

# Techniques for the non-destructive and continuous analysis of sediment cores. Application in the Iberian continental margin

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## ABSTRACT

Sediment sequences are the most valuable record of long-term environmental conditions at local, regional and/or global scales. Consequently, they are amongst the best archives of the climatic and oceanographic history of the Earth. In the last few decades a strong effort has been made, both in terms of quantity and quality, to improve our knowledge regarding the evolution of our planet from marine and lake sediment records, and also from other records such as ice cores. Such an effort requires reinforcing the geographical coverage and achieving the highest possible robustness in the reconstruction of past environments. Such a target requires the optimization of the time resolution of the records and reconstructions so that fast, high frequency shifts, such as those occurring nowadays due to the on-going global warming, can be disentangled. Beyond paleoenvironmental research, other disciplines have also contributed significantly to the fast growing number of sediment cores already available worldwide. Knowing the physical state and the chemical composition of sedimentary deposits is essential for land management purposes and for many industrial applications. A number of key technological developments are now allowing the acquisition for the first time of massive amounts of multiple parameters from sediment cores in a non-destructive, fast, continuous, repetitive and high-resolution form. In this paper we provide an overview of the state-of-the-art continuous and non-destructive analytical techniques used by the geoscientific community for the study of sediment cores and we present some examples of the application of these methods in several studies carried out around the Iberian Margin.

Key words: chemical properties, high-resolution records, non-destructive techniques, physical properties, sediment cores.

## ***Técnicas para el análisis no destructivo y en continuo de testigos de sedimento. Aplicación en el Margen Continental de Iberia***

### RESUMEN

*Las secuencias sedimentarias constituyen el registro más valioso de las condiciones ambientales del pasado a largo plazo, a escalas local, regional y global. Son, por tanto, uno de los mejores archivos de la historia climática y oceanográfica de la Tierra. En las últimas décadas se ha hecho un gran esfuerzo, tanto cuantitativo como cualitativo, para mejorar el conocimiento de la evolución de nuestro planeta a partir de registros sedimentarios marinos y lacustres, y también de otros registros, como los testigos de hielo. Dicho esfuerzo pasa por alcanzar una amplia cobertura geográfica y conseguir la mayor consistencia posible en las reconstrucciones ambientales del pasado. Para ello es necesario optimizar la resolución temporal de los registros y de las reconstrucciones, de modo que se puedan discriminar los cambios rápidos, de alta frecuencia, como los que se están sucediendo a raíz del calentamiento global rápido actual. Más allá de la investigación paleoambiental, otras disciplinas han contribuido también de manera significativa a acrecentar el número de testigos de sedimento ya disponibles a escala global. El conocimiento del estado físico y de la composición química de los depósitos sedimentarios es, por ejemplo, esencial para la gestión del uso de la tierra y para aplicacio-*

nes industriales. Un buen número de desarrollos tecnológicos clave está permitiendo adquirir por primera vez cantidades masivas de múltiples parámetros a partir de testigos de sedimento de forma no destructiva, rápida, continua, repetitiva y con alta resolución. En esta contribución se hace un repaso del estado del arte de las técnicas analíticas continuas y no destructivas utilizadas por la comunidad geocientífica para el estudio de testigos de sedimento y presentamos algunos ejemplos aplicados del uso de estos métodos en diversos trabajos realizados en el Margen Ibérico.

*Palabras clave:* propiedades químicas, registros de alta resolución, técnicas no destructivas, propiedades físicas, testigos de sedimento.

## VERSION ABREVIADA EN CASTELLANO

### **Introducción**

La medición continua y con carácter no destructivo de las propiedades físicas y químicas de testigos de sedimento a resoluciones altas y muy altas (cm- $\mu$ m) es la base de cualquier investigación avanzada sobre el contexto ambiental en que se formó el registro objeto de examen, así como los procesos y mecanismos involucrados. Las técnicas correspondientes proveen una enorme cantidad de datos de gran valor para las investigaciones relacionadas con el estudio de las condiciones climáticas y ambientales del pasado. Además, dichas técnicas son también muy importantes en campos de actividad tan distintos como la geotecnia, el análisis de riesgos geológicos (p.ej., estabilidad de taludes), la exploración petrolífera, el estudio de la contaminación de suelos o las obras públicas (Rothwell and Rack, 2006). El uso de estas técnicas ha revolucionado la investigación en el campo de las Geociencias, puesto que permiten obtener una gran cantidad de información en muy poco tiempo, con un coste relativamente bajo, y con una resolución que en muchas ocasiones es inalcanzable con las técnicas tradicionales.

Los registros de las propiedades físicas y químicas del sedimento son esenciales para determinar la estratigrafía y correlacionar testigos, para obtener una primera valoración sobre la composición y la textura de los sedimentos y para disponer de series temporales continuas de alta calidad. Además, proporcionan información determinante en la toma de decisiones sobre la estrategia de muestreo para análisis destructivos (St-Onge et al., 2007). En este trabajo presentamos una breve descripción de las principales técnicas no destructivas utilizadas en el campo de las Geociencias, algunas de las cuales están ya operativas en algunos laboratorios españoles.

### **Perfiladores de testigos multisensoriales**

Los perfiladores multisensoriales (MSCL) para el análisis en continuo, no destructivo y de alta resolución de las propiedades físicas y geoquímicas de testigos de sedimento se han desarrollado durante los últimos 20 años, en particular por la empresa Geotek. Estos perfiladores permiten medir la susceptibilidad magnética, la densidad aparente, la velocidad de las ondas P, la resistividad eléctrica o la radiación natural gamma en testigos de sedimento sin abrir o abiertos longitudinalmente (MSCL estándar). Los modelos más recientes cuentan también con sensores para la toma digital en continuo de imágenes fotográficas, térmicas y de rayos X en 2D (radiografías) y en 3D (Tomografía Axial Computerizada, TAC), así como con sensores de fluorescencia de rayos X y colorimetría (Figs. 1 y 2).

En España, diversos organismos públicos cuentan con el MSCL estándar, como el Instituto de Ciencias del Mar en Barcelona y el Instituto Andaluz de Ciencias de la Tierra en Granada, ambos del CSIC, el Instituto Geológico y Minero de España (IGME), o la Unidad de Tecnología Marina del CSIC en un laboratorio contenedor para poder ser instalado en buques de investigación oceanográfica. Además, la Universidad de Barcelona tiene prevista la adquisición de un nuevo MSCL estándar en breve.

### **Radiografía digital 2D y 3D**

#### **Radiografías digitales en 2D**

La utilización de rayos X para la obtención de radiografías de testigos se basa en la diferencia de transmisión de la radiación X en el sedimento debido a la atenuación por absorción y dispersión producida por las partículas y otras sustancias que lo forman (Bouma, 1969; Principato, 2004). La atenuación de los rayos X depende principalmente de la densidad relativa del sedimento (Axelsson, 1983; Holyer et al., 1996), la cual también

está afectada por el tamaño de grano y la composición, así como por variaciones en el contenido de agua, en la compactación o en la porosidad (St-Onge et al., 2007).

Durante muchos años las radiografías de testigos de sedimento se realizaban con sistemas médicos no específicos para materiales geológicos. Actualmente existen varios sistemas de radiografía digital en 2D diseñados para el trabajo con testigos de sedimento, como el sistema llamado SCOPIX, desarrollado por la Universidad de Burdeos 1 (Fig. 3), el MSCL-XCT de Geotek, o el sistema incorporado en el escáner de testigos por FRX de ITRAX. En España, el MSCL de la Unidad de Tecnología Marina del CSIC tiene incorporado este sistema.

### **Tomografía axial computarizada (TAC)**

La tomografía axial computarizada (TAC) permite la visualización y el análisis no destructivo de las estructuras internas del sedimento a resoluciones muy elevadas (~0.1-1 mm). La técnica se basa en el mismo principio de atenuación de los rayos X que las radiografías 2D, con la diferencia que los equipos de TAC permiten cuantificar y mapear el testigo (u otro objeto), facilitando así la reconstrucción tridimensional del mismo. Las imágenes de TAC ilustran, como las radiografías, pero en 3D, los cambios en las propiedades densitométricas del sedimento (Fig. 4).

Hasta hace poco los análisis de TAC en testigos de sedimento se realizaban con equipos para uso médico y veterinario no específicos para materiales geológicos, con una resolución máxima de voxel de 200-300  $\mu\text{m}$ . En España, el equipo "Multitom CORE X-ray CT system" instalado en el laboratorio CORELAB de la Universidad de Barcelona, fue diseñado para poder analizar testigos de sedimento largos y otro tipo de muestras, permitiendo obtener tomografías computarizadas con resolución variable (entre 5 y 300  $\mu\text{m}$ ).

### **Cámaras fotográficas digitales y en color de barrido lineal**

Durante la apertura de los testigos, el sedimento entra en contacto con el aire y puede oxidarse rápidamente. Por este motivo es necesario obtener fotografías digitales de los testigos inmediatamente después de su apertura, las cuales servirán de referencia exacta del aspecto del sedimento en sus condiciones originales (Fig. 5). En la actualidad existen sistemas de fotografía digital en continuo que proporcionan imágenes con una gran resolución de pixel (entre 25 y 100  $\mu\text{m}$ ), a partir de las cuales, con un software adecuado, se pueden extraer los datos numéricos del color en varios formatos, generalmente RGB o CIE-L\*a\*b\*. Este tipo de cámaras han sido incorporadas en algunos de los equipos más utilizados para el análisis de las propiedades físicas y químicas de los sedimentos, como el MSCL de Geotek, y los escáneres de testigos por FRX de AVAA-TECH y ITRAX (Fig. 6).

### **Escáneres de fluorescencia de rayos X**

Los escáneres de testigos por fluorescencia de rayos X (FRX o XRF en inglés) permiten obtener la composición química elemental, para los elementos con pesos atómicos entre el Al y el U, en secciones abiertas, de manera continua, no destructiva y relativamente rápida, con una resolución que oscila entre 1 cm y 100  $\mu\text{m}$  (Jansen et al., 1998; Rothwell and Rack, 2006). En Europa hay actualmente dos compañías fabricantes de estos sistemas: Cox Analytical Systems que comercializa el sistema ITRAX, y AVAA-TECH que comercializa el sistema del mismo nombre (Fig. 7).

Las variaciones en la composición elemental de los sedimentos contienen información clave sobre cambios en el área fuente, los procesos y mecanismos de transporte, y los patrones de sedimentación de las partículas, generalmente relacionados con fluctuaciones ambientales y climáticas, incluidos los resultantes de la influencia humana (Löwemark et al., 2011). Por esta razón, los registros elementales de FRX se usan como indicadores (proxies) de las condiciones ambientales que determinan el registro sedimentario (p.ej., Rothwell and Rack, 2006; Calvert and Pedersen, 2007).

En España, la Universidad de Barcelona cuenta con un escáner de XRF de AVAA-TECH, y la Universidad de Vigo dispone de un escáner de XRF de ITRAX. Además, el IGME tiene prevista la adquisición de un escáner de XRF para la mejora del equipo de MSCL estándar.

