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The contribution of fluorescence to measurements of light scattering in oil-in-water emulsions

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Abstract

The paper presents some results of research on the fluorescence properties of petroleum oil-in-water emulsions. The research addresses the question of measuring and modelling light fields in an oil-polluted marine environment. The fluorescence of the emulsion manifests itself in the spectral range from 260 to over 400 nm and is much less intensive in the visible light region. Fluorescence is always weaker than the radiation scattered in an emulsion. The measurement and modelling of ultraviolet radiation scattering requires fluorescence to be taken into consideration, but in the visible spectral region this phenomenon is negligible.

1. Introduction

Remote sensing based on optical measurements makes it possible to collect continuous data from inaccessible places and is used in different areas of the earth sciences. Orbital platforms or aircraft collect and transmit data from different parts of the electromagnetic spectrum, which provide information for monitoring natural phenomena. Remote sensing works on the principle of the inverse problem: although the parameter of interest (for example: temperature) may not be directly measurable, there exists some optical variable that can be measured, which may be related to that parameter through the use of a data-derived computer model (Parkinson et al. (eds.) 2006). Such a model should be based on the real physical relationship between the parameter of interest and the measured optical variable. Moreover, any model should take many different phenomena into consideration and should be corroborated with experimental data. This is what has happened in the modelling of light fields in a structure of such complexity as the sea (e.g. McKee et al. 2008, Piskozub et al. 2008). Seawater often contains many different constituents and the presence of many of them is manifested by optical phenomena (Dera 2003). Petroleum is one of the most common pollutants of the marine environment; indeed, in some basins it is an almost constant component of seawater (GESAMP 1993, 2007). Petroleum occurs in various forms in seawater (Kaniewski 1999). Each of these forms exerts its own individual influence on the environment and modifies the optical properties of the polluted water (Otremba 1997, Otremba et al. 2003). An oil-water emulsion is one of the forms of oil pollution. The average concentration of emulsion particles in seawater is assumed to range from 10^9 m^{-3} in oceanic water to over 10^{13} m^{-3} in such basins like Pomeranian Bay (Gurgul 1991).

An emulsion is a turbid medium, and light scattering is the main optical phenomenon through which it makes its presence felt in deep water. Light scattering[1] can be described by the volume scattering function β (Jerlov 1976). This function characterizes the optical properties of any medium, including seawater (Dera 2003). The function β is calculated by averaging the intensity functions[2] on the basis of the size distribution of the emulsion particles and their concentration (Bohren & Huffman 1983). Because an emulsion is a suspension of spherical oil droplets with a radius of several micrometers, the Mie solution is suitable for describing the scattering of light by them (van de Hulst 1957, Król 1984, 1985). A description of the phenomenon with the volume scattering function assumes the single scattering model (a particular photon does not interact with more than

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[1] Light scattering is a general physical process in which the photons of radiation passing though a medium are forced to deviate from a straight trajectory. In this paper – if it is not stressed that the opposite case holds true – this term refers to elastic scattering, in which no photon changes its wavelength.

[2] The volume scattering function β depends on the wavelength of the radiation and on the angle between the illuminating flux and the direction of observation. Whereas β is a measurable quantity, the intensity function is only a theoretical quantity suitable for modelling β. This function describes light scattering at a single particle – here, an emulsion droplet – and depends on its diameter, the scattering angle and the radiation's wavelength.

one particle of emulsion). The correctness of such a description of a real phenomenon has been tested for light scattering at right angles in a Baltic crude oil – seawater emulsion (Stelmaszewski et al. 2009). The spectral dependence of the calculated function for wavelengths from 380 nm to 730 nm was compared with the measured scattering spectrum. This test has shown that the scattering function β corresponds to experimental results and that the single scattering model does provide an adequate description of the phenomenon. Application of this model under natural conditions to the scattering of solar radiation in polluted seawater needs to take into consideration the fluorescence of the emulsions. This is important because petroleum is a fluorescent medium. Emulsion particles are fluorescent objects and, moreover, dissolving the fluorescent compounds can accompany emulsifying oil in water. The test mentioned above was carried out for monochromatic radiation (the scattered light measured had the same wavelength as the illuminating radiation), and fluorescence remained undetected in these measurements. In the case of polychromatic radiation like natural sunlight, the separation of fluorescence from scattering appears to be impossible.

The foregoing indicates that any investigation of light scattering in an oil-in-water emulsion should be supplemented by a study of its fluorescence properties. This is the subject of this paper: it discusses the fluorescence of emulsions of seven different oils representing the main petroleum types. These emulsions were tested in the spectral range from 220 nm to 720 nm. The important question was to determine how photoluminescence can influence light scattering measurements. To this end, fluorescence and scattering spectra were measured and the intensities of these phenomena compared.

