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Palynology and geochemistry of the Frasnian global transgression in the Parnaiba Basin, Brazil

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Abstract :

An integrated palynological and geochemical approach has been initiated to date and to precise the geochemical events of the Pimenteiras Formation, in the Late Devonian from the Parnaiba Basin, Brazil. Based on good biostratigraphic markers and presence of index species among miospores, acritarchs and chitinozoa, the investigated sequences are referred to the Middle to early Late Frasnian. Among Miospores, Chelinospora concinna and Verrucosisporites Witt-eras are likely to be from the BPi Interval Zone defined in the Amazon Basin. Chitinozoans of the Early to early Late Frasnian Angochitina (Lagenochitina) avelinoi zone of the Parnaiba Basin are present, and acritarch assemblages arc also consistent with a Middle to early Late Frasnian age, by comparison to assemblages previously described in other Brazilian basins. Some important accompanying species of palynomorphs are illustrated. In the studied outcrops, black shale intervals are referred to top of the Radioactive Sale B and Radioactive Shale C according to Rodrigues (1995) definitions. Top of Radioactive Shale B shows good concentration of terrestrial organic matter, but low potential source of gas. By comparison Radioactive Shale C of the upper part of the Pimenteiras Formation, with important concentration of marine organic matter have low to high potential as source of gas and condensate. This study shows that a progressive flooding took place during the Devonian period in the Parnaiba Basin with maximum marine transgression in the Frasnian. These global events of interest for hydrocarbon exploration, are regarded as markers for Gondwanan regional chronostratigraphic correlations. (C) 2020 Published by Elsevier B.V.

Highlights

► Miospores, acritarchs and chitinozoans are relatively abundant and well preserved. ► Palynomorphs provide a concordant age corresponding to the mid-Frasnian. ► Miospores are correlated with the

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conodont biostratigraphic scale from the Boulonnais. ► Palynomorphs are characteristic of the Gondwana and more precisely from Brazil. ► Effective generation of hydrocarbon is dependent of the igneous effect.

Keywords : Miospores, Acritarchs, Chitinozoan, Geochemistry, Frasnian, Brazil

1. Introduction

The Brazilian Palaeozoic basins contain thick Devonian shaley sections. These sections often display interlaminated mudstones/siltstones/very fine sandstones deposited in offshore conditions. The chronology of Palaeozoic sea-level changes suggested by Haq and Schutter (2008) demonstrated that a new long-term sea-level rise began in the Middle Devonian and reached its peak in the early Late Devonian (Frasnian). In the Parnaíba Basin, black-shale intervals with relatively high Total Organic Carbon (TOC) values (> 4.0%) are frequently detected in the Devonian section. These geochemical signatures are likely related to the Late Devonian worldwide transgression described in the literature (Lüning et al., 2004; Souza et al. 2013, Bond and Wignall, 2008; Trindade and Carvalho, 2018). Therefore, the present work intends to describe the TOC content excursions recorded in the Devonian section of the Parnaíba Basin and date these excursions by means of palynology, to fit the intervals to the global sea-level curve.

The complementary geochemical data used in this work detailed has made possible for a better understanding of the control of the anoxic / dysoxic conditions of the organic matter preservation, which would typically have taken place within the flooding surfaces associated with the transgression events. In the Devonian section of the Parnaíba Basin, these surfaces—which show the highest TOC content and the highest Hydrogen Index (HI)—represent the most important hydrocarbon generation intervals.

Rodrigues (1995) identified three potentially generating shale intervals within the Pimenteiras Formation, calling them Radioactive Shales A, B and C. According to Rodrigues (1995), the Radioactive Shale A is positioned approximately at the Eifelian / Givetian boundary; has a maximum thickness of 20 m and exhibits TOC contents ranging from 1.0 to 3.0% and contains type III organic matter. The Radioactive Shale B, Middle Givetian in age, has a maximum thickness of 20 m, TOC contents ranging from 1.0 to 3.5% and type II and III organic matter. According to Rodrigues (1995), the Radioactive Shale C, deposited during the Frasnian, is correlatable to the main source rock intervals in the Solimões (Jandiatuba Formation) and Amazonas (Barreirinha Formation) basins. It is the main interval of radioactive shale layer in the basin, reaching a thickness of 40 m. It has TOC contents ranging from between 1.0 and 5.0% and type II organic matter.

The gas accumulations related to these intervals in the Parnaíba Basin normally display a dry composition. Dry composition is normally indicating a generation at high maturation stage. However, in Parnaíba Basin, this condition is not reached by burial, suggesting an atypical process of generation related to the heat effect of dolerite intrusions (Rodrigues, 1995; Rodrigues et al., 1995, 2012; Zambrano et al., 2017; Miranda et al., 2018; Cioccari and Mizusaki, 2019).

The palynological samples has revealed a rich assemblage of acritarchs, chitinozoans and miospores. Despite their relative abundance, they all agree with regard to dating the geochemichal events of an equivalent Frasnian age. New observations in Brazil and new data from the Boulonnais Area, Northern France (Streel et al., in press) have made it possible to correlate the Brazilian outcrops with the European conodont zones and therefore with the stratotypes of the Frasnian.

Finally, for the first time, a high precision palynological study has dated the most important source rock intervals of the Parnaíba Basin, the geochemical aspects of which have been described in detail. These integrated results are particularly important and will greatly contribute to more precise future basin modelling studies.

2. Geological background

2.1. Overview

The Parnaíba Basin is currently distributed over an approximately circular area, with about 600,000 km² of which is located in north-eastern Brazil (Fig. 2), where it covers part of the states of Piauí, Maranhão, Tocantins, Pará, Ceará and Bahia (Pereira et al., 2012). Due to its great stratigraphic similarity with other basins (Gabon, Ghana, Northeast Brazil), some researchers (Milani and Thomaz Filho, 2000; Almeida and Carneiro, 2004; Pereira et al., 2012) suggest that the depositional basin scenario could have occupied an area two to three times greater than the basin’s current area during the Palaeozoic era. The thickness of the sedimentary column is about 3,500 m in the depocenter, while the average thickness of the basin is about 2,000 m.