### **Magnetómetros**

El estudio de las propiedades magnéticas de los sedimentos en un sentido amplio puede resultar también de gran interés, tanto con fines de datación como para obtener información paleoambiental, aprovechando el carácter ubicuo de los minerales magnéticos, especialmente óxidos de hierro, puesto que este es uno de los

*elementos más abundantes en la corteza terrestre (Evans and Heller, 2003). La información que aporta el análisis de las propiedades magnéticas de los sedimentos, cuya riqueza en magnetita puede variar notablemente, se utilizó inicialmente en el estudio de los cambios del campo magnético terrestre como útil de datación y puede ser utilizada también como indicador de procesos paleoambientales, como la acumulación de cantidades significativas de polvo del desierto o loess, entre otros (p. ej., Thouveny *et al.*, 2000; Moreno *et al.*, 2002; Evans and Heller, 2003; Larrasoña *et al.*, 2003; Stoner and St-Onge, 2007; Larrasoña *et al.*, 2008; Channell *et al.*, 2013). El sistema más usado en el análisis de sedimento es el magnetómetro superconductor SRM, que permite impartir y medir magnetizaciones de diversos tipos, como la magnetización remanente anhistórica (ARM), la magnetización remanente isotérmica (IRM) y la susceptibilidad magnética volumétrica.*

*En España, el Laboratorio de Arqueomagnetismo del Centro Nacional de Investigación sobre la Evolución Humana (CENEH) en Burgos y el Laboratorio de Paleomagnetismo del Institut de Ciències de la Terra Jaume Almera (ICTJA-CSIC) en Barcelona están dotados con diversos equipos (principalmente magnetómetros y desmagnetizadores de diversos tipos) que permiten la obtención de información relevante para la realización de estudios sobre datación magnetoestratigráfica de secuencias de sedimento, datación arqueomagnética, paleomagnetismo aplicado al estudio de cinturones orogénicos y tectónica de placas y estudios magnéticos ambientales.*

## Introduction

A rather common problem when dealing with sediment cores is the limited volume of sediment that is available per unit length, which depends on the diameter of the core. This imposes a limitation to the amount of sediment ready for redistribution to the interested parties and, consequently, to the number of analyses that can be carried out. Classical analytical procedures involve treating and destroying the samples as a result of the analysis itself. The reality is that sediment availability exerts a main control over the resolution that can be achieved in every study, in the same way that sedimentation rates limit the temporal resolution. Furthermore, high to very high-resolution studies are also hampered because of (i) the practical difficulties for obtaining sediment slices thinner than 1-0.5 cm yielding the minimum volume needed for subsequent analyses, (ii) the high amount of time and effort required for high-frequency sub-sampling of sediment cores, and (iii) the increase of the costs associated with any improvement of the sub-sampling resolution followed by subsequent analyses (i.e. the larger number of samples to be analysed).

The last few decades have seen the development of several large-scale programmes such as the *International Marine Past Global Change Study (IMAGES)*, the *International Ocean Discovery Program (IODP)* and its predecessors, and the *International Continental Drilling Project (ICDP)*, which together with numerous national and industry driven projects have provided an unprecedented amount of sediment cores. As a consequence of the necessity to quickly and reliably analyse such an enormous number of cores at the highest possible resolution, the development of non-invasive and non-destructive techniques has been boosted during this period.

These techniques have revolutionized geoscientific

research as they provide high amounts of highly valuable information in a rapid and continuous way, at relatively low cost, and also save operator time, with a resolution that in most occasions is not achievable through classical, destructive techniques (Rothwell and Rack, 2006). These techniques are particularly useful in fields such as paleoclimatology and paleoceanography, sedimentology, geotechnics, geohazard assessment (e.g., slope stability), hydrogeology, hydrocarbon exploration, mining, environmental assessment, soil pollution, in public works and for military purposes. Today the continuous, non-destructive measurement of the physical and chemical properties of sediment cores at high and very high resolution (cm- $\mu$ m) is a basic requirement for investigating the environmental setting where the sediment records under study were formed, and on the processes and mechanisms involved, and also to assess the state of sedimentary deposits for multiple applications.

The continuous record of the physical and chemical properties of the sediment is crucial to establish the stratigraphy of the cores and for core correlation, to get a first insight on sediment composition and texture, and to obtain high-quality time logs. Furthermore, they provide valuable information for defining the best sub-sampling strategy for subsequent destructive analyses on discrete volumes of sediment (St-Onge *et al.*, 2007). Taking of all these reasons into account, non-destructive techniques for the continuous analysis of sediment cores now constitute an essential toolkit that is widely used by scientists and professionals of numerous disciplines.

An additional advantage of these techniques when sea-going work is involved is that most of the instruments used to carry out the measurements can be installed onboard oceanographic vessels, thus allowing the analyses to be performed immediately after the sediment cores are recovered, i.e. when they are



still undisturbed due to transport, storage and ageing. However, in most occasions, and also for land work, cores still are transported to specialised laboratories where the analyses are performed with the shortest delay.

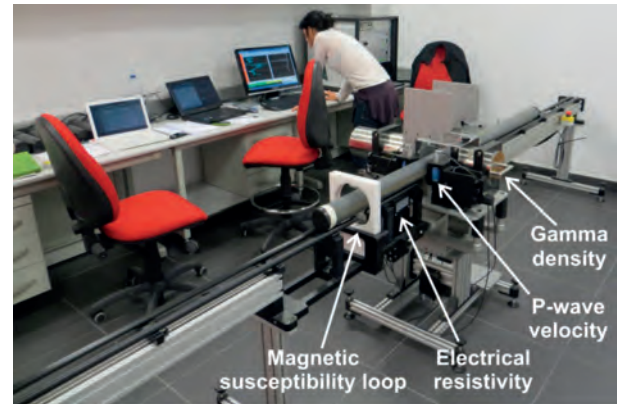
In this paper we summarize the main continuous and non-destructive techniques and instruments that are increasingly used in the broad area of Geosciences, namely multisensory core logging, digital core imaging and colour data acquisition, digital 2D and 3D X-radiography, X-ray fluorescence profiling by means of core scanners, and the measurement of magnetic properties. Several of these techniques are fully operational in Spanish laboratories. For a more detailed review on non-destructive methods and measurement protocols the reader is referred to the works of Blum (1997), Rothwell and Rack (2006) and St-Onge *et al.* (2007).

### Multi-sensor core loggers

Over the last twenty years multi-sensor core loggers (MSCLs) have been developed, in particular by the Geotek company, for the continuous, high-resolution and non-destructive measurement of the physical and chemical properties of sediment cores. MSCLs allow the simultaneous, automatic measurement of the magnetic susceptibility, the wet bulk density (by the gamma attenuation method), the P-wave velocity, the electrical resistivity and the natural gamma radiation of the sediments in whole or split sediment cores (Figs. 1 and 2). The most modern MSCL systems can also be equipped with sensors for line-scan photography, thermal, and 2D (radiography) and 3D (computerized axial tomography) X-ray imagery, and also with sensors for X-ray fluorescence and colour spectrophotometry.

The standard system (MSCL-S) can log sediment cores of 50-150 mm in diameter at rates of 12  $\text{m h}^{-1}$  at sampling intervals down to 1 mm. Core sections normally are up to 1.5 m long and measurements are done with cores in a horizontal position, though a vertical configuration (MSCL-V) is available too, which is mostly used for measurements of the sediment-water interface, and is particularly well suited for laboratories or vessels where available space is limited. MSCLs are often installed onboard survey and drilling vessels allowing the measurements to be carried out immediately after core retrieval.

In addition to their robustness, the Geotek MSCLs are very versatile since they can be equipped with sensors for most of the non-destructive methods currently applied to the analysis of sediment and rock



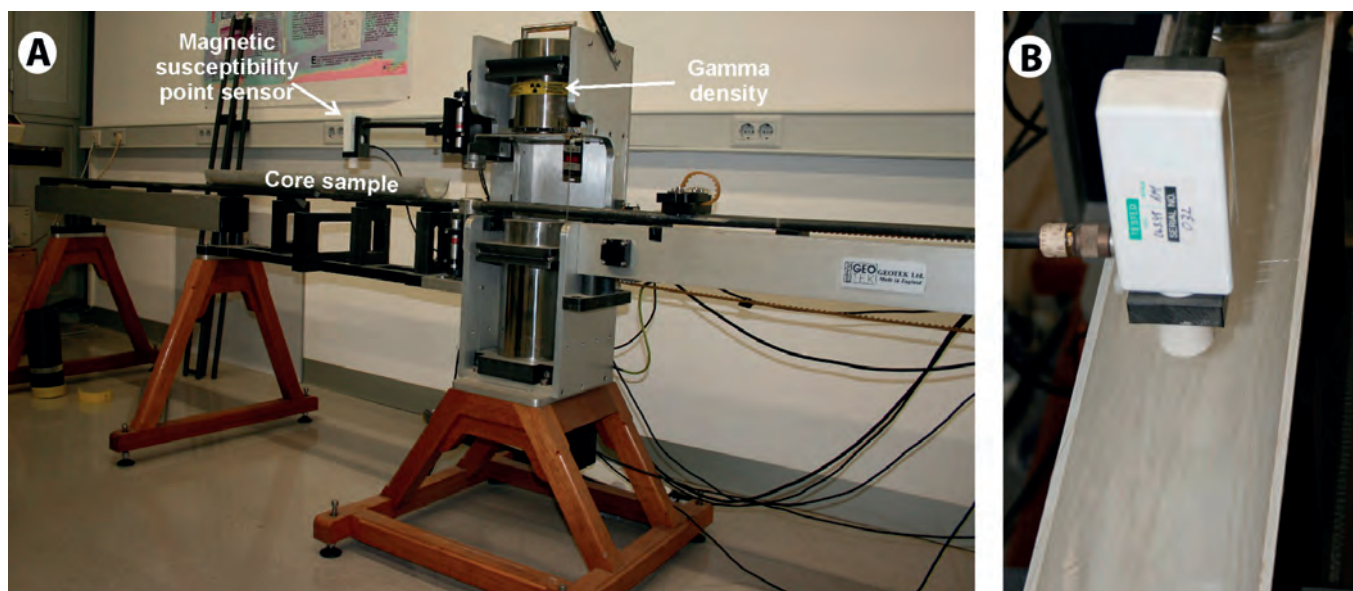
**Figure 1.** Geotek MSCL system installed in the Instituto Andaluz de Ciencias de la Tierra (IACT) of the Spanish National Research Council (CSIC), Granada ([www.iact.csic.es/](http://www.iact.csic.es/)). This system incorporates sensors for measuring, from left to right: the magnetic susceptibility, the electrical resistivity, the P-wave velocity and the gamma density. In the picture the system is set for the analysis of whole cores.

