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Dissolved organic carbon in coral-reef lagoons, by high temperature catalytic oxidation and UV spectrometry

Jean Pagès, Jean-Pascal Torréton and Richard Sempéré

Abstract

Two surveys were carried out on ten atolls in the Tuamotu archipelago (French Polynesia, Pacific Ocean).

In vitro UV (250-400 nm) spectra of water samples gave absorption at 254 nm, \( \Lambda_{254} \) and spectrum slope, \( S^* \) (computed from \( \ln \Lambda \) versus \( \lambda \)). These two descriptors are negatively correlated, and data points are arrayed along a hyperbola spanned between an oceanic pole (high \( S^* \), low \( \Lambda_{254} \)) and a confined pole (low \( S^* \), high \( \Lambda_{254} \)).

Dissolved organic carbon (DOC) concentrations, [C], as assessed by HTCO, exhibit a narrow range (0.7-1.0 mg C L\(^{-1}\) for most lagoons) contrasting with the wide diversity of optical characteristics. [C] and \( \Lambda_{254} \) are positively correlated, with a significant intercept (0.5 mg C L\(^{-1}\)) representing non-chromophoric DOC. Carbon-specific absorption, \( \varepsilon_{254} \), increases (from 0.4 to 1.3 m\(^2\) g\(^{-1}\)) with increasing [C], mainly according to the literature (owing to increased average molecular weight (MW) of the chromophoric DOC fraction, which also lowers \( S^* \).

Our optical data thus illustrate a gradient of confinement (or residence time) that corresponds to a continuum in DOC nature, especially in MW and hence in bioavailability. Optical methods are confirmed as quick and effective means of assessing DOM distribution.

Keywords: Coral reef lagoons, Dissolved organic carbon, Specific UV absorption, Tuamotu, France.

Resume

Carbone organique dissous dans des lagons d’atoll, par combustion catalytique à haute température et par spectrophotométrie

Deux campagnes ont prospecté dix lagons d’atoll des Tuamotu (Polynésie française, océan Pacifique).

Sur des échantillons d’eau, le spectre UV (250-400 nm) in vitro est décrit par l’absorption à 254 nm, \( \Lambda_{254} \), et la pente, \( S^* \) (régression de \( \ln \Lambda \) versus \( \lambda \)). Ces deux descripteurs sont négativement corrélés. Les points s’organisent le long d’une hyperbole entre un pôle océanique (fort \( S^* \), faible \( \Lambda_{254} \)) et un pôle confiné (faible \( S^* \), fort \( \Lambda_{254} \)).

Le carbone organique dissous (COD) présente des concentrations ([C], déterminées par HTCO) peu variables (0.7-1.0 mg C L\(^{-1}\)), contrastant avec la large diversité des propriétés optiques. [C] et \( \Lambda_{254} \) ont une corrélation positive, avec une ordonnée à l’origine significative (0.5 mg C L\(^{-1}\)) correspondant à une fraction « non chromophorique » du COD. L’absorption spécifique ramenée au carbone, \( \varepsilon_{254} \), croît (de 0.4 à 1.3 m\(^2\) g\(^{-1}\)) pour des [C] croissantes, surtout d’après la littérature (en relation avec une augmentation du poids moléculaire moyen, qui abaisse les valeurs de \( S^* \)).

Nos mesures optiques décrivent ainsi un gradient de confinement (ou de temps de séjour) qui correspond à un continuum dans la nature du COD, dont son poids moléculaire et donc sa susceptibilité à une action biologique. Cette méthode rapide confirme son efficacité pour la description de la distribution de la MUO.

Mots clés : Lagons d’atoll, Carbone organique dissous, Absorption spécifique UV, Tuamotu, France.

Note

Présentée par Georges Pédro.

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I. INTRODUCTION

It is well accepted that dissolved organic matter (DOM) represents the main organic compartment in the global aquatic carbon stock. Despite this importance, several authors have explicitly underlined that only a small part (5-30%) of marine DOM can be chemically characterized (Collins and Williams, 1977; Chen and Bada, 1992; Benner et al., 1992; Lee and Henrichs, 1993; Amon and Benner, 1996). This has a direct consequence on the perspectives of assessing DOM’s bioavailability, which is at least as important as knowing total DOC concentration (see example in Benner et al., 1995). Describing “bulk characteristics” of marine DOM will thus remain a plausible aim, while it would be...
useful to recognize some broad categories (Hobbie, 1992), for instance through non-chemical methods (Coble et al., 1990).