2.2. Stratigraphic framework

Kegel (1953) was the first to suggest the Late Devonian age for sediments in the Parnaíba Basin. These sediments make up the upper part of the Pimenteiras, Cabeças, and lowermost Longá formations (Góes and Feijó, 1994; Grahn et al., 2006). The Pimenteiras Fm (than Member) was established by Small (1914) for a 20 m thick shale sequence near the town of Pimenteiras, in the State of Piauí. Plummer (1948) divided the Pimenteiras Formation into an upper sandy member and a lower shaly member. Andrade and Daemon (1974) assigned a Late Eifelian to Late Famennian age span to the Pimenteiras Formation, whereas Loboziak et al. (2000) do not consider the fully developed part of the top of the unit to be younger than Early or Middle Famennian.. The upper part of the formation is partly developed into finely laminated and radioactive dark shales, which are best developed in the subsurface of the basin and only exposed in outcrops on its western margin (Loboziak et al., 2000). The maximum thickness of the formation is over 500 m in the central part of the basin. A gap corresponding to the Late Givetian and earliest Frasnian is present all over the basin, and the upper part of the Pimenteiras Formation is entirely of Late Devonian age (Rodrigues et al., 1995; Grahn et al., 2006). This younger interval of the formation is hitherto unknown in the eastern outcrop belt of the Parnaíba Basin (Melo, 1988). Similar ages for the upper levels of the Pimenteiras Formation were also obtained recentlty by Andrade et al. (2009) on the western edge of the basin.

The overlying Cabeças Formation was established by Plummer (1948). Beurlen (1965) included its lowermost part in the Pimenteiras Formation, and Aguiar (1971) placed the upper part in the overlying Longá Formation. The Cabeças Formation is here treated in its original sense (Melo and Loboziak, 2003; Grahn et al., 2006) as defined in its type area near Dom Expedito Lopes (formerly the town of Cabeças), State of Piauí. Daemon (1974) and Andrade and Daemon (1974) found evidence placing the formation in the western part of the Parnaíba Basin in the Late Famennian age. Throughout the basin an erosional hiatus separates the latest

Famennian glaciogenic strata of the upper Cabeças from variably older sediments belonging to the Pimenteiras and lower Cabeças formations, and towards the western margin the former also overlap directly onto metamorphics of the Precambrian basement (Loboziak et al., 2000). The lithology in the lower Cabeças lacks diamictites and differs from the Pimenteiras Formation in that it has a higher content of sandstone beds. In its upper part the Cabeças Formation features a regressive progradation and fast intergradation of alluvial fan, fan delta and fan delta front systems and glacial lobes at the top (Caputo and Crowell, 1985). The maximum thickness (on the eastern flank of the basin) is about 300 m. While no in-situ fossils older than the Famennian have been established in the Cabeças Fm, shelly faunas from the Middle Devonian occur in outcrops traditionally attributed to the lowest part of the formation along the eastern margin of the basin (Melo, 1988). These fossiliferous sandstones correspond to Kegel’s (1953) “Passagem Member”, now dated as late early or middle Givetian by Breuer and Grahn (2011), and consist of alternating tempestites, and sigmoidal lobes that are genetically related to the Pimenteiras Formation. The Longá Formation was introduced by Albuquerque and Dequech (1950) for a section in the valley of the Longá River, and along the road between Campo Maior and Castelo in the State of Piauí. Kegel (1953) and Lange and Petri (1967) considered it Frasnian in age. Kegel (1953) reported a shelly fauna, including rare Malvinokaffric elements (Melo, 1988), and possible tentaculitids in basal Longá Formation outcrops at the Barreiras Farm, on the eastern flank of the basin. Malvinokaffric faunas became extinct during the early Givetian (Bosetti et al., 2011), and tentaculitids are unknown later than the Frasnian. However, closer inspection of the latter (Melo in Grahn and Melo, 2005) revealed that the alleged "tentaculitids" are in fact serpulitid worm tubes characterized by annular structures. The lower (latest Famennian) part of the Longá Formation (lateral facies to the uppermost Cabeças Fm.) features rhythmites including micaceous, sideritic and well laminated greenish-grey shale with bioturbated interbedded siltstone. The formation above (Tournaisian) contains argillaceous, cross-laminated sandstones with conglomeratic horizons, and silty shales and siltstones in the upper part. The total thickness of the formation is about 150 m (Caputo, 1984). Reworked palynomorphs were found in the lower Longá Formation, in the central and western parts of the basin. This corresponds to chitinozoans occurrences likely to have been reworked and which were reported by Cruz (in Lima and Leite, 1978) from the Valença do Piauí region, on the eastern flank of the Parnaíba Basin.

3. Material and methods

The samples that support the geochemical and palynological analysis of this study were colleted from outcrops located on the western boundary of the Parnaíba Basin.

The samples were collected from outcrops along the roads in Tocantins State (Fig. 1). Three outcrops (sites 10, 11, 12) were described in detail at the edge of the road and were stratigraphically positioned (Fig. 2), producing a composite stratigraphic section of around 70 meters. Fourteen samples were collected from these sections in the Pimenteiras Formation. In an additional 20 metre thick section (Site 35) of the Pimenteiras Formation thirty-one samples were collected (Fig. 3). Twelve other samples were collected in site 37 (Fig. 4). They are distributed as follows in the sites: Site 10: samples 5-8; Site 11: samples 9-12; Site 12: samples 13-18; Site 35: samples 48-78; Site 37: samples 80-91. All the samples were processed for geochemical (Tbl. 1) and palynological analysis.

Before sending the samples for TOC and Rock-Eval pyrolysis analyses, they were macerated and powdered at 80 mesh (0.177 mm), and then acidified with hydrochloric acid, for removal of carbonates.

The TOC of the samples was obtained via Leco SC-632 equipment and reported as a relative weight percentage of the original sample. The Rock-Eval pyrolysis analyses were made in a Vinci Technologies pyrolyzer, in accordance with to the procedures of Espitalié et al. (1977). The S1 (free liquid hydrocarbons present in the rock, in mg HC/g rock); S2 (hydrocarbon generating potential of the organic matter present in the rock, in mg HC/g rock); S3 (CO2 generated by the organic matter present in the rock, in mg CO2/g rock) and Tmax (maximum hydrocarbon generation temperature in °C, measured at the maximum height of the S2 peak) were then read. The hydrogen indices [HI = (S2/TOC)x100, in mg HC/g TOC] were also calculated.

The geochemical analyses were carried out in the Laboratory of Chemical Stratigraphy and Organic Geochemistry of the Faculty of Geology of the Rio de Janeiro State University.

All samples were also processed for palynological studies. This processing was performed by Palynological Laboratory Services, U.K. The samples were first scraped and washed before being crushed to <1mm fragments. A weight of 35g was then used for processing.

Samples were treated with 20% hydrochloric acid (HCl) until any reactions had ceased. The samples were then left in the HCl for approximately one hour, to ensure that carbonate digestion was complete. They were then topped up with water and allowed to settle for at least one hour, after which time they were decanted. This stage was repeated to bring the samples to a neutral pH.

