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Outcrop gamma-ray spectrometry: Applications to the Sinemurian–Pliensbachian organic-rich facies of the Lusitanian Basin (Portugal)

Espectrometría de rayos gamma: Aplicaciones a las facies orgánicas carbonatadas del Sinemuriense–Pliensbachiense de la Cuenca Lusitánica (Portugal)

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Abstract

This work provides detailed information of outcrop gamma-ray logging from the Sinemurian–Pliensbachian organic-rich units (Água de Madeiros and Vale das Fontes formations) of the Lusitanian Basin, which are recognized as one of the most important potential oil source rocks interval of Portugal. The study included total and spectral gamma-ray measurements in outcrop and laboratory, Total Organic Carbon (TOC) content and whole-rock mineralogical characterization by X-ray diffraction, carried out in the two most important outcrop areas of the Western Lusitanian Basin (S. Pedro de Moel and Peniche). The outcrop data was compared with subsurface information.

The results show high variability of the gamma radiation (26 to 210 cps) and radioactive elements associated with the lithological, mineralogical and geochemical differences. The TOC data yielded the organic matter enrichment of both formations, where the highest values are observed in the Água de Madeiros Formation (maximum=20.4%). The highest U concentration (11 ppm) is consistent with the highest Total Organic Carbon peak mainly supported by the precipitation of authigenic U (10 ppm). The majority of the analysed samples show an obvious authigenic U enrichment, especially in the Água de Madeiros Formation where it represents the main component of the total U content. A highly significant U/TOC correlation (r up to 0.87) with a low ratio (0.3–0.7 ppm/wt.%TOC) was obtained, confirming that U content can be used as a proxy for organic richness in the studied depositional system. The gamma-ray tool was also useful in the identification of 2nd order Transgressive–Regressive facies cycles (and maximum flooding surfaces) and in the correlation between outcrop and subsurface data.

Keywords: gamma-ray spectrometry; organic-rich carbonates; Sinemurian, Pliensbachian; Lusitanian Basin

Resumen

Este trabajo proporciona información detallada de espectrometría de rayos gamma de las unidades ricas en materia orgánica del Sinemuriense–Pliensbachiense de la Cuenca Lusitánica, reconocidas como los depósitos con más potencial para generar petróleo en Portugal. El estudio incluye mediciones de espectrometría de rayos gamma en afloramiento y laboratorio, cuantificación del carbono orgánico total (COT) y caracterización mineralógica por difracción de rayos X, llevada a cabo en dos áreas de la Cuenca Lusitánica (S. Pedro de Moel y Peniche). Los datos de afloramiento se han comparado con información de pozos.

Los resultados muestran una alta variabilidad de la radiación gamma (26 a 210 cps), de los elementos radioactivos asociados a las distintas litologias, y de las características mineralógicas y geoquímicas. Los datos de COT evidencian un enriquecimiento en materia orgánica de toda la secuencia estudiada, en particular de la Formación de Água de Madeiros (máximo=20.4%). La concentración más elevada de U (11 ppm) es consistente con el pico principal de COT causado por la precipitación de U autigénico (10 ppm). La mayoría de las muestras analizadas presentan un marcado enriquecimiento en U autigénico, especialmente en la Formación de Água de Madeiros que representa el principal componente del contenido total de U. Se ha obtenido una correlación U/COT muy significativa (que alcanza valores de r=0.87) con una proporción baja (0.3 a 0.7 ppm/%COT), lo que confirma que el valor de la concentración de U puede ser utilizado para calcular el contenido en materia orgánica en el sistema deposicional estudiado. La técnica de rayos gamma también ha sido útil en la identificación de ciclos Transgressivo-Regressivos de 2º orden, de superficies de máxima inundación y en la correlación de datos de superficie con datos de pozos.

Palabras clave: espectrometría de rayos gamma; carbonatos ricos en materia orgánica; Sinemuriense, Pliensbachiense; Cuenca Lusitánica.

1. Introduction

Organic-rich deposits, particularly with dark and laminated marls (commonly named as black shales), are of great importance for petroleum exploration, since they may represent major source rock intervals (e.g., Bodin *et al.*, 2011; Riediger, 2002; Lüning *et al.*, 2000). The outcrop Gamma-ray logs allows the characterization of source rocks and their organic content estimation, being also a very important correlation tool to better understand the subsurface data (Bessa and Hesselbo, 1997). Gamma-ray (GR) spectrometry measures the total gamma radiation and the individual contribution from the three main radioactive elements: potassium (K), thorium (Th) and uranium (U) (see a detailed discussion in Ellis, 1987). In carbonate sequences (the focus of this work), the K and Th are mainly concentrated in the minerals that constitute the insoluble residue (e.g., Lucia, 2007). The U also occurs in the detrital clay fraction however, and unlike Th, it is also partly carried in solution as uranyl carbonate complexes ($UO_2(CO_3)_2^{2-}$) (Langmuir, 1978). Under oxygen depleted conditions, it may be precipitated to enrich the sediment in authigenic (nondetrital) U, fixed at the sediment-water interface accumulating together with the organic matter (Wignall and Myers, 1988). In this systems, it is possible to obtain a linear relationship between the total organic carbon (TOC) and U (Lüning and Kolonic, 2003). This technique, when associated with the direct analysis of the rocks (biostratigraphy, sedimentology, petrography, geochemistry) can be very helpful, not only to characterize a potential source rock (e.g., Bodin *et al.*, 2011; Lüning *et al.*, 2005; Lüning and Kolonic, 2003; Myers and Wignall, 1987), but also as an important tool for basin analysis, particularly in sequence stratigraphy studies (e.g., Catuneanu, 2006; Collins *et al.*, 2006; Pawellek and Aigner, 2003; Parkinson, 1996), lithostratigraphic characterization (e.g., Raddadi *et al.*, 2005; Martinius *et al.*, 2002; Hadley *et al.*, 2000; Bessa and Hesselbo, 1997) and correlation with subsurface data (e.g., Aigner *et al.*, 1995; Leslie *et al.*, 1993).

Here we studied the Lower Jurassic organic-rich units deposited during the Late Sinemurian–Pliensbachian time interval, one of the main potential source rock intervals of the Lusitanian Basin (e.g., Duarte *et al.*, 2012; Oliveira *et al.*, 2006). More than one hundred wells have been drilled and thousands of kilometres of seismic lines were obtained in the Lusitanian Basin. However, no significant oil or gas field with commercial interest was found in this basin or in Portugal.

Both field and laboratory spectral GR techniques were applied in different key-sections of the western sector of the Lusitanian Basin near S. Pedro de Moel and Peniche (Fig. 1), supported by stratigraphic, geochemical and mineralogical investigations. One of the main targets of this data set is to correlate the U content with the organic richness and test their relationship, in order to use the U results as a proxy to estimate the TOC. The geochemical analysis, normally more complex and expensive, can be replaced using indirect measurement of U content by GR spectrometry in wells and outcrops. This work also highlights the importance of these methods in the delimitation of the 2nd order Transgressive-Regressive (T-R) facies cycles (sensu Jacquin and de Graciansky, 1998) and the identification of maximum flooding intervals (maximum flooding surfaces in Galloway, 1989) already presented for the Lusitanian Basin (e.g., Duarte *et al.*, 2010). Final-

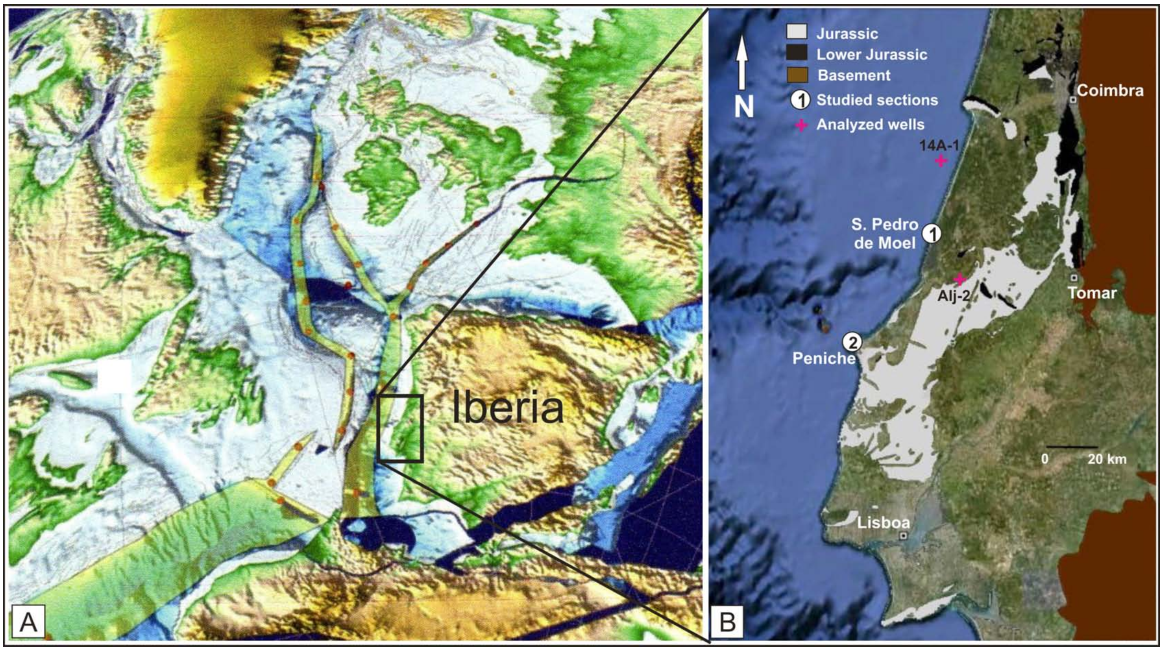

Fig. 1 – A) Plate kinematic model of the North Atlantic early rifting (180 Ma) with epicontinental seas to the West (light blue) and the Tethys sea to the East (dark blue) (modified from Skogseid, 2010); B) Simplified geological map and spatial distribution of the carbonate units of Lower Jurassic age in the Lusitanian Basin, with the locations of the studied sections and wells. 1- Polvoeira and Água de Madeiros section; 2- Portinho da Areia Norte section.

