# When the Syn-Depositional Climatic Variations Influence My Source Rock Properties – The Case Study of the Vaca Muerta*

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## Abstract

The Vaca Muerta Formation is a world class source rock developed in the Neuquén Basin of Argentina at the Jurassic Cretaceous boundary. Several continuous high-resolution acquisitions were performed on the Vaca Muerta Formation in order to thoroughly characterize this heterogeneous source rock.

Core spectral gamma-ray and density as well as LIPS (Laser Induced Pyrolysis System for TOC acquisitions at a resolution of one centimeter) were acquired over complete sections of cores. This set of data enabled a cyclostratigraphy study that revealed the control of orbital forcing on the TOC quantity and quality of the source rock. Imbricated sequences from 1.2 Myr down to 20 Kyr (around a meter thick) were successfully defined on many wells where this set of data was acquired.

Besides these previous acquisitions, automated micro-XRF spectrometer and Automated Fourier Transform Infra-Red spectroscopy (FTIR) were tested on portions of cores for high resolution continuous mineralogical determinations. An impulse hammer probe was also used alongside the FTIR for elastic stiffness determination.

The results show that all rock properties vary along with the paleo-climatic variations, independently of the cycle duration. In general, the carbonate content is negatively correlated with the silica and clay contents. Next to Maximum Flooding Surfaces, the TOC is the highest whereas the calcite content is the lowest. The corresponding source rock facies is a mudstone with few radiolarians. During subsequent progradational periods (or HST), the TOC is gradually decreasing whereas the calcite is increasing until the Transgressive Surface is reached. The mudstones become wackestones incorporating gradually more radiolarians, bioclasts or also develop cementation (nodules). During

transgressive periods (TST), the inverse variations are observed with increasing TOC and decreasing carbonate content. As a consequence, the Brittleness Index (BRI) computed from these high-resolution mineralogical acquisitions also regularly varies with the sequence stratigraphy. Vertical trends independently obtained with the Impulse Hammer exhibit strong similarities with this BRI. The integration of all this data set helps us to derive a detailed geological model of the Vaca Muerta along with all its heterogeneities, facies, and associated properties.

## Introduction

The Vaca Muerta Formation is a world class source rock with average present-day TOC of at least 5% and initial Hydrogen Index (HI) values rather high for a marine source rock, above 650 mg HC/g TOC. This productive source rock is deposited in the Neuquén Basin, a back-arc basin during Late Jurassic to Early Cretaceous times (Figure 1). The Vaca Muerta Formation is characterized by well-defined northwestwards prograding and retrograding sequences filling the basin during a period of high subsidence rate (Pose et al., 2014 and Raijenstein et al., 2015). Whereas the organic rich source rock, referred to as the Vaca Muerta Formation, accumulates in the bottomsets of these sequences, the Quintuco and Mulichinco Formations, which are mixed carbonated siliciclastic deposits, accumulate in the foresets and topsets.

Currently, the Neuquén Basin is a foreland basin, and, to the east, the Vaca Muerta Formation is explored and is being produced for its shale gas and oil resources. Total has acquired numerous cores from vertical exploration wells representative of all stratigraphic sequences and all maturity ranges, from the oil to dry gas windows. About 500 m of cores, ranging from 36 m to 160 m in thickness, have been thoroughly studied with different innovative techniques. The acquisitions performed on these cores can be split between continuous (or semi-continuous) and discontinuous (i.e., punctual ones). Continuous measurements were performed ahead of the discontinuous ones and preferentially ahead of the core slabbing. They usually comprise core logs (spectral gamma-rays and density), CT-scans and scratch tests for geomechanical studies. For gas wells only, they also comprise a continuous TOC measurement with the LIPS method (Laser Induced Pyrolysis System; Elias et al., 2013) at a resolution of 1 cm spacing. For oil or gas condensate cores, standard Rock-Eval were preferred and were performed on homogenized rock powder obtained every meter after the scratch test analyses. This method enables TOC profiles to be compatible with log scale resolution. Once these continuous measurements are done, cores are sampled for various punctual measurements such as Quantitative Mineralogy or also Quantax or Qemscan mineralogical mappings (Fialips et al., 2013). Porosity and permeability measurements, geomechanical testing (such as tri-axial tests or Brinnel Hardness for proppant embedment tests) and characterization of the pore network with FIB-SEM acquisitions are also systematically performed. Once the core was slabbed, a sedimentological description was performed based on micro-facies previously defined on thin sections.

The Vaca Muerta Formation consists of black, organic rich mudstones that are characterized by a variable content in radiolarians that drive their mineralogical compositions and their geomechanical properties (Mathieu et al., 2016). Other facies are characterized by their allochem content, such as crinoïds and bioclasts. A simplified facies model is shown on Figure 2, along with their mineralogical composition. Two main opposite trends are observed: in one hand, the carbonate and radiolarian contents increase towards the slope of the basin and in another hand, the TOC, clays, and silicates globally increase towards the basin's center. On top of these depositional variations, the Vaca Muerta Formation is punctuated by thin, frequent heterogeneities accounting for at least 18% of the entire Vaca Muerta. These heterogeneities include different types of ash beds, clay rich or calcite cemented (Rutman et al., 2017), cemented concretions or nodules, calcitic microbialites and bed parallel veins filled by fibrous calcite or beef (Lejay et al., 2017). However, despite all these acquisitions, it was still unsure that the sampling plan was representative of all the various heterogeneities encountered in the Vaca Muerta Formation, the main goal being the prognosis of source rock properties from existing proxies (such as TOC, core density, sedimentological descriptions …).

In order to answer this question, different studies, among them a study of the Milankovitch astronomical cycles aiming at understanding the apparent cyclic recurrence of some of the properties such as TOC, were launched. Cyclic variability of TOC was shown by the initial study, which suggested that, maybe, orbital forcing could also influence other properties such as facies, mineralogy, or geomechanical parameters. On this regard, a semi-continuous measurement of mineralogy and of a geomechanical parameter on an automated bench named Autoscan and provided by NER (New England Research, Boitnott et al., 2018) was tested. These acquisitions are respectively the Fourier Transformed Infra-Red spectroscopy (FTIR) and Impulse Hammer (IH). Whereas the FTIR spectroscopy provides the simplified mineralogical composition of rocks after calibration, the IH provides an elastic stiffness E* commonly referred to as the reduced Young's Modulus. These data were tested on 15 meters of cores at a resolution of 5 mm.