Samples were then treated with 40% hydrofluoric acid (HF) and warmed on a hotplate at 50°C for two hours. Next, they were removed from the hotplate and left in the HF for approximately 12 hours to complete the silicate digestion, during which time they were stirred occasionally.

Samples were then topped up with water and sieved over a 10-micron sieve cloth; the collected fraction was brought to a neutral pH and placed in a Pyrex glass beaker. The samples were then simmered on a hotplate at 100°C for approximately 1 hour, before being topped up and re-sieved over the 10-micron sieve. The residue was collected and brought to a neutral pH.

The residues were then re-sieved to separate and provide a 10-53µ and a >53µ fraction. The >53µ was then placed in a glass vial, as this would not require any oxidation. A representative sample of the 10-53µ residue was placed in a glass vial to provide a kerogen (pre oxidation) slide.

Most of the samples required a short period of cold concentrated nitric acid, ranging in time from 5 minutes to 10 minutes. One or two of the samples required a very short sonication of about 10 seconds, to reduce the amount of organic amorphous residue present.

Both the 10-53µ and >53µ fractions were mounted in Norland Optical adhesive No61. The slides are housed at the University of Liège, Department of Geology, EDDy Lab/Palynology.

The different palynomorphs observed during this study have been photographed and are illustrated on plates I to XV. They have been grouped by biological affinities: acirtarchs (Pl. I-II), chitinozoans (Pl. III-IV) and miospores (Pl. V-XV). A list of all species with their author(s) is provided in the appendix.

In accordance with the recommendations of the Subcommission on Devonian stratigraphy, Newletter N° 22 (2007, p.2), in this paper we used the subdivision of the Early Frasnian into Early and Middle Frasnian, to replace of Early and late Early Frasnian.

4. Results and discussion

4.1. Geochemistry data

According to Rodrigues' (1995) definitions, the geochemical characteristics (Tbl. 1) of the lithological and sedimentological aspects of the outcrops studied (Fig. 2-4), allow for the recognition of the top of Radioactive Shale B in sites 10 and 37. The Radioactive Shale C—of the lower part of the upper Pimenteiras Formation—is recognised in site 12 and in the totality of the section recorded in site 35, as well as in the shales of the upper part of the Pimenteiras Formation in site 11. Theses radioactive shales were originally described by Rodrigues (1995) using geochemistry data and log profiles from wells located in the center of the Parnaíba Basin.

The top of Radioactive Shale B, recorded in site 10, and the lower part of site 37, is characterised by a TOC of around 1%, which implies a good concentration of organic matter. In most of the samples, the S2 values of between 0.69 and 3.00 mg HC / g rock, and the hydrogen index of between 70 and 200 mg HC / g COT, indicate low source potential for gas.

The data of the hydrogen index shows that, in thermally immature samples (values of $T_{max} \leq 435$ °C), the organic matter ranges from type IV to type III. This suggests the predominance of degraded terrestrial organic matter according to Jones (1987). This interpretation concurs with the works of Rodrigues (1995) and Rodrigues et al. (1995), who concluded that the shales below Radioactive Shale C are characterised by a greater influence of higher plants in terms of organic matter composition.

The radioactive Shale C was recorded in sites 12 and 35 and the middle section of site 37. This interval is characterised by TOC contents of 1.0 to 6.95%, implying a moderate to high concentration of organic matter. The S2 values of between 1.17 and 11.28 mg HC / g rock, and the hydrogen index of between 100 and 336 mg HC / g COT, indicate a low to high potential source of gas and condensate.

Considering that the samples are thermally immature (values of $T_{max} < 430$ °C), the hydrogen index data suggests that the organic matter of this interval is heterogeneous, showing a mixture of type IV to type II. However, the literature (Rodrigues, 1995; Rodrigues et al., 1995) shows that the organic matter of Radioactive Shale C is predominantly composed of marine algae. As marine organic matter predominates, it is possible to infer that the lower values of the hydrogen content are casually related to the higher proportion of oxidised organic matter. As the radioactive shales C contains the highest proportion of marine organic matter and the highest total organic carbon contents (sites 12 and 35)—when compared to the Radioactive Shale B and to the upper shales of the Pimenteiras Formation— it can be concluded that they may represent deposits related to the maximum marine flood surface (Rodrigues, 1995) and may therefore represent deeper and more distal areas of terrestrial organic matter influx.

The shales of the upper part of the Pimenteiras Formation are recorded in site 11, where they come into contact with the Cabeças Formation. The total organic carbon contents range from 1 to 2%, representing a high concentration of organic matter. The S2 values of between 2 and 5 mg HC / g rock, and the hydrogen content of between 150 and 230 mg HC / g COT, characterise a moderate potential source of gas.

Since the samples analysed are less thermally developed (values of T_{max} < 430 °C), the values of the hydrogen index—at around 150 mg HC / g COT—characterise a predominance of type III organic matter. This aligns with the findings of Rodrigues (1995) and Rodrigues et al. (1995), which describe a greater proportion of higher plants in the composition of the organic matter of the shales above the Radioactive Shale C.

The geochemistry results indicate that the Radioactive Shale B of the Pimenteiras Formation—recorded in the outcropping—exhibit a good concentration of terrestrial organic matter, with a low potential source of gas. The Radioactive Shale C of the Pimenteiras Formation, described in sites 12 and 35, has a good to excellent concentration of marine organic matter and is a low to high potential source of gas and condensate. Lastly, the shales of the upper part of the Pimenteiras Formation, recorded in site 11, show a moderate concentration of terrestrial organic matter with a moderate potential source of gas. However, the pyrolysis analysis shows that the organic matter contained in the Pimenteiras Formation is thermally immature in all outcrops studied in the southwestern area of the Parnaíba Basin.

4.2. Acritarchs and other microalgae (fig. 5)

Müller's (1962) palynozonal scheme, based on the combination of major palynomorph groups (acritarchs, miospores and chitinozoans) for the Silurian-Mississippian interval of the Parnaíba Basin in northeastern Brazil, represents the first attempt at dating the pre-Tertiary sedimentary sequence. Müller's pioneering work was followed by other palynological investigations and the proposition of regional zonation by Brito (1967b, 1971); Andrade and Daemon (1974); Daemon (1974, 1976); and Quadros (1982, 1988). Since that time, these studies have remained largely unrevised. However, the opportunity offered by a long collaboration with Petrobras in recent years, based mostly on the study of subsurface materials, has enabled us to systematically analyse acritarch assemblages from the Amazon, Parnaíba and Solimões basins, to define biostratigraphic subdivisions based on these marine and possibly freshwater planktonic microfloras, and to evaluate their stratigraphic value for the Palaeozoic succession in comparison with chitinozoans and miospores. This work is, for the most part confidential. Yet the experience acquired is used here to discuss the biostratigraphic dating of the outcrops analysed, complementing the data and chitinozoans and miospores.