Among these, optical characteristics have been widely used, perhaps more often in continental waters (partial review in Moore, 1987) than in open-sea waters. We applied spectrophotometry to characterize the waters of several coral-reef lagoons, with the initial aim of a typological, semi-quantitative classification (Pagès et al., 1997). We could thus describe the distribution of qualitative characters of DOM (or at least of its "chromophoric" part) among different lagoons. Here, we compare these results with those of the classical HTCO (high temperature catalytic oxidation) assessment of DOC concentration, to determine whether i) there is a relationship between optical properties and DOC concentration, and ii) we can predict DOC from optics.

II. MATERIAL AND METHODS

Our observations were part of several programs studying the general biological production of atoll lagoons, and more specifically the processes in the water column. The main series of samples was taken during surveys (the "Typatoll" cruises) carried out in November 1995 ("Typatoll 3") and March 1996 ("Typatoll 4"), during which we studied a total of ten atolls in the Tuamotu archipelago (15-18°S, 141-146°W). Open-sea samples were also collected. Some characteristics of the lagoons studied here are given in Table 1.

Sampling was performed within a few hours on each atoll, at 5-8 stations (see details in Pagès et al., 1997).

Spectrophotometry was performed on board during the surveys. We used a Milton Roy "Spectronic 1201" fitted with a 100 mm quartz cell. We measured absorption (A252) at 12 discrete wavelengths between 250 and 400 nm against blanks of distilled de-ionized water. Reproducibility of absorption values is good. Mean coefficient of variation on 71 duplicates (either successive sub-samples or duplicate samples) is 0.019 (i.e. less than 2%).

For DOC assessment, samples (11-mL) were not filtered (De Baar et al., 1993), but preserved with HgCl₂ (200 μL of a 0.5 g L⁻¹

| Lagoon | Area | Z | A254 | S⁺ | DOC | 254 | POC |
|--------|------|---|------|----|-----|-----|-----|
| Harai | 10.4 | 8.3 | 0.80 | 0.93 | 0.018 | 0.015 | 0.88 | 0.78 | 0.91 | 1.19 | 0.11 | 0.18 |
| Yuki | 82.5 | 18 | 0.67 | 0.74 | 0.023 | 0.025 | 1.05 | 0.96 | 0.65 | 0.77 | 0.05 | 0.11 |
| Hiti | 15.3 | 8 | 0.79 | 0.75 | 0.020 | 0.019 | 0.96 | 0.95 | 0.83 | 0.79 | 0.11 | 0.12 |
| Kauchi | 315 | 32 | 0.47 | 0.53 | 0.022 | 0.024 | 0.92 | 0.91 | 0.52 | 0.65 | 0.05 | 0.09 |
| Morokau | 217 | 20 | 0.61 | 0.65 | 0.024 | 0.021 | 0.76 | 0.88 | 0.80 | 0.74 | 0.09 | 0.11 |
| Nihiru | 79 | 18 | 0.74 | 0.71 | 0.021 | 0.018 | 0.075 | 0.74 | 0.99 | 0.97 | 0.06 | 0.11 |
| Rekareka | 0.74 | 1.0 | 1.43 | 1.28 | 0.016 | 0.015 | 1.20 | 1.03 | 1.18 | 1.25 | 0.22 | 0.23 |
| Taiaro | 11.8 | 12 | 2.58 | 2.13 | 0.022 | 0.025 | 1.91 | 1.72 | 1.36 | 1.24 | 0.18 | 0.20 |
| Tekokota | 5.1 | 3 | 0.45 | 0.43 | 0.022 | 0.019 | 0.75 | 0.74 | 0.61 | 0.56 | 0.05 | 0.09 |
| Tepoto Sud | 1.6 | 5 | 0.68 | 0.47 | 0.018 | 0.023 | 0.90 | 0.76 | 0.77 | 0.62 | 0.13 | 0.12 |
| Ocean | — | — | 0.40 | 0.37 | 0.036 | 0.043 | 0.98 | 1.03 | 0.42 | 0.36 | 0.05 | 0.05 |

Each figure is based on six separate stations. For each characteristic, two columns give the results from Typatoll 3 (T3) and Typatoll 4 (T4) surveys: Lagoon area in km², averaged depth (Z) in m; absorption A254 in m⁻¹; spectrum slope S⁺ in nm⁻¹; carbon-specific absorption B254 in m² (g C)⁻¹; DOC and POC in mg C L⁻¹.