## Cyclostratigraphy of the Vaca Muerta Formation

The first studies linking high resolution sequences with orbital forcing were performed by Scasso et al. (2005) based on $\delta^{13}C$ variations interpreted as being influenced by long eccentricity climatic variations (405 Kyr). From different outcrops descriptions of the Neuquen Basin, Kietzmann et al. (2015) also showed the role of orbital forcing in Vaca Muerta Formation sequence stratigraphy. These results are consistent with magnetostratigraphy studies. More recently, also based on paleomagnetism, a new cyclostratigraphy study performed on proximal deposits of the Vaca Muerta Formation has shown the role of astronomic cycles in sequence architecture (Kohan Martinez et al., 2017).

Regarding the analysis of Milankovitch cycles from Total subsurface data, Martinez (2014) has developed a methodology to show the role of orbital forcing in the Vaca Muerta sedimentation, however from wireline logs and not from high resolution semi-continuous core data as in this present case study.

Our analysis of orbitally driven sequences in the Vaca Muerta is based on several parameters, usually high resolution, most of them being semi-continuously acquired on cores. These parameters are LIPS or "Laser Induced Pyrolysis System" (Elias et al., 2013) for a measurement of TOC every 0.01 m, the core density with a 0.01 m spacing, the spectral core gamma-rays (U, TH, and K) with a 0.08 m spacing. Other parameters were also successfully tested (Monkenbusch et al., 2017) and are issued from Rock-Eval Pyrolysis such the HI or Hydrogen Index which reflects the quality of the source rock. In this case, the resolution is lower, varying from 0.3 m to 1 m regular spacing, depending of the studied well.

The method to identify sedimentary cycles as Milankovitch cycles is a spectral analysis, based on the Fourier Transform, computed using the Astrochron Package in the R scripting language (R core Team, Meyers, 2014). The analysis is targeting periods from Waltham (2015).

All signals are detrended using a LOWESS smoothing (Cleveland, 1981). The power spectral analysis on all signals was performed using the multi taper method (MTM; Thomson, 1990) with red noises modeled for each signal for the calculation of spectral peak significance (Figure

3A). To identify variabilities in sedimentation rate, evolutionary fast Fourier transform (FFT) spectrograms were calculated. The period ratio was used to identify links between observed sedimentary cyclicities and astronomical forcing. Taner low pass filters were designed to filter out low frequencies to unmask significant high-frequency peaks and Taner bandpass filters were constructed to extract frequency bands that were linked to astronomical forcing (Taner, 2000). The *spectrum* of Figure 3A highlights 405 Kyr cycles (long eccentricity) with an average thickness of 18 m, 100 Kyr cycles (short eccentricity) with a thickness varying between 3.6 and 7 m, 40 Kyr cycles (obliquity) and also 20 Kyr cycles (precession) with an average thickness of about 1m. For each well, all studied parameters were tested in order to check the consistency of the results. On Figure 3B, the same well is shown along with the high-resolution TOC (LIPS) and the resulting low pass filters, from 400 Kyr and its modulation at 1200 Kyr down to the 20 Kyr filter. As already shown by previous studies (Kietzmann et al., 2016), for low frequency sequences and especially by Gonzalez Tomassini et al. (2016) but demonstrated in this study at any order of sequences, the TOC is maximum close to the Maximum Flooding Surface and decreases toward the Transgressive Surface where it is minimal.

This study on cyclostratigraphy, mainly performed from organic matter proxies, lead us to build a stratigraphic framework tested between studied wells and matching the Vaca Muerta stratigraphy as published in the regional Rosetta stone stratigraphic chart (Gonzalez Tomassini et al., 2016).

## Fourier Transform Infra-Red Spectroscopy (FTIR) and Impulse Hammer (IH) Acquisition

Both FTIR and IH probes are part of an automated petrophysical scanner also named Autoscan bench (Figure 4) and set up by NER (Boitnott et al., 2017.

As described by Boitnott et al. (2018), the FTIR spectrometer is configured as a non-contacting probe measuring the Infra-Red Reflectance over a broad spectral range. Each measurement with the FTIR results in the acquisition of an absorbance spectrum, the interpretation of which requires careful calibrations and modeling. In this study, the resulting mineralogical interpretation corresponds to a mixture of three mineralogical poles: Quartz, Carbonates, and Clays.

The Impulse Hammer probe (or IH) was originally designed to provide a nondestructive, semi-continuous mechanical profile along a core, at the very same depth as the FTIR acquisition (Boitnott et al., 2017). The IH primary output is the elastic stiffness E*, commonly referred to as the reduced Young's Modulus. This measurement was conducted using a sensor head with small mass (~60 g), a medium tip curvature (radius 3.8 mm), and a low drop height (12.7 mm). The measurement attributes are chosen to stay within the elastic regime of the rock.

The results of both FTIR and IH acquisitions performed over nearly 15 m of Vaca Muerta cores are summarized in Figure 5 and illustrate the very high frequency variations of the source rock composition (TOC, carbonates, silicates, and clays) as well as of the geomechanical properties. On the first track, we have displayed the high-resolution TOC profile from the LIPS technique, which the data set the Milankovitch analysis was performed. On tracks 2 to 4, we have displayed the mineralogical results (raw and smoothed results) along with the Quantitative Mineralogy results. We observe a very good fit between the two data sets meaning that the FTIR interpretation is reliable. Comparing tracks 1 and 2, it looks as though the carbonates vary vertically inversely to the TOC variations. We also observe inverse vertical evolutions between carbonates on the one hand and silicates and clays on the other. The result of the Impulse Hammer is shown on the 6th track and, for comparison purposes, we have also plotted a mineralogical Brittleness Index (BRI) on track 5. The TOC from the LIPS has been added to the sum of the three analyzed mineralogical poles and scaled to 100%; the mineralogical BRI corresponds to the sum of Carbonates and Silicates over the Total mineralogical composition including the TOC. This BRI, computed with FTIR data, matches well the BRI computed from the Quantitative Mineralogy data (red dots). We observe, in most of the studied sections, similar vertical trends between the two data sets, i.e., the Impulse Hammer elastic stiffness follows the variations of BRI derived from the hardness of the rock and apparently mostly linked to the carbonate variations. In the last column, we have displayed the sedimentological facies.