2. Material and methods

The test was carried out on seven different types of petroleum: two crude oils of differing properties (Baltic and Romashkino), two fuels, as well as lubricating, hydraulic and transformer oils. Samples of each oil were emulsified in seawater. Because the water should be assumed to be a non-fluorescent and fully transparent medium, it was prepared by dissolving the principal sea salts in demineralized water to achieve an ionic composition similar to that of natural water of salinity 7.5 PSU. The emulsion was prepared as follows. An aliquot of oil (3 cm^3) was dissolved in n-hexane (2 cm^3), and this solution was stirred with water (3 dm^3) in a stainless steel vessel at 600 rpm for 3 hours. The emulsion was then allowed to stabilize at 20°C for 24 hours. After this initial stabilization a half-litre sample of the emulsion was drawn from the middle of the vessel volume by an outlet valve in its sidewall and stored in darkness at constant temperature 5°C. This method of preparation yields emulsions, the constitutions of which resemble those encountered in natural waters. The diameter of the majority of petroleum droplets was < 3 μm. The emulsion particle radii formed a size distribution described by a log-normal function (Stelmaszewski et al. 2009) corresponding to the size distribution characterizing the emulsions present in natural basins (Staroń 1999, Mikłaszewicz 2006).

The fluorescence and scattering spectra of the emulsions were measured using a *Fluorat-02 Panorama* spectrofluorimeter. In this device a narrow flux of illuminating radiation runs through the centre of the 1 cm long quartz-glass cell. The illuminating beam is about 1 mm in diameter and its half-intensity width does not exceed 5 nm. For comparing fluorescence with light scattering, the measurements were carried out under the same conditions. The measured radiation coming from the test sample – light scattered or emitted by the emulsion – was restricted by a diaphragm with a circular hole 2 mm in diameter. The measured radiation thus came from the centre of the cell at right angles to the illuminating flux, and its solid angle did not exceed 0.12 sr. The fluorimeter measures two non-dimensional values: F and T. F is proportional to the ratio of the radiation reaching the receiver (fluorescence channel) and the intensity of the illuminating flux:

$$F_{\mathrm{ij}} = \frac{I_{\mathrm{ij}}{}^{\mathrm{f}}}{I^{\mathrm{o}}{}_{\mathrm{i}}^{\mathrm{ex}}}\,,$$

where $I_{\mathrm{ij}}{}^{\mathrm{f}}$ denotes the luminescence intensity of wavelength $\lambda_{\mathrm{j}}{}^{\mathrm{f}}$ ($\lambda^{\mathrm{f}} =$ 'j-wavelength'), $I^{\mathrm{o}}{}_{\mathrm{i}}^{\mathrm{ex}}$ the intensity of the exciting radiation of wavelength $\lambda_{\mathrm{i}}{}^{\mathrm{ex}}$ ($\lambda^{\mathrm{ex}} =$ the 'i-wavelength'). The second value T – the transmission[3] – is the intensity of the light I after having passed through the sample in relation to the intensity of the radiation incident on the cell I^{o}:

$$T = \frac{I}{I^{\mathrm{o}}}\,.$$

The transmission was measured to take into consideration the light attenuation in an emulsion. The fluorescence function w was determined from these measurements according to the following formula:

$$w_{\mathrm{ij}} = \sqrt{\frac{T^{\mathrm{o}}_{\mathrm{i}}\,T^{\mathrm{o}}_{\mathrm{j}}}{T_{\mathrm{i}}\,T_{\mathrm{j}}}}F_{\mathrm{ij}} - F^{\mathrm{o}}_{\mathrm{ij}}\,, \tag{1}$$

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[3] This quantity is also called *transmittance*, whereas the term *transmission* refers to the process of light passing through a medium.

where F denotes the measurement for the emulsion tested, F^{o} the background measurement (radiation scattered in pure water), T the transmission of radiation through the emulsion, and T^{o} transmission through the water. The fluorescence function w is proportional to the internal energy efficiency of fluorescence. It represents the total spectrum of the luminophore and describes real fluorescence at the point where this phenomenon occurs, allowing for various effects like light attenuation or Raman scattering. This function also represents an ordinary fluorescence spectrum (luminescence as a function of λ^{f} for any defined wavelength $\lambda_{\mathrm{i}}^{\mathrm{ex}}$ of exciting radiation), but generally, w is a function of two variables: the exciting radiation wavelength λ^{ex} and the luminescence wavelength λ^{f}: $w = w(\lambda^{\mathrm{ex}}, \lambda^{\mathrm{f}})$. A discrete set of function values creates the matrix $[w_{\mathrm{ij}}]$. The matrix diagonal corresponds to a synchronous spectrum. In the special case when $\lambda_{\mathrm{i}} = \lambda_{\mathrm{j}}$, the function w represents the scattering spectrum: its value is proportional to the volume scattering function β and to the scattering coefficient b; then formula (1) can be written in a simpler form and without any subscripts:

$$w = F\frac{T^{\mathrm{o}}}{T} - F^{\mathrm{o}}. \tag{2}$$

The fluorescence was measured every 5 nm in the spectral range from 260 to 720 nm. These spectra were excited by monochromatic radiation of wavelength every 20 nm in the range from 220 to 400 nm. The emulsion of no oil emits radiation of wavelength shorter than 260 nm. At the same time, radiation of wavelengths longer than 400 nm causes very slight luminescence, so the spectra excited by such light are not given. Scattering of radiation at right angles was measured in the range from 220 to 720 nm.