Fig. 1 – A) Esquema cinemático de las placas del *rift* del Atlántico Norte (180 Ma), con los mares epicontinentales hacia el oeste (azul claro) y el mar de Tethys hacia el Este (azul oscuro) (modificado de Skogseid, 2010); B) Mapa geológico simplificado con la distribución espacial de las unidades carbonatadas del Jurásico Inferior de la Cuenca Lusitánica, con las localizaciones de los perfiles estudiados y los pozos analizados. 1 – Sección de Polvoeira y Água de Madeiros; 2 – Sección Portinho da Areia Norte.

ly, the obtained data are also used to characterize the radiometric signature of the different lithologies and lithostratigraphic units in outcrop and to correlate the results with the natural GR data collected in some exploratory wells drilled in the surrounding areas.

2. Geological Setting

The Lower Jurassic is particularly well represented in the Lusitanian Basin, one of the Atlantic basins bordering the Variscan Iberian Massif (Fig. 1A). It comprises a thick carbonate succession consisting of up to 550 m of mostly marl-limestone alternations characterizing a great part of the Late Sinemurian–Toarcian series (Duarte *et al.*, 2010; Duarte, 2007; Duarte and Soares, 2002). These facies were deposited in the Upper Triassic–Callovian 1st-order cycle (Azerêdo *et al.*, 2003; Soares *et al.*, 1993; Wilson *et al.*, 1989), resulting in the progressive flooding of the basin, controlled by an epicontinental sea and sustained by a low-gradient carbonate ramp dipping NW (Duarte *et al.*, 2010; Duarte, 2007; Dommergues and Mouterde, 1987).

The studied late Sinemurian–Pliensbachian interval is characterized by the occurrence of organic-rich facies (Duarte *et al.*, 2011a, b, 2010; Oliveira *et al.*, 2006). This study is focused on the Água de Madeiros and Vale das Fontes formations, defined by Duarte and Soares (2002), although other units were partially analysed, for example, the Coimbra and Lemede formations (Fig. 2). As a result from the ramp morphology and controlled by regional tectonics and sea level changes, the thickness of these units increases towards the west, which are biostratigraphically well constrained due to the abundance of benthic and nektonic fauna, namely ammonites and calcareous nannofossils (Silva *et al.*, 2011; Duarte *et al.*, 2010; Oliveira *et al.*, 2007; Dommergues and Mouterde, 1987; Mouterde, 1953, 1967) (Fig. 2).

The top of the Coimbra Formation (Fm), consists of a series of thick limestone beds with some argillaceous interbeds with abundant benthic macrofauna (e.g., Duarte and Soares, 2002). The age equivalent Água de Madeiros Fm, belonging to the Oxynotum–Jamesoni Zone, consists of marl-limestone alternations with frequent dark and laminated marls and is very well exposed in the S. Pedro de Moel coastal area, where its type-section is defined (Duarte and Soares, 2002). This unit consists of two members: the Polvoeira Member (Mb), which contains organic-rich dark, laminated marls (mostly black shales), and the overlying Praia da Pedra Lisa (PPL) Mb, with a predominance of limestones, especially at the base of the succession.

The overlying Vale das Fontes Fm represents the return to a marly sedimentation, which characterizes almost the entire Pliensbachian (Jamesoni–Margaritatus Zone) of most of the Lusitanian Basin. This formation is subdivided into three members, but only the uppermost one was analysed, the Marl-Limestone with Organic-Rich Facies (MLOF) Mb, due to the abundance of dark marls. This interval belongs to the Davoei-Margaritatus Zone and was previously also referred to an Organic Matter Preservation Interval (Late Pliensbachian OMPI sensu Silva *et al.*, 2011).

The uppermost Pliensbachian is represented by the Lemede Fm (Duarte, 2007; Duarte and Soares, 2002) which comprises a carbonate dominated sequence.

3. Materials and Methods

3.1. Field Procedure

Field work concentrated on the two mentioned stratigraphic units which were studied in three sections where recent geological studies had been conducted and detailed stratigraphic logs constructed (used in this study): Polvoeira (Duarte *et al.*, 2011a; 2012), Água de Madeiros (Duarte *et al.*, 2010, 2012) and Portinho da Areia do Norte (Silva *et al.*, 2011). A high resolution spectral GR survey was carried out with a portable GR spectrometer (model GR-130G from Exploranium®) equipped with a NaI (Tl) detector 38 mm x 51 mm in size, integrating a 256 channels system and with a spectral range of 0–3 MeV. This equipment measures the character of about 0.3 m^3, in a hemispheric shape, behind the outcrop face. Each section was logged by measuring the total gamma-radiation every 20-30 cm (or each bed), over a time integration of 10 s. Around 700 valid readings were obtained, covering a stratigraphic interval of approximately 85 m. This equipment also allows the quantification of the major radioactive elements: U, Th and K. Based on their sedimentary and radiometric characteristics, a total of 60 different beds were GR-measured. The obtained U, Th and K concentrations are the average result of three valid measurements, each one with a duration of 100 s. This methodology follows the recommendations of the manufacturers and has been widely used in other works with similar logging procedures (e.g., Bodin *et al.*, 2011; Pereira *et al.*, 2003; Svendsen and Hartley, 2001). Field tests carried out at the beginning of the study, using measurements with higher time integration intervals (20 and 30 s for the total GR and 200 s for the U, Th and K), do not show a significant difference, indicating that the used measurement duration resulted in an acceptable data reproducibility. The portable equipment was calibrated after each hour of use with a ^{137}Cs radioactive source. As a cross-check, several test analyses were undertaken using an alternative portable scintillation counter, Saphymo SPP2, equipped with a NaI (Tl) detector that is 38 x 25 mm in size. All the gamma-radiation data were measured in counts per second (cps), U and Th in parts per million (ppm) and K in percentage (%). All measurements followed a strict procedure to avoid significant errors mainly related with the heterogeneity of the outcrops and the sample volume. A detailed description of these procedures can be found in Svendsen and Hartley (2001).

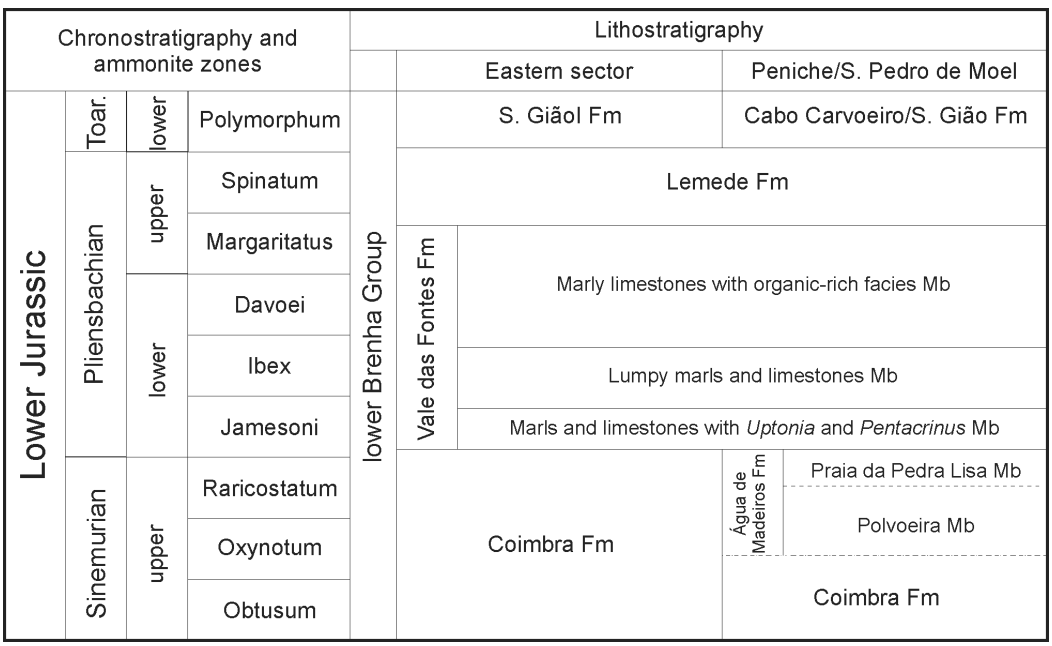

Fig. 2 – Lithostratigraphic chart for the upper Sinemurian–lowermost Toarcian series of the Lusitanian Basin (modified after Silva *et al*., 2011; Duarte *et al*., 2010; Duarte and Soares, 2002). These units constitute the lower part of the Brenha Group (Witt, 1977).

Fig. 2 – Unidades litoestratigráficas del Sinemuriense superior–Toarciense inferior de la Cuenca Lusitánica (modificado de Silva *et al*., 2011; Duarte *et al*., 2010; Duarte and Soares, 2002). Estas unidades constituyen la parte inferior del Grupo Brenha (Witt, 1977).