In Figure 6A, a carbonate vs TOC cross plot illustrates an inverse correlation with a poor correlation coefficient (less than 0.5). We also note that radiolarian rich facies in blue are characterized on this plot by overall lower TOC and higher carbonate content compared to the other facies. In Figure 6B, the inverse correlation between carbonates and silicates is much clearer with a correlation coefficient of 0.8. The facies breakdown is also characteristic with the clear distinction of nodules (turquoise), the radiolarian and carbonate rich facies (dark blue) and all other facies poorer in radiolarians. The plot of Figure 6C illustrates the positive correlation between clays and silicates, showing that, despite existing quartz cement, they reflect terrigenous influxes, along with the organic matter.

## Cyclicity of the Vaca Muerta Formation Properties

All these high frequency variations of the Vaca Muerta Formation properties are compared with the existing stratigraphic layering interpreted from the Milankovitch cycle study detailed here above. On Figure 7, starting to the left, the 405 Kyr sequences fit a smoothed TOC profile with high TOC value close to Maximum Flooding Surfaces (MFS), decreasing towards the top of the progradations (up to the Transgressive Surfaces or TS during Highstand System Tracts) and increasing again above towards the next MFS. Such sequences are about 20 to 40 m thick, depending of the stratigraphic location within the well. When zooming in towards the right of Figure 7, the 405 Kyr sequences are composed of 100 Kyr sequences, the thickness of these vary from about 5 to 10 m. The same type of variations of TOC is clearly observed at this resolution. At the scale of the 20 Kyr sequences, further right of the figure, one gets to the 15 meters studied interval with FTIR and IH but here, we focus at the lower half of this interval. The thickness of the 20 Kyr sequences is in the range of 1 m. And again, at this scale, one clearly observes TOC variations following a similar tendency with the highest values close to MFS and lowest values close to TS. Similarly, and in agreement with the cross-correlations between source rock properties (Figure 6), one observes also cyclic variations of all mineralogical components that fit within the existing stratigraphic layering. At this scale of observation, the inverse relationship between TOC and carbonate is very clear. Similarly, the inverse correlation between carbonates and silicates and clays is also well expressed. Some of these relationships between mineralogical components were already observed by previous authors, such as Gonzalez Tomassini et al. (2016) for instance, but based on acquisitions with a lower sample spacing. At this same scale of observation, the mineralogical Brittleness Index (BRI) and Impulse Hammer (IH) results also fit with these observations: close to MFS, the BRI and IH are minimal, they increase towards the TS where they are maximal. Therefore, one observes a kind of fractal pattern of source rock properties, including the geomechanical properties, which vary at all scales from the 20 Kyr (about 1 m) up to the 405 Kyr sequences (several tens of meters). This cyclostratigraphy analysis also shows the accuracy of macro-facies description which evolve accordingly with the defined stacking pattern. This statement sounds evident; however, one has to realize that the sedimentary facies in this sedimentary setting were first defined from sparse thin sections before being propagated over the entire cores, implying a necessary visual and sometimes difficult interpolation between the defined micro- facies. In complete agreement with the simplified model of Figure 2, facies 2, the TOC richest is mainly found close to the MFS, whereas, facies 4 and 8, poorer facies in TOC, are mainly found associated with the TS. These two facies are also richer in carbonates. Some particular facies, such as facies 6, the TOC richest mudstone with micro-beef (i.e. mm thick discontinuous fibrous calcitic veins), seems to be mainly found within Transgressive System Tracts.

## Conclusions

The high-resolution acquisition of mineralogy and of one geomechanical parameter enables assessment of the highly heterogeneous nature of the Vaca Muerta Formation source rock. However, based on an existing study of Milankovitch cycles performed from high resolution TOC acquisitions (LIPS), it was observed that these high frequency variations of mineralogy and geomechanical properties were also related to the existing stacking pattern summarized in Figure 8. These cyclic variations are influenced by the variations of the earth orbital parameters known as the 20, 40, 100 and 405 Kyr cycles. These cycles influence the global climate as well as the global sea level variations (eustatism) of the planet. The cyclic variations of these parameters influence the succession of facies and thus the succession of properties such as the mineralogical contents and therefore the geomechanical properties. Regarding the effects on the TOC, the initial study on cyclostratigraphy (Monkenbusch et al., 2017) has shown that, not only the astronomical cycles influence the quantity of TOC, but also the quality of the organic matter as shown by the cyclic fluctuations of the Hydrogen Index (HI from Rock-Eval measurements).

All these observations will help us build refined geological models at the scale of a production pad for example. Based on a high-resolution layering and based on the evidence of relationships between various parameters of the source rock, we are able to predict vertical trends of the source rock properties.