The fluorescence spectra of petroleum surfaces were also measured. Only the quantity F was obtained here: the layer of oil was illuminated by a monochromatic exciting beam and the radiation emitted by the oil measured. The oil surface was positioned at an angle of $\pi/4$ to both the exciting beam and the direction of the luminescence channel.

Raman scattering was measured in pure seawater in the spectral range of exciting radiation from 220 to 440 nm. The Raman effect was very less intensive for radiation of wavelength over 400 nm and was non-measurable for light of wavelength longer than 450 nm.

The oil concentration in an emulsion was determined by the fluorescence method. A hexane extract was prepared for each sample of emulsion, and a reference solution of each oil was made up. Fluorescence and transmission was measured for both the extract and the reference solution, after which the respective values of the function w were determined according to formula (1). The measured luminescence had a wavelength ${\lambda_j}^f = 320$ nm and was excited by radiation of wavelength ${\lambda_i}^{ex} = 240$ nm. The concentration C of petroleum in the emulsion was determined by comparing the w of its extract with w_{ref} of the reference solution, according to the formula

$$C = \frac{w}{w_{ref}} \frac{m}{M} C_{ref} \,, \tag{3}$$

where m denotes the mass of hexane used for extraction, M the mass of the emulsion tested, and C_{ref} the oil concentration in the reference solution.

The concentration of oxygen dissolved in the emulsion was measured at 20°C using a *CyberScan PCD 650* multimeter equipped with a membrane sensor.

3. Results

Table 1 shows the concentration of oil and dissolved oxygen in the emulsions tested. Further results are illustrated graphically in the following figures. Figure 1 presents the intensity of fluorescence with respect to the oil concentration in the emulsion. This test was carried out for emulsions of hydraulic oil (a) and of Baltic crude (b). The wavelengths of fluorescence (λ^f) and of exciting radiation (λ^{ex}) are given at the respective plots. The initial concentration C of oil in the emulsion was determined by the fluorescence method described in the previous section, after which the emulsion was diluted with water to obtain samples of particular concentrations: $1/5\,C$, $2/5\,C$ etc. This figure also shows the intensity of light scattered at right angles in a hydraulic oil-in-water emulsion with respect to the oil concentration. Scattering was measured for radiation of wavelengths 400 nm (c) and 600 nm (d)

Table 1. Concentration of oil and of dissolved oxygen in water emulsions of the respective oils

Emulsion of oil	Oil concentration [mg dm^{-3}]	Dissolved oxygen [mg dm^{-3}]
diesel oil 1	133	8.75
diesel oil 2	143	8.33
Baltic crude	53	8.99
Romashkino crude	28	8.89
hydraulic oil	323	8.20
transformer oil	90	9.01
lube oil *Helix*	4.4	8.71

