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An optical fibre technique for measuring optical absorption by chromophores in the presence of scattering particles

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Abstract. The evanescent radiation field associated with a suitably-prepared silica optical fibre, whereby the outer cladding has been removed and the core immersed in a liquid, has been investigated for its applicability as a device for measuring the spectral absorption of a liquid containing both chromophore and suspended scattering material. It is found that the evanescent radiation field adjacent to the core-liquid boundary interacts with chromophores in a liquid in much the same way as radiation does when directed, unbound, through a cuvette containing the same liquid in an optical spectrophotometer. However, the small penetration depth of the evanescent field, of the order of one micron, results in a sensor which is found to be insensitive to light-scattering particulate matter than was present in the liquid.

1. Introduction

Optical fibres are generally cylindrical dielectric waveguides, made of fused silica or plastics where a central fibre core, of higher refractive index is surrounded by concentric cladding of lower refractive index, and a plastic, protective jacket. The difference in refractive indices of the core and cladding is such that light, when directed (or coupled) into the core of an optical fibre, is trapped and propagates along the fibre core via total internal reflection at the core-cladding interface. Typical optical fibres have cores ranging from 10 to 1000 µm in diameter and are most widely known for their role in telecommunications. However, optical fibres also find many uses, both as extrinsic and intrinsic components of chemical and physical sensors [1, 2].

At the point of reflection at the core-cladding boundary (figure 1), optical interference between the incident and reflected rays produces a standing wave. Although all the power is reflected from the core-cladding interface back into the core, the electric field associated with the standing wave penetrates some distance into the cladding, diminishing with distance from the boundary. This field carries no energy away from the interface [3, 4] and is termed the evanescent field.

The evanescent field decays exponentially in z direction according to the expression [5, 6]

\[ E = E_0 e^{-\beta z} \]  (1)
where $z$ is the distance normal to the core interface, $\beta$ is the attenuation factor or attenuation coefficient, which is expressed as

$$\beta = k_0 \sqrt{(n_1^2 \sin^2 \phi - n_2^2)}$$

and $k_0$ is the free-space propagation factor $(2\pi/\lambda)$ where $\lambda$ is the free-space wavelength and $\phi$ is the angle of incidence at the interface (figure 1).

![Figure 1. An evanescent field is generated at the point of total internal reflection. An oscillatory standing wave is set up in the medium of higher refractive index, $n_1$, (fibre core) and decays exponentially into the medium of lower refractive index, $n_2$, (fibre cladding) [5].](image)

The depth of penetration, $d_p$, is defined as the distance over which the evanescent field is reduced to $1/e$ of its interface-value, and can be calculated from the solution of Maxwell’s equations [7] at the interface using

$$d_p = \frac{\lambda}{2\pi \sqrt{n_1^2 \sin^2 \phi - n_2^2}}$$

In the case where the core is fused silica, $n_1 \approx 1.50$, and, instead of fused silica, the cladding is aqueous with $n_2 \approx 1.33$, the depth of penetration ranges from approximately $\lambda/5$ at an incident angle of close to $90^\circ$, to approximately $\lambda$ for an incident angle within $1^\circ$ of the critical angle. However, virtually no radiation is incident at the critical angle because of light leakage and scattering resulting from the "coarseness" of the interface surface.

A sensor based on evanescent field absorption relies on the interaction of a target substance with the evanescent field adjacent to the fibre core. Removing the cladding from a portion of an optical fibre permits the evanescent field to interact with the substances in-which the fibre is immersed. This interaction with a chromophore, a term we use here to describe a material that absorbs light at selective wavelengths, will result in attenuation of the intensity of the radiation travelling down the fibre core. The alteration of this intensity may be measured and associated with the concentration of the material in contact with the fibre core surface in the same fashion as traditional optical absorption measurements.