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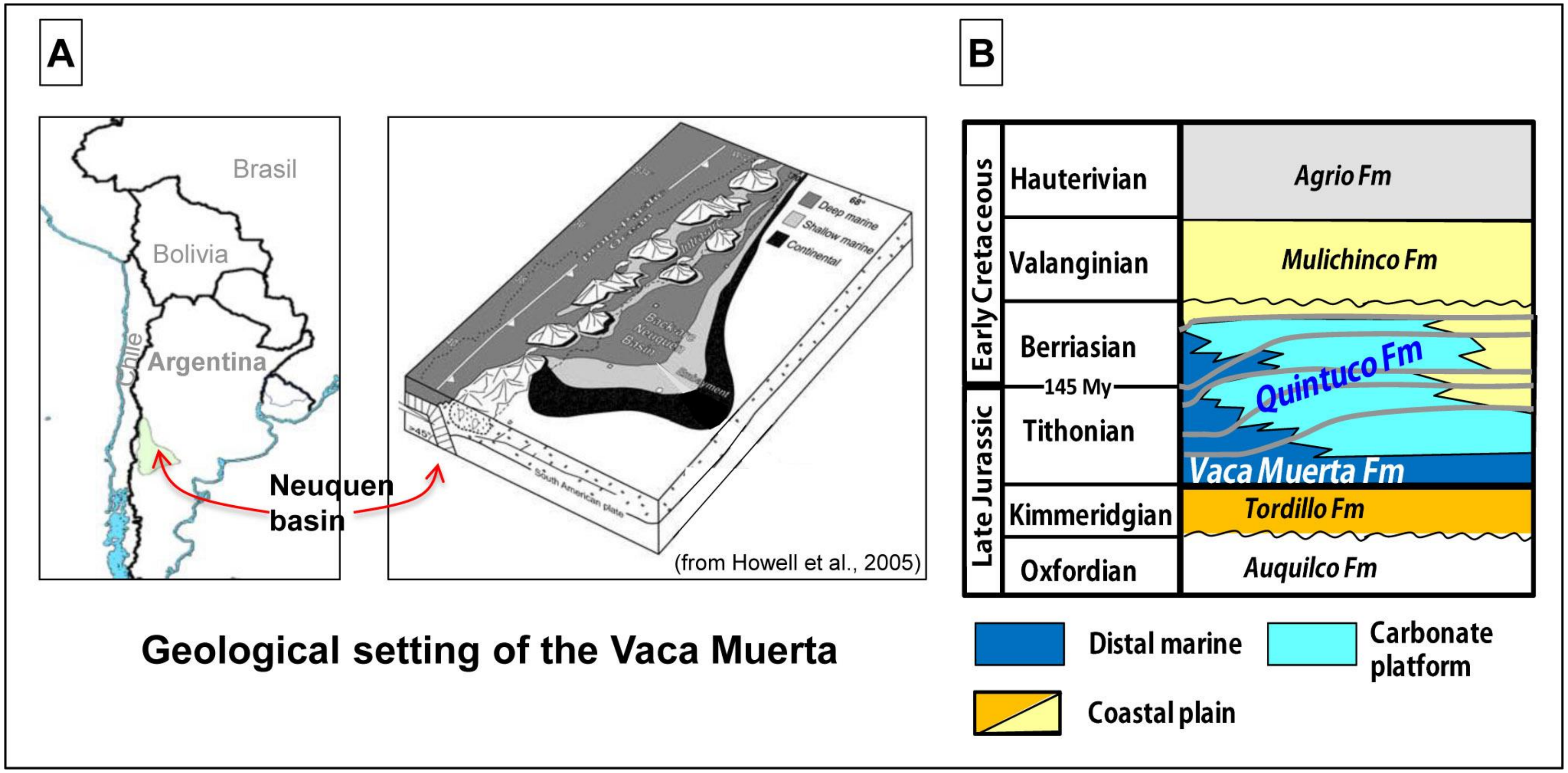


Figure 1. A) Location map of the Neuquen Basin, a back-arc basin during the deposition of the Vaca Muerta. B) Stratigraphy of the Vaca Muerta Formation source rock dated Tithononian-Berriasian and deposited in the distal setting of the Quintuco Formation platform.

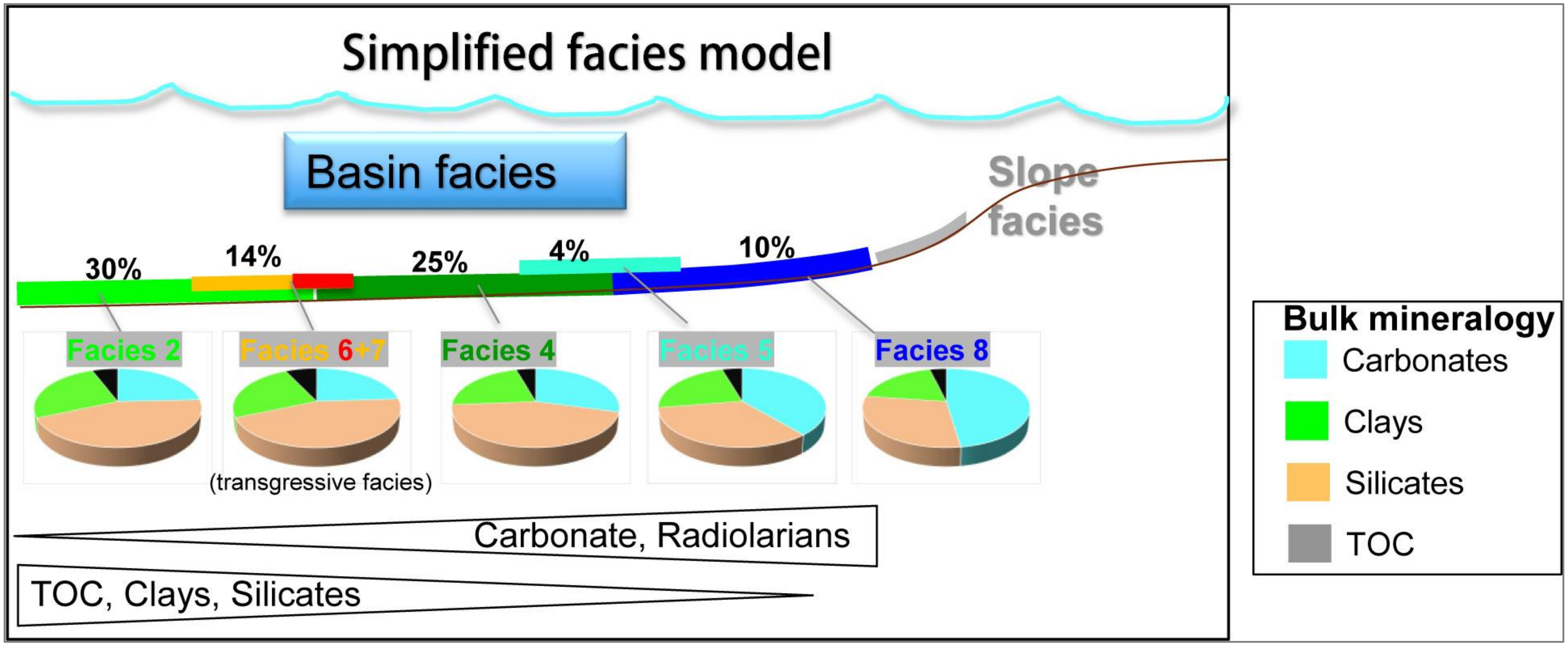


Figure 2. This simplified facies model shows the main matrix facies of the Vaca Muerta with their relative proportion. Their mineralogical content is summarized by pie charts. Two opposite trends are observed. In one hand, the carbonates and radiolarians content increase from the basin's center towards the slope of the basin. In another hand, the TOC, clays, and silicates increase towards the basin's center.