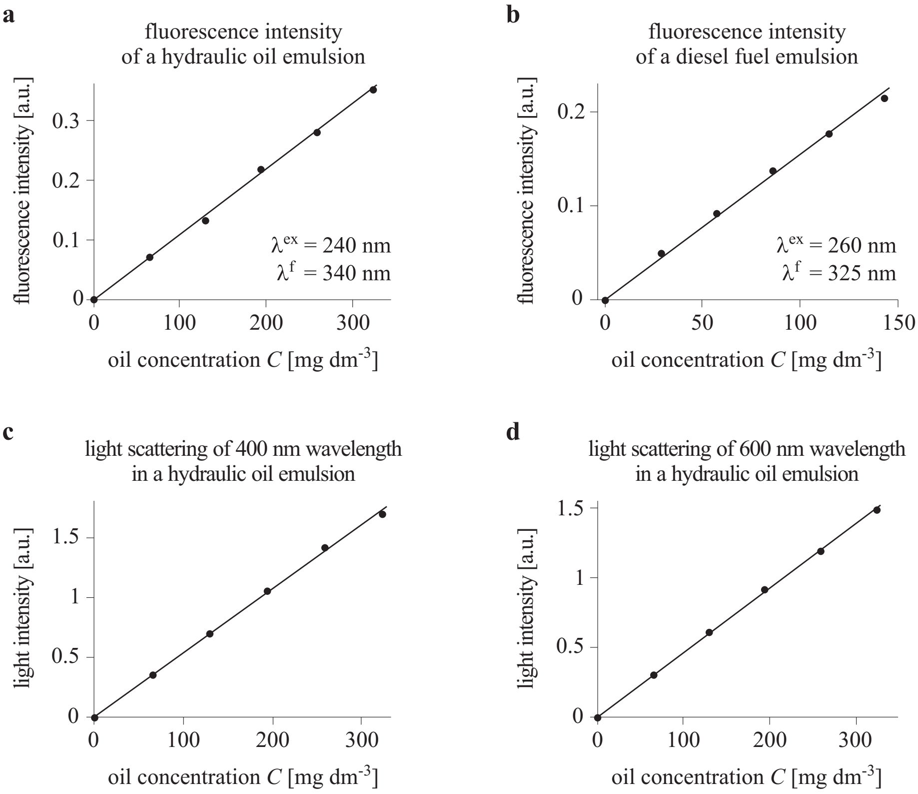

Figure 1. Fluorescence intensity of a hydraulic oil emulsion (a) and a diesel fuel emulsion (b) with respect to the oil concentration and light scattering of wavelength 400 nm (c) and 600 nm (d) in a hydraulic oil-in-water emulsion with respect to the oil concentration

Figure 2 shows a number of fluorescence spectra of the emulsions. The type of emulsified oil is stated above each plot. The spectra were excited by radiation of wavelengths 220 nm, 240 nm, 260 nm, 300 nm and 340 nm, and the colour of a particular line corresponds to the relevant excitation. Fluorescence decreases with wavelength if the exciting radiation is longer than 300 nm and visible light causes very weak luminescence, so the rest of the measured spectra are not presented.

Figure 3 depicts selected fluorescence spectra of the emulsions in comparison with the spectra of the corresponding oils. Petroleum strongly absorbs illuminating radiation, the level of absorption depending on the kind. Both crude oils absorbed so much radiation that the fluorescence was not measurable. The intensity of fluorescence from the emulsion and that from the oil surface were not comparable because these measurements were carried out in different ways; only the shapes of the spectra could be