Acritarchs and other microalgae are well represented in the samples studied, even though miospores are dominant in some samples. A total of 70 species have been identified (see list in the appendix). Among those definitively identified, are some characteristic species of the Late Devonian. The data obtained enables the subdivision of that interval into two assemblages: the oldest from the Middle to earliest Late Frasnian age; the youngest from the early Late Frasnian age. The palynological results with acritarchs are correlated and largely calibrated by available miospores and chitinozoans zonations (Melo and Loboziak, 2003; Grahn et al., 2006) in use in the same regions. As previously mentioned, we take account of the recent subdivision of the Early Frasnian, into Early and Middle Frasnian. This has affected the dating of the local Bpi miospore Zone of Melo and Loboziak (2003), with the upper part now being referred to as the Middle Frasnian instead of late Early Frasnian. This data makes it possible to correlate the assemblages, including acritarchs and prasinophytes, with Late Devonian assemblages documented from the Amazonas Basin (Le Hérissé, 2001), and other regional studies providing information of the stratigraphical distribution of some species (Brito and Quadros, 1984 ; Oliveira and Burjack, 1996 ; Oliveira and Burjack, 1997 ; Quadros, 1997). We also refer to the unpublished thesis by Oliveira (1997) and unpublished personal data. The difficulty lay in working with different outcrops, since we have observed variations in the suites of acritarchs and other microalgae on a site by site basis —suggesting some variations in their biostratigraphy, as previously suggested from the study of miospores. However, the first occurrence of some species, and the associations of species within the different sites, has allowed us to suggest quite an effective resolution in the biostratigraphic analysis.

4.2.1 First assemblage

A first assemblage of the Middle to earliest Late Frasnian age is suggested, in the material studied. This is due to the association of the following species—*Crucidia camirense* (Pl. 1, fig. 7), *Duvernaysphaera cruciformis* nov. sp. (Pl. 1, figs 14-16), *Leiofusa bisubulata* (Pl. 2, fig. 6), *Mediocorpore conspicuus* (Pl. 2, fig. 1), *Petrovina connata* (Pl. 1, fig. 5), *Pseudolunulidia micropunctata* (Pl. 1, fig. 8) and *Puteoscortum limai* (Pl. 2, fig. 5) —in sites 10 and 12, and to the occurrence of *Leiofusa bisubulata*, *Mediocorpore conspicuus, Pseudolunulidia micropunctata* and *Puteoscortum limai* in the lower two-thirds of site 35.

Comparisons and correlations were made with coeval assemblages known to exist within subsurface successions in the Parnaíba and Amazon basins (Le Hérissé unpublished data). *Leiofusa bisubulata* which we used in association with other species represented here to characterise the Middle Frasnian is an index species of interest in the eponymous zone. This zone existed prior to the Late Frasnian and overlaps with it and is marked by a consistent occurrence of *Maranhites* spp. with internal dots. The first specimens of this group appear sporadically in the upper part of the *Leiofusa bisubulata* zone.

The *Leiofusa bisubulata* Interval Zone encompasses the main part of the radioactive shale interval of the Parnaíba Basin (Frasnian anoxic event of Rodrigues, 2001; Rodrigues et al., 1995), with equivalents in the lower Frasnian of the Amazonas Basin (Barreireinha Formation). However, even though good components of the radioactive shales, such as the large Tasmanites and leiospheres are well represented in some samples, such as in site 12 but also higher, we never have an over-representation of these elements compared to the Amazonas basin. This fact along with some of the other biostratigraphical results detailed below, might suggest that we are above the interval with a maximum of radioactive shales.

The occurrence of *Crucidia camirense* and *Petrovina connata* at the base of site 10 suggests that these samples are not of earliest Frasnian age. This is because the oldest records of the two species are in the upper part of the *Leiofusa bisubulata* biozone in other sections of the Parnaíba Basin (based on unpublished data), and this is also reflected in the reference core RSP-1 of the Paraná Basin, where *Petrovina connata* was defined (Burjack and Oliveira, 1996)

Important additional species in the first interval, which were distinguished in this study include: *Advenasphaeridium australis* (Pl. 2, fig. 4), *A. acerosum, ?Chomotriletes* sp. (a new form, Pl. 2, figs 7-9), *Diaphorochroa gracile* (Pl. 2, fig. 11), *Duvernaysphaera cruciforme* nov. sp. (Pl. 1, figs 14-16) associated with *D. angelae/tessella* (Pl. 1, figs 17) and well represented, *D. radiata* (Pl. 2, fig. 2) , *D. tenucingulata, Estiastra spinireticulata* (Pl. 2, fig. 16), *Exochoderma arca, Geron elegans, Hemiruptia legaultii, Leiofusa fastidiona*, common *Maranhites* (without dots) with *Maranhites brasiliensis* (Pl. 1, fig. 1), *M. lobulatus, M. magnus, M. primus, M. stockmansii* (Pl. 1, fig. 3), some *Navifusa bacilla* (Pl. 1, fig. 9) with the crescentic variants and the long and fine *N. bacilla* var. *procera, Naevisphaeridium sprucegrovense* (Pl. 2, fig. 12), *Polyedryxium fragosulum, P. pharaone, Pterospermopsis pernambucensis, Tinacula simplex* (Pl. 2, fig. 19), *Umbellasphaeridium deflandrei* (Pl. 1, fig. 11), *U. saharicum, Unellium ampullium* (Pl. 2, fig. 10), *U. piriforme* and some triapsidate *Veryhachium.*

4.2.1.1. Correlation with previous regional zonation

In the Parnaíba Basin, the *Leiofusa bisubulata* zone encompasses the combined upper part of the *Samarisporites triangulatus* (Trg), which is not represented here, the *Verrucosisporites bulliferus-Geminospora piliformis* (Bpi) and the lower part of the *Rugospora bricei-Diducites mucronatus* (BMu) miospore zones of Melo and Loboziak (2003). It corresponds to the *L. avelinoi* chitinozoan zone (Grahn et al., 2006). The higher part of the local Bpi miospore zone is now reassigned to the Middle Frasnian. The Early to early Late Frasnian strata of the Parnaíba Basin correspond to Müller's (1962) palynozone Q, at least—as well as to the transition between interval V and VI, sensu Daemon and Contreiras (1971), Andrade and Daemon (1974) and Daemon (1974, 1976).

