Geophysical Research Abstracts
Vol. 12, EGU2010-4851, 2010
EGU General Assembly 2010

Comparison of infrared spectroscopy techniques: developing an efficient method for high resolution analysis of sediment properties from long records

Annette Hahn (1), Peter Rosén (2), Pierre Kliem (1), Christian Ohlendorf (1), Per Persson (3), Bernd Zolitschka (1), and the PASADO Science Team ()

(1) Geomorphology and Polar Research (GEOPOLAR), Institute of Geography, University of Bremen, Celsiusstr. FVG-M, D-28359 Bremen, Germany (anhahn@uni-bremen.de), (2) Climate Impacts Research Centre, Umeå University, SE-981 07 Abisko, Sweden, (3) Department of Chemistry, Umeå University, SE-901 87 Umeå, Sweden

The analysis of sediment samples in visible to mid-infrared spectra is ideal for high-resolution records. It requires only small amounts (0.01-0.1g dry weight) of sample material and facilitates rapid and cost efficient analysis of a wide variety of biogeochemical properties on minerogenic and organic substances (Kellner et al. 1998). One of these techniques, the Diffuse Reflectance Fourier Transform Infrared Spectrometry (DRIFTS), has already been successfully applied to lake sediment from very different settings and has shown to be a promising technique for high resolution analyses of long sedimentary records on glacial-interglacial timescales (Rosén et al. 2009). However, the DRIFTS technique includes a time-consuming step where sediment samples are mixed with KBr. To assess if alternative and more rapid infrared (IR) techniques can be used, four different IR spectroscopy techniques are compared for core catcher sediment samples from Laguna Potrok Aike – an ICDP site located in southernmost South America. Partial least square (PLS) calibration models were developed using the DRIFTS technique. The correlation coefficients (R) for correlations between DRIFTS-inferred and conventionally measured biogeochemical properties show values of 0.80 for biogenic silica (BSi), 0.95 for total organic carbon (TOC), 0.91 for total nitrogen (TN), and 0.92 for total inorganic carbon (TIC). Good statistical performance was also obtained by using the Attenuated Total Reflectance Fourier Transform Infrared Spectroscopy ATR-FTIRS technique which requires less sample preparation. Two devices were used, the full-sized Bruker Equinox 252 and the smaller and less expensive Bruker Alpha. R for ATR-FTIRS-inferred and conventionally measured biogeochemical properties were 0.87 (BSi), 0.93 (TOC), 0.90 (TN), and 0.91 (TIC) for the Alpha, and 0.78 (TOC), 0.85 (TN), 0.79 (TIC) for the Equinox 252 device. As the penetration depth of the IR beam is frequency dependent, a firm surface contact of the sample is necessary. This could not be accomplished, therefore absorbance in higher wavelengths was not recorded correctly. As a result of the poor spectral quality no calibration model was established for BSi using the Equinox device. Since this is by far the most time-consuming and elaborate conventional measurement, results give clear advantages for the Alpha device.

Further calibration models were developed using spectra from the Visible Near Infrared Spectroscopy (VNIRS) region (400-2500 nm). Sample preparation for VNIRS analysis also is faster than for DRIFTS. However, FTIRS calibrations seem to perform better than those for VNIRS which show an R of 0.75 (BSi), 0.93 (TOC), 0.93 (TN), and 0.89 (TIC). NIRS primarily measures overtones of molecular vibrations and is typically used for quantitative measurement of organic functional groups. FTIRS is similar to NIRS, but uses longer wavelengths and directly monitors molecular vibrations. As a consequence, FTIRS allows more detailed structural and compositional analyses of both organic and inorganic compounds. Statistical analysis of the FTIRS-PLS models shows that the calibration depends on specific wave numbers, which compare well with spectra of pure compounds. The VNIRS technique gives rise to a spectrum with broad peaks and many overlapping signals which makes interpretation difficult without statistical analyses.

In conclusion, the DRIFTS technique shows the best statistical performance for the analysis of biogeochemical properties. However, the VNIRS techniques and especially the ATR-FTIRS Alpha device show comparable results and can also be used as a rapid screening tool when time and costs are limiting factors.

Kellner R, Mermet J-M, Otto M, Widmer HM (1998) Analytical chemistry. Wiley-VCH, Weinheim, etc.

Rosén P, Vogel H, Cunnigham L, Reuss N, Conley DJ, Persson P (2009) Fourier transform infrared spectroscopy, a new method for rapid determination of total organic and inorganic carbon and biogenic silica concentration in lake sediments. J Paleolimnol