Chaque valeur est fondée sur six stations distinctes. Pour chaque paramètre, deux colonnes donnent les résultats des deux campagnes, Typatoll 3 (T3) et Typatoll 4 (T4).
solution) and HCl (50 µL of a 6 N solution), and kept at 5 °C on board. Measurements were made (at least in duplicate) with standard HTCO techniques on a Shimadzu TOC 5000. Mean standard error on duplicates was 0.046 mg C.L⁻¹, i.e. 4.3%. These TOC figures were then corrected for particulate organic carbon (POC), which we determined with a Leco CHN analyzer.

III. RESULTS

1. Spectral parameters and qualitative aspects

Most of this section has been expounded elsewhere (Pagès et al., 1997), but must be briefly recalled for comprehension of the following.

Absorption spectra show the classical linear relation between ln A and λ (Bricaud et al., 1981). Spectrum slope (S*) values range between 0.009 and 0.030 nm⁻¹ in atolls, while oceanic samples exhibit higher values, up to 0.040 nm⁻¹ (table I). Absorption at 254 nm (A₂₅₄) mostly ranges between 0.5 and 0.8 m⁻¹, with lower values (0.4 m⁻¹) in oceanic samples, and higher values (up to about 2 m⁻¹) in some confined lagoons (Rekareka and Tāiāro).

A plot of A₂₅₄ against S* for all lagoon and seawater samples (fig. 1) shows an inverse correlation between these two descriptors. The waters from Tāiāro, a closed lagoon with abundant terrestrial vegetation, are conspicuous outliers. Their abnormally high S* values stem from the presence of lignin degradation products and humic acids (ms in prep.). Excluding this lagoon, the best fit (r² = 0.60) is given by the regression of (1/S*) against (1/A₂₅₄) (see table II).

Data points are not randomly distributed along this hyperbolic regression curve. Spatially detailed observations in Tikahau lagoon (see details in Pagès et al., 1997) show that data points are arranged between an “oceanic” pole, with high S* values (~0.04 nm⁻¹) and low A₂₅₄ (0.4 m⁻¹), and a “eutrophic” pole, with low S* (<0.01 nm⁻¹) and high A₂₅₄ (>2 m⁻¹). By mixing different water types, we could see that the observed hyperbola corresponds to mixtures, in varying proportions, between two main types of water reflecting the above description of the two poles. We are thus dealing with a continuum of waters between which there is no basic difference, but only a continuous variation of proportions.

2. Quantitative aspects: DOC and carbon-specific absorbance

(a) DOC concentrations

For most lagoon samples, DOC concentrations [C] exhibit a narrow range, from 0.7 to 1.0 mg C.L⁻¹ (table I), against [C] values of ~1.0 mg C.L⁻¹ in oceanic samples. Relatively high DOC concentrations are only found in Rekareka and Tāiāro (averaging 1.11 and 1.82 mg C.L⁻¹). Intra-lagoon variability is very low in most cases. Coefficient of variation (CV) for a given lagoon on a given survey generally amounts to ~5%, and this includes the (low) analytical variability. If we consider only “normal” lagoons and oceanic samples,
they exhibit a 1.5-fold amplitude for [C], against a two-fold one for $A_{254}$ (see Table I).

Absorption, $A_{254}$ (in m$^{-1}$), is correlated with DOC concentration (Fig. 2 and Table II). The overall regression ($r^2 = 0.76$) is slightly improved ($r^2 = 0.84$) when oceanic samples exhibiting "too much" DOC are excluded. We can note that this correlation is acceptable only after including the "high DOC" lagoons (Taiaro and Rekareka); with "normal" lagoons only, the correlation is statistically significant ($r = 0.521$, $n = 107$, $P > 0.001$) but the regression is worthless in terms of predicting [C] from $A_{254}$. The other salient point is the intercept, amounting to about 0.5 mg C.L$^{-1}$ of DOC without optical activity.