4.2.1.2. International correlations

Umbellasphaeridium saharicum, also recently registered by Andrade et al. (2020) in the upper levels of the Pimenteiras Formation in the southwestern border of the Parnaíba Basin, while never abundant, is of interest in the zone—as a complement to the eponymous species and other key taxa—because of its stratigraphic value as an Upper Devonian index in the Brazilian Palaeozoic basins (see also Melo, 2005). It is consistent with the Frasnian through Strunian (Zones L5-L7) range of the species in the Algerian Sahara (Jardiné, 1972; Jardiné et al., 1974). In Ghana, in some successions which are somewhat equivalent to the succession in the Parnaíba and Maranhão Basin, the species is recorded in the Frasnian (Bär and Riegel, 1974). It is also an important species present in the Frasnian assemblage SA3 of Argentina (Noetinger and Di Pasquo, 2011). The first record of the species was certainly by Regali (1964), in the Tucano-Jatobá Basin of northeastern Brazil. The said occurrence, first dated as Middle (?) Devonian by Regali (1964), was later reinterpreted as Frasnian by Brito (1976) and as Strunian by Quadros (1980) and Dino et al. (2011). Another historical record is in the Mac-Mahon Basin, in the upper Famennian-Strunian, as “acritarche à entonnoirs” N°441-33 by Lanzoni and Magloire (1969). The accurate stratigraphic distribution of *Umbellasphaeridium saharicum* in the U.S.A. is more complex, since the material is associated with reworked elements in the Late Devonian (Wood, 1979).

There is a good correlation with the Late Devonian of Argentina, particularly the Los Monos Formation assemblages described in the Tarija Basin. Many of the species recorded by Ottone (1986) occur in the Leiofusa bisubulata biozone, such as: *Leiofusa bisubulata* (as *Leiofusa* sp. Pl.7, figs. 7, 8 in Ottone) (Pl. 2, 6), *Maranhites mosesii* (Pl. 1, 4) and *Pseudolunulidia* spp. The re-description of *Crucidia camirense* (Pl. 1, fig. 7) in Argentina is also noteworthy, after the species was first illustrated in Bolivia by Boneta (1975). With the presence of *Crucidia camirense* in Argentina, among other elements in Argentina, we consider the age of these Argentinian assemblages as most likely early Late Frasnian, rather than Late Givetian to Early Frasnian as suggested by Ottone (1996).

As discussed earlier, direct comparisons are also possible with the Paraná Basin and the reference RSP-1 borehole, previously studied by Burjack et al. (1987), Loboziak et al.

(1988), and Oliveira (1997). Many of the species found in the *Leiofusa bisubulata* biozone were described for the first time in the RSP-1 core-drill (Oliveira and Burjack, 1996; Oliveira and Burjack, 1997), yet many species are still *nomen nudum*. The equivalent of the Dc-Lb Zone is the interval between 60.50 m and 200 m. Species in common include *Advenasphaeridium acerosum* (nomen nudum), *A. australis* (nomen nudum) (Pl. 2, fig. 4), *Crucidia camirense* (Pl. 1, 7), *Diaphorochroa gracile* (*nomen nudum*) (Pl. 2, fig. 11), *Naevisphaeridium hercyniana, Leiofusa bisubulata, Petrovina connata* (Pl. 1, fig. 5), the netromorphs *Navifusa bacilla crescentis* (Pl. 1, fig. 9) and *Pseudolunulidia micropunctata* (Pl. 1, fig. 8).

4.2.2. Second assemblage

The stratigraphically higher assemblage is defined in the last three samples of site 35 the last four samples of site 11 and up to sample 86 in site 37. It contains significant species that are unrepresented in the lower assemblage, including *Horologinella quadrispina* (Pl. 1, figs 12-13), *Maranhites mosesii, Pseudomaranhites densus, Pterospermopsis crassimarginata* (Pl. 1, fig. 6), *Veryhachium capitata* nov. sp. (Pl. 2, fig. 17), *Veryhachium insanum* nov sp. (Pl. 2, fig. 18) and *V. pannuceum.*

In the Amazon Basin, the succession of *Veryhachium insanum, Bipolarisvelata accreta* and the introduction of *Horologinella quadrispina* is observed in the reference core Caima PH-2 (Le Hérissé, 2001; Le Hérissé, Longhim, Melo, in prep.), at the boundary between Middle and Late Frasnian. It corresponds also to an interval marked by the appearance of *Maranhites mosesii.*

The order of appearance of some of these species is also the same in the upper part of the RSP-1 core in the Paraná Basin (see Oliveira, 1997, Fig. 18). But the material does not include the introduction of *Horologinella quadrispina* (Pl. 1, figs 12-13).

Pterospermopsis crassimarginata is also a common component of the zone, but it can be mentioned as a precocious appearance of this species in the Paraná Basin, where it has been defined for the first time.

Puteoscortum williereae is the index species of Zone Pw (Vanguestaine et al., 1983) and is characteristic of the lower Famennian in Belgium. Our data points to an earlier introduction of the species in the Frasnian of Brazilian successions, whereas its regional exit is in the Middle Famennian.

In terms of correlation with previous regional zonation, the higher assemblage recognised here will be equivalent to the lower part of the *Rugospora bricei-Diducites mucronatus* (BMu) zone of the Late Frasnian age, according to Melo and Loboziak (2003) and Grahn and Melo (2006).

4.3. Chitinozoans

Devonian chitinozoans from the Parnaíba Basin were first discussed by Müller (1962). His Silurian through Lower Carboniferous Biozones were divided into seven zones, ranging from N (upper Visean) to T (upper Silurian), based on chitinozoans, spores and acritarchs. The Pimenteiras Formation corresponds to his Zone Q (upper Zone R–P by Brito, 1967, 1971 and Q–P by Quadros, 1982). These zones were, until recently, regarded as a reference palynomorph biozonation for the Parnaíba Basin (e.g. Brito, 1967, 1971; Daemon, 1974, 1976; Quadros, 1982, Molyneux et al., 1996). Daemon refined the Silurian – early Carboniferous sequence into 12 biozones based on miospores. In his scheme, the upper zones 5 and 6 correspond to the Pimenteiras Formation. Sommer and Van Boekel (1964) described Middle Devonian chitinozoans from outcrops of the lower Pimenteiras Formation on the western margin of the Parnaíba Basin. Concurrently, Daemon (1964) formulated a generalised chitinozoan biostratigraphy for the basin using Müller's zonal scheme as a framework. Lange

(1967) briefly mentioned chitinozoans from the Parnaíba Basin, and Cruz and Quadros (1985) described a new chitinozoan species from the latest Frasnian – early Famennian. More recently, chitinozoans have been discussed and correlated with miospores in papers by Grahn et al. (2001, 2005, 2006, 2008) and Breuer and Grahn (2011). The Pimenteiras Formation consists of a lower Middle Devonian section, separated by a hiatus from the upper Late Devonian section.