The ability of the evanescent field to interact with liquids containing chromophores has been investigated by numerous researchers [3, 8]. However, what has not been fully investigated is the fact that the limited penetration depth of the evanescent field, while sensitive to chromophores in liquids, may be insensitive to scattering by any suspended scattering particles that may be present if the inter-particle or particle-fibre surface distance is greater than the penetration depth. If so, the evanescent field absorption technique offers the potential to constitute an optical absorption sensor for
chroomophores in liquids in the presence of suspended scattering particles without the need for centrifugation and/or filtering of the sample to achieve clarity. One example whereby such a technique may be employed, is for measuring colour of freshwaters in the presence of significant amounts of suspended sediment. In this context, this paper reports on a preliminary investigation of the evanescent field absorption technique involving liquids containing a chromophore plus significant concentrations of suspended particulate matter.

2. Materials and methods

Due to the potential importance of this investigation to the measurement of water quality in the context of environmental monitoring, chlorophyll (hitherto referred to as chlorophyll-related pigments) was used as the water-borne pigment and finely-ground, ashed clay was used as a suspended sediment.

A stock solution of chlorophyll-related pigments was prepared from samples of domestic spinach plant following [9]. The concentration of chlorophyll-related pigments used in subsequent analysis ranged from 0.83 to 4.15 mg/l.

The preparation of sediment for suspension in the chlorophyll samples was completed following [10]. The sediment sample was collected and oven-dried at a temperature of 80 °C for 3 days. A sub-sample was then crushed using a mortar and pestle and fired in a muffle furnace (Ceramic Engineering) for 5 hours at 500 °C to dispose of all the organic content of the sediment. The clay samples were sieved to a grain size no larger than 120 μm.

The complete optical apparatus used for the spectral absorption measurements is depicted in figure 2. The apparatus comprised a regulated light source, collimating optics, a sample cell, optical fibre cable and a spectrometer. All optical measurements were conducted in a dark room.

The regulated white light source comprised an OSRAM quartz-tungsten-halogen bulb (250 W, 24 V) mounted in a Series Q Lamp Housing (ORIEL Model 60000), complete with an integral condensing/collimating lens. The light source was driven by a constant current power supply (Oriel Model 68830). The collimated beam from the regulated white light source was directed into a precision optical fibre coupler (Newport, Model F-915), which contained one end of the selected fibre used for the evanescent sensor. The fibre used was a cylindrical, pure core silica (PCS) optical fibre of 600μm diameter (F-MBD, Newport, USA). Approximately 3 mm of the jacket and cladding were stripped from both ends of the PCS optical fibre using a scalpel. Both ends of the fibre were cleaved with a fibre scribe (Newport, Model F-CL1), and ground and polished using three, progressively finer, grades of sand paper on a glass plate submerged in water. The jacket/cladding from an 8 mm segment of the middle-portion of the fibre was stripped by burning with a butane pencil torch (Blue Blazer, China) with a maximum flame temperature of approximately 1316 °C. The exposed core was subsequently cleaned with isopropyl alcohol using lint-free tissue paper (Kimwipes, Kimberly-Clark Australia Pty. Limited) to remove any residual carbonisation that was produced by combustion of dust or debris on the cladding.

The prepared fibre was inserted into the centre of a sample cell constructed from perspex measuring 4 cm (width) x 4 cm (depth) x 16 cm (length) (figure 3). The sample cell was designed to hold the chromophore/suspension-containing liquid. The output-end of the sensing fibre (downstream of the sample cell) was butt-coupled to a 200-μm core-diameter glass optical fibre collection cable using a precision optical fibre positioner (Newport, Model FP-1). The latter optical fibre formed the input slit of the Ocean Optics SD1000 spectrometer (Ocean Optics Inc., Dunedin, Florida, USA) and was connected to the spectrometer via a SMA 905 connector. The internal diffraction grating was ruled to 600 lines/mm and blazed at a wavelength of 500 nm. Light reflected from the grating was directed onto a linear 1024-element charged-couple-device (CCD) array. The CCD array, in conjunction with the input fibre and grating, recorded spectral information in the wavelength range of 390 – 890 nm with a spectral resolution of 1.4 nm. The SD1000 was connected to a lap-top Toshiba computer running the software Spectra-Solve™ Junior.
Figure 2. Schematic diagram of the complete apparatus for measuring chlorophyll-related pigment in water containing suspended sediment using the evanescent field absorption technique.

Figure 3. Photograph of the perspex sample cell with the PCS optical fibre inserted.