Inspection of the results indicates that $A_{254}$ allows a better discrimination between lagoons than does [C]. To quantify the discriminative potential of $A_{254}$ and [C], we tested the null hypothesis of a homogeneous population comprising all stations in all lagoons and the ocean during the two surveys (22 sets of six data each), using the Kruskal–Wallis one-way analysis by ranks (two-by-two comparison on rank sums of adjacent sets; 21 comparisons). Significance of the differences between lagoons was also tested using the Mann–Whitney test (two-by-two comparison on all data; 213 (i.e. 21 + 20 + 19 + ...) comparisons). Results of the two tests confirm that optics are more able to discriminate between lagoons than chemical (HTCO) DOC measurements.

We computed carbon-specific absorption as $e_{254} = A_{254}/[C]$, with $A$ in m$^{-1}$ and [C] in mg C.L$^{-1}$. Values of $e_{254}$ (see Table I) range between 0.5 and 1.0 m$^2$.g$^{-1}$ in "normal" lagoons, but are higher for Rekareka and Taiaro lagoons (1.2–1.3 m$^2$.g$^{-1}$), while oceanic samples exhibit low $e_{254}$ values ($< 0.4$ m$^2$.g$^{-1}$).

Comparison between $e_{254}$ and spectrum slope $S^*$ shows a general inverse trend (high $e_{254}$ values for low $S^*$), with Taiaro samples as prominent outliers (figure not shown). Excluding Taiaro, the best fit is given by a hyperbolic ($1/e_{254}$ versus $1/S^*$) equation with significant correlation ($r^2 = 0.62$, $P < 0.001$ for $n = 130$). Data points appear too widely scattered (figure not shown) for a satisfactory prediction of $e_{254}$ from $S^*$.
(c) Prediction of DOC concentration

The various correlations seen above should allow determination of DOC concentration, [C], from optical properties, in particular from the regression between $A_{254}$ and DOC concentration. We obtain a set of computed values, [C], which we compare with actual (measured) values [Cm] (see fig. 3).

In the narrow range of "normal" lagoons ($n = 96$), mean values ($\pm$ s.d.) agree very well: $0.852 \pm 0.096$ for [Cm], against $0.827 \pm 0.083$ for [Cz]. For oceanic samples, [Cz] values are underestimated ($0.68$ mg C L$^{-1}$ instead of $1.00$), in accordance with the divergence between [Cm] and $A_{254}$ (section (a)). Considering now the whole range, with the two "eutrophic" lagoons (Rekareka and Taiaro), we obtain a high correlation ($r^2 = 0.857$) between [Cm] and [Cz] (see fig. 3 and table II).

IV. DISCUSSION

The relations we found between optical properties and DOC concentration, [C], have two aspects: i) quantified prediction of [C] and ii) semi-quantitative evaluation of the nature of the DOC.

Optics, and especially absorption at a given wavelength, have been widely used as a proxy estimator (or a "surrogate parameter" (Summers et al., 1987)) of DOM concentration, in the laboratory or in the field. Even if caution is necessary in some particular environments, such as hard-water lakes (Stewart and Wetzel, 1981), optical measurements provide an interesting method for DOC assessment, once duly calibrated. In our case, agreement between measured [Cm] and computed [Cz] is reasonable ($r^2 = 0.86$) across the whole range studied here (including Rekareka and Taiaro).

Excluding Rekareka and Taiaro, the "normal" lagoons show a small relative variation in total DOC concentration (range 0.74-1.05 mg C L$^{-1}$). This contrasts with the much wider variation in absorption ($A_{254}$ ranging between 0.43 and $0.93$ m$^{-1}$). We have seen that $A_{254}$ discriminates better between lagoons than does bulk DOC concentration. The sensitivity of optical characteristics (both $\varepsilon_{254}$ and $S^*$) to minute variations in DOM nature and/or concentration has been shown in a previous paper (Pagès et al., 1997).

The DOC concentrations we found are quite normal for oligo- to mesotrophic waters (Martin and Fitzwater, 1992; Guo et al., 1994; Carlson and Ducklow, 1995). In these "normal" lagoons, then, the purely quantitative aspect of optical measurements is useful only as a preliminary estimation of bulk DOC concentration, especially when considering the cost/benefit ratio (see the analogous conclusion reached by Moore, 1987).