**Figura 1.** Sistema MSCL de Geotek instalado en el Instituto Andaluz de Ciencias de la Tierra (IACT) del Consejo Superior de Investigaciones Científicas (CSIC), Granada ([www.iact.csic.es/](http://www.iact.csic.es/)). Este sistema incorpora los sensores para medir, de izquierda a derecha: la susceptibilidad magnética, la resistividad eléctrica, la velocidad de las ondas P y la densidad gamma. En la imagen el equipo está configurado para el análisis de testigos cerrados.

cores. Geotek MSCL systems are now the most widely used instruments for the continuous measurement of the physical properties of sediment cores. Later on in this section we will focus on three key physical properties that are usually measured with MSCL-S, which are gamma density, P-wave velocity and magnetic susceptibility. Other measurements, such as line scan imaging, colour spectrophotometry, X-ray fluorescence and X-ray core imaging are described individually in other sections of this paper. For further information on natural gamma radiation and electrical resistivity we refer the reader to Breitzke (2006), Jackson *et al.* (2006), Rothwell and Rack (2006) and St-Onge *et al.* (2007). In Spain, MSCL-S are operating at the *Instituto de Ciencias del Mar*, the *Instituto Andaluz de Ciencias de la Tierra* or in a containerised lab to be installed on oceanographic vessels at the *Unidad de Tecnología Marina*, all of them belonging to the CSIC, and at the *Instituto Geológico y Minero de España*-IGME. In addition, the University of Barcelona will acquire a new MSCL-S shortly.

### Gamma density

The density of a material is a measure of how tightly the matter within it is packed together and is given by



**Figure 2.** A) MSCL system of Geotek installed in the Institut de Ciències del Mar (ICM) of the Spanish National Research Council (CSIC), Barcelona ([www.icm.csic.es/geo/gma/](http://www.icm.csic.es/geo/gma/)). In the picture the system is set for the analysis of split cores; and B) a detail of the point sensor Bartington for magnetic susceptibility measurements on split-sediment cores.

**Figura 2.** A) Sistema MSCL de Geotek instalado en el Instituto de Ciencias del Mar (ICM) del Consejo Superior de Investigaciones Científicas (CSIC), Barcelona ([www.icm.csic.es/geo/gma/](http://www.icm.csic.es/geo/gma/)). En la imagen el equipo está configurado para el análisis de testigos abiertos; y B) detalle del sensor de puntero Bartington MS2E para la medida de la susceptibilidad magnética en testigos de sedimento abiertos.

the ratio of its mass to its volume. The bulk density of soils and sediments is greatly dependent on their mineralogy and degree of compaction, and can give information about grain-size, porosity and water content. In water-saturated materials such as seafloor sediments, bulk density is closely related to porosity and water content, so that the measurement of one of these properties provides information on the others (Avnimelech *et al.*, 2001; Breitzke, 2006). Bulk density is, therefore, one of the most important properties describing the physical state of marine sediments, as it enables the characterization of the lithology of sediments, inferring their depositional history and establishing correlations, both amongst cores and with other physical properties (Gerland and Villinger, 1995).

Traditionally, wet bulk density was determined by measuring the weight and volume of small sediment samples, usually obtained with syringes from the central part of split sediment cores. Currently, the most widely used non-destructive technique for wet bulk density and porosity is the measurement of the attenuation of gamma rays (GRA) after going radially through the sediment core (Blum, 1997; Breitzke, 2006). MSCL systems use a  $^{137}\text{Ce}$  source and a NaI (TI) scintillation detector, in conjunction with a universal counter, for measuring the GRA on whole or split cores. This measure relates to the GRA coefficient

that is a function of the detected gamma-ray energy, which in turn depends on every individual device and on the characteristics of the sediments themselves. Due to this reason, this procedure requires a good calibration of the GRA using a standard of known density in distilled water. Such a calibration is normally done with a telescoping aluminium rod of varying diameters mounted inside a section of the same core liner holding the sediments to be measured, filled with distilled water (Best and Gunn, 1999; Rothwell and Rack, 2006). In this standard two-phase system model the aluminium rod, with an attenuation coefficient similar to most common minerals, represents the mineral phase standard, whilst pure water is used as the interstitial water phase standard. This calibration allows the establishment of a linear regression equation between the GRA and the density from where calibration coefficients are extracted thus allowing the determination of density variations in the sediments (Blum, 1997).

### ***P-wave velocity***

The measurements of P-wave velocity transmission in sediments are normally used in combination with density values to calculate acoustic impedance (i.e. the product of density and P-wave velocity), and

reflection coefficients, which are needed to determine the depth of reflectors observed in seismic reflection profiles and to construct synthetic seismic profiles. Variations in P-wave velocity within a sedimentary sequence are caused by changes in lithology, porosity, bulk density, lithostatic pressure, degrees of fracturing, consolidation and lithification, and the occurrence and abundance of free gas and gas hydrate (Blum, 1997; St-Onge *et al.*, 2007). The combination of P-wave velocity logs with density records in sedimentary sequences is a widely used and very useful tool to correlate the reflectors in seismic reflection profiles with changes in the physical properties in the sediment cores, thus allowing researchers to establish the true depth of seismostratigraphic units (e.g., Reeder *et al.*, 1998).

Geotek MSCL systems include a sensor for continuously measuring P-wave velocity in sediment cores. The sensor consists of a pair of transducers acting as transmitter and receiver. Original systems with two "piston" type transducers were static and required the addition of a fluid, commonly water, between the transducers and the core liner. At present, MSCLs use Acoustic Rolling Contact (ACR) transducers with a stationary active transducer element made of a material that combines high coupling with relatively low acoustic impedance. ACR transducers have several advantages compared to the old ones: 1) direct coupling to the core liner without the necessity of adding any coupling fluid; 2) a large contact area that, together with the lower frequency used, improves the signal to noise ratio, enabling accurate velocity measurement even in difficult materials such as coarse sand, and 3) wider bandwidth and pulse characteristics that, together with consistent coupling, enable meaningful sediment characterization from spectral analysis.

### **Magnetic susceptibility**

The magnetic susceptibility is the degree to which a material can be magnetized per unit volume in response to an applied external magnetic field. Since both magnetization per unit volume and magnetic field are measured in  $\text{Am}^{-1}$ , magnetic susceptibility ( $\kappa$ ) is dimensionless, and is strictly called "volume susceptibility". To obtain the "mass susceptibility" it is necessary to consider the sediment density, which can be measured simultaneously in most MSCLs (Blum, 1997; Evans and Heller, 2003). The dimensions of mass susceptibility are, therefore,  $\text{m}^3\text{kg}^{-1}$ . Magnetic susceptibility is commonly measured at very low fields, usually not exceeding 0.5 mT, and for this reason it is often referred as low-field susceptibility.

Magnetic susceptibility provides a first order estimate of ferromagnetic mineral abundance in sediments (e.g., magnetite and other members of the iron-titanium solid solution series), but is also sensitive to grain-size variations (St-Onge *et al.*, 2007). As these minerals are common in nearly all rocks and their weathering products and magnetic susceptibility can be regarded as a proxy for minerogenic input to the sediment (Zolitschka *et al.*, 2001). This allows magnetic susceptibility in marine or lacustrine sequences to be used as an indicator of temporal variations in the concentration and size of terrigenous particles accumulated in the basin. Whilst magnetic susceptibility has been widely applied to core correlation on the basis of lithology and to the identification of missing or disturbed core sections, it holds great potential for investigating physical processes and studying the provenance of terrigenous particles entering a sedimentary environment, such as ice rafted debris (IRD), turbidites, aeolian dust or tephra particles (Verosub and Roberts, 1995; Reeder *et al.*, 1998; Hounslow and Maher, 1999; Nowaczyk, 2001; de Fontaine *et al.*, 2007; Larrasoana *et al.*, 2008; Sáez *et al.*, 2009).

The Geotek MSCLs allow the measurement of magnetic susceptibility on whole cores thanks to a Bartington loop sensor (MS2C), which is available at different diameters adjustable to the core diameter. One disadvantage of using a magnetic susceptibility loop on whole cores is the rather large response function, resulting in several centimetres of smoothing depending on the loop and core diameters (St-Onge *et al.*, 2007). To achieve the maximum resolution in magnetic susceptibility measurements the loop-diameter/core-diameter ratio should be as small as possible when using this method. To increase the resolution a point-source sensor on split cores is recommended. Measurements on split cores are normally made by using a Bartington point sensor (MS2E) mounted on an arm of the MSCL, which allows the sensor to be placed on the core surface for every measurement (Fig. 2B). The MS2E sensor uses the same electronics as the loop sensor and gives much higher spatial resolution, although it is less sensitive. The sensitive area (i.e., the area receiving 50% maximum response) in the MS2E probe consists of a rectangle 3.8 mm x 10.5 mm. Another option to increase resolution is performing continuous measurements on U-channels (normally 2x2 cm, 1.5 m long plastic liners holding the sediment) by means of a smaller diameter loop coupled with a cryogenic magnetometer or by using a separate track with a kappa bridge (St-Onge *et al.*, 2007).



## 2D and 3D digital radiography

### 2D digital radiographs

The use of X-rays to obtain radiographs is based on the differential transmission of X-rays in the sediment due to attenuation by absorption and scattering caused by the particles and other substances forming the sediment (Bouma, 1969; Principato, 2004). The attenuation of the X-ray beam depends mainly on the bulk density of the sediment (Axelsson, 1983; Holyer *et al.*, 1996), which itself is affected by grain-size and lithology, including carbonate and silica contents. In addition, bulk density can also be affected by changes in water content, compaction and porosity (St-Onge *et al.*, 2007). Higher sediment density leads to stronger X-ray absorption, which translates into the grey scale intensity of radiographs, which is proportional to X-ray attenuation. Therefore, the spatial distribution of the grey scale in the X-ray radiograph of a sediment core is considered to reflect the spatial structure of sediment density (Holyer *et al.*, 1996). However, variations in the grey levels of a sediment radiograph should be properly calibrated or interpreted together with complementary records such as grain-size or mineralogy (St-Onge *et al.*, 2007). Since absorption of X-rays is also dependent upon the wavelength of the radiation and the density and thickness of the sample, inhomogeneities in the sediment cores, such as thickness variations, and will result in variable degrees of darkening of the X-ray radiograph (Axelsson, 1983).

Traditional radiographs obtained by using a chemical film, and eventually converted to digital images, have been applied to geosciences for many years as a non-destructive technique for investigating sedimentary rocks, and paleontological specimens, amongst many others aspects (Ojala, 2004; Principato, 2004 and references therein). Nowadays, different systems allow researchers to obtain digital radiographs of sediment cores or slabs, both onboard and onshore, which are subsequently used directly for high-resolution inspection of the sedimentary sequences. These radiographs often allow the identification of sedimentary structures that are unrecognizable to the naked eye (St-Onge *et al.*, 2007).

For many years radiographs of sediment cores were taken with medical and industrial systems without any particular adaptation to X-radiograph geological materials. In these cases the sediment sample had to be manipulated and prepared for the systems. It usually consisted of performing 37 cm long by 1 cm thick sediment slabs on methacrylate holders. This scenario has changed in the last decades so that at

present there are several systems designed to obtain high quality X-ray radiographs of sediment cores. For instance, the EPOC laboratory of the Université Bordeaux 1 has developed a digital X-ray imaging system named SCOPIX, which combines conventional X-ray equipment with a radiology instrument developed by the Cegelec company (Migeon *et al.*, 1999; Lofi and Weber, 2001) (Fig. 3). The SCOPIX system consists of a lead box holding the X-ray source and a platform with two motorized lateral sleeves to move the sediment samples. It allows the X-radiographing of whole and split cores and U-channels, although more commonly the X-radiograph is performed after placing 1 cm thick sediment slabs on 7 cm wide by 1.5 m long aluminium holders. A uniform slab thickness prevents variations in the attenuation due to changes in sediment thickness. The X-ray spectrum is recorded by a 4096 grey level Hamamatsu ORCA camera (1,280 x 1,024 pixels) giving a resolution of 125  $\mu\text{m}$ . Devoted acquisition software converts the images into grey level values while allowing their reconstruction. Image processing is done with another software that includes several numerical filtering and extracting options (Migeon *et al.*, 1999; Lofi and Weber, 2001).