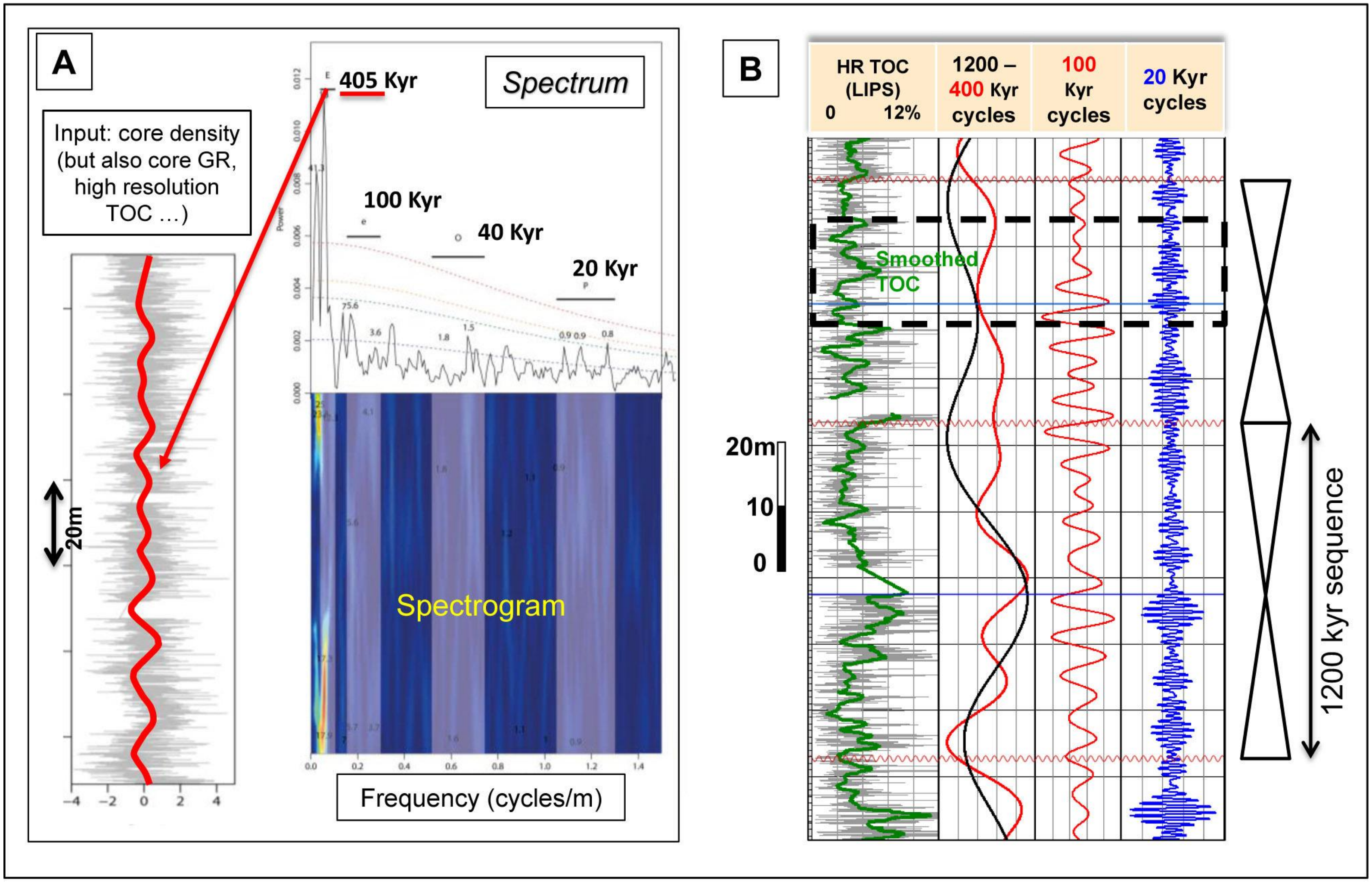


Figure 3. Principles and results of the Milankovitch cycles analysis. A) The spectral analysis consists in detrending the raw data, performing a spectral analysis which is the MTM (Multi taper method) and evolutive spectra, calculating and comparing the ratios and in testing using amplitude modulations. B) Example of resulting filters on a complete section of Vaca Muerta based on LIPS curve (« Laser Induced Pyrolysis System », 1 cm vertical resolution). The dashed rectangle indicates the 15 m interval studied in detail with FTIR (Fourier Transform Infra-Red) and IH (Impulse Hammer).