3.2 Laboratory procedure

During the field work, 45 rock samples with an average weight of 1 kg were collected for later laboratory analysis. All parameters (gamma-ray spectrometry, geochemical and mineralogical) were obtained from the same portion of rock avoiding errors associated with lateral and vertical variation. For each sample, the U, Th and K were quantified in the Natural Radioactivity Laboratory (University of Coimbra) using a Gamma-Ray spectrometer Ortec®, with a 3x3" NaI (Tl) detector, integrating a 1024 Multichannel system with a NaI spectral analysis software ScintiVision-32. The higher precision of this equipment is related to the fact that all these measurements were performed in standardized conditions, allowing the cross-check and validation of the radiometric data obtained with the field instrument. As mentioned earlier, quantification of the different components of the U is important when studying organic-rich intervals. For this purpose, and taking into account that the minimum Th/U ratio of normal mudstones where U is assumed to be entirely detrital is 3, the formula introduced by Wignall and Myers (1988) to estimate the detrital and authigenic U fractions in mudstones was used (Eq. 1):

$$U_{authigenic} = U_{total} - Th/3 \qquad \text{(Eq. 1)}$$

The mineralogical analyses were performed by X-ray diffraction (XRD – model PW3710, with a PW1830 generator, Cooper ampoule and PW1050 goniometer) in the X-Ray Laboratory of the Earth Sciences Department of the University of Coimbra. The data was grouped and semi-quantifications were projected in the graphic logs as carbonates (calcite and dolomite) and siliciclastics (quartz, feldspar and muscovite/biotite - mica). Due to the difficulty of accurately semi-quantifying the abundance of the clay minerals, these were not incorporated in the data set.

The TOC and insoluble residue content were determined with a SC-144DR LECO® analyser in the LAFO Laboratory of the Geosciences Institute of the Federal University of Rio de Janeiro with an analytical precision of ±0.1%.

Taking into account that the GR curve obtained in outcrop can be used to calibrate and better understand the subsurface GR-character, the logs of exploration wells 14 A-1 and Alj-2 drilled in the Lusitanian Basin were acquired from the Research and Petroleum Exploration Division of Portugal for correlation (see location in Fig. 1B).

4. Results

4.1. Polvoeira section

This section is situated 4 km south of the S. Pedro de Moel village, in the cliffs of the Polvoeira beach (WGS84 coordinates: 39º 43' 26.76''N; 09º 02' 51.66''W) (Fig. 1B). A total of 30 m were logged (interrupted only by one small inaccessible sector), belonging to the Oxynotum–Raricostatum Zone, Late Sinemurian (Duarte *et al.*, 2011a, 2012; Duarte and Soares, 2002) (Fig. 3A). The analysed section includes the last 4 m of the Coimbra Fm and the transition to the Água de Madeiros Fm, where a big portion of the Polvoeira Mb was logged, in a sequence with a great predominance of the organic-rich levels towards the top (Fig. 3B).

Regarding the total GR values, this section reveals a generalized tendency to increase towards the top, from 30 to 60 cps registered in the Coimbra Fm to 180–190 cps obtained in some black shales of the Povoeira Mb (Fig. 4). The highest GR value (210 cps) is associated with one of the thickest black shale horizons. The spectral GR data collected in the field and in the laboratory are quite similar, although it is emphasized that in the field method, the U concentrations are normally higher and the Th and K lower when compared with the laboratory data. Specially the U and partly also the Th values, show a similar trend to the total GR, where the low values (< 1 ppm of U and Th) observed in the Coimbra Fm, contrast with the highest values observed in the black shales of the Polvoeira Mb (11.6 ppm of U, 7.6 ppm of Th and 210 cps of total GR). On the other hand, the K values did not show any particular trend using the field equipment, ranging from 0.5% to 2%. In contrast, in the laboratory measurements K has a similar trend as the Th, ranging from 1 to 3.1% (Fig. 4).

The variability of TOC generally follows the U trend, where the highest TOC value (20.4 wt.%) corresponds to the highest U concentration (11 ppm). Insoluble residue always shows a good correlation with the mineralogy, where the lowest value (35%) corresponds to the highest carbonate/siliciclastic ratio and to the highest concentrations of Th and K. The XRD analysis also registered the presence of dolomite and the highest peaks of pyrite or uraninite were observed in this section, coincident with the black shale levels with high U content. The uraninite and pyrite share the same spectral interval becoming very difficult to distinguish them.

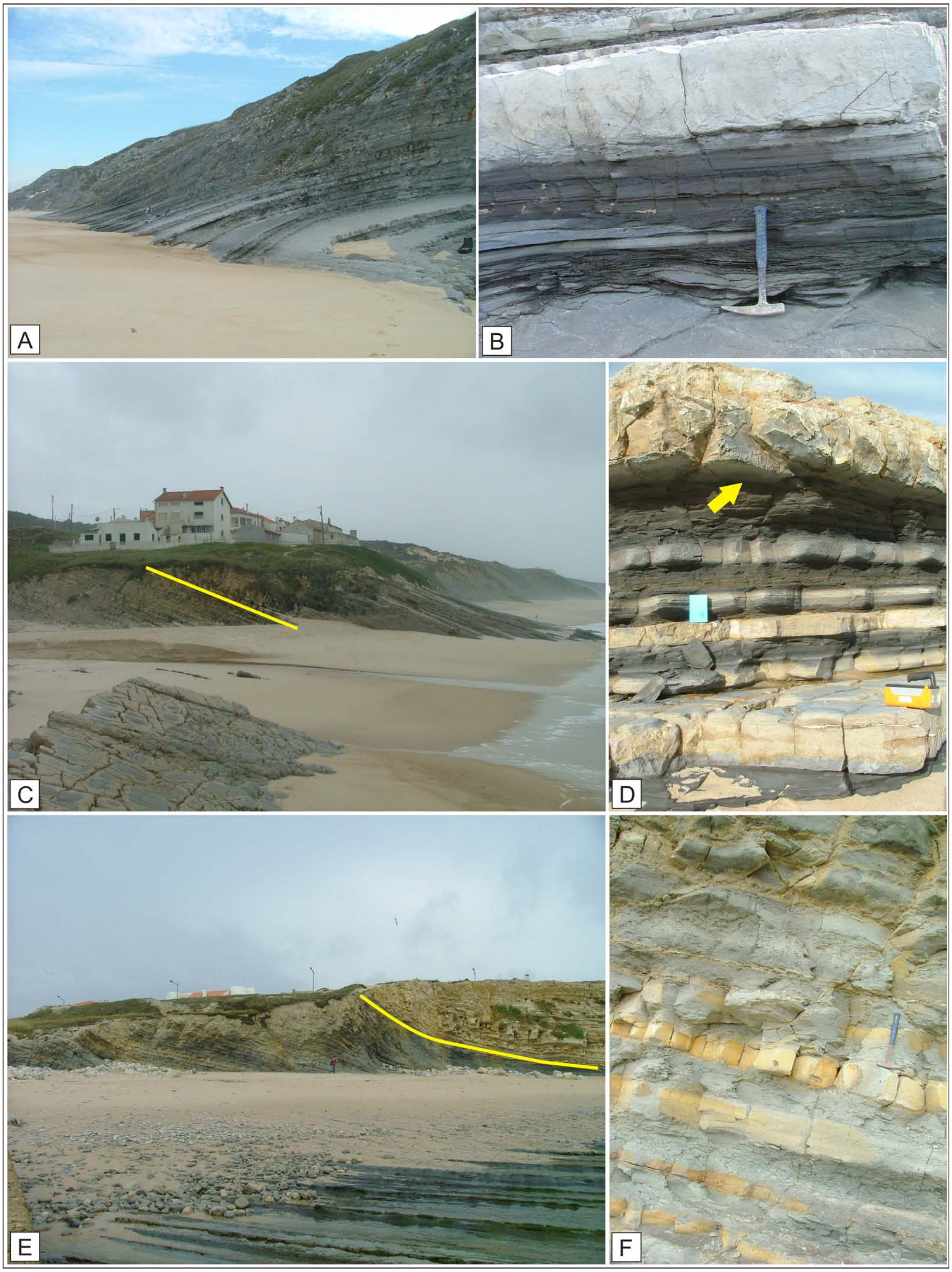

Fig. 3 – Field photographs of several key aspects of the studied sections; A) View of the Polvoeira section showing the marl-limestone alternations that characterise the Polvoeira Mb; B) Detail of one of the most expressive black shale interval of the Polvoeira section; C) View of the Água de Madeiros section, with the Polvoeira Mb at the base, and the calcareous PPL Mb to the top (the yellow line divides both units); D) Detail of the stratigraphic transition (arrow) between the two members of the Água de Madeiros Fm (Água de Madeiros section); E) Portinho da Areia Norte outcrop including the top of the MLOF Mb and the base of the Lemede Fm (the yellow line divides both units); F) Detail of the marly–limestone alternations that characterize de MLOF Mb (Portinho da Areia Norte section).

Fig. 3 – Fotografías de varios aspectos clave de los perfiles estudiados; A) Aspecto general de la sección de Polvoeira con la alternancia de marga y caliza que caracteriza el Mb Polvoeira; B) Detalle de uno de los intervalos de *black shale* más expresivo de la sección de Polvoeira; C) Aspecto general de la sección de Água de Madeiros, con el Mb de Polvoeira en la base, y el Mb PPL en la parte superior (la línea amarilla separa las dos unidades); D) Detalle de la transición estratigráfica (flecha) entre los dos miembros de la Fm de Água de Madeiros (sección de Água de Madeiros); E) Afloramiento de Portinho da Areia Norte que incluye la parte superior del Mb margo-calcáreo con facies bituminosas (MCFB) y la base de la Fm de Lemede (la línea amarilla separa dos unidades); F) Detalle de la alternancia de margas y calizas que caracteriza el Mb MCFB (sección de Portinho da Areia Norte).