The chitinozoans encountered in the present study constitute a characteristic Frasnian assemblage, although not earliest or latest Frasnian. They are typical for the early to early Late Frasnian *Angochitina* (*Lagenochitina*) *avelinoi* (Pl. 3, figs 1-2) regional interval range zone of the Parnaíba Basin, which corresponds to the lower *Hoegisphaera glabra* global interval range zone (Paris et al., 2000). *Angochitina mourai* (Pl. 3, figs 3, 4) and *Angochitina avelinoi* (Lange, 1952) are recorded in the Late Devonian of the major intracratonic basins of Brazil. The latter species has a more restricted range in the Paraná (early Late Frasnian) and Amazonas Basins (early Frasnian). In the Parnaíba Basin, there is a variety of *Angochitina mourai* with a long neck, common in the early Late Frasnian. *Lagenochitina* sp. A *in* Grahn and Melo (2005) (Pl. 4, figs 5-6), along with similar specimens, are recorded from the early Frasnian but are also common in the early Late Frasnian. *Fungochitina pilosa* (Collinson and Scott, 1958) (Pl. 4, figs 1-2) is something of a vast basket, and specimens like *Fungochitina pilosa* have been recorded from the late early Eifelian to the Frasnian. *Lagenochitina sommeri* (Lange, 1952) (Pl. 4, figs 3-4) was first described from the early Frasnian in the Amazonas Basin, but also occurs in the early Late Frasnian of the Parnaíba Basin. Both *Angochitina* sp. C and *Fungochitina microspinosa* (Grahn and Melo, 2005) (Pl. 3, figs 9-10) are common Frasnian species in the Parnaíba Basin. Although an early Frasnian age cannot be excluded, the chitinozoans present are altogether more similar to an early late Frasnian age.

4.4. Miospores

4.4.1 Results

Several studies on miospores are available for the Frasnian of the Parnaíba Basin. Most have been carried out by Loboziak and his co-authors (e.g., Burjak et al., 1987; Dino et al., 1996; Grahn et al., 2006, 2008; Loboziak et al., 2000; Loboziak et al., 1994; Loboziak et al., 1992b). Few papers describing the whole Frasnian assemblage of outcrops or cores from the western Gondwana are available.

The miospores observed here are well preserved, and yellow to pale brown in colour. The assemblages are rich and diverse. The most prolific samples in miospores are the N° AM7, AM8, AM9, AM89 and AM95. Samples AM50, AM53, AM55, AM60, AM65, AM70, AM80, AM85 and AM88 contain rare miospores. In all the samples, miospores are more abundant than marine palynomorphs, especially in the upper part of the section where acritarchs are rarer. This decrease in the quantity of acritarch input has also been noted in the upper part of the Pimenteiras Formation of the Well 2-PM-1-MA (Loboziak et al., 1992b), probably corresponding to a regression.

Weak variations in the composition of the assemblages have been observed throughout the sequence, except for very minor differences. Moreover, as the different sections are being separated by outcrop gaps, their lateral geometrical relationship is somewhat difficult to achieve.

The richest levels in miospores contain more or less the same assemblage in which the most helpful species for biostratigraphy are known from the classical Boulonnais area from Northern France (Streel et al., 1987, Loboziak and Streel, 1988). Among them, the following species have been observed: *Samarisporites triangulatus* (pl. 15, figs 1-3), *Chelinospora concinna* (Pl. 7, figs 6-9), *Verrucosisporites bulliferus* (Pl. 15, figs 4-6), *Lophozonotriletes media* (Pl. 13, figs 9-10), etc. These suggest the Frasnian age. The oldest sample from the site

10 (AM7) already contains, among others, *Verrucosisporites bulliferus* (Pl. 15, figs 4-6) and *Lophozonotriletes media* (Pl. 13, figs 9-10)

In the Boulonnais area of northern France (Streel et al, 2000), miospores occur in the same sections as conodonts. The chronostratigraphic limits of the Frasnian stage, fixed on conodonts in the Montagne Noire area, can thus be transferred to the Boulonnais area. In this area, *Chelinospora concinna* and *Samarisporites triangulatus*—which characterise the TCo Zone— first occur in the upper Givetian but are still present in the Early and Middle Frasnian.

Verrucosisporites bulliferus first occurs in the early Frasnian and *Lophozonotriletes media* in the Middle Frasnian (BJ and BM Zones), but both are still present in the upper Frasnian and the Famennian. *Rugospora bricei, Grandispora gracilis* and *Diducites plicabilis (*BA Zone or Regional "IV" Zone) first occur successively in the upper Frasnian but are still present in the Famennian (Streel, 2009).

A Middle to Late Frasnian (but not latest) age is therefore suggested for all the studied samples.

4.4.2. Discussion

4.4.2.1. Correlations with Brazilian biostratigraphic scale (Fig. 6)

Miospores from the Givetian to the Tournaisian have been studied by Loboziak et al. (2000) in the Tocantins Valley of the Parnaíba Basin located some 120 km away from the sections studied here. The biozones from the Boulonnais area (Streel et al. 1987), ranging from TCo to BM-"IV"(or BA Zone), were used in this paper as well as in Loboziak and Melo (2002). New biozone names (BPi –BMu-TP) were introduced later by Melo and Loboziak (2003) in the Amazon Basin. Among the Tocantins Valley samples from the Pimenteiras Formation, 12 are shown to belong to the Frasnian. A total of 32 taxa have been identified in these 12 samples including 9 taxa which are restricted to the Frasnian. From our samples there are only 15 taxa in common with these 32 taxa recorded in the Tocantins Valley. 7 of the 32 taxa are also known in the Cabeças Formation belonging to the Latest Famennian where they are “ascribed to reworking on the basis of differential preservation or color” (Loboziak et al., 2000, p. 308). However, it is possible to correlate the two areas, thanks to the species *Lophozonotriletes media*, *Grandispora tabulata* (Pl. 13, figs 1-2), *Verrucosisporites bulliferus* and *Samarisporites* sp. C (synonym of S. sp. E in Melo and Loboziak, 2003) (Pl. 14, figs 10-12).

In the samples studied herein, *Verrucosisporites bulliferus* is one of the eponymous species of the BPi Interval Zone—along with *Geminospora piliformis*—according to the biozonation established in Brazil by Melo and Loboziak (2003). This biozone is marked by the FOB of *Verrucosisporites bulliferus*. The analysed samples should thus belong to the BPi Interval Zone in Brazil, despite the absence of observation of *Geminospora piliformis*—a species that is poorly scattered and controlled. However, Melo and Loboziak (2003) note that in the Paraná Basin’s Ponta Grossa Formation, *Geminospora piliformis* appears with *Lophozonotriletes media* in the upper part of the zone, allowing the zone to be subdivided into two parts, and demonstrates a correlation of this higher interval with the BM Oppel zone of Western Europe. The BPi Zone has also been observed in the Amazon Basin, and probably also in the Solimões Basin within the Jandiatuba and Uerê Formations.