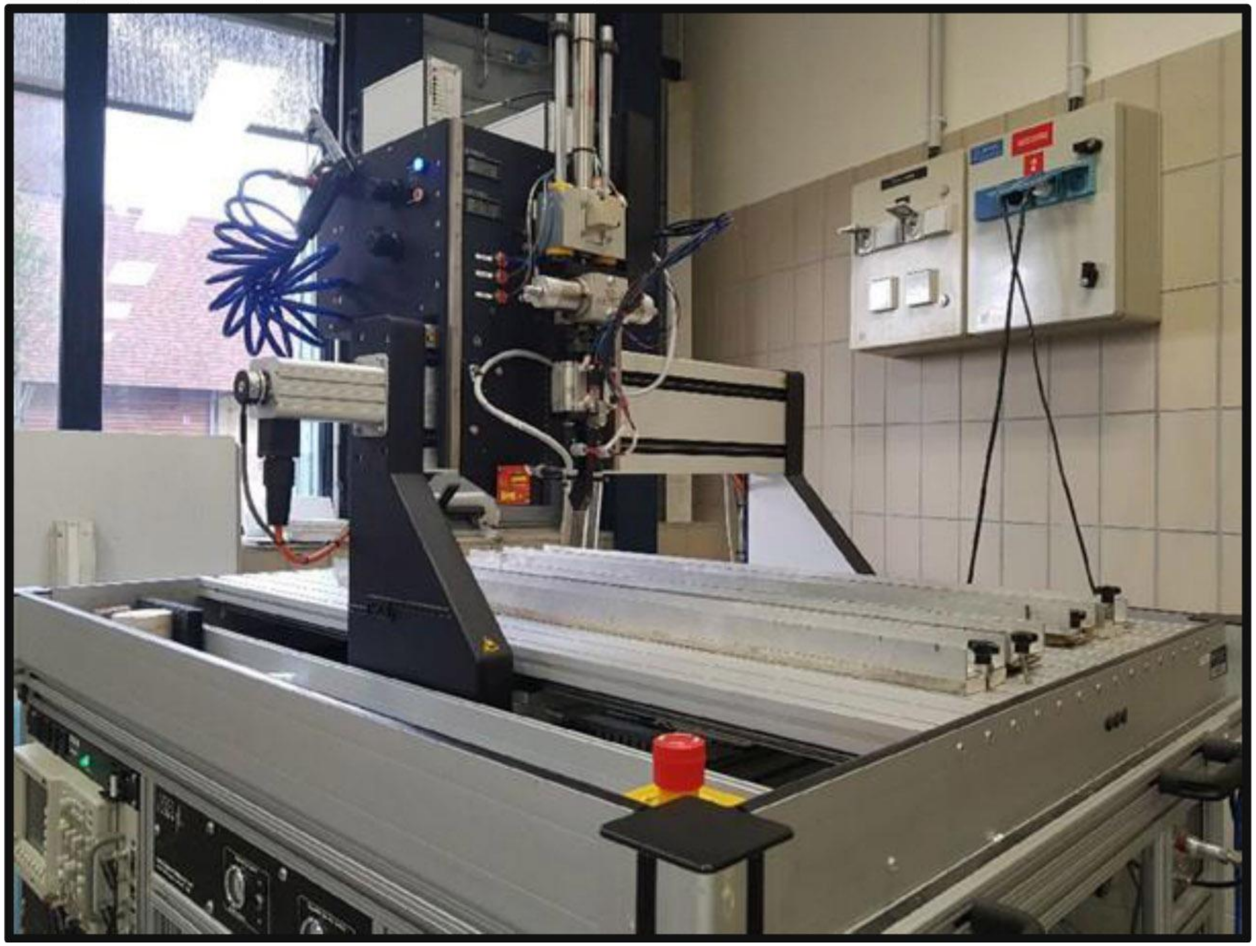

Figure 4. Picture of the automated Autoscan bench set up with the FTIR (Fourier Transform Infra-Red) probe.

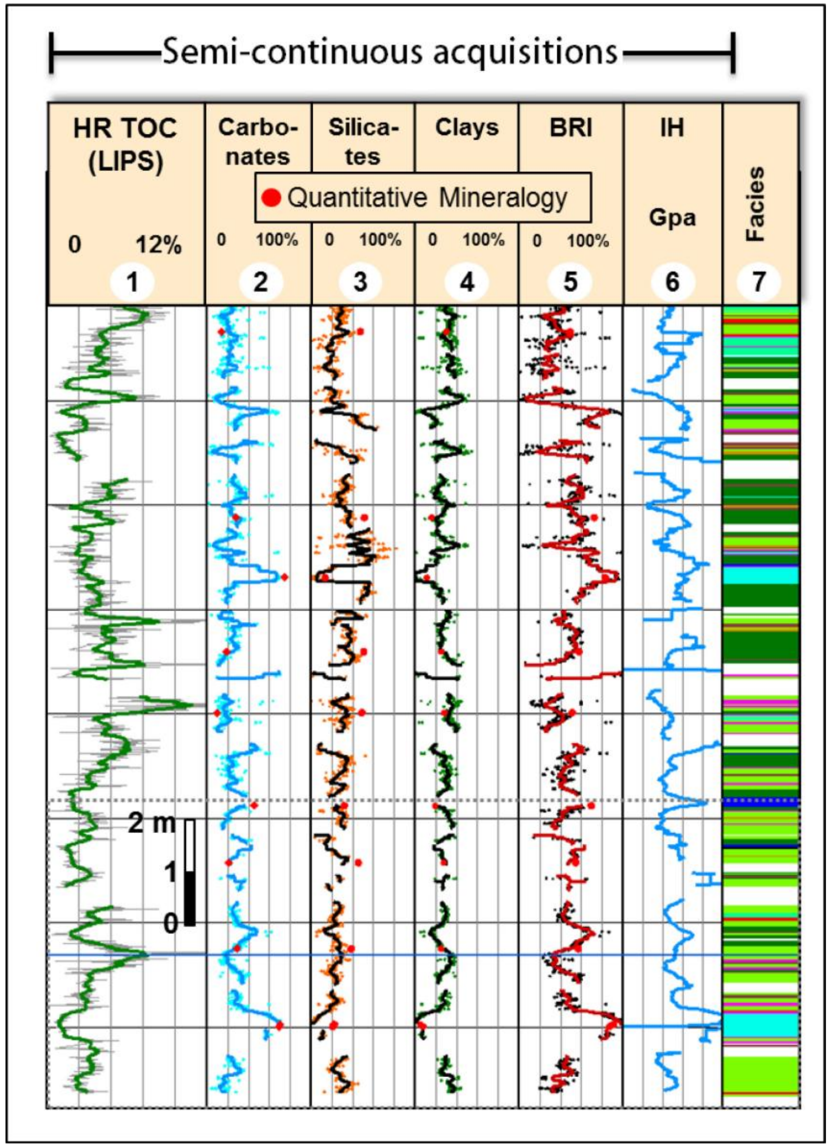


Figure 5. Results of the semi- continuous acquisitions of nearly 15 m of Vaca Muerta cores. In column 1, is displayed the TOC profile acquired with the LIPS at a resolution of 1 cm. In columns 2, 3, and 4 are plotted the NER interpretations of carbonates (including dolomite), silicates (including feldspars), and clays contents. FTIR was acquired at a resolution of ½ cm. These high-resolution values are compared with the punctual Quantitative Mineralogy results. A mineralogical Brittleness Index, column 5, illustrates how quickly should vary the geomechanical properties. The results of the Impulse Hammer are shown on column 6 with vertical variations very close to the computed BRI. The detailed sedimentological description is displayed on column 7.