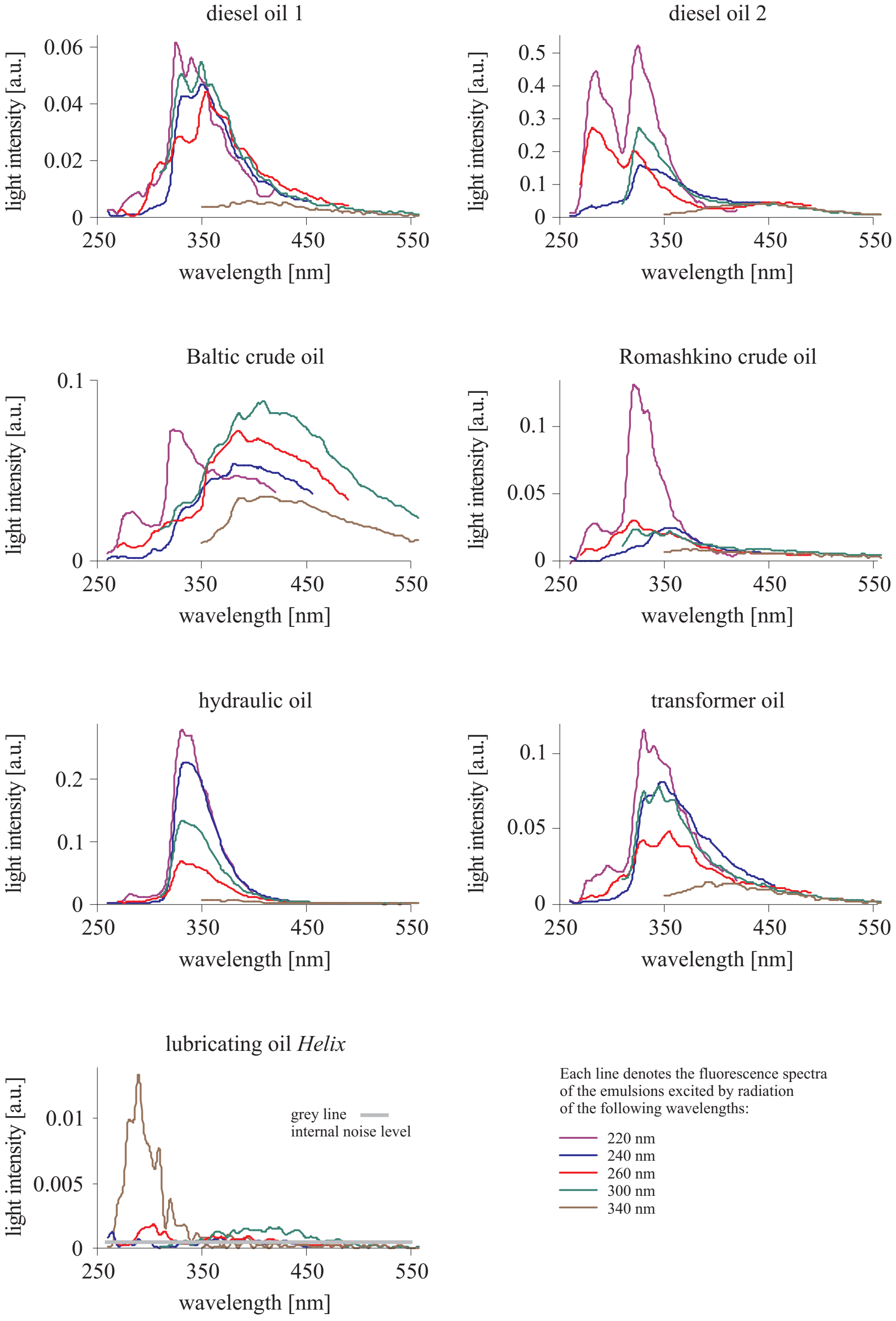

Figure 2. Fluorescence spectra of petroleum-in-water emulsions. The type of emulsified oil is indicated on the relevant plot. The exciting radiation was of wavelength 220 nm, 240 nm, 260 nm, 300 nm and 340 nm (the colour of a line corresponds to the excitation wavelength)

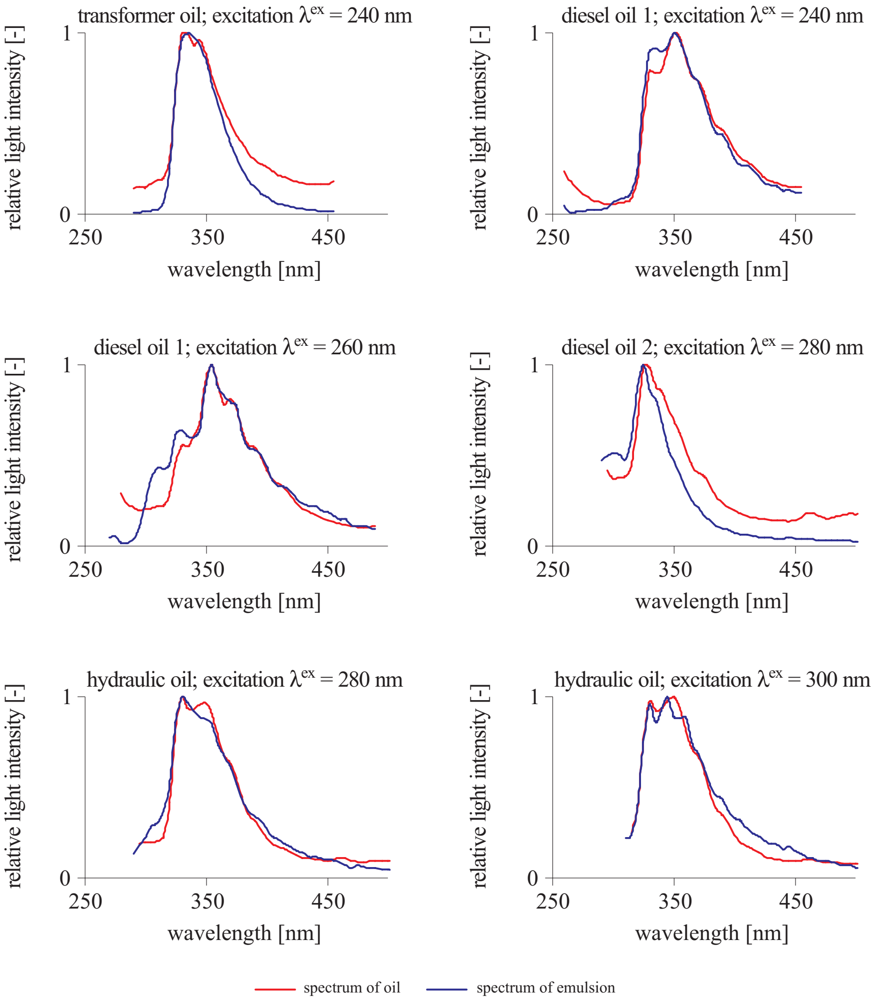

Figure 3. Fluorescence spectra of oil-in-water emulsions (blue lines) in comparison with the spectra of the respective oils (red lines). Each spectrum was normalized to its maximum value

compared. Thus, all the spectra presented here were normalized to their maximum values.

Figure 4 presents scattering spectra of the emulsions. Some plots also show the Raman effect in pure water (marked as a dotted line) with respect to the wavelength of the scattered radiation.