Rugospora bricei is a species appearing in Biozone BA (ex “IV” Refional Zone) in Western Europe (Streel et al., 1987). This species is also known in Brazil and appears close to the base of the BMu Biozone (Melo and Loboziak, 2003). The species has been identified in the Parnaíba, Amazon, and Paraná Basins (Burjack et al., 1987; Grahn et al., 2006; Melo and Loboziak, 2003). Two specimens like *Rugospora bricei* have been observed in site 10, samples 7 and 8. However, the nature of the convolute proximal muri is different to those of the original species; they are too coarse. Thus, the two specimens are here considered as

Rugospora cf. Rugospora bricei (Pl. 14, figs 7-8)~~.~~ *Rugospora bricei* is also close to *Rugospora radiata*. The main differences are in the size and arrangement of the rugulae in *Rugospora flexuosa* are much coarser (Higgs et al., 2013). These last two species are known in the Famennian.

Among other important species for the biostratigraphy, *Camarozonotriletes concavus* (Pl. 7, figs 4-5) first appears in the Eifelian and is supposed to have disappeared close to the BJ-BM boundary (Loboziak and Melo, 2002). *Grandispora tabulata* first appears just below *V. bulliferus* and is known up to the Famennian (Loboziak et al., 1988). *Auroraspora macra* (Pl. 7, figs 1-3), which has been known since sample 7, first appears at the boundary between Biozones BM and BA (ex "IV" Regional Zone) (Loboziak et al., 1988) and is known up to the Tournaisian (Zwan, 1980).

Most of the spores are geographically cosmopolitan, except for some species that are restricted to the Gondwana like *Geminospora piliformis, Grandispora gabesensis, Grandispora libyensis* (Pl10, figs 6-7) and *Grandispora tabulata.*

4.4.2.2. Correlation with the European biostratigraphic scales (Fig. 6)

Givetian to Lower Famennian deposits, containing miospores, are widespread on the territory of the Pripyat Depression in SE Belarus and the Timan –Pechora Province in Russia. The biostratigraphy of these deposits is based also on conodonts in the Timan-Pechora (see in Streel et al., submitted).

Correlations during the Late Givetian to Middle Frasnian between Boulonnais and Eastern Europe are shown to be obvious from *varcus* to *punctata* conodont Zones. They are less obvious from the Middle Frasnian to the Early Famennian (Obukhovskaya, 2000; Streel et al., 2000; Telnova, 2008; Telnova et al., 2019)

Geminospora lemurata (Pl. 9, figs 5-6) is well known and distributed worldwide, appearing just above the Eifelian / Givetian boundary. This species characterises the Lem Interval Biozone (Streel et al., 1987). *Samarisporites triangulatus* first appears in the late Givetian and is the eponymous species of the TA and TCo Biozones (Streel et al., 1987) in Europe. The species may reach the Tournaisian in both Brazil and Tunisia (Loboziak et al., 1992a; Loboziak et al., 1988; Melo and Loboziak, 2003). *Chelinospora concinna*, the second eponymous species of the TCo Biozone, appears below the Givetian / Frasnian boundary. *Verrucosisporites bulliferus* appears above the base of the Frasnian and characterises Biozones BJ and BM (Streel et al., 1987), which are both included in the lower part of the Frasnian. Finally, *Lophozonotriletes media* is characteristic of the BM Biozone (Streel et al., 1987), around the Middle Frasnian.

The synthesis of miospore based on Givetian to Famennian biostratigraphy in the Boulonnais (Loboziak and Streel 1988) did not provide detailed analysis of the data accumulated by Loboziak et al. (1983) in the Upper Frasnian. Restudy of this data and new samples collected in boreholes from neighboring Flanders (Belgium) allow for better characterization of the transition from Middle to Lowermost Famennian and a comparison with contemporary Eastern Europe miospores based zonations. (Streel et al., 2020 submitted). The correlation at the BM/BA transition dated Upper Frasnian by the *rhenana* conodont Zone in Eastern Europe points to the inaccuracy of the Ferques succession and Hydrequent Formations with regard to tracing the base of the Upper Frasnian in the Boulonnais. A first look at the many recorded ranges of taxa immediately suggests, in the first instance, a sampling gap between these formations. Obviously, it also suggests a deep change in the vegetation cover occurring at that level corresponding more or less to the Lower Kellwasser Event (LKW) starting in the lower *rhenana* conodont Zone (Becker et al. 2016).

If the Melo and Loboziak's (2003) correlation of the Brazilian BPi Zone with the West European BJ and BM Zones makes sense, alignment of the base of the Brazilian BMu Zone with the base of the West European BA (ex "IV") Zone is not secure. Nevertheless the FOB in the Pimenteiras Formation of *Auroraspora macra* , *Samarisporites* sp C in Loboziak, Streel and Vanguestaine 1983 (synonym of S. sp. E in Melo and Loboziak 2003) and possible *Rugospora bricei* occurring within the lowermost part of BA Zone (ex IV a-b or BA pre-gracilis subzone in Streel 2009) confirm the presence of an early part of the Upper Frasnian.

5. Geochemistry and maximum flooding surface

Some authors (Myers, 2004; Sutton et al., 2004; Lüning et al., 2004; Rodrigues, 2005; and references therein) have described the relationship between TOC content and the stratigraphy of sequences. Organic matter preservation in the sedimentary record depends on many factors. Myers (2004) pointed out that the most important factors are the physiogeography of the basin, climate, terrestrial organic productivity, marine aquatic organic productivity, oceanic circulation, sedimentation rate and water depth. With the exception of climate and oceanic circulation, the other factors described by Myers (2004) are influenced by relative sea level change.

Under lowstand conditions, the high sediment influx product, the dilution of the organic marine matter content and the organic terrestrial matter are highly oxidised. In this context, the amount of organic matter preserved in the sediments is low and the consequent TOC values are inexpressive.

In the transgressive system tract, the rapid increase in relative sea level caused the shoreline to retreat landward. It resulted in a progressive extension of the shallow marine shelf deposition, and in a reduction of the clastic sediment supply. According to Lüning et al. (2004), the transgression has likely led to sediment starvation because the detritus became trapped in river mouths, thus preventing the dilution of the organic matter on the shelf. These conditions led to the progressive the expansion of the deep distal area of the basin until it reached the maximum flooding surface (MFS). In this basin, therefore, there was an extension of the anoxic conditions, which permitted an increase in the preservation of organic matter. The retreat of sediments at the shoreline decreased the dilution of organic matter in the depositional setting. In contrast with the lowstand conditions, the TOC values increased, reaching a maximum at MFS.