The semi-quantitative evaluation of DOC nature has higher potentials. Our argumentation is based on (i) the $A_{254}$-versus-$S^*$ distribution, and (ii) the correlation between [C] and $A_{254}$. This latter shows the existence of a "residual" colourless DOC fraction amounting to about $0.5$ mg C L$^{-1}$. Such an optically inactive fraction (appearing as the intercept in the [C]-versus-$A_{254}$ regression) has been found in other environments, at concentrations ranging between 0.4 and $0.9$ mg C L$^{-1}$ in oceanic samples (Amador et al., 1990; Vodacek et al., 1995) and between 2 and $6$ mg C L$^{-1}$ in coastal or continental waters (De Haan and De Boer, 1987).

The relative increase in the ratio between active and inactive C at increasing [C] leads to the mathematical artifact of increasing $\varepsilon_{254}$, even if the "true" specific absorption ($\varepsilon^*$) were constant, which is not necessarily true (see below).

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**Fig. 3** DOC calculated from $A_{254}$ compared to measured values. Same symbols as in figure 1.

COD calculé à partir de $A_{254}$ par rapport aux valeurs déterminées expérimentalement. Symboles : voir la figure 1.
Our $\varepsilon_{254}$ varies between 0.6 and 1.4 m$^2$ g$^{-1}$ in the lagoons studied. We found only one comparable value (0.29 m$^2$ g$^{-1}$), obtained on marine fulvics (Carder et al., 1989). Most other published values range between 3 and 11 m$^2$ g$^{-1}$, but were obtained with isolated fractions (humics or fulvics) of various aquatic origins (Summers et al., 1987; De Haan et al., 1988; Blough et al., 1993).

Among the identified factors of variation of $\varepsilon$ (at any wavelength) are ionic strength (Summers et al., 1987; De Haan et al., 1987) and pH (De Haan et al., 1983), which are irrelevant for our measurements on natural seawater. We deal here with marine DOM, which is an undetermined mixture of molecules (Amon and Benner, 1996). The chief factor controlling $\varepsilon$ in natural samples will be average molecular weight, MW, or "molecular size". Several studies show, more or less explicitly, a positive correlation between $\varepsilon$ and log MW, and a negative correlation between spectrum slope $S^*$ (or the equivalent ratio E2/E3 (De Haan et al., 1988)) and MW (Stewart and Wetzel, 1980; De Haan et al., 1983; Summers et al., 1987; Senesi et al., 1989).

The "confined" pole, with high $\Lambda_{254}$ and high [C] corresponds to gradually increased average MW (as evidenced by the low $S^*$ values), as found in other environments (Tanzi, 1990; Guo et al., 1994). The increase in $\varepsilon_{254}$ in this chromophoric portion may be also due to a parallel (and slight) increase in the proportion of high-$S^*$ molecules (Blough et al., 1993), such as lignin derivatives, or UV-protecting compounds (Shick et al., 1992).

The continuity that we observe between lagoons suggests a continuum of MW, such as is described by several authors (Moran et al., 1991; Guo et al., 1994; Amon and Benner, 1996). This array of MWs can result in part from bacterial heterotrophic activity, which leads to decreased $S^*$ (Blough et al., 1993). Bacterial uptake induces very small variations in bulk properties (Brophy and Carlson, 1989), but may alter the more sensitive optical properties of DOM.

V. CONCLUSIONS

Optical characteristics allow the prediction of DOC concentration with an accuracy of about ±0.1 mg L$^{-1}$ (average absolute difference between [Cm] and [Cv]). The main potential of optics lies though in the ability to detect minute variations, or alterations, in the nature of DOC, or at least of its "chromophoric" portion.

We have noted that our data points (our lagoon samples) are arrayed in the $\Lambda_{254}$ versus $S^*$ plane, between two poles. A distance along the regression curve (measured on the graph from the data points for open-sea samples) would then also correspond to a factual distance (in space and/or in time) between the open sea and a water body. Optical characteristics would then estimate confinement. We could verify their good correlation with chlorophyll concentration Pagès et al., (1997), which has been shown to be related to water residence time (Turnas et al., 1990; Delcaselle and Sournia, 1992).

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