Figure 5 is the most significant because it shows both the fluorescence and the scattering spectra of the emulsions. The luminescence and scattering intensities are presented on a logarithmic scale. The black line represents the scattering spectrum, and the coloured lines show

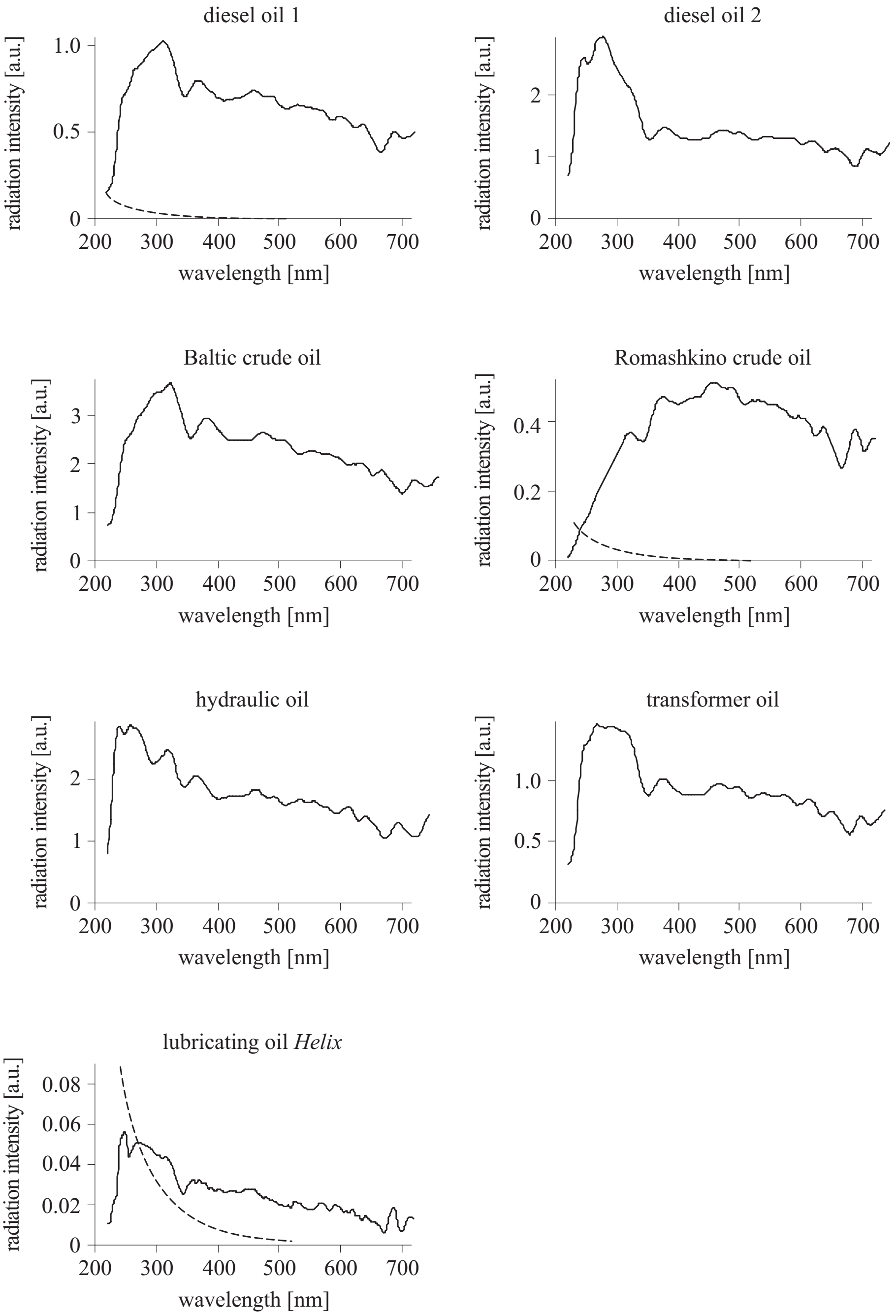

Figure 4. Scattering spectra (solid lines) of petroleum-in-water emulsions. For comparison, some plots also depict the intensity of the Raman effect in seawater (dotted line) as a function of scattered radiation wavelength