Commercially, the two most extended X-ray radiography systems are the Geotek MSCL-XCT and the one in the ITRAX XRF core scanner from COX Analytical Systems. The MSCL-XCT includes an X-ray core imaging device that performs linear digital X-ray images on whole, split or slabbed core sections with a typical resolution of 120  $\mu\text{m}$  on core sections up to



**Figure 3.** Digital X-ray imaging system SCOPIX in the EPOC laboratory of the Université de Bourdeaux 1 (source: <http://www.epoc.u-bordeaux.fr>).

**Figura 3.** Sistema de radiografías digital SCOPIX del laboratorio EPOC en la Universidad de Burdeos 1 (fuente: <http://www.epoc.u-bordeaux.fr>).



155 cm in length and 15 cm in diameter. The ITRAX XRF core scanner can mount a charge-coupled (CCD) line camera located below the core section being scanned that allows digital continuous-strip X-radiograph 20 mm in width and variable lateral resolution down to 20x20  $\mu\text{m}$  to be obtained. One additional advantage of these two commercial systems is that both allow previewing X-radiograph information together with the other parameters being measured at every operation, which is often very helpful for the interpretation of the results.

### **Computerized axial tomography**

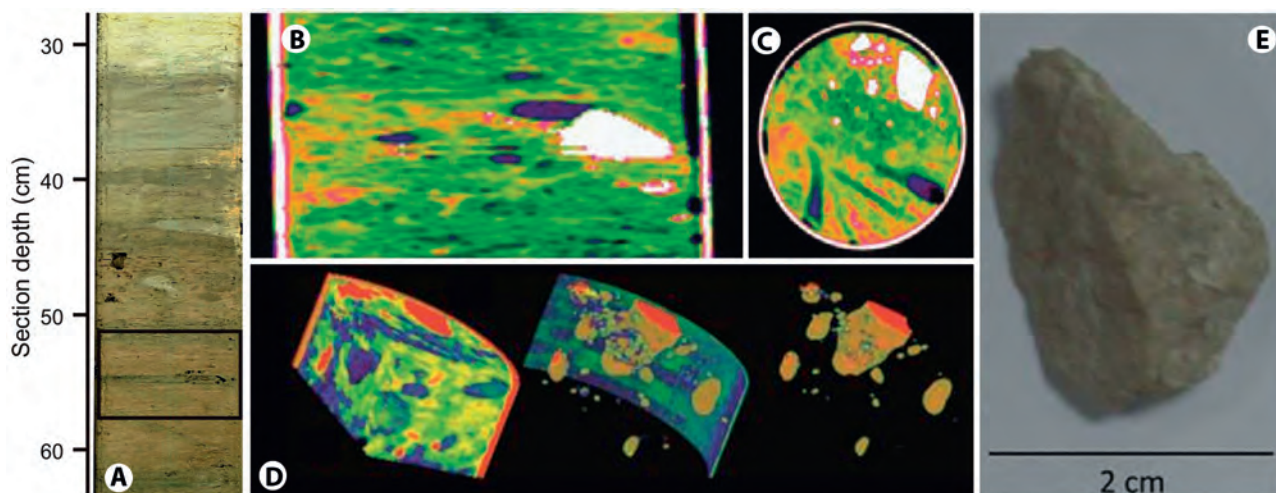
Computerized axial tomography (CAT-scan) is a non-destructive technique that allows the 3D visualization and analysis of the internal structure of objects. In sediment cores, the CAT-scan is normally applied to whole cores and sometimes to discrete samples too, for which it provides detailed information at very high-resolution ( $\sim 0.1\text{-}1\text{ mm}$ ). This technique is based on the quantification and mapping of the X-ray attenuation by the analysed object as recorded in longitudinal (topogram) or transverse (tomogram) images. The images are scaled in grey levels where darker and lighter zones represent lower and higher X-ray attenuation, respectively. Following the same principle as X-ray radiographs, the grey scale of CAT-scan images indicates changes in the density of the analysed object. Grey values are expressed as Hounsfield units or CT numbers, which result from comparison of the attenuation coefficient to the one of water. The CT number is a complex unit that relates to sediment bulk density, mineralogy and porosity (St-Onge *et al.*, 2007). While X-ray radiographs project the object in a 2D detection plane, which implies losing information in the depth direction, the acquisition of images from different directions and planes with a CAT-scan allows 3D volumetric reconstructions by means of dedicated algorithms (Cnudde and Boone, 2013).

The ability to image the internal structure of any kind of object at sub-millimetre scale without altering it has revolutionized the study of the physical properties of materials in most fields, including geosciences. Amongst many other applications, this technique has been used to investigate sequence stratigraphy, sedimentary and biogenic structures, sediment composition and fabrics, and fossil and fluid contents (Mees *et al.*, 2003; St-Onge *et al.*, 2007; Mena *et al.*, 2012; Cnudde and Boone, 2013 and references therein) (Fig. 4).

For many years medical CT systems have been used for geoscientific purposes, including the study

of sediment cores. Some advantages of medical CTs are their suitability to scan long core sections and their availability, as many hospitals have these instruments. Whilst technological progress has led to the development of new, better performing and faster CT scanners, their spatial resolution is limited to several hundreds of micrometres (typically around 200-300 $\mu\text{m}$  voxel size), as they are conceived for the size and behaviour of the human body (Cnudde and Boone, 2013). In recent years, high-resolution X-ray tomography, commonly called micro-CT, is becoming more popular since it yields resolutions much better than 50  $\mu\text{m}$  and it is more cost-efficient than other methods, such as those based on synchrotron radiation, which are clearly superior in terms of achievable spatial resolution but also very expensive in terms of operational costs. Contrary to medical CTs, in micro-CT systems the object under investigation is the element that shifts position (normally by rotation) to ease the multidirectional cuts, whereas the X-ray source and detector remain stationary. This results in mechanical stability and allows true resolutions below 10  $\mu\text{m}$  to be obtained.

In the setup of a standard micro-CT, the object is located at any position between the source and the detector, which allows geometrical magnification and, therefore, the highest achievable resolution that is mainly limited by the focal spot size of the X-ray source (Cnudde and Boone, 2013). However, a major limitation of micro-CT systems is the sample size, usually not more than some tens of centimetres, making them useless for long sediment cores. A new CT-scan system using the technology of micro-CTs also allowing the 3D imaging of long sediment cores is at present under development and was installed at the CORELAB laboratory of the University of Barcelona in 2014. The new system is called the "Multitom Core X-ray CT System", as it will allow multi-resolution tomographic scanning. The new Multitom is both a micro-CT, since it will provide a resolution better than 5  $\mu\text{m}$ , but it can also behave as a standard CT allowing the analysis of objects at lower resolutions (i.e. down to 300  $\mu\text{m}$ ). To sum up, it combines the advantages of large systems, as it can scan objects with lengths greater than 150 cm, with the precision of the best micro-CT systems for small objects, whilst releasing optimum quality imagery for every object. The Multitom will also allow quantitative information from the X-ray CT-scan data to be obtained by means of 3D volume analysis using a dedicated software package. All the above characteristics will give the Multitom a strong, unprecedented potential for the study of a wide range of samples of different nature and very different objectives.



**Figure 4.** CAT-scan analysis on a marine sediment core from the NW Iberian margin used for the identification of ice rafted debris (IRD) (Mena *et al.*, 2011 and 2015). A) Digital and colour photography of a 30 cm fragment of sediment core FSG09-16. The black square marks the area shown in the CAT images B, C and D; B) CAT 2D image of the selected area; C) CAT 2D image of an axial section within the selected area; D) CAT 3D image of the selected area (left) and products by removing information with different density thresholds (centre and right). Note that changes in the colour of the CAT-scan images b, c and d show changes in the material density, where lighter colors represent denser materials. Thus, the lightest areas allowed the identification of IRD accumulation in the selected area; and E) a digital image of an IRD found within the selected area.

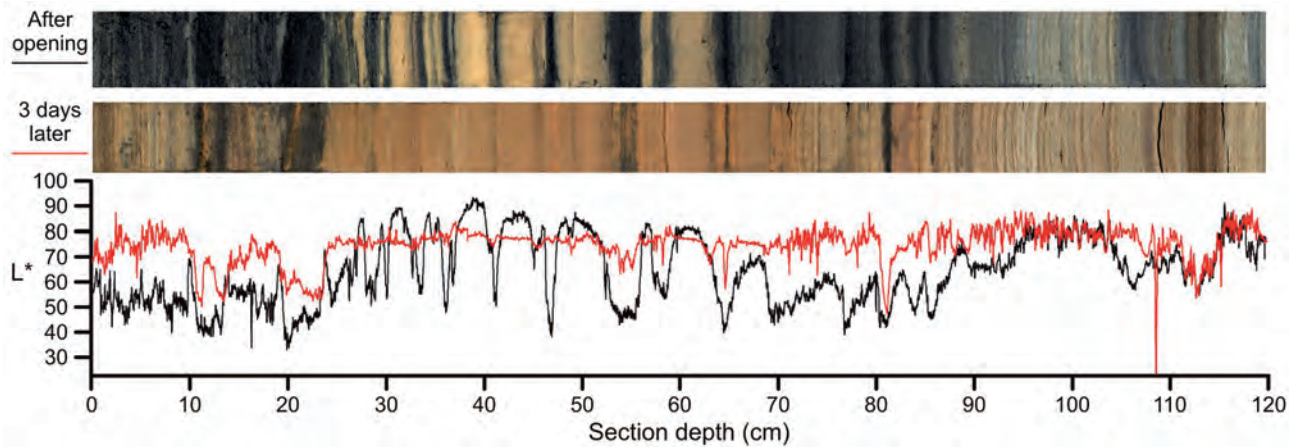
**Figura 4.** Análisis de TAC en un testigo de sedimento del margen NW de la Península Ibérica usado para la identificación de detritos procedentes de la fusión de icebergs a la deriva (IRD, del inglés Ice Rafted Debris) (Mena *et al.*, 2011 y 2015). A) Fotografía digital a color de un fragmento de 30 cm del testigo de sedimento FSG09-16. El recuadro negro señala el área representada en las imágenes de TAC en B, C y D; B) imagen de TAC en 2D del área seleccionada; C) Imagen de TAC en 2D de la sección axial en el área seleccionada; D) imagen de TAC en 3D del área seleccionada (izquierda) y productos obtenidos tras la eliminación de información densitométrica mediante la aplicación de diferentes umbrales de densidad (centro y derecha). Notar que los cambios en los colores de las imágenes de TAC en b, c y d corresponden con cambios en la densidad del material, siendo los colores más claros representativos de materiales más densos. En consecuencia, las áreas más claras permiten la identificación de la acumulación de IRD en el área seleccionada; y E) imagen digital de un IRD encontrado dentro del área seleccionada.