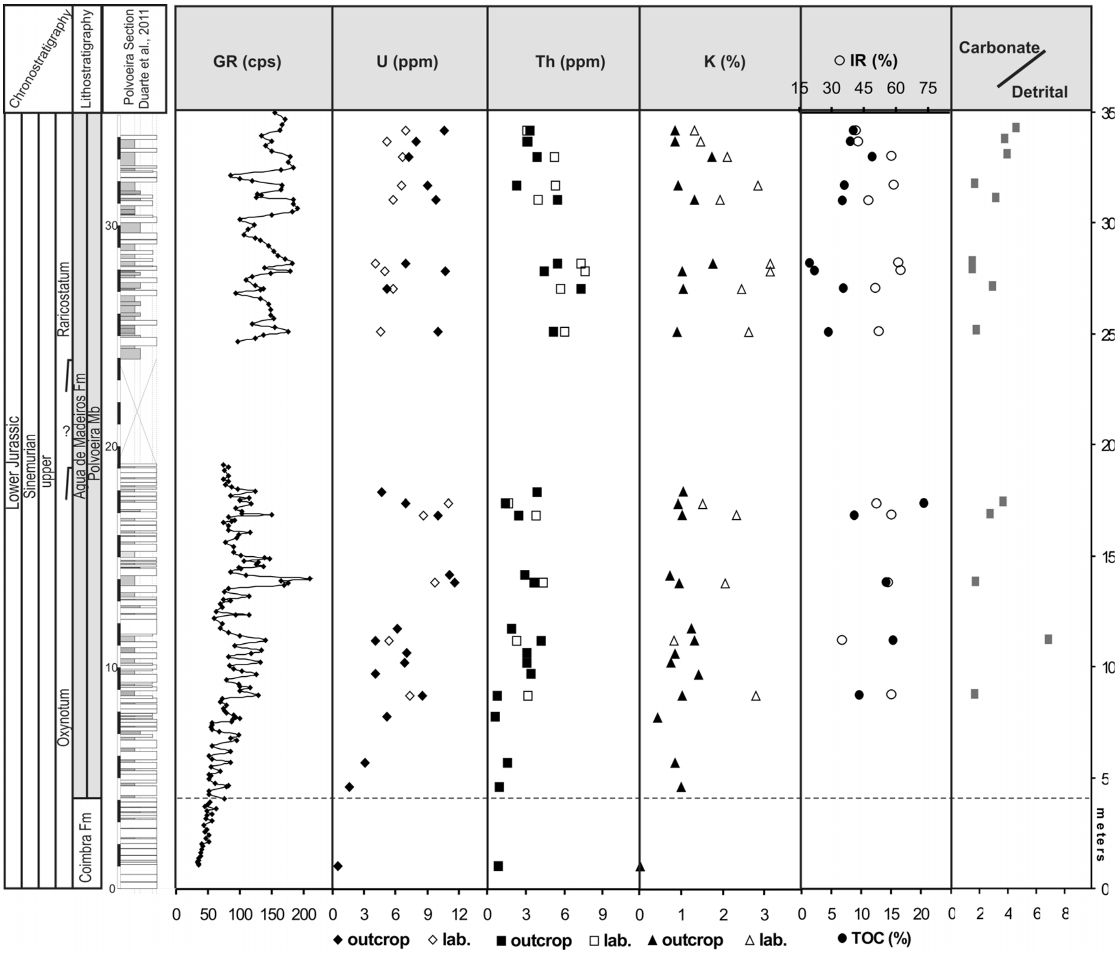

Fig. 4 – Lithostratigraphic log (Duarte *et al.*, 2011c), spectral gamma-ray measurements (field and laboratory), IR (insoluble residue), TOC content and carbonate/siliciclastic (Calcite + Dolomite/ Quartz + Mica + Feldspar) ratio of the Polvoeira section.

Fig. 4 – Columna litoestratigráfica (Duarte *et al.*, 2011c), perfiles de espectrometría de rayos gamma (mediciones de campo y laboratorio), residuo insoluble (IR), carbono orgánico total (TOC) y proporción carbonato/ siliciclástico (calcita + dolomita/cuarzo + mica + feldespato) de la sección de Polvoeira.

4.2. Água de Madeiros section

This sector is located 2 km north to the previous section (Coordinates: 39º 44’ 28.66’’N; 09º 02’ 21.52’’W). A total of 32 m were logged, in a sequence that belongs to the Raricostatum–base of Jamesoni Zone (Duarte *et al.*, 2010). The base of this section is stratigraphically equivalent to the top of the Polvoeira section described previously. This section includes the last 13 m of the Polvoeira Mb and almost the whole Praia da Pedra Lisa (PPL) Mb (15.5 m) (Fig. 3C). The transition between these two members is very expressive and visible in outcrop, materialized by a thick limestone bed deposited over a decimetre thick black shale bed (Fig. 3D). It also contains a 3 m section of the Marly Limestone with *Uptonia* and *Pentacrinus* (MLUP) Mb of the Vale das Fontes Fm with a clearly predominance of grey marls (Fig. 5).

The lithological transition between the two members of the Água de Madeiros Fm is also well marked in the radiometric data, characterized by the abrupt decrease from 150 to 54 cps, which can be considered a good benchmark in the correlation with the well logs. From the 22 m of the profile towards the top, the GR values start a positive and irregular incursion (33 to 120 cps) coincident with the more frequent and expressive occurrence of dark/argillaceous levels (Fig. 5).

Regarding the concentration of the different radioactive elements, the abrupt transition mentioned before is also observed, namely in the U concentrations (9.3 to 1.7 ppm). From the bottom of the PPL Mb to the top of this section, including the small analysed interval correspondent to the MLUP Mb, the content in U, Th and K increases gradually. Comparing the field with the laboratory data, the U values are similar but the Th and K content were higher in the laboratory analyses. Nevertheless, the variability associated to both techniques is quite similar (Fig. 5).

Regarding the geochemical data, the levels analysed in the top of the Polvoeira Mb show high TOC percentages (2.0 to 9.6 wt.%), although slightly lower when compared with the top of the Polvoeira section. For the insoluble residue, the highest amounts (> 60%) were obtained in the levels with the most important argillaceous fractions. The two samples easily observed at the 14 m and 28 m of the log by their high carbonate/siliciclastic ratio correspond to thick limestone beds characterized by low GR intensities (< 50 cps).

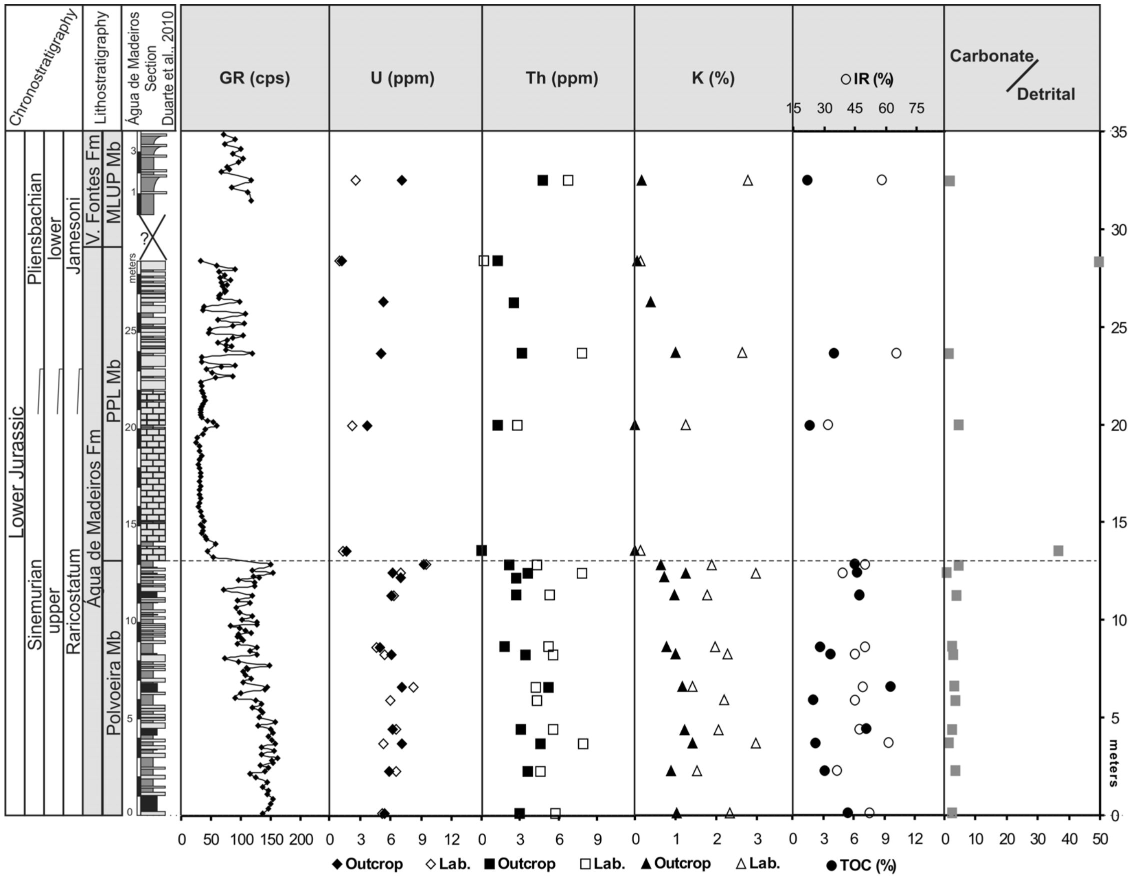

Fig. 5 - Lithostratigraphic log (Duarte *et al*., 2010), spectral gamma-ray measurements (field and laboratory), IR (insoluble residue), TOC content and carbonate/ siliciclastic (Calcite + Dolomite/ Quartz + Mica + Feldspar) ratio of the Água de Madeiros section.