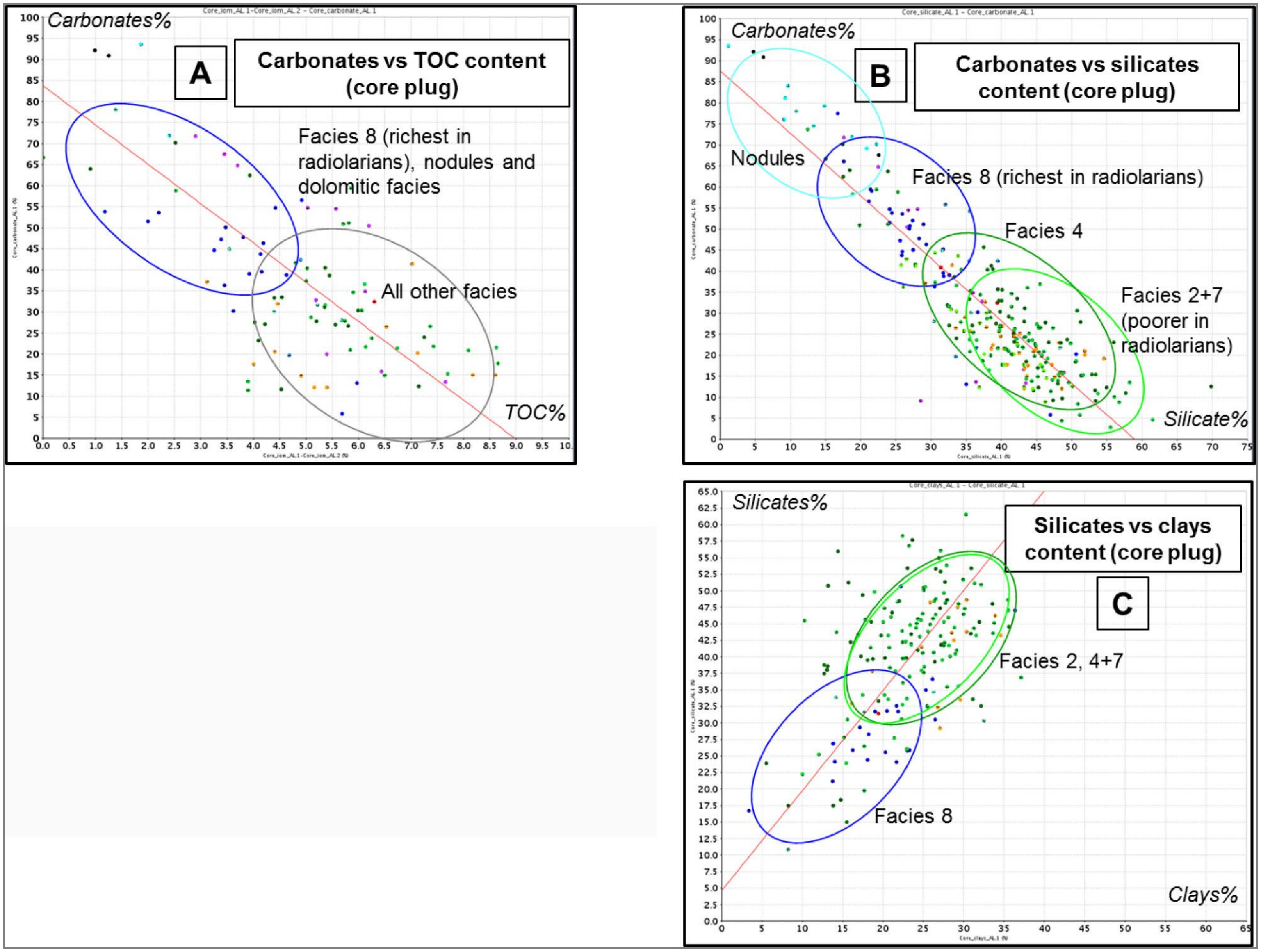


Figure 6. Examples of cross relationships between mineralogical poles based on Quantitative Mineralogy results from several wells chosen within similar stratigraphic intervals. The data points are coloured with their corresponding sedimentological facies (see Figure 2 for facies colour legend). A) Rough, but existing, inverse cross correlation between carbonates and TOC. B) Clearer inverse correlation of carbonates and clays content with a clear facies distribution. C) Positive correlation between clays and silicates contents (although diagenetic imprints can be important).