Absorbance spectra were recorded using the following protocol; integration time, 50 ms; measurement of dark current spectrum, DARK(λ); measurement of radiation spectrum using sample cell filled with clear distilled water, REFERENCE(λ); drop wise addition of chlorophyll-related pigment and sediment (with agitation) to water in the sample cell and subsequent measurement of radiation spectrum, SAMPLE(λ); calculation of absorbance spectrum using

$$\text{Abs}(\lambda) = -\log_{10} \left[ \frac{\text{SAMPLE}(\lambda) - \text{DARK}(\lambda)}{\text{REFERENCE}(\lambda) - \text{DARK}(\lambda)} \right]$$ (4)

3. Results and discussion

Absorption spectra (relative to clear distilled water) were acquired for chlorophyll-related pigments of concentrations 0.83 mg/L, 1.66 mg/L, 2.49 mg/L, 3.32 mg/L and 4.15 mg/L. An example of a set of absorbance spectra (for five concentrations of chlorophyll-related pigment) is given in figure 4.
Figure 4. Evanescent field absorbance spectra of chlorophyll-related pigments in clear distilled water for an 8 cm length of exposed fibre core. Fibre diameter = 600 µm.

The absorbance spectra of figure 4 exhibits prominent features characteristic of chlorophyll-related pigments, including the peaks at approximately 430 and 670 nm which are attributable to chlorophyll-a [11, 12]. Absorption spectra (relative to clear distilled water) for a single concentration of chlorophyll-related pigment in distilled water (4.15 mg/l), but this time with the addition of five concentrations of clay as suspended sediment (2, 4, 6, 8 and 10 g/l) are given in figure 5.

Figure 5. Evanescent field absorbance spectra of 4.15 mg/L chlorophyll-related pigments in water containing various concentrations of clay for an 8 cm length of exposed fibre core. Fibre diameter = 600 µm.
Figure 5 demonstrates that the absorption spectra is not modified by the presence of suspended sediment particles up to 10 g/L.

Using equation 3 with $\lambda = 670$ nm, a value of $n_2$ corresponding to a chlorophyll-related pigment concentration of $4.15 \times 10^{-3}$ kg/m$^3$ (4.15 mg/L), and a light launch angle close to the critical angle ($35^\circ$), the penetration depth achievable using the PCS fibre is approximately 1.77 $\mu$m. If the clay particles used as suspended sediment are assumed to be spherical, their average diameter, $d$, is 120 $\mu$m, and the density of silica, taken to be $\rho = 2.33 \times 10^3$ g/L [13], the approximate distance between the centres of adjacent clay particles is 0.61 mm. Allowing for a particle diameter of 120 $\mu$m, this equates to an approximate free-space separation of 0.49 mm. This distance is approximately 277 times greater than the calculated achievable penetration depth of the optical radiation from the PCS fibre core into the liquid.

If we further extend this simple comparison between the penetration depth of probing radiation and average free-space distance between clay particles, it is possible to crudely estimate the concentration of clay particles which could influence the evanescent absorption of any pigment in the water sample. For the average free-space distance between adjacent clay particles to be comparable to the penetration depth associated with the PCS fibre core (1.77 $\mu$m), the concentration would need to be approximately 1.20 kg/L. This is an enormous level of sediment loading in any water sample. If we return to the context of freshwater bodies, this would be approximately two thousand-times higher than values reported in typical inland rivers worldwide [14, 15, 16, 17, 18, 19].

4. Conclusion

This paper has evaluated an optical fibre technique as a possible method of measuring optical absorption of a chromophore in samples containing suspended material; a technique which would not require sample pre-treatment (filtering or centrifugation). The optical fibre evanescent field absorption technique relies on the pigmented sample absorbing radiation from the evanescent field of light propagating within the exposed core of a short length of optical fibre. Simple measurements were performed on samples of clear distilled water containing chlorophyll-related pigment and suspended sediment. The technique was found to be sensitive to absorption by the chlorophyll-related pigments in the water, yet insensitive to the presence of suspended clay particles up to, and including, the maximum introduced concentration of 10 g/L. An empirical calculation, based on the evanescent field absorption apparatus used, subsequently estimated that the apparatus would remain insensitive to the introduction of suspended sediment up to a concentration of 1.2 kg/L.

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