### Digital and colour line-scan cameras

Once a core section is cut and split in two halves the sediment is in contact with air and can oxidize rapidly, changing its colour and rendering the identification of original features difficult. It is, therefore, crucial to acquire digital photographs of the cores immediately after splitting, which will serve as a reference of the original appearance of the sediment. Furthermore, these photographs can provide complementary quantitative information about the sediment colour that cannot be obtained from the traditional Munsell colour charts for sediment description (Fig. 5). After some years the most common platforms for continuous, non-destructive sediment cores analysis, such as the Geotek MSCL and the AVAATECH and ITRAX XRF core scanners (see later on in the text), mount high-resolution digital colour line-scan cameras (Fig. 6).

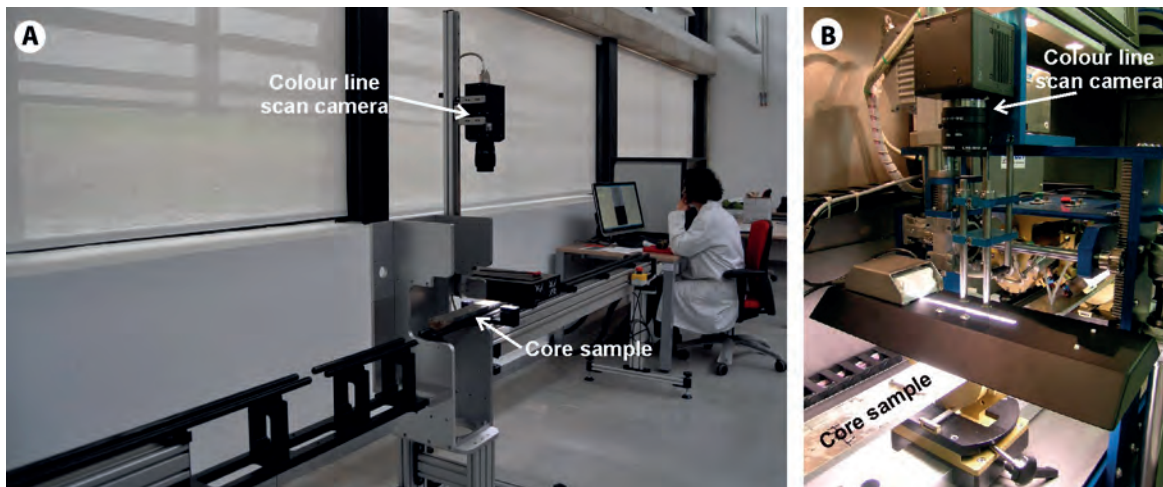
Line-scan cameras allow a rapid acquisition of ready to use images (TIFF, JPG and other formats) at very high resolution without some typical problems that occur when taking photographs with still area cameras, such as uneven lighting, spherical distor-

tion, montage or “stitching” effects (Nederbragt and Thurow, 2004). All line-scan cameras have similar technical characteristics based on 3 Charge Coupled Device (CCD) sensors, one for each RGB colour (red, green, blue), with 2048 pixels each. However, some differences may exist in: 1) the way photos are taken (in Geotek and ITRAX systems the core moves towards the apparatus, whilst in AVAATECH the camera moves towards the core); 2) the maximum achievable resolution (photos with the Geotek MSCL can be collected at resolutions of 100, 50 or 25  $\mu\text{m}$  pixel size, whilst ITRAX allows photos at 200  $\mu\text{m}$  or 50  $\mu\text{m}$  pixel size and AVAATECH at 70  $\mu\text{m}$  to be obtained); and 3) the field of view of the images (10 cm for Geotek MSCL and ITRAX, and 14 cm for AVAATECH). All the systems include a processing software to extract quantitative information of RGB and/or CIE-L\*-a\*-b\* colour data of the sediment at the same resolution as the photographs (i.e., greater than 100  $\mu\text{m}$ ). The AVAATECH system can also provide UV luminescence images of corals, speleothems, shells and carbonates in general, and also of other materials, by using a 380nm UV source (Grove *et al.*, 2010).



**Figure 5.** Digital images and colour data information of a varved sediment core from Montcortés Lake, Lérida (Spain), obtained with the colour line-scan camera of the AVAATECH XRF core scanner at two different moments: immediately after opening, and 3 days later (Corella *et al.*, 2014). Oxidation of the organic matter by contact with the air produced a significant change in the appearance of sediments. c) Colour data, here expressed as reflectivity ( $L^*$ ), allows a quantitative identification of colour changes in the sediment at 70  $\mu\text{m}$  resolution. Differences in reflectivity of both records denote the great impact that oxidation has on the sediment.

**Figura 5.** Imágenes digitales y datos de color de un testigo de sedimento varvado del Lago Montcortés, Lérida (España), obtenido con la cámara de barrido lineal de color integrada en el sistema de XRF de AVAATECH en dos momentos diferentes: inmediatamente después de la apertura del testigo, y 3 días después (Corella *et al.*, 2014). La oxidación de la materia orgánica por el contacto con el aire produjo un profundo cambio en la apariencia de los sedimentos. c) Los datos de color, expresados aquí mediante la reflectividad ( $L^*$ ), permiten la identificación cuantitativa de los cambios de color en el sedimento a 70  $\mu\text{m}$  de resolución. Las diferencias en la reflectividad de ambos registros muestran el gran impacto que la oxidación produce en el sedimento.



**Figure 6.** Colour line-scan camera from: A) the MSCL system of Geotek installed in the Instituto Pirenaico de Ecología (IPE) of the Spanish National Research Council (CSIC), Jaca (<http://www.ipe.csic.es/>); and B) the AVAATECH XRF core scanner system in the CORELAB laboratory of the University of Barcelona (<http://www.ub.edu/depqm/>).

**Figura 6.** Sistema de fotografía digital en continuo de: A) equipo MSCL de Geotek instalado en el Instituto Pirenaico de Ecología (IPE) del Consejo Superior de Investigaciones Científicas (CSIC), Jaca (<http://www.ipe.csic.es/>); y B) escáner de FRX de AVAATECH instalado en el laboratorio CORELAB de la Universidad de Barcelona (<http://www.ub.edu/depqm/>).

Changes in sediment colour have been considered for years as indicative of composition and preservation state. Certainly, colour variations are reliable indicators of shifts in the content of carbonate, free and bound iron, Fe minerals and clays, as demonstrated recently (Mix *et al.*, 1992; Nederbragt and Thurow, 2004; Rogerson *et al.*, 2006; Debret *et al.*,

2011). Therefore, sediment colour data is a very useful tool for sedimentological studies at millimetre and sub-millimetre scales, thanks to the high-resolution given by modern digital colour line-scan cameras. This is well illustrated by the reconstruction of paleoclimatic records from laminated and varved deposits, the cross-correlation of overlapping cores, wiggle-



matching amongst paleoclimatic records, the establishment of chronostratigraphic markers, the analysis of the cyclicity of lithological changes, the estimation of the abundance of specific compounds, or the study of coral grow bands and speleothem laminas ([Balsam \*et al.\*, 1999](#); [Nederbragt and Thurow, 2004](#); [Nederbragt \*et al.\*, 2006](#); [Froelich \*et al.\*, 2007](#); [Grove \*et al.\*, 2010](#); [de Jong \*et al.\*, 2013](#); [Hodell \*et al.\*, 2013a](#); [Vanni re \*et al.\*, 2013](#)).

In addition to reflectance spectroscopy in the visible range, imaging of sediment cores with hyperspectral systems in the VIS and near IR range (400-1000 nm) has a great potential for the study of changes in the sediment composition, e.g., photopigments and clay minerals, at very high spectral and spatial resolutions. A dedicated device, the Specim Ltd. Scanner, has been recently developed in the Paleolimnology Laboratory of University of Bern (Switzerland), which consists of a hyperspectral camera and a sample tray that moves under the illumination unit and the camera. This system allows reflectance spectra from the sediment surface in the range 400-1000 nm to be obtained with a spectral resolution of 0.8 nm and a spatial resolution (pixel size) of 38  $\mu\text{m}$ . The first results obtained with these cameras are still being evaluated, but they present great potential in the identification and quantification of specific substances and minerals within the sediments by attributing specific spectral properties, and therefore in the calibration between proxies ([Grosjean \*et al.\*, 2014](#)).

### X-ray fluorescence core scanners

X-ray fluorescence (XRF) core scanners allow rapid, non-destructive, continuous and high-resolution determination of the elemental chemical composition of the sediments on split cores ([Jansen \*et al.\*, 1998](#); [Rothwell and Rack, 2006](#)). They have been applied successfully to a wide range of lacustrine and marine sediments from tropical to polar environments, and also to soils, peats, speleothems and other materials ([L wemark \*et al.\*, 2011](#)).

The technique is based on the excitation of the chemical elements in the sediment with an X-ray source causing the ejection of the electrons from the inner shell of the atoms. An electron falling back from an outer shell subsequently fills the vacancy thus generated which results in an energy surplus that is emitted as an electromagnetic radiation (i.e., the energy difference between the vacant inner and the outer shells). The wavelength(s) of the radiation emitted is characteristic of each element, and the peak

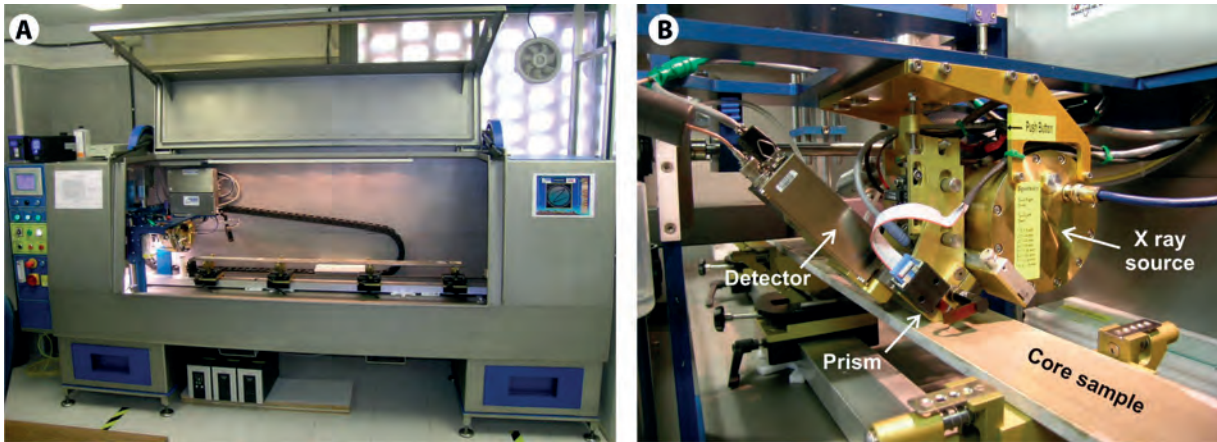
amplitudes in the XRF spectrum are proportional to the concentration of every given element in the sediment being analysed ([Richter \*et al.\*, 2006](#)).