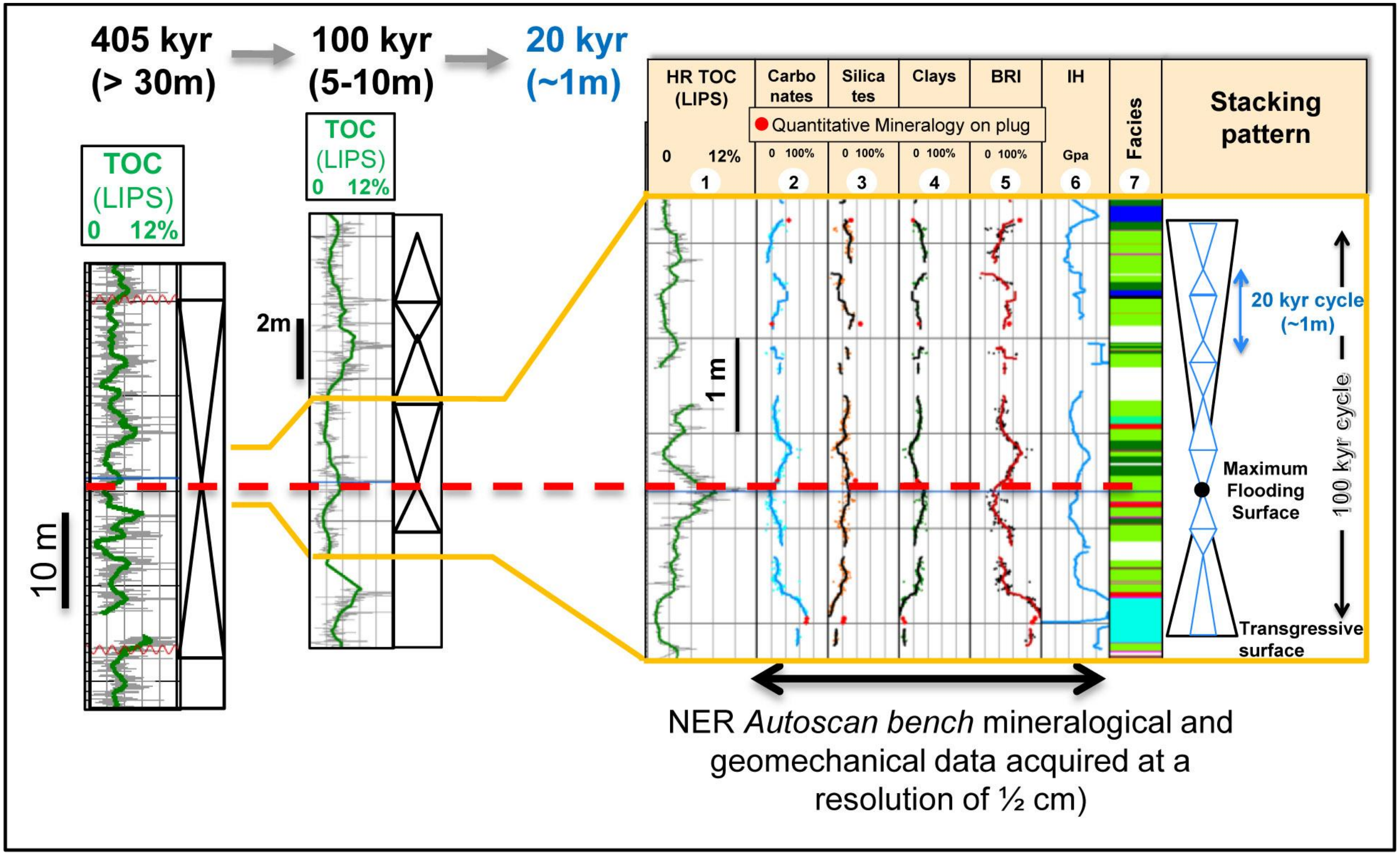


Figure 7. « Fractal » source rock properties. Semi-continuous acquisitions on cores (*Autoscan bench* from NER) reveal the cyclic pattern of source rock properties, from 1 m thick sequences (20 Kyr) to several tens of meters thick sequences (405 Kyr). In the distal part of sedimentary basins, often unperturbed, source rock characteristics, such as sedimentary facies and mineralogy (including TOC) and associated geomechanical properties (Brittleness Index or E* from Impulse Hammer), can be directly correlated to paleo-climate variations derived from the astronomic Milankovitch cycles.

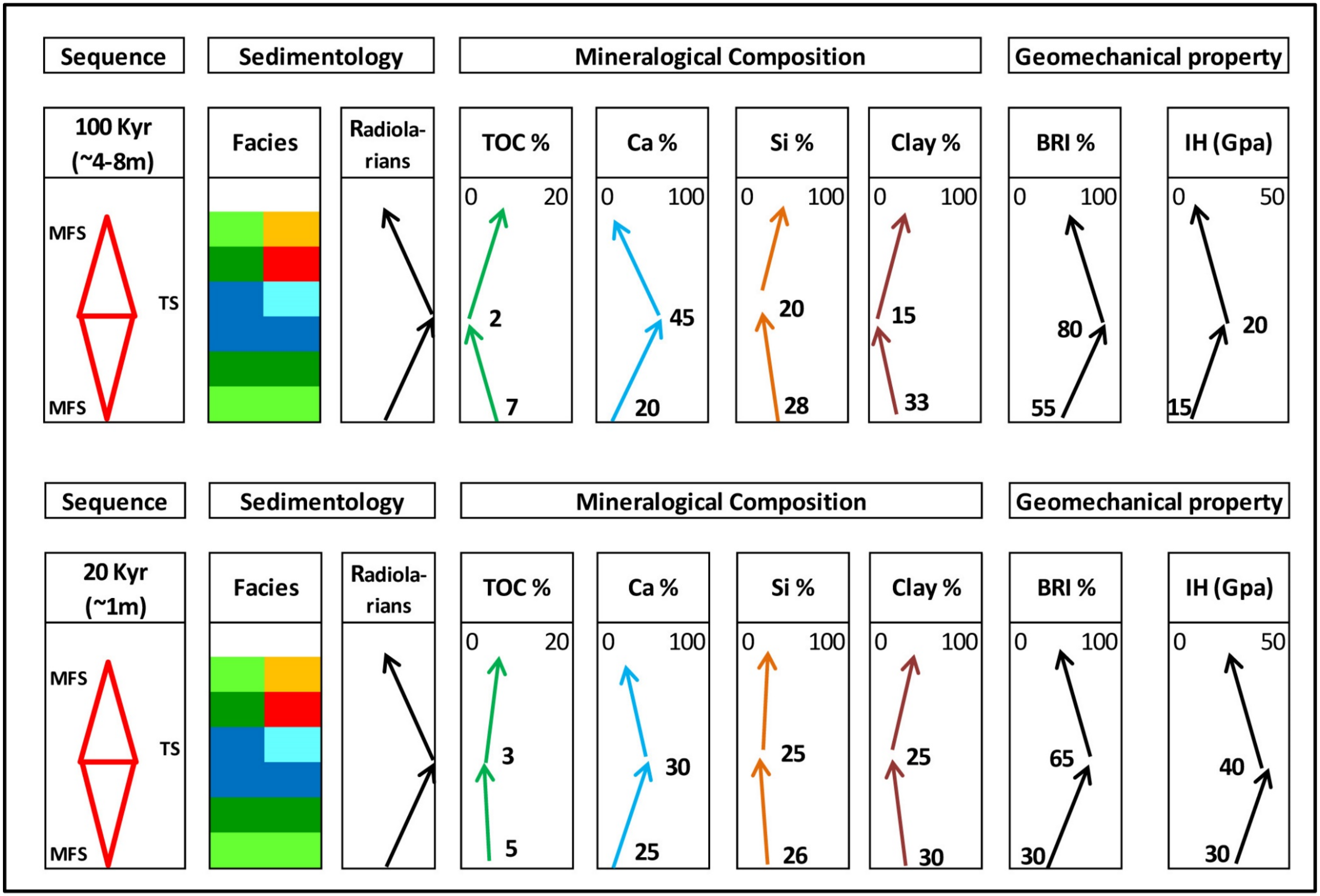


Figure 8. Sedimentological, mineralogical, and geomechanical average variations within a 100 Kyr sequence of the Vaca Muerta (5-10 m thick).