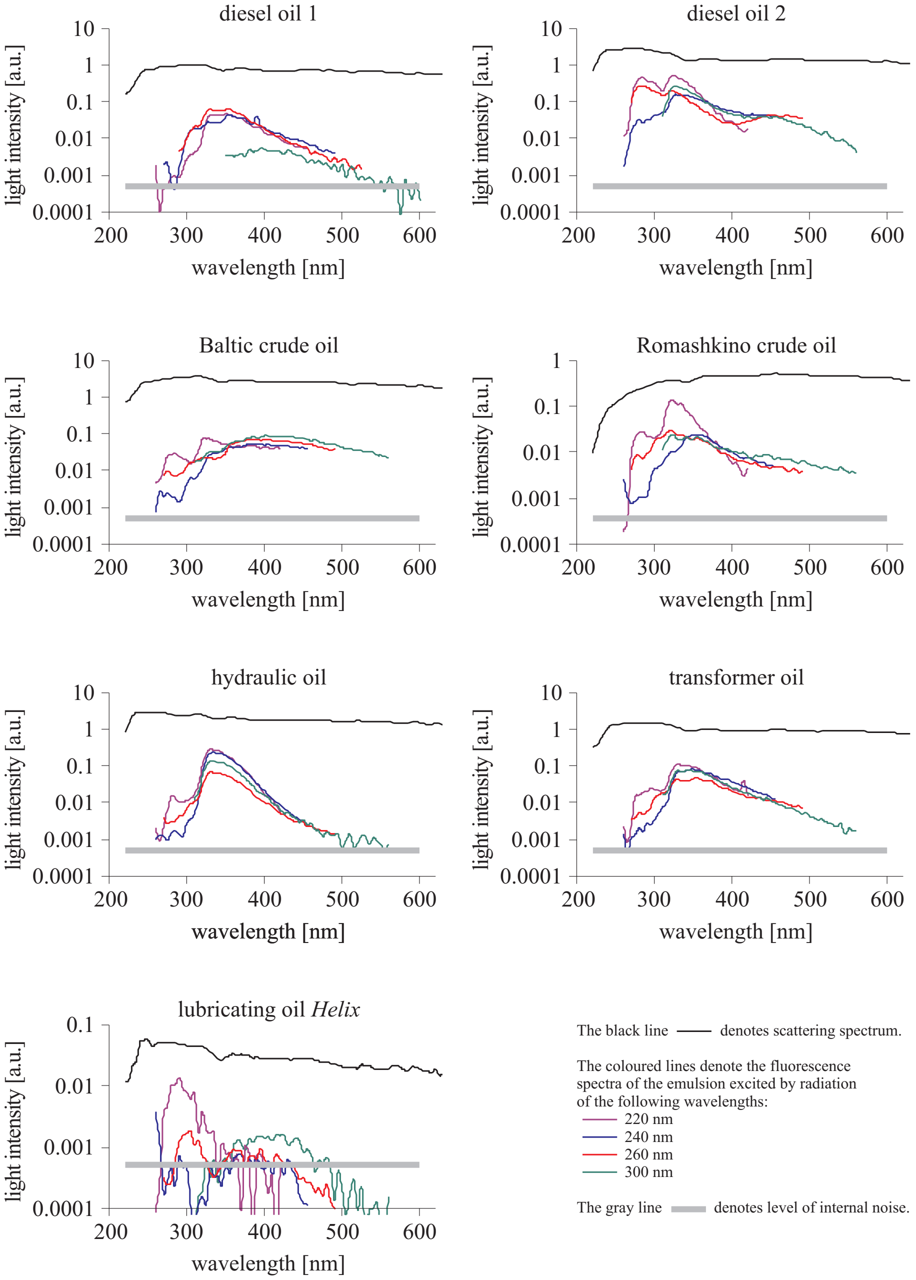

Figure 5. Fluorescence spectra of the oil-in-water emulsions (colour lines) and spectra of light scattered through a right angle (black lines) measured under the same conditions. The exciting radiation was of wavelength 220 nm, 240 nm, 260 nm and 300 nm (the colour of a line corresponds to the excitation wavelength). The intensities of fluorescence and scattered light (y-axis) are presented on a logarithmic scale

the fluorescence spectra excited by radiation of the corresponding wavelengths.

4. Discussion

Above all, the results demonstrate the great diversity of petroleum oils and their properties. This diversity manifests itself in the emulsification of particular oils in water and in the stability of the emulsions. The final result was that the oil concentration in 1 dm^3 of emulsion varied from 4.4 mg of lubricating oil to over 300 mg of hydraulic oil. Comparison of the spectra of the various emulsions shows that both scattering and fluorescence reflect the diversity of the oils. Only the saturation of the emulsions varies within narrow limits from 8.2 mg to 9.0 mg of dissolved oxygen in 1 dm^3 of water. Such results are similar to the saturation of natural seawater.

The dependences of light scattering in emulsions and their fluorescence on the oil concentrations were the key point of the study. Both the intensity of fluorescence and light scattering in the emulsion are proportional to the oil concentration (Figure 1). The result of light scattered in a hydraulic oil-in-water emulsion was similar to that for Baltic crude (Stelmaszewski et al. 2009).