At present, there are two main commercially available XRF core scanner systems in Europe, which are the ITRAX core scanner from Cox Analytical Systems and the AVAATECH core scanner manufactured by a spin-off of the Royal Netherlands Institute for Sea Research (NIOZ) ([Jansen \*et al.\*, 1998](#)), recently purchased by the Dutch corporation Doeschot BV (Fig. 7). Both systems have similar analytical capabilities since they are able to determine the elemental composition of sediments for elements ranging from Al to U at resolutions from 1 cm to 100  $\mu\text{m}$  on split sediment cores up to 1.8 m long.

However, the two systems present some noticeable differences in design. One first difference is the movable element, either the sediment core or the source-detector unit. The ITRAX XRF core scanner consists of a fixed central measuring tower and two projections on either side of the tower. The central tower includes an X-ray focusing unit and a range of sensors: a colour line scan camera, the X-ray detector, a laser topographic scanner, and an X-ray line camera, whilst the side projections are part of the motorized split-core transport bed ([Croudace \*et al.\*, 2006](#)). This design allows cores and other samples to be moved through the central measuring tower where the measurements are carried out, thus reducing the radiation protection to this area but increasing the length of the whole system. By contrast, in the AVAATECH XRF core scanner the X-ray source-detector unit moves over the core, which is kept in a fixed position over a platform. Due to these design differences, the AVAATECH XRF core scanner is heavier than the ITRAX one, as the entire system is protected for X-ray radiation. Up to a point this has been minimised by reducing the overall size of the instrument. Nonetheless, these differences in the design of both systems neither influence their measuring capabilities nor their installation on land-based or onboard laboratories.

A more relevant difference is the way they carry out the XRF measurements, which influences detection limits and the overall quality of the data. Each company implemented different solutions to avoid or reduce the problem of X-ray fluorescence attenuation by air absorption, which is particularly critical for the lightest elements, mainly Al and Si. With the ITRAX XRF core scanner measurements are carried out in normal atmospheric conditions, it is in the presence of air, while keeping a constant distance between the detector and the sediment based on a previous control laser topographic scanner of the core surface. Since air should absorb part of the fluorescence emit-





**Figure 7.** A) AVAATECH XRF core-scanner system installed in the CORELAB laboratory of the University of Barcelona (<http://www.ub.edu/depqm/>). B) A detail of the main measurement structure showing the x-ray source, the detector, the prism flushed with He and the location of the core sample.

**Figura 7.** A) Escáner de FRX de AVAATECH instalado en el laboratorio CORELAB de la Universidad de Barcelona (<http://www.ub.edu/depqm/>). B) Detalle de las principales partes de medida mostrando la fuente de rayos X, el detector, el prisma aireado con He y la localización de la muestra.

ted by the sediment, a high-power X-ray generator (3000W) is used to significantly increase the fluorescence signal. Consequently, a strong fluorescence signal in counts per seconds is obtained for major elements with a relatively high atomic mass (Z), especially those above Si. However, the sensitivity for lighter elements, especially Al, can be critically low, depending on the sediment type (Croudace *et al.*, 2006). This is probably due to the fact that weaker fluorescence energies are more susceptible to scatter and absorption effects (Potts, 1987). An advantage of the ITRAX XRF core scanner is that it can perform XRF analysis on sediment cores with irregular surface, though the quality of the results improves significantly on flat smooth surfaces.

The AVAATECH XRF core scanner performs the XRF measurements in contact with the surface of the sediment by using a prism flushed with helium to remove the air in the measuring area, thus avoiding the problem of radiation absorption by air (Richter *et al.*, 2006). This allows the AVAATECH XRF core scanner to do the analyses with a low power X-ray source (100W) that is easily cooled with a simple water-cooling unit. The main advantage of the AVAATECH system is that once the air is removed from the measuring area the transmission of the radiation emitted by the sediment elements has no obstacles, which leads to a very good detection of all the elements with an atomic weight between Al and U, including the lightest ones (Richter *et al.*, 2006). One disadvantage of this system is that it needs flat, smooth surfaces for the precise landing and coupling of the prism to the sediment surface, which prevents measurements

over irregular sediment surfaces. In this case, the presence of air bubbles between the sediment and the special plastic foil used for XRF analysis should be removed to avoid radiation absorption by the air.

Variations in the elemental composition of the sediment provide key information about changes in the source area, and on the transport and accumulation processes and mechanisms, which generally relate to environmental and climatic changes, also including potential anthropogenic influences on sedimentary environments (Löwemark *et al.*, 2011). XRF elemental records are thus widely used as proxies of environmental conditions controlling the development of the sedimentary record and its lithogenic, biogenic and diagenetic phases (e.g., Rothwell and Rack, 2006; Calvert and Pedersen, 2007). However, some care has to be taken when dealing with chemical element records from XRF core scanners taken on undisturbed "fresh" sediment sections. A first word of caution refers to the fact that elemental variations from XRF core scanners cannot be considered quantitative since they depend on the excitation conditions used in the measurements and they are expressed as counts per second, rather than concentrations. Calibrations with quantitative analytical data of the bulk sediment chemistry with other techniques might help to overcome this problem (Löwemark *et al.*, 2011). Another relevant issue is surface roughness and grain-size variations influencing the measurement by XRF core scanners, eventually producing significant decreases in the total counts due to increases in porosity and air absorption. The sediment might also incorporate phases that are not measurable by

the XRF core scanners, such as water and organic matter, which will affect the elemental chemistry signal leading to the well-known closed-sum effect (Calvert, 1983; Rollinson, 1993; Tjallingii *et al.*, 2007).

Since one of the strengths of the XRF-scanning method lies in the relative variations of the elements (Croudace *et al.*, 2006), it is highly convenient to normalize the records resulting from measurements so that an environmentally meaningful signal is obtained. Otherwise, interpretations may prove wrong because of the mirroring effect of records with respect to specific components of the sediment, mainly organic matter and carbonates (Löwemark *et al.*, 2011). A common practice to solve this issue is the normalization of individual elemental counts to the Al record, thus taking advantage of the conservative behaviour of this element (Calvert and Pedersen, 1992; Rollinson, 1993; Piper and Perkins, 2004; Calvert and Pedersen, 2007). However, if not applied with extreme care, this practice could lead to biased results due to spurious correlations amongst uncorrelated variables or to seeming correlations between normalized element contents as related to the closure effect (Van der Weijden, 2002).

Normalization is in any case required to obtain useful qualitative information on element enrichments and depletions and in correcting the elemental records for dilution effects. In some cases, elemental/Ti normalization is also applied by assuming that Ti is a lithogenic proxy. However, the use of Ti for normalization is somewhat problematic as its content varies to a large extent in different rock types compared to Al (Calvert and Pedersen, 2007). Other ratios are also commonly used to compare elemental records with the aim of differentiating the dominance of opposing processes or situations. This is the case of the Ca/Fe and Ca/Ti ratios to discriminate glacial/interglacial cycles, such as pelagic sediments deposited during interglacial periods having higher carbonate contents than those deposited during glacial periods (Rothwell and Rack, 2006; Hodell *et al.*, 2013a), or as indicative of endogenic calcite precipitation in lakes (Francus *et al.*, 2013). Another example is the use of the Zr/Rb ratio for tracking changes in sediment grain-size, as on average Zr usually comes with coarser particles than Rb does (Calvert and Pedersen, 2007; Kylander *et al.*, 2011). The K/Ti ratio has been explored to trace the relative importance of acidic vs. basaltic source rocks for the terrigenous fraction of sediments, whilst the Sr/Ca ratio has been applied to discriminate aragonite vs. calcite content (Richter *et al.*, 2006 and references therein). However, the use of element ratios and their meaning still are under debate. Establishing log-ratios of element intensities

is a recommended practice to easily interpret the signals of relative changes in chemical composition throughout the sediment core. This is due to their value to provide accurate and precise predictions of sediment composition after XRF core-scanner output data (Weltje and Tjallingii, 2008).

In Spain, two XRF core scanners are available at present, an AVAATECH system installed in the CORE-LAB of the University of Barcelona and an ITRAX system in the University of Vigo. In addition, the IGME will shortly acquire a XRF-core scanner to be mounted on the MSCL-S.

## Magnetometry

The study of the magnetic properties of sediments provides valuable information that is often combined with data obtained with the above-described methods. This is why we include here a brief description of magnetic properties and the techniques used to measure them in sediments. More detailed information can be obtained from Evans and Heller (2003) and Stoner and St-Onge (2007).

Magnetic minerals, particularly iron oxides, occur more or less universally, with iron being one of the most common elements in the Earth's crust (Evans and Heller, 2003). Initially, the analysis of the magnetic properties of the sediments focused on the study of the phases of the magnetic field in the past by taking advantage of the fact that magnetization records are indicative of the condition of the geomagnetic field and its variations through time (Stoner and St-Onge, 2007). This led to the development of paleomagnetism and paleomagnetic stratigraphy. Magnetic reversals are globally synchronous at geological timescales and, in addition, they are environmentally independent events that can be recorded both in deposited and thermally cooled materials. The dating of reversals and anomalies, and the inter-calibration of the reversal record with other chronostratigraphic evidence deserved much attention as a consequence of the interest to understand time in the geologic past and to establish a global geological time scale. The pioneer work by Shackleton and Opdyke (1973) is the first example of modern marine stratigraphy, as it includes an inter calibration of magnetic reversals and oxygen isotopes. The study of magnetic properties in marine sediments can also provide information on source areas and transport processes, as is the case for desert dust and loess, and on the grain-size of magnetic minerals, amongst others (Thouveny *et al.*, 2000; Moreno *et al.*, 2002; Evans and Heller, 2003; Larrasoana *et al.*, 2003; Stoner and St-Onge, 2007; Larrasoana *et al.*, 2008; Channell *et al.*, 2013).

In the simplest case, the natural remanent magnetization (NRM) of the sediment is aligned with the geomagnetic field and is a function of its intensity and direction at the time of deposition. However, many factors can modify or totally remove the original geomagnetic signal. When this occurs, only in some cases can the original signal be recovered by isolating or avoiding some of these factors and their effects. The most critical requirement to retrieve a reliable paleomagnetic record is that the primary vector magnetization locked in the sediment is preserved in its original orientation, both during core extraction from the seafloor and throughout laboratory analyses (Stoner and St-Onge, 2007).

When sub-sampling is needed to measure the magnetic properties of sediment, a U-channel is normally taken from the central part of the core. Once in the U-channel, the sediment is kept intact during and after the measurements, so that it can be stored as permanent archive or used for further non-destructive, or destructive analyses. Technological innovation is also being applied to the development of more capable magnetometers, such as superconducting rock magnetometers (SRM) that combine high sensitivity and speed with flexibility. The large throughput of most SMRs makes them particularly suitable for the analysis of long sediment cores and U-channels (Stoner and St-Onge, 2007). This allows the measurement of several types of magnetization including an hysteric remanent magnetization (ARM), isothermal remanent magnetization (IRM), and volumetric magnetic susceptibility.

Statistical techniques are commonly applied to the analysis of the huge datasets produced by the above-described methods. For instance, the Principal Component Analysis (PCA) technique has been applied to the statistical analysis of large XRF core scanner datasets and other variables. PCA proved useful for the statistics-supported interpretation of elemental composition records by grouping elements according to their behaviour (Giralt *et al.*, 2008; Martín-Puertas *et al.*, 2009; Moreno *et al.*, 2010; Nieto-Moreno *et al.*, 2013). The inclusion of a wide range of variables into PCA analyses normally facilitates a more integrated approach for the investigation of the environmental signatures held in sediment records.