Fig. 5 - Columna litoestratigráfica (Duarte *et al*., 2010), perfiles de espectrometría de rayos gamma (mediciones de campo y laboratorio), residuo insoluble (IR), carbono orgánico total (TOC) y proporción carbonato/ siliciclástico (calcita + dolomita/cuarzo + mica + feldespato) de la sección de Água de Madeiros.

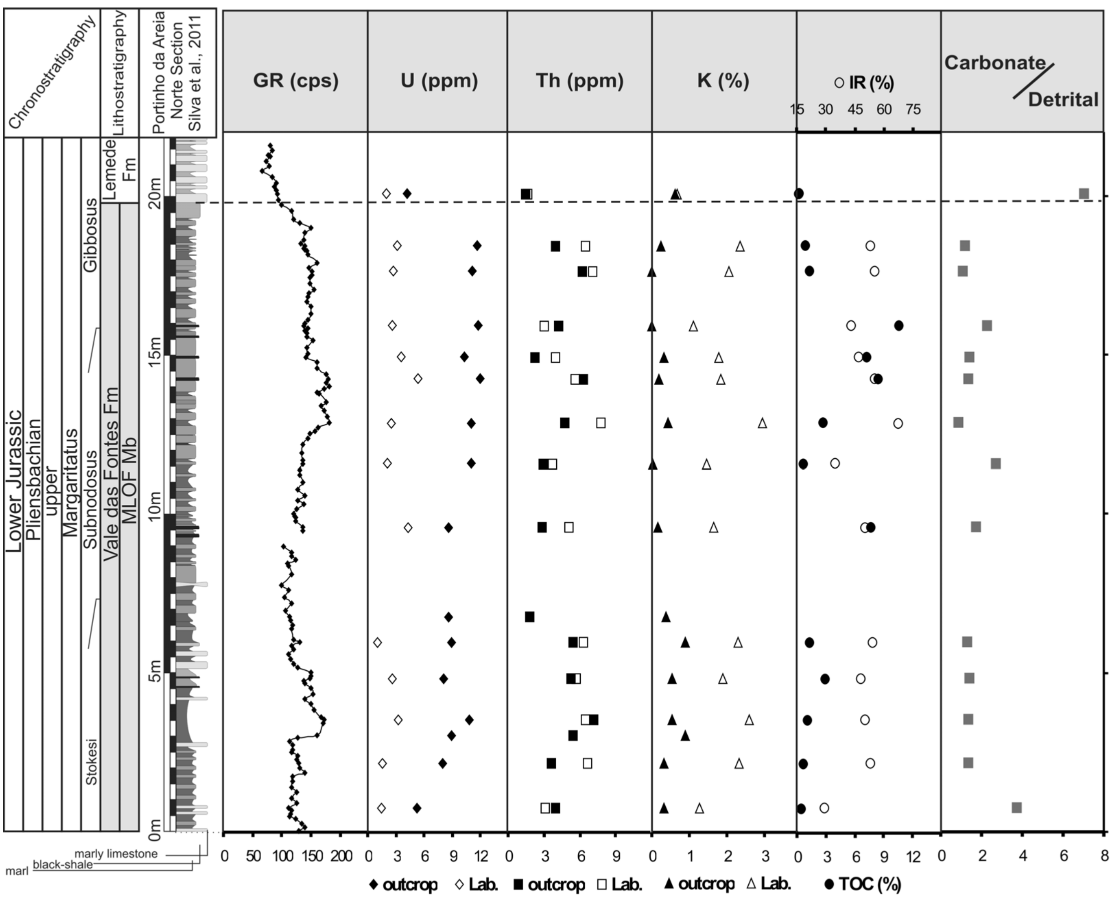

Fig. 6 - Lithostratigraphic log (Silva *et al*., 2011), spectral gamma-ray measurements (field and laboratory), IR (insoluble residue), TOC content and carbonate/ siliciclastic (Calcite + Dolomite/ Quartz + Mica + Feldspar) ratio of the Portinho da Areia Norte section.

Fig. 6 - Columna litoestratigráfica (Silva *et al*., 2011), perfiles de espectrometría de rayos gamma (mediciones de campo y laboratorio), residuo insoluble (IR), carbono orgánico total (TOC) y proporción carbonato/ siliciclástico (calcita + dolomita/cuarzo + mica + feldespato) de la sección de Portinho de Areia Norte.

4.3 Portinho da Areia do Norte section

This section is located in the cliffs of the Portinho da Areia do Norte beach, in the Peniche Peninsula (Coordinates: 39º 22’ 06.60’’N; 09º 22’ 42.98’’W). The logged section covers the last 20 m of the MLOF Mb from the Vale das Fontes Fm and the transition to the Lemede Fm (Fig. 3E), all dated from the Margaritatus Zone (Silva *et al.*, 2011). This sequence is the most argillaceous and organic-rich of the entire Vale das Fontes Fm (Oliveira *et al.*, 2006) (Fig. 3F). The transition to the Lemede Fm is easily observed by the colour change of the rock (yellow), announcing a predominant calcareous interval.

The GR data reveal high intensities, increasing progressively towards the top (100 to 181 cps), with two main peaks observed at 3 m (173 cps) and 13 m (181 cps) of the log (Fig. 6) (see also Parkinson, 1996). Comparing with the S. Pedro de Moel section, the obtained curve is more regular and concordant with a marly sedimentary sequence. From the 18 m of the profile (top of the MLOF Mb), the GR-values decrease rapidly from 160 cps to 90 cps, characteristic of the carbonate-dominated Lemede Fm.

The radioactive elements, namely the U, show a slight increase to the top of the section accompanying the GR-response, ranging between 8 and 12 ppm. The Th and K concentrations do not reveal any particular trend. Some differences were found in the laboratory analysis. First, the U content is much lower (around 1.5–5.3 ppm), but the trends defined between both analyses continue to be quite similar. The Th readings are similar (1.6–7.8 ppm) and the K, contrarily to the U, show higher values, ranging between 1–3% (Fig. 6).

Regarding the geochemical data, the average TOC is 3 wt.%, far from the 7 wt.% observed in the Água de Madeiros Fm at S. Pedro de Moel. It must be highlighted the occurrence of dolomite in trace amounts in some samples, and pyrite/uraninite in the richest U samples.

5. Discussion

5.1 Implications in the lithological characterization

The obtained GR values range between 26 to 210 cps (Fig. 7), which can be considered a large variation considering that only carbonated rocks were analysed (e.g.Pawellek and Aigner, 2003, 2004; Boyle, 1982). Those differences can be easily observed, not only when distinguishing different formations and members, but also within a single member (e.g. Polvoeira and PPL members).

The units with the lowest GR values and U, Th, K content, correspond to the sections with the highest carbonate content and lower clay fractions, with a predominance of limestone sequences such as the Coimbra Fm and the first meters of the PPL Mb (average values < 50 cps). The GR data in these units normally assumes blocky patterns, proved by the lower standard deviations ($\delta < 10$). On the other side, the highest peaks (> 150 cps) were obtained in the organic rich sections such as the Polvoeira and MLOF members (Fig. 7), mainly supported by the precipitation of authigenic U (lowest Th/U and K/U ratios, Fig. 8). The curves of those members are characterized by irregular patterns due to the marl and limestone alternations, showing higher standard deviations ($\delta > 20$). The highest Th concentrations were observed in the MLOF Mb and are consistent with a more abundant siliciclastic fraction and higher Th/U ratio (2.2) when compared to the Polvoeira Mb (Fig 8). This statement is also confirmed by their higher quartz, mica and feldspar content (obtained by XRD).

A correlation matrix for total GR and the three radioactive elements measured in outcrop is shown in Table 1. The total GR values are dominated by the U content shown by the strong correlation between the U log and the total GR counts (r = 0.77). The Th and K values show lower, but still significant correlation (r = 0.64 and 0.46 respectively). Most of the correlations with K values were not significant due to the restrictions of the portable spectrometer.

r	U_{field}	Th_{field}	K_{field}	Total GR
U_{field}	1			
Th_{field}	0.50	1		
K_{field}	0.08	0.19	1	
Total GR	0.77	0.64	0.46	1

Table 1 – Correlation matrix for radioactive elements determined by gamma-ray spectrometry in outcrop. At 99% confidence level a correlation coefficient of 0.339 (n = 57) is considered to be a significant result (Sachs, 1984).

Tabla 1 – Matriz de correlaciones de los elementos radiactivos determinados por espectrometría de rayos gamma en afloramiento. Un coeficiente de correlación de 0.339 (n = 57) es un resultado significativo para un nivel de confianza del 99% (Sachs, 1984).

