into 3 parts according to the oil pollution detected on the water surface: oil film thickness and the standard error (a); fluorescent factors of DOM and chlorophyll *a* (b)

On the other hand, the fluorescent factors of DOM and chlorophyll *a* were the smallest at the open sea stations (P13, P14 and the others from the ‘open sea I’, Fig. 5), hence the open seawater is relatively clean or well mixed.

The oil film thickness on the sea surface was measured at the same stations. According to the results (Fig. 7a), the largest amounts of oil in the surface layer of the sea were found in the vicinity of the Vistula (Zn2 – 2.25 μm) and Łeba (Z – 1.55 μm) river mouths. The quantity of oil decreased with distance from the Gulf of Gdańsk and river mouths towards the open sea (P13 – 0.5 μm), the resultant thickness fluctuating from 2.25 μm in bay and coastal waters to 0.5 μm in the open seawater. Nevertheless, the quantities of oil in the study area were generally insignificant.

5. Discussion

The application of the KLS–10 lidar – CCD-camera system in the laboratory experiment enabled every fluorescent component in the natural water fluorescence spectrum to be recorded, after which the changes in particular spectral bands that vary due to oil pollution could be examined. The presence of the oil appears to increase the intensity of the 370–500 nm spectral band, characteristic of both DOM and Petrobaltic oil fluorescence, and to decrease the water Raman line. Moreover, the intensity of the

370–500 nm spectral band is slightly higher for the oil-water emulsion than an oil film thinner than $9\ \mu\text{m}$, which results from the superposition of the DOM and oil fluorescence emissions. At sea, the effect of oil on the fluorescence spectrum of seawater was analysed on the basis of the fluorescence characteristics of clean water, water polluted by a Petrobaltic oil film, and an oil-water emulsion.

The FLS-12 lidar system allows the lidar-induced seawater fluorescent emission spectrum, known as ‘real signals’ to be obtained from the lidar-induced return and background signals. The fluorescence factors obtained from calculations and approximations based on the fluorescence spectrum provide information about the concentrations of fluorescing substances (DOM and chlorophyll *a*) in the water column, the length of the column being defined by the maximum penetration of the lidar beam. There are several factors that distort the shape of the fluorescence spectrum as well as other photoluminescence properties of seawater, so it seems reasonable to study them in order to reduce the defects in the active remote sensing methods. Oil pollution affects seawater fluorescence spectra significantly, so the detection of oil spills, and the investigation of the fluorescence properties of oils and the dependence of the lidar-induced return seawater signal on the thickness of the oil film on a water surface are most important tasks.

According to the results of these experiments, the cleanest area of the southern Baltic Sea, only slightly polluted by $0.5\text{--}1.1\ \mu\text{m}$ thick oil films, is in the open sea, whereas the worst polluted waters are near the Vistula and Łeba river mouths, and the ports, where the oil films were $2.2\text{--}2.25\ \mu\text{m}$ thick (Fig. 7).

Petrobaltic oil, the most typical crude oil for the Baltic Sea, was used in both the laboratory experiments and the calculations of oil film thickness. The 3D spectrum of Petrobaltic oil (Fig. 2) was obtained by excitation with UV and blue light, regions in which the oil absorbs and then fluoresces the most strongly. The emission spectrum of the oil at excitation wavelengths of 308 and 440 nm is characterised by a broad band located in the 400–450 nm and 530–630 nm ranges, respectively, that is they coincide with the DOM fluorescence spectrum (Figs. 1 and 6b).

The results of the laboratory experiment (Fig. 3) show that the DOM-oil fluorescence band increases in intensity by about 30% when the thickness of the oil film on the sea surface reaches $1\ \mu\text{m}$, and nearly twice (or 2.5 times) when the oil film thickness exceeds $2\ \mu\text{m}$ (or $4\ \mu\text{m}$), respectively. On the basis of the above results, oil pollution is also expected to increase the DOM-oil fluorescence intensity in the VIS spectrum.

In addition, the oil film covering the sea surface attenuates the water Raman scattering signal as well as the DOM and chlorophyll *a* fluorescence

emissions, so that errors enter into the lidar-data interpretation, that is, into the final distribution of the DOM and chlorophyll *a* fluorescence factors.

It can therefore be assumed that at the stations where the oil film was about 2 μm thick, the DOM fluorescence factor was overestimated and chlorophyll *a* fluorescence factors were underestimated (open sea I, II and Gulf of Gdańsk; Figs. 5 and 7).

6. Conclusions

The most important achievements of these experiments were the attempt to create a database of seawater fluorescence spectra in various regions of the Baltic Sea and to investigate the effect of natural factors distorting the lidar results. It seems that the detection and monitoring of oil pollution in seawater can explain the different values of the fluorescent factors of fluorescing components of seawater such as DOM and chlorophyll *a*.

Moreover, as the laboratory experiment showed, the presence of oil on the water surface affects the lidar-induced seawater emission spectrum. The oil fluorescence emission, located as it is in the blue and blue-green parts of the visible spectrum (Fig. 2), distorts mainly DOM fluorescence, while the oil film attenuates the laser and water Raman signals, and also the fluorescence of chlorophyll *a*. Since the oil pollution of seawater alters the backscattered water Raman signal P_R and the fluorescence signal P_F , the results of the fluorescence factors obtained in the worst polluted waters ('open sea II' and Gulf of Gdańsk, Fig. 5) are encumbered by the biggest errors. The oil distorts the remote sensing readings of seawater fluorescence spectra because it modifies both the lidar signal entering the water and the return signal. Therefore, additional methods for detecting oil pollution must be applied in order to correct the remote sensing data.

The oil contents in the sea surface layer as well as the concentration of DOM and chlorophyll *a* are highest in coastal waters and lowest in the open sea (Figs. 5 and 7).

The relationships between the other optical and physical parameters of seawater (e.g. temperature, salinity, and light attenuation coefficient) and lidar measurements of the fluorescence spectra of the surface seawater layer requires further study.

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