The fluorescence of petroleum oils and their water emulsions covers a spectral area from 260 to over 500 nm (see Figures 2 and 3) but the phenomenon is most significant in the 270–400 nm range. Fluorescence is greatest if it is excited by radiation of wavelength < 280 nm. If wavelength of this radiation is longer than 300 nm and further increases, then the fluorescence decreases and visible light causes very slight luminescence. Both the spectral range and shapes of the spectra depend on the kind of oil. At the same time the spectrum of the emulsion is very similar to the spectrum of the corresponding oil (Figure 3), although the shapes of these spectra are not absolutely identical. This may indicate that the observed changes undergone by petroleum during its emulsification in water are responsible for the optical properties of emulsion particles differing only slightly from those of the initial oil (Stelmaszewski & Toczek 2007). The situation is different in the case of the dissolved phase. The emulsification of oil is accompanied by the dissolution of its individual components. The solubility of the components of petroleum is generally very small (Verschueren 1983, Pereda et al. 2009), but molecules can pass from the oil layer covering the water surface into the water column. The fluorescence spectra of the dissolved phase are quite different from those of oils (Stelmaszewski 2001). This is not surprising because the individual components of any oil are dissolved in water to different degrees, while the properties of emulsion particles do not differ significantly from the properties of the initial oil. The resemblance between the emulsion and oil spectra suggest that the fluorescence of an emulsion derived mainly from oil particles and the contribution of the dissolved phase appear to be negligible.

The scattering spectra of emulsions differ from each other (Figure 4). In general, light scattering (at right angles) in an emulsion increases with wavelength to a certain maximum in the range from 300 to 500 nm, depending on the kind of oil, then decreases slightly with wavelength. Apart from this, each spectrum is characterized by numerous relative extremes in the whole spectral area. The same spectral dependence with numerous extremes also characterizes the scattering function β calculated on the basis of the optical properties of oil and the size distribution of the oil droplets.[4]

The results of radiation scattered inelastically in water are consistent with theoretical data. The bathochromic shift corresponds to an oscillation energy of 6.2×10^{-20} J (3330 cm^{-1}) and is near the reference value for the O–H group oscillation[5], which is ca 3400 cm^{-1} (Walrafen & Pugh 2004, Pershin et al. 2007). In addition, the spectral dependence of the Raman effect conforms with the theoretical dependence – the scattering intensity is proportional to λ^{-4}. Because of this, Raman scattering was distinct in the ultraviolet area and non-measurable for visible light of wavelength longer than 450 nm. The Raman effect should always be taken into consideration in light scattering measurements; this is especially important in the case of emulsions with a low oil content.

Comparison of the fluorescence of oil-in-water emulsion with light scattering was a significant aspect of this research (see Figure 5). The fluorescence intensity is always less than the intensity of scattered radiation, even in ultraviolet spectral areas, where fluorescence is the most significant. While fluorescence, though less intensive, is comparable with the scattering of ultraviolet radiation, the difference between the intensities of these phenomena is more than an order of magnitude for light of wavelength longer than 400 nm and increases with increasing wavelength for any kind of oil. Consequently, fluorescence makes only a small contribution to the scattering flux of the visible light coming from an emulsion.

The above remarks refer to the situation where the intensity of the illuminating radiation of any wavelength is equal – this follows directly from the fact that the intensity of light scattered or fluoresced by an emulsion is measured in relation to the intensity of the illuminating radiation. These

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[4] A close similarity between the spectrum of function β and the measured scattering spectrum has been found for a Baltic crude oil-in-water emulsion (Stelmaszewski et al. 2009), but this similarity should be confirmed for emulsions of other oils.

[5] There are three major O–H bands near wavenumber 3400 cm^{-1}; they depend on external conditions (Pershin et al. 2007).

remarks are all the more valid for emulsion illuminated by solar light. Taking into consideration the spectrum of the solar radiation reaching the Earth's surface (Dera 2003), one can assume that fluorescence plays a negligible part in the radiation scattered in an emulsion. The possible quenching of fluorescence by dissolved oxygen does not change this conclusion. Oxygen is natural component of seawater, and the saturation of subsurface water often exceeds 100% and is greater than the saturation of the samples tested.

The results are limited to scattering at right angles, but this does not alter the above conclusion. An oil-in-water emulsion is an isotropic medium and its fluorescence does not depend on the angle of illumination, in contrast to scattering. The index of scattering reaches a minimum at 90° and observations at an angle other than 90° will cause fluorescence to be even less than the scattered radiation.

These comments refer to the scattering of unpolarized light. Illumination of an emulsion with polarized radiation causes the scattering-to-fluorescence ratio to be different.

5. Summary

The results can be summed up as follows:

- Emulsions fluoresce in the spectral region from 260 to > 400 nm; the range of fluorescence and shapes of the spectra depend on the kind of oil.
- The great diversity of oils manifests itself in the already known variety of their fluorescence spectra as well as in the variety of scattering spectra of their emulsions.
- The fluorescence of an emulsion is derived mainly from the fluorescence of oil droplets, whereas the luminescence of the dissolved phase is negligible.
- Fluorescence and scattering are proportional to the oil concentration in an emulsion.
- The fluorescence of an emulsion is always less intense than the light scattering.
- The scattering spectra are characterized by many local extremes.

These investigations lead to the following conclusions concerning natural seawater containing emulsified petroleum:

1. The measurement and modelling of ultraviolet radiation scattering require the fluorescence of an emulsion to be taken into consideration.
2. Fluorescence caused by solar radiation under natural marine conditions is negligible in comparison with scattering.

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