It is possible to observe in Table 2 significant differences between the outcrop and laboratory measurements. An example is observed in the Polvoeira log at 17.5 m (Fig. 4). To the highest U concentration (11 ppm) obtained among all the laboratory analysis corresponds a significantly lower radiometric signature in the outcrop survey (GR = 125, U = 6.9, Th = 1.4). This is a consequence of the lithological sequence and the outcrop geometry where a marl horizon 11 cm thick occurs between two limestone beds (with 14 and 30 cm). As the used portable

Lithologies	Formations	U_{field}	Th_{field}	K_{field}	n	U_{lab}	Th_{lab}	K_{lab}	n
Limestone	All	1.9	0.9	0.18	4	1.7	0.8	0.40	2
Marly limestone	All	6.3	2.2	0.20	4	2.4	3.1	1.29	5
Marls	Água de Madeiros Fm	6,3	2.9	1.00	16	6.7	5.2	2.37	11
	Vale das Fontes Fm	9.6	4.8	0.43	9	2.8	7.0	2.52	5
Black Shales	Água de Madeiros Fm	7.6	3.6	1.06	19	6.4	4.9	2.04	15
	Vale das Fontes Fm	10.1	4.2	0.25	5	3.7	4.7	1.66	5

Table 2 – Relationship between lithology and the radioactive elements concentrations obtained through field and laboratory measurements.
Tabla 2 – Relación entre la litología y las concentraciones de elementos radiactivos obtenidos mediante mediciones de campo y laboratorio.

equipment measures the character of approximately 0.3 m^3 of rock, the obtained value in the marl horizon will be depleted by the limestone beds with lower GR values. Another example is the Vale das Fontes Fm with big discrepancies between the U values obtained in the field and in the laboratory. Again, the lithological sequence seems to be the main reason. For instance, the Vale das Fontes Fm is mainly a marly sequence characterized by high GR values. The expected trend is to obtain super-estimated values. On the contrary, the Água de Madeiros Fm consists of marl-limestones alternations. The measurements performed in the marly horizons are frequently affected and depleted by the proximity of the limestone beds. Also the internal calibrations of both equipments are different. Nevertheless, we emphasize the similar curves/trends between field and laboratory data. These differences highlight the importance of the laboratory measurements performed in standardized conditions (resulting in significantly stronger correlations), and the restrictions input to the GR logging in wells and outcrops.

A correlation matrix for the radiometric, mineralogical and IR data measured in laboratory (except the GR) is shown in Table 3. The majority of the correlations are statistically significant (see Sachs, 1984). The total GR shows a good correlation with the calcite (r = 0.71) higher than with the carbonate fraction (r = 0.56) mainly because the few samples with dolomite disturb this relation. The radioactive elements, in particular Th and K, show a highly significant correlation between them (r = 0.87). These two elements are also strongly correlated with the mineral fraction, increasing together with the siliciclastic fraction and decreasing with the precipitation of carbonate confirming their tight relationship with a continental source materialized by clays, feldspar and micas (see Boyle, 1982). Based in highly significant correlations, the GR, U, Th and K values can be used as a proxy for the carbonate or siliciclastic content (see Pawellek and Aigner, 2004). However, in the studied interval, the U is the exception without any significant correlation with the different elements, due to the precipitation of authigenic U.

The carbonate and siliciclastic fractions show highly significant correlations between them (r = 0.85–0.96), except the mica where the decrease of the correlation quality is substantial. However, when the samples from the Água de Madeiros and Vale das Fontes formations are analysed and correlated separately, two different and highly significant relationships become clear (see the example between the mica and the Th in Fig. 9). This is a consequence of different sediment supply sources for the Água de Madeiros and Vale das Fontes formations and also independent depositional environments between S. Pedro de Moel and Peniche regions, previously sug-

r	Total GR	U_{lab}	Th_{lab}	K_{lab}	Carbonate	Calcite	Quartz	Mica	IR
Total GR	1								
U_{lab}	0,47	1							
Th_{lab}	0.56	0.02	1						
K_{lab}	0.64	0.20	0.87	1					
Carbonate	0.56	0.06	0.84	0.79	1				
Calcite	0.71	0.18	0.74	0.84	0.86	1			
Quartz	0.55	0.04	0.83	0.72	0.96	0.85	1		
Mica	0.50	0.18	0.57	0.49	0.71	0.64	0.53	1	
IR	0.55	0.20	0.65	0.78	0.62	0.75	0.69	0.30	1

Table 3 – Correlation matrix for radioactive elements determined by gamma-ray spectrometry (laboratory), total gamma-ray counts obtained in the field and other elements determined by whole rock XRD and geochemistry. At 99% confidence level a correlation coefficient of 0.386 (n = 44) is considered to be a significant result (Sachs, 1984).

Tabla 3 - Matriz de correlaciones de los elementos radiactivos determinados por espectrometría de rayos gamma (en laboratorio), el total de rayos gamma obtenidos en el campo y otros elementos determinados por difracción de rayos X y geoquímica. Para un nivel de confianza del 99%, un coeficiente de correlación de 0.386 (n = 44) es un resultado significativo (Sachs, 1984).

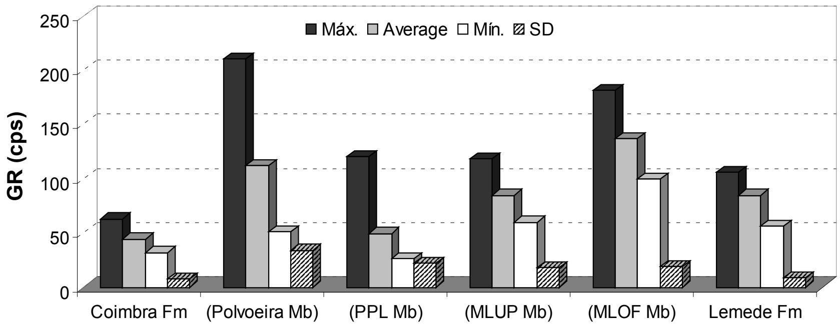

Fig. 7 – Maximum, minimum, average and standard deviation values of the total gamma-ray counts for each Formation/Member.

Fig. 7 – Valores máximos, mínimos, media y desviación estándar de los valores de rayos gamma obtenidos para cada Formación/Miembro.

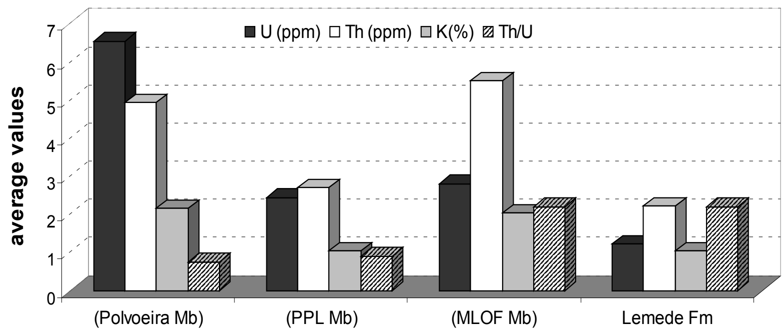

Fig. 8 – U, Th, K average content and Th/U average ratio for each Formation/Member.

Fig. 8 – Contenido promediado de U, Th y K y proporción promedio de Th/U para cada Formación/Miembro.

gested by Duarte et al. (2010). The less significant correlations between the siliciclastic fraction (specially the mica) and the Th and K observed in Peniche (r = 0.54 and 0.49 respectively) when compared to S. Pedro de Moel (r = 0.74 and 0.90), suggest different and intermittent sediment supplies from the eastern mainland and from the granitic Berlengas Horst to the west.

5.2 Sequence stratigraphy and subsurface data correlation

The studied interval corresponds to two 2nd order T-R facies cycles (SUS and SP in Duarte *et al.*, 2010; Duarte, 2007) and its correlation with the surface GR log is illustrated in the figure 10. In addition, two well logs were selected (14 A-1 and Alj-2) for further analysis and correlation with the surface data (see location in Fig. 1B). The GR spectrometry data collected at the surface allowed the subdivision of the lower Brenha Group (Witt, 1977) into four units (numbers 2, 3, 4 and 5, with the first one corresponding to the top of the Coimbra Fm), providing a better understanding of these data set (Fig. 10). The Brenha Group, described in the well reports, comprises a thick (> 1500 m) marine sequence deposited during the late Sinemurian–late Callovian time interval (see Alves *et al.*, 2002) which is subdivided into several equivalent units at the surface (Azerêdo, 2007; Azerêdo *et al.*, 2003; Duarte and Soares, 2002).

Starting from the top of the Coimbra Fm (unit 1) it is possible to observe a deepening-upward phase in the surface and subsurface GR logs, indicating a decrease in the energy of the depositional environment, linked with the development of a transgressive phase (Catuneanu, 2006; Pawellek and Aigner, 2003), that continues through a great part of the Polvoeira Mb (unit 2). The Maximum Flooding Interval (MFI) of the upper Sinemurian T-R facies cycle occurs around the 30 m of the Polvoeira section, where the high GR values (180 to 190 cps) coincide with high Th and K content, indicating maximum condensation (minimum sand input) (Fig. 4 and 10). This interval is also coincident with expressive thick black shale layers very rich in organic matter (TOC > 10% and high U). Using only total GR and U can be risky because these parameters indicate anoxia, not necessarily sea level change or condensation. This GR peak can be also correlated with the 2350 m deep of the 14 A-1 well. From this point, the regressive phase of the upper Sinemurian T-R facies cycle is characterized by the gradual decrease of the GR, suggesting an increased availability of oxygen in the depositional system, or, as suggested by Catuneanu (2006), a gradual increase in the energy. After the abrupt GR-decrease between the Polvoeira and PPL members (limit between unit 2 and 3), the GR assumes a typical blocky trend associated with a well stratified limestone sequence. This trend suggests constant (and probably low) energy levels during the sedimentation (Catuneanu, 2006; Pawellek and Aigner, 2003), although higher than in the Polvoeira Mb.