### **Application of non-destructive and continuous analysis techniques on the study of sediment cores from the Iberian continental margin**

Advances in marine coring techniques have allowed the recovery of long sediment sequences from the

seafloor worldwide. Specifically, the Iberian Continental Margin has been the subject of study within national and international projects, providing unique sedimentary and rock core samples for the study of a wide range of geological processes. International collaborative projects, namely the Integrated Ocean Discovery Program (IODP) (previously Ocean Drilling Program, ODP, and initially the Deep Sea Drilling Project, DSDP) and the International Marine Past Global Change Study program (IMAGES), provided a huge amount of sediment sequences around the Iberian Margin, which in most cases were routinely analyzed with non-destructive and continuous techniques for the acquisition of the physical properties. In parallel, nationally funded research projects have also provided a huge amount of sediment sequences during the last two decades, which in addition have also contributed to the acquisition of new equipment and in some cases to the development of new laboratories devoted to non-destructive and continuous analyses. Here, we present some examples of research representative of the effort devoted during the last decades to the study of the Iberian Continental Margin sediments by using some of the non-destructive and continuous techniques described in the previous sections.

### ***The Galician margin***

After the disaster of the sinking of the Prestige tanker and the subsequent oil spill in November 2002, the marine geology community devoted a great deal of effort in the characterization of the Galicia Bank region in order to quantify and monitor the resulting impact (Ercilla and Vilas, 2008). Sediment gravity cores recovered around the Prestige wreck area were analyzed by means of non-destructive techniques in order to characterize the sedimentary environment and processes that have occurred in the spoil area since the late Pleistocene to Holocene. MSCL, XRF core scanner and magnetic properties data allowed defining lithological facies and stratigraphy, and to study the detrital and diagenetic processes that characterize the sediments surrounding the area affected by the oil spill (Alonso *et al.*, 2008; Rey *et al.*, 2008).

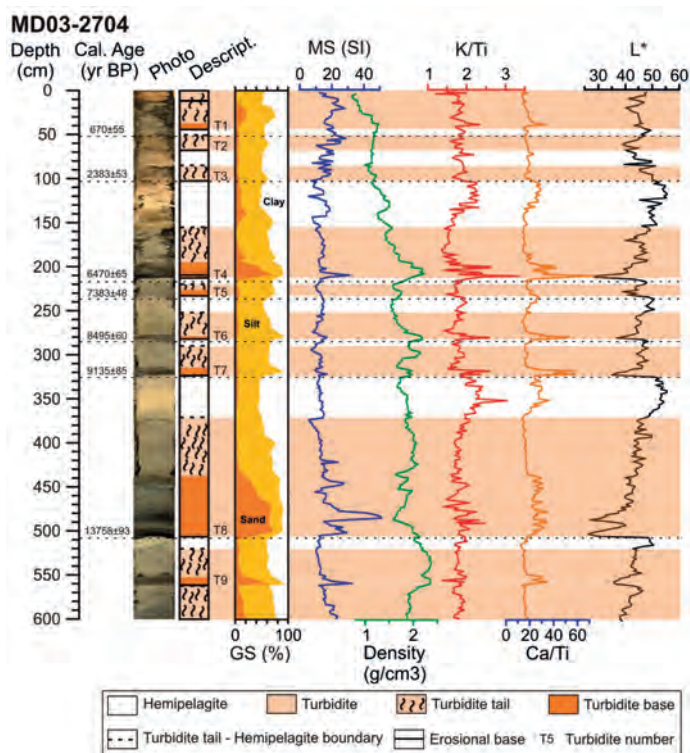
Sediment cores from the Galicia Interior Basin have also been studied by means of computerized Tomography (CT) by Mena *et al.* (2011) for the characterization of sedimentary processes in Upper Pleistocene sediments, resulting especially useful for the identification of ice rafted detritus (IRD) (Mena *et al.*, 2012 and 2015). And Martins *et al.* (2013b) also used magnetic susceptibility, together with other



records, to identify and describe the sources of glacial IRD in the NW Iberian Continental Margin over the last 40 ka. The multi-proxy study carried out by [Bender \*et al.\* \(2012\)](#) on sediment cores from the Galician continental slope integrated XRF and MSCL data to describe the late Quaternary association between slope morphology, sea-level variations and sediment dynamics on the NW Iberian slope. [Andrade \*et al.\* \(2011\)](#) and [Mohamed \*et al.\* \(2010\)](#) used non-destructive methods to study paleoclimatic, sedimentologic and diagenetic processes in the sediments of the Ría de Muros and on the Galician Continental Margin, respectively. Evaluation of the ITRAX XRF data with discrete samples analyzed by means of ICP-MS carried out by [Rodríguez-Germade \*et al.\* \(2013\)](#) using a sediment core from the transitional zone between the Galicia Bank and Galicia Interior Basin suggested the need for a re-evaluation and moving average smothering of the XRF data to improve the quality of the lighter element profiles and the correlation with discrete samples.

### The Portuguese margin

The Portuguese Margin has been the focus of study for multiple disciplines, margin stability and paleoclimate related studies being those that are more likely to have benefited from the use of non-destructive and continuous techniques of analyses. [Lebreiro \*et al.\* \(1997\)](#) studied X-radiographs of sediment-core slabs from the Horseshoe abyssal plain to characterize the texture and distribution of turbidites on the southern Iberian Margin. XRF core-scanner data resulted very useful for the study of the depositional frequency of distal turbidites off the Lisbon–Setúbal canyons within core MD03-2698, bringing new understanding on the response of sediment instability under abrupt glacial climate changes ([Lebreiro \*et al.\*, 2009](#)). The study of turbidite layers through the use of the XRF core scanner and physical properties data allowed [Gracia \*et al.\* \(2010\)](#) to define synchronous turbidite events in separated basins, thus resulting in a turbidite event chronology for the SW Iberian Margin (Fig. 8). Magnetic susceptibility, XRF core-scanner data and X-ray scanning, together with other proxies, served for defining instantaneous deposits associated with river discharge increases that occurred during the Little Ice Age off Lisbon ([Abrantes \*et al.\*, 2005](#)) and to identify “instantaneous deposits” associated with the 1969 and 1755 earthquake-caused tsunamis ([Abrantes \*et al.\*, 2008](#)). Likewise, variations of magnetic parameters measured in U-channels from several cores of the Portuguese Margin were interpreted in terms of



**Figure 8.** Continuous and non-destructive geophysical and geochemical data of core MD03-2704 compared with other destructive data used for the identification and characterization of turbidite layers (T) in the Horseshoe Abyssal Plain. From left to right: image, lithological description, grain-size distribution (GS: clay, silt and sand percentages), Geotek MSCL magnetic susceptibility (MS) and density (blue and green, respectively), AVAATECH XRF core-scanner geochemical composition expressed by K/Ti (red) and Ca/Ti (orange) ratios, and lightness (L\*, black) (modified from Gracia *et al.*, 2010).

**Figura 8.** Datos geofísicos y geoquímicos obtenidos con técnicas analíticas no destructivas y en continuo del testigo MD03-2704, comparados con otros datos de origen destructivo, usados para la identificación y caracterización de capas turbidíticas en la Llanura Abisal de Horseshoe. De izquierda a derecha: imagen, descripción litológica, distribución de tamaño de grano (GS: porcentajes de arcillas, limos y arenas), susceptibilidad magnética (MS) y densidad obtenidas con un sistema MSCL de Geotek (azul y verde, respectivamente), composición geoquímica obtenida con un escáner de FRX AVAATECH expresada con las relaciones K/Ti (rojo) y Ca/Ti (naranja), y la reflectividad (L\*, negro) (modificado de Gracia *et al.*, 2010).

environmental and climatic variations ([Thouveny \*et al.\*, 2000](#); [Moreno \*et al.\*, 2002](#); [Thouveny \*et al.\*, 2004](#)).

[Martins \*et al.\* \(2013a\)](#) studied the sediments of the lagoon of Aveiro (Portugal) in order to distinguish between anthropogenic vs climatic influences through the analysis of the XRF core scanner. They observed increases in the Zn, Pb and Cu records at the top of the sequence that clearly reflect human contributions and show the usefulness of the XRF



core-scanner analysis in the study of human induced contamination. Similar results are being obtained for lacustrine sediments in the Iberian Peninsula where Pb anomalies detected by XRF core-scanner show human pollution since Roman times (Mata *et al.*, 2013).

After the work of Shackleton *et al.* (2000), in which they showed the phase relationships between millennial-scale events of both hemispheres as registered in the same samples within core MD95-2042 off southern Portugal, this area has become very attractive for the paleo-community and several sediment cores have been recovered. Most of them were recovered within the IMAGES program aboard the RV Marion Dufresne (MD95, MD99, MD01 and MD03), which established the study base for the drilling of the 'Shackleton site' with the RV Joides Resolution within IODP Expedition 339 down to 1.45 Ma, for establishing a marine reference section of Pleistocene climate (Stow *et al.*, 2013). The recent works published by Hodell *et al.* (2013a and 2013b) represent good examples of the use of continuous and non-destructive methods for the study of past climate conditions in the Portuguese margin. Thus, variations in XRF Ca/Ti, representing mixing ratios of biogenic (Ca) and detrital (Ti) sediment, and changes in sediment redness, responding to low-latitude wind-driven processes (e.g., dust transport, upwelling, precipitation), are highly responsive to climate change on both millennial and orbital time scales, as the spectral analysis of both records has corroborated.

### **Gulf of Cadiz**

The Gulf of Cadiz has also been extensively studied from different perspectives during the last decades, with special focus on the study of the Plio-Pleistocene sedimentary and tectonic evolution, and for the investigation of past oceanographic conditions and its influence on global circulation and climate. Several studies have taken advantage of the use of non-destructive techniques, such as line-scan imaging, X-radiographs, multi-sensor core loggers and XRF core scanners for describing hydrodynamic regimes and sediment composition that together with sediment texture provide important sedimentological information regarding contourite systems, past climate and oceanographic conditions, or to define diagenetic processes related to the presence of methane in the sediments (Llave *et al.*, 2006; Voelker *et al.*, 2006; Toucanne *et al.*, 2007; Kolganova *et al.*, 2009; Mulder *et al.*, 2009; Hanquiez *et al.*, 2010; Marchès *et al.*, 2010; Mata *et al.*, 2010 and 2012; Antón *et al.*, 2013; Lebreiro

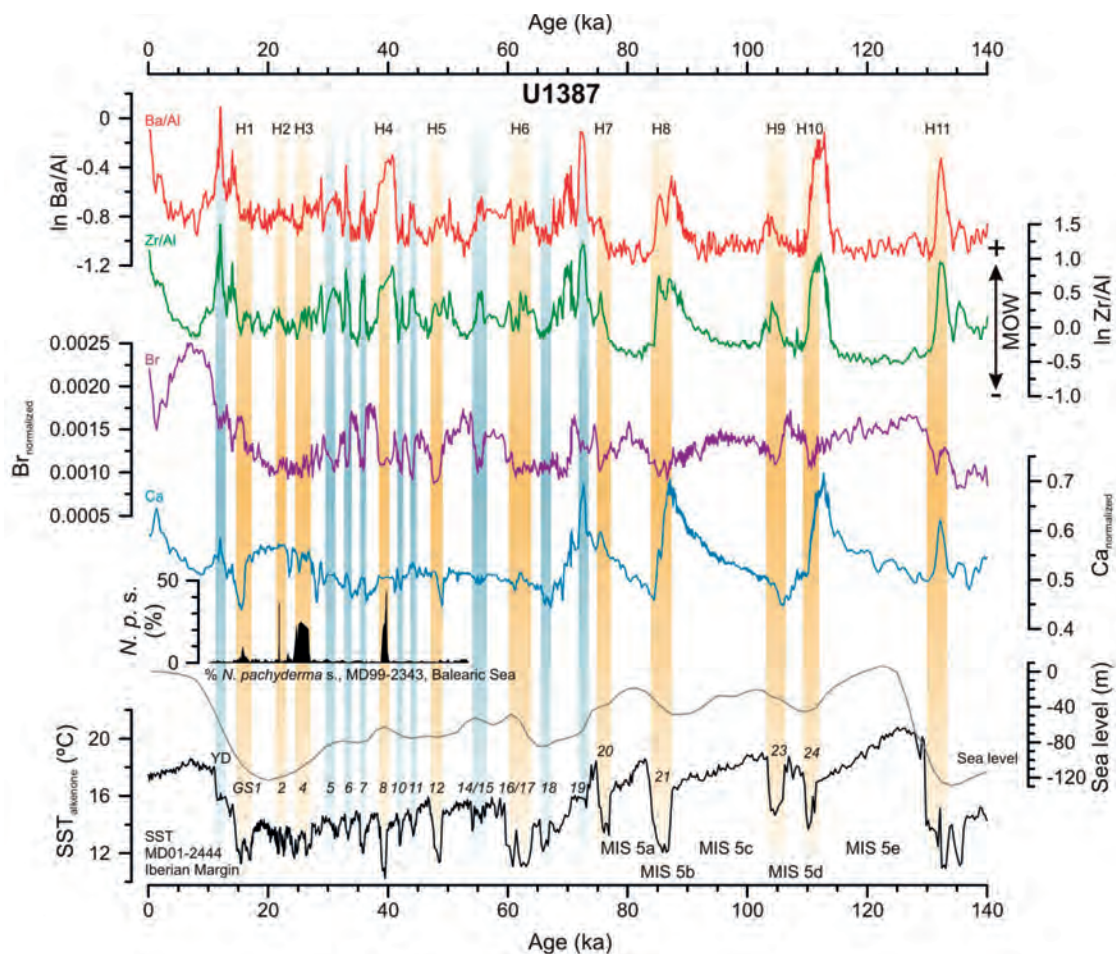
*et al.*, 2013; Vanneste *et al.*, 2013; Bahr *et al.*, 2014) (Fig. 9).

Thus, the Gulf of Cadiz was targeted for drilling by Expedition IOPD 339 (November 2011-January 2012) as a key location for the investigation of Mediterranean Outflow Water (MOW) through the Strait of Gibraltar (Gibraltar Gateway) and its influence on global circulation and climate, and also for understanding the effects of tectonic activity on evolution of the Gibraltar Gateway and margin sedimentation (Stow *et al.*, 2013). The first analysis carried out by means of continuous and non-destructive techniques on the drilled sediments has already revealed important results on the sedimentation pattern of the sites and has provided a preliminary time constrain of the sequences covered (Hodell *et al.*, 2013b; Stow *et al.*, 2013).

### **Western Mediterranean Basin**

On the Mediterranean side of the Iberian Peninsula the Alboran Basin is probably the region which has been more intensively studied. Most of the major achievements carried out in the Alboran Basin, mainly related with the study of paleoclimatic and paleoceanographic conditions, resulted from the study of discrete samples (Cacho *et al.*, 1999, 2000, 2001, 2002 and 2006; Martrat *et al.*, 2004; Moreno *et al.*, 2005; Jimenez-Espejo *et al.*, 2007b and 2008 among others). However, non-destructive techniques for the study of sediment sequences are being applied in recent years, not only in the Alboran Basin, but also in materials from the Algero-Balearic Basin, and the XRF core scanner data is especially giving very promising results.

In the Alboran Basin, Jimenez-Espejo *et al.* (2007a) carried out a multiproxy analysis, including XRF core-scanner data, describing paleoenvironmental changes in the Western Mediterranean Basin at the time of the Neanderthal extinction, and complementing a previous work by Finlayson *et al.* (2006) in which XRF core scanner data was applied in the excavation of Gorham's Cave, in Gibraltar. Also with XRF core scanner data, Fink *et al.* (2013) described changes in productivity and aridity/humidity conditions during the last deglaciation in sediment cores from the Melilla Coral Province, Cacho *et al.* (2013) described centennial-scale aridification events during the present interglacial with sediment cores from the Malaga slope and Alonso *et al.* (2014) described fluctuations in bottom currents during the Pleistocene and Holocene in the Djibouti Ville Seamount. Using MSCL data, Bozzano *et al.* (2009) described the influence of



**Figure 9.** XRF scanning records for U1387, from the Gulf of Cadiz, for the past 140 ka with a chronostratigraphy based on the tuning of the bromine record to the *G. bulloides*  $\delta^{18}\text{O}$  record of core MD01-2444 (Hodell *et al.*, 2013). Oscillations of Zr/Al ratio are interpreted as a proxy for Mediterranean Outflow Water (MOW) variability; Ba/Al (representing barite) is related at this site to the accumulation of heavy minerals (such as zircon) due to gravitational sorting and winnowing under high bottom current speed (note the great similarity between Zr/Al and Ba/Al records). Br is interpreted as an indicator of primary productivity whilst the Ca at this site cannot be used as an indicator of carbonate production since it appears to be almost completely overprinted by bottom current processes. Br and Ca records were normalized to the total counts of all processed elements excluding Ag (slit) and Rh (X-ray tube). Positive excursions in Zr/Al relate well to SST<sub>alkenone</sub> cooling at Iberian Margin site MD01-2444 (Martrat *et al.*, 2007) and variations in global sea level (Waelbroeck *et al.*, 2002). Invasion of cold, fresh North Atlantic waters into the Alboran Sea during Heinrich Stadials is indicated by high abundances of *Neogloboquadrina pachyderma* (*s*) in core MD99-2343 (Sierro *et al.*, 2009). All XRF records are three-point running means. Major cool events (Greenland Stadials) not associated with major ice rafting are indicated by italics and blue shading, Heinrich Stadials (H) are marked by orange bars. All data are plotted on the time scale of MD01-2444 (Hodell *et al.*, 2013). YD, Younger Dryas; MIS, Marine Isotope Stage; GS, Greenland Stadial (modified from Bahr *et al.*, 2014).

**Figura 9.** Registros de escáner de FRX del testigo U1387, del Golfo de Cádiz, para los últimos 140 ka con una cronoestratigrafía basada en el ajuste del registro de bromo con el registro de  $\delta^{18}\text{O}$  de *G. bulloides* del testigo MD01-2444 (Hodell *et al.*, 2013). Las oscilaciones en la relación Zr/Al se interpretan como indicadores de variabilidad en el Agua de Salida del Mediterráneo (MOW); el registro de Ba/Al (barita) está relacionado en este testigo con la acumulación de minerales pesados (como el zircón) debido a procesos gravitacionales de clasificación y lavado bajo la acción de corrientes de fondo con elevadas velocidades (notar la gran similitud entre los registros de Zr/Al y Ba/Al). El registro de Br se interpreta como indicador de productividad primaria mientras que el registro de Ca no se corresponde con la producción carbonatada en este testigo puesto que su señal está casi totalmente sobreimpresa por procesos de corrientes de fondo. Los registros de Br y Ca han sido normalizados en este trabajo con las cuentas totales de todos los elementos procesados excluyendo el Ag (presente en la máscara que delimita el haz de rayos X) y el Rh (del tubo de rayos X). Excursiones positivas en la relación Zr/Al se correlacionan con enfriamientos de la temperatura superficial del agua (SST<sub>alkenone</sub>) observados en el testigo MD01-2444 del margen ibérico (Martrat *et al.*, 2007) y variaciones globales en el nivel del mar (Waelbroeck *et al.*, 2002). Invasiones de agua menos salina y fría del Atlántico Norte en el Mar de Alborán durante los Estadales Heinrich (H) están indicados con incrementos en la abundancia de *Neogloboquadrina pachyderma* (*s*) en el testigo de sedimento MD99-2343 (Sierro *et al.*, 2009). Todos los registros de FRX están promediados a tres puntos. Los eventos fríos de mayor grado (Estadales de Groenlandia) no asociados con descargas masivas de icebergs se marcan en cursiva y sombreado azul. Los Estadales Heinrich (H) están señalados con barras naranjas. Todos los datos se han representado usando la escala de tiempo del testigo MD01-2444 (Hodell *et al.*, 2013). YD, Dryas reciente; MIS, Estadio Isotópico Marino; GS, Estadial de Groenlandia (modificado de Barhet *et al.*, 2014).

sea-level variations on deep-sea sedimentation in the Almeria turbidity system.

In the Algero-Balearic Basin, Frigola *et al.* (2013) used XRF core scanner and colour data to correlate and establish a preliminary chronostratigraphy in several cores recovered in the Eivissa Channel, allowing the description of changes in the sedimentation pattern on this region related to orbital changes in summer insolation, and Ca-XRF data from a 300 m sequence drilled in the upper slope of the Gulf of Lion, together with grain-size oscillations, allowed Frigola *et al.* (2012) to identify five regressive units stacked during the sea-level lowering phases of the last five glacial-interglacial 100-kyr cycles.

## Conclusions

The use of non-destructive, continuous, high-resolution techniques for the analysis of the properties in sediment records by the geoscientific community has experienced a significant growth during the last decades. These techniques are now essential to any state-of-the-art study of sedimentary deposits because of their capacity to quickly provide vast amounts of high-quality data at a relatively low cost, with resolutions higher than 1 mm, and in some cases close to 1  $\mu\text{m}$ . Most instruments can be installed both in land-based and onboard laboratories. Both approaches, and especially the latter, still have a long way to go, as their use on research vessels will probably be developed further in the future. Obtaining the information immediately after cores are recovered should be given priority as it provides the opportunity of characterizing sediments in an optimum state before they are exposed to handling, transport, storage and post-splitting. A battery of new instruments especially adapted to the needs of the geoscientific community and to the characteristics of the samples to be investigated is now available and will develop further. The newest developments in CAT-scans, for instance, will optimize core analysis at a very to extremely high resolution. Other techniques, such as the hyperspectral imaging of sediment cores have an enormous potential. Some portable systems are now also available (e.g., the Single Core Scanner, Channel Systems Inc.).

Last but not least, secure, accessible and user-friendly databases, where the enormous flow of information generated by non-destructive sediment core can be stored and shared, unless protected by confidentiality clauses, are now mandatory for the progress of geoscientific research and also to achieve the best possible cost/benefit ratio.

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