The increase of the GR and radioactive elements content starting from the 22 m of the Àgua de Madeiros log

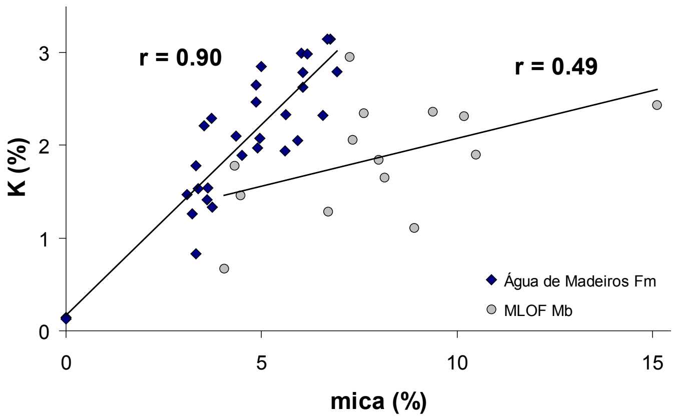

Fig. 9 – K_{lab}/mica crossplot with separated correlations for the Água de Madeiros and Vale das Fontes formations.

Fig. 9 – Gráfico mostrando la relación K_{lab}/mica con distintas correlaciones para las formaciones de Água de Madeiros e Vale das Fontes.

(Fig. 5 and 10) dated from the uppermost Sinemurian indicates the base of the Pliensbachian T-R cycle. This trend continues in the few surface measurements done in the MLUP Mb of the Vale das Fontes Fm. In the well data the deepening-upwards trend is well expressed. The transgressive phase corresponds to almost the entire Vale das Fontes Fm (unit 4) as suggested by Duarte *et al.* (2010), and is also observed in the two well logs. At Peniche, Parkinson (1996) suggests two cycles of upwards-increase in clay content during the Pliensbachian interval, with the most clay-rich sediments being found at the base of the Ibex Zone and at the top of the Margaritatus Zone. A similar pattern is also observed in the GR well logs, even if not so pronounced, with the limit between the two cycles of upwards-increase being found at 2260 and 2615 m of the 14 A-1 and Alj-2 wells respectively. The MFI of this T-R facies cycle is observed at 14 m of the Portinho da Areia do Norte log, where the highest GR, U, Th and K values were obtained (Fig. 6 and 10). This interval corresponds to the last black shale package coincident with a TOC peak of 10% (see also Oliveira *et al.*, 2006; Silva *et al.*, 2011 and references therein). In subsurface, the correlative GR peak occurs at the 2185 m and 2555 m of the 14 A-1 and Alj-2 wells, respectively. From this point onwards, all the determined parameters start to decrease, characteristic of the regressive phase that will last until the extreme base of the Toarcian. This phase, mainly coincides with the deposition of the Lemede Fm (unit 5), which is characterized by a regular radiometric background (70 to 90 cps), represented by well stratified limestone beds.

Besides the 2nd order T-R facies cycles, 3rd order cycles could be also tentatively identified along the studied sections. For example, the GR peaks obtained at 14 m of the Polvoeira log (figure 4) and between the 3–4 m of the Portinho da Areia do Norte section (figure 6) probably correspond to 3rd order MFI. However, for a more detailed analysis of these 3rd order cycles, a dataset with a higher resolution and a better understanding of the lateral facies variation and bathymetric changes across the basin is needed.

5.3. The uranium as a proxy for the total organic carbon content

The studied interval (upper Sinemurian–Pliensbachian), is currently regarded as one of main potential source rocks of the Lusitanian Basin. Based on its geochemical characteristics this thick marl-limestone sequence has a great potential for hydrocarbons generation. However, thermal maturation studies concluded that in these regions and in outcrop, the organic matter is immature or very early mature (Duarte *et al.*, 2011b, 2012; ; Silva *et al.*, 2011; Ferreira *et al.*, 2010; Oliveira *et al.*, 2006). Nevertheless, in the offshore sectors of the Lusitanian Basin and in the Peniche Basin (deep offshore) the petroleum system may work because the studied interval is buried more deeply.

The TOC curve shows a similar pattern to the U content (Fig. 10). The Polvoeira Mb at S. Pedro de Moel is characterized by the highest average TOC (7%) and U (6.5 ppm) values while the MLOF Mb analysed in Peniche, represents the second most important organic rich section (TOC - 3% and U - 3.3 ppm).

The U peaks of the entire interval are in part decoupled from the Th and K curves. It is also possible to observe that the Th/U ratio drops below 2.2, particularly in the Polvoeira Mb where this ratio is 0.8 (Fig. 8). After applying the formula introduced by Wignall (1994) to the U content measured mainly in marl and black shale levels, it is evident that the authigenic U assumes an important percentage in the total U content. In the Água de Madeiros Fm at S. Pedro de Moel, the authigenic U is the main component of U (4.8 ppm of authigenic U and 1.7 ppm of detrital U). In the Vale das Fontes Fm at Peniche, the detrital U persists as the main component (1.9 ppm of detrital U against 1.3 ppm of authigenic U). The reduction and precipitation of authigenic U, especially in the Água de Madeiros Fm, is responsible for the significant increase of the total U content in the sediments, being also the main reason for the increasing total GR counts (r = 0.77 in Table 1). Several authors (e.g., Bodin *et al.*, 2011; Lüning and Kolonic, 2003; Wignall and Myers, 1988) assume that the precipitation of authigenic U suggests that oxygen depleted conditions prevailed during the time of deposition. This constitutes one more indicator of a possible deep depositional environment that leads

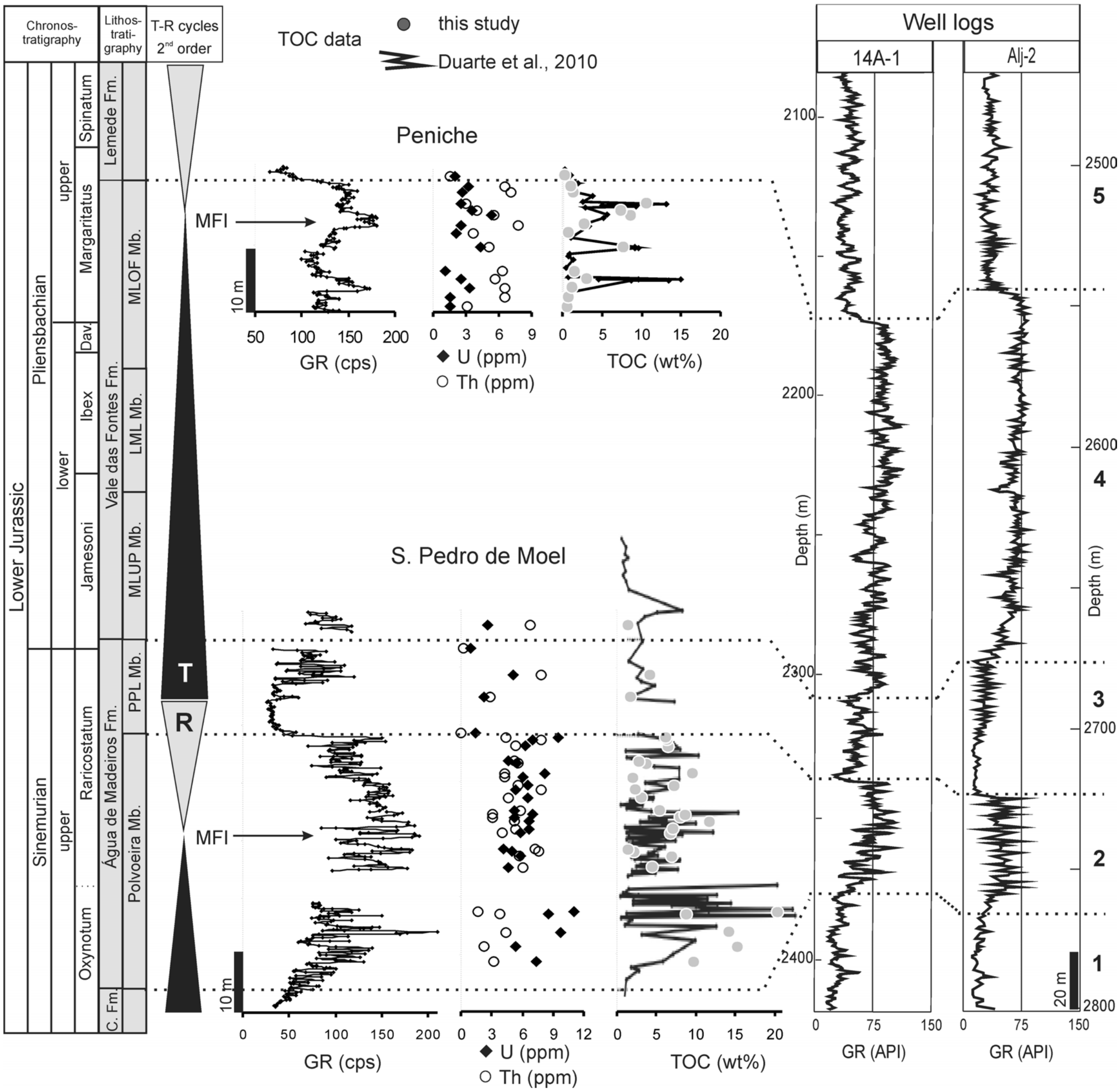

Fig. 10 – Comparison of the total gamma-ray, U and TOC content, 2nd order T-R facies cycles and partial gamma-ray well logs (14 A-1 and Alj-2). The dashed lines represent the stratigraphic limits of each unit.

Fig. 10 – Comparación de los rayos gamma, U y el contenido de COT, ciclos T-R de 2º orden y perfiles parciales de rayos gamma de dos pozos (14 A-1 y Alj-2). Las líneas discontinuas representan los limites estratigráficos de cada unidad.

to an oxygen depleted period, more severe during the upper Sinemurian in the S. Pedro de Moel region than in the Pliensbachian of Peniche.

In table 4, the correlation coefficients between the total U, authigenic U, Th and the TOC are listed. In the Agua de Madeiros Fm and MLOF Mb it is noticiable a strong U_{total}/TOC correlation ($r = 0.72$ and $r = 0.64$ respectively). Specifically in the Polvoeira Mb, the Th/TOC ratio also shows a significant correlation ($r = 0.72$, Fig. 11B). In the 44 rock samples analysed in both sections the correlation is highly significant ($r = 0.72$) (see Correia *et al.*, 2011). If only the calculated authigenic U is taken into account, the correlation $U_{authigenic}$/TOC becomes stronger, confirming the preferred precipitation of the authigenic U with the organic matter (Lüning and Kolonic, 2003; Wignall and Myers, 1988). In Figure 11A, it is possible to observe three samples with anomalous U/TOC relationships that diminish the quality of the correlations. If these values are excluded, the quality of the $U_{authigenic}$/TOC correlation increases significantly ($r = 0.88$). The XRD analyses of the mineral fractions show that both samples are characterized by their high content in carbonate, which Lüning and Kolonic (2003) refers as one of the main disturbing factors in the U/TOC correlations.

In both sections, the U/TOC ratios are low (Fig. 11A), although slightly lower in the Vale das Fontes Fm (0.3 ppm/wt.%TOC, Fig. 11D) when compared with the same ratio in the Água de Madeiros Fm (0.7 ppm/wt.%TOC, Fig. 11C). These results are similar to others obtained by different authors (compiled by Lüning and Kolonic, 2003) in formations dated from the Lower Jurassic of Western Europe. These low ratios are consistent with

Formations	U_{total}/TOC r	$U_{authigenic}$/TOC r	Th/TOC r	N
All	0.72	0.79	0.60	44
Agua de Madeiros Fm	0.72	0.79	0.64	29
Vale das Fontes Fm	0.64	0.80	0.45	15

Table 4 – U_{total}/TOC, $U_{authigenic}$/TOC and Th/TOC correlations obtained for the entire studied interval and for the Agua de Madeiros and Vale das Fontes formations separately. At 99% confidence level a correlation coefficient of 0.386 (n = 44), 0.471 (n = 29) and 0.641 (n = 15) is considered to be a significant result (Sachs, 1984).

Tabla 4 – Correlaciones U_{total}/COT, $U_{autigénico}$/COT y Th/COT obtenidas para todo el intervalo estudiado y para las formaciones de Água de Madeiros y Vale das Fontes por separado. Para un nivel de confianza del 99%, coeficientes de correlación de 0.386 (n = 44), 0.471 (n = 29) y 0.641 (n = 15) son resultados significativos (Sachs, 1984).

carbonate-dominated systems, but also with the thermal immaturity of the source rocks as suggested by Lüning *et al.* (2005).

6. Conclusions

1. The GR spectrometry performed in the laboratory had a great importance in all correlations established in this study due to their accuracy. Even so, the portable equipment is a quick, simple and low cost technique, with multiple applications.

2. The spectral GR logs show great similarity to the observed sedimentological changes in the studied units. The Th and K values show a strong correlation with the siliciclastic fraction. In the particular case of the mica, the correlation indicates different sediment supply sources between S. Pedro de Moel and Peniche. During the Pliensbachian, the influence of the Berlengas Horst was already present in the Peniche sectors.

3. The outcrop gamma-ray techniques are in good agreement with the Sinemurian–Pliensbachian T-R cycles helping in the identification of maximum flooding intervals. After the characterization of the radiometric signature of each unit and lithotype, these techniques are also a powerful tool to integrate subsurface and surface geology, making these techniques an important tool in oil exploration.

4. Part of the Late Sinemurian–Pliensbachian interval of the Lusitanian Basin, namely the Polvoeira and the MLOF members, have a high potential as a source rock. This is in good agreement with the high estimated contents of authigenic U (average Th/U ratio of 0.8 and 2.2, respectively) and TOC (average values of 7 wt.%TOC

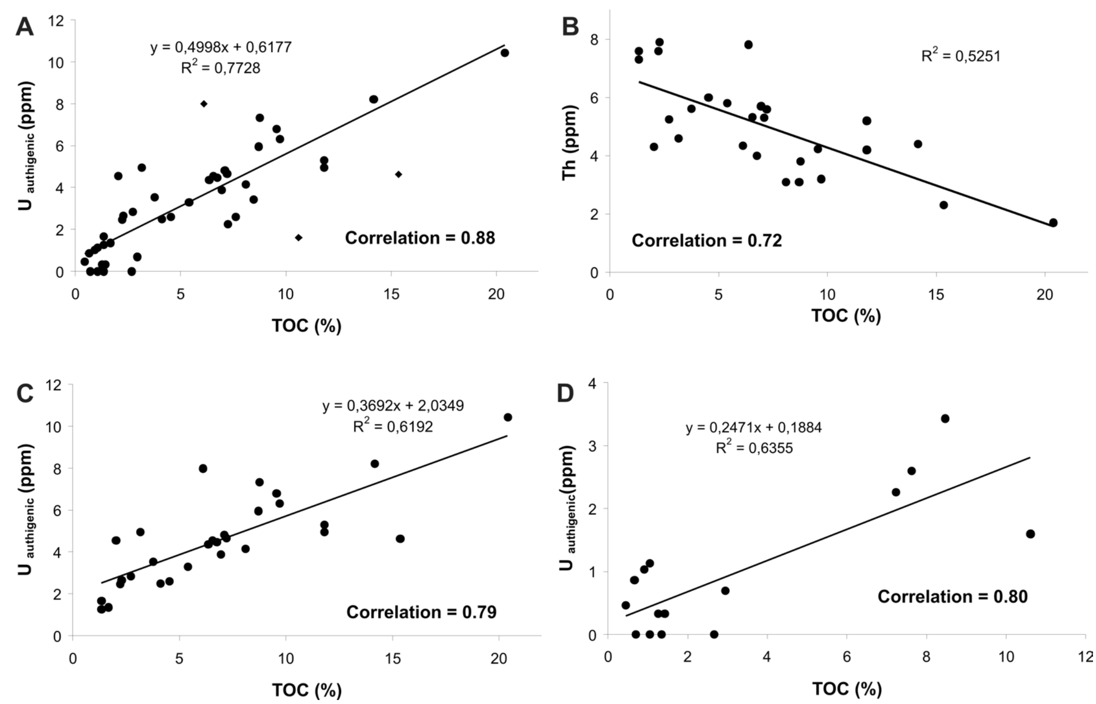

Fig. 11 – Correlations determined by GR spectrometry. At 99% confidence level a correlation coefficient of 0.393 (n=44), 0.471 (n=29) and 0.641 (n=15) is considered to be significant (Sachs, 1984). A) $U_{authigenic}$/TOC crossplot of selected samples from the Água de Madeiros and Vale das Fontes formations, excluding the samples with the highest carbonate content; B) Th/TOC crossplot of selected samples from the Polvoeira Mb. C) $U_{authigenic}$/TOC crossplot from the Água de Madeiros Fm (n=29). D) $U_{authigenic}$/TOC crossplot from the Vale das Fontes Fm (n=15)

Fig. 11 – Correlaciones obtenidas por espectrometría de rayos gamma. Un coeficiente de correlación de 0.393 (n=44), 0.471 (n=29) y 0.641 (n=15) se considera que es significativo para un nivel de confianza del 99% (Sachs, 1984). A) $U_{autigénico}$/COT de las muestras seleccionadas de las formaciones de Água de Madeiros y Vale das Fontes; B) Th/TOC de las muestras seleccionadas del Mb de Polvoeira; C) $U_{autigénico}$/COT de la Fm de Água de Madeiros; D) $U_{autigénico}$/COT de la Fm de Vale das Fontes (n=15).

and 3 wt.%TOC, respectively), suggesting the deposition under oxygen depleted conditions.

5. For the entire studied interval a highly significant U/TOC correlation (r = 0.72) was obtained allowing the U content to be used as a proxy to predict the TOC in these sectors of the Lusitanian Basin. Time and resources consuming geochemical analysis can be replaced using indirect measurement of the U by GR spectrometry, not only in these outcrops, but in other sedimentary basins where similar depositional systems occur. The quality of the correlation increases (r = 0.79) if only the authigenic U component is considered. It was also confirmed that the high carbonate content can interfere with the development of a stable U/TOC relationship.

6. The U/TOC ratio of the studied samples is low (0.3–0.7 ppm/wt.%TOC), similarly to other case studies of Western Europe basins. The low U/TOC ratios are consistent with Jurassic carbonate systems, but also with the thermal immaturity of the source rocks.

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