As also observed in the section studied in this work, Creaney and Passey (1993) noted that many marine source rocks are characterised by an initial abrupt upward increase in organic content, which, in contrast with background values, and a subsequent gradual decrease in organic content (Myers, 2004). The authors attributed this pattern to the control of the organic carbon contents by the clastic sedimentation rate, under anoxic bottom water conditions. The rapid increment of TOC results from the retreat of the sediments in the source, during the transgression. The subsequent gradual decrease in TOC reflects the increase in the clastic sediment supply and the dilution of organic carbon during highstand progradation. In this context, the MFS is positioned at the turning point of the TOC curve. Most authors consider it to be partly contained in the lower highstand, and partly in the upper transgressive systems tracts (Sutton et al., 2004; Posamentier and Allen, 1999).

This succession is well defined in the section studied, where the TOC ranges from low to moderate in site 10 (Figure 03). In the subsequent interval (site 12), associated with Radioactive Shale C, the TOC shows an abrupt increase, defining the improved conditions for organic matter preservation. These conditions mark the maximum flooding surface. This interval is also observed at the base of site 35. In the interval above (site 11), the TOC decreases towards the Cabeças Formation, which indicates the start of the regression system and the consequent increased inflow of sediment toward the basin.

6. Conclusions

The palynological results—based on acritarchs, chitinozoans and miospores—are relatively well concordant, and correlatable with Devonian palynozones in use in northern Brazilian basins.

The occurrence of several species of acritarchs—such as *Leiofusa bisubulata, Mediocorpore conspicuus* and *Pseudolunulidia micropunctata*—in sites 10 and 12, and in the lower 2/3 of site 35, have made it possible to distinguish a first assemblage of Middle to earliest Late Frasnian age.

In the last three samples of site 35 and the last four samples of site 11, and from site 37 up to sample 86, we observe the occurrence of *Horologinella quadrispina, Maranhites mosesii, Pseudomaranhites densus, Pterospermopsis crassimarginata, Veryhachium capitata* nov. sp., *Veryhachium insanum* nov. sp. and *V. pannuceum.* This points to a late Frasnian age, quite equivalent to that of the miospore Biozone *Rugospora bricei-Diducites mucronatus* (BMu).

The chitinozoans that were identified in the different slides belong to a common assemblage characteristic of the Frasnian and, more precisely, neither the earliest nor the latest Frasnian. They belong to the early-early Late Frasnian *Angochitina (Lagenochitina) avelinoi* regional interval range zone of the Parnaíba Basin. This is equivalent to the lower *Hoegisphaera glabra* global interval range zone, which is the second and youngest global chitinozoan biozone of the Frasnian. The miospores do not show variations in the different samples and have been studied as a whole and unique assemblage. Species like *Verrucosisporites bulliferus* and *Lophozonotriletes media* clearly demonstrate a mid-Frasnian age. The analysed samples would belong to the BPi Interval Zone and possibly an early part of the BMu Zone in Brazil.

The present work has characterised the global marine flooding surfaces (FS) recorded in the Devonian section of the Brazilian Palaeozoic basins as seen in the Parnaíba Basin, using the geochemistry parameter Total Organic Carbon content (TOC) and Pyrolysis data.

Two intervals of interest were recorded, according to their TOC content, in the Devonian section we studied. These were Radioactive Shale B and Radioactive Shale C, recorded in the Pimenteiras Formations (latest Eifelian to late early Givetian in the lower part and Frasnian, but not earliest, to Famennian in the upper part), which are richest in organic matter concentration. The proxies studied in this work show a regional distribution in the basins—probably associated with a coeval anoxic / dysoxic depositional condition over a large area of the basin.

The Late Devonian sections presented in this study demonstrated that progressive flooding took place during Devonian era in the Parnaíba Basin—which, in the Frasnian, shows the maximum marine transgression. These global events are regarded as markers for regional chronostratigraphic correlations in Gondwana.

This global event is also an object of interest for hydrocarbon exploration, as it relates to the identification of source rock intervals. However, the conditions for oil and gas generation for this basin are closely related to the specific evolutionary model, for which the atypical model is the most effective.

APPENDIX

List of species encountered in the material and/or cited in the text

Acritarchs

Advenasphaeridium acerosum Burjack and Oliveira *apud* Oliveira, 1997, *nomen nudum*

Advenasphaeridium australis Burjack and Oliveira 1997, *nomen nudum*

Arkonites bilixus Legault 1973

Bimerga bensonii Wood, 1995

Bipolarisvelata accreta Burjack and Oliveira *apud* Oliveira, 1997, *nomen nudum*

Chomotriletes sp.

Crucidia camirense (Lobo Boneta) Ottone, 1996

Diaphorochroa gracile Burjack and Oliveira *apud* Oliveira, 1997, *nomen nudum*

Disparifusa urariaensis Le Hérissé, 2013, *nomen nudum*

Duvernaysphaera angelae (=D. tessella) Deunff, 1964

Duvernaysphaera capitana (Wicander) Le Hérissé nov.comb

Duvernaysphaera cruciformis Le Hérissé, 2001, *nomen nudum*

Duvernaysphaera heliocentrica Burjack and Oliveira *apud* Oliveira, 1997

Duvernaysphaera radiata Brito, 1967a

Duvernaysphaera stellata Deunff, 1964b

Duvernaysphaera tenuicingulata Staplin, 1961

Eisenackidium triplodermum (Cramer) Eisenack et al., 1973.

Estiastra spinireticulata Oliveira and Burjack, 1997

Exochoderma arca Wicander and Wood, 1981

Exochoderma irregulare Wicander, 1974

Geron elegans Oliveira and Burjack, 1997

Gorgonisphaeridium winslowiae Staplin et al., 1965

Hemiruptia legaultii Ottone, 1996

Hologinella horologia (Staplin) Jardiné, Combaz, Peniguel and Vachey, 1972

Horologinella quadrispina Jardiné, Combaz, Magloire, Peniguel and Vachey, 1972

Leiofusa bisubulata Brito and Quadros, 1984

Leiofusa fastidiona (Cramer and Díez) nov. comb.

Maranhites brasiliensis Brito, 1965 emend Burjack and Oliveira 1989

Maranhites britoi Stockmans and Willière, 1969 R5+R6+R7+R8

Maranhites lobulatus Burjack and Oliveira 1989

Maranhites magnus Burjack and Oliveira 1989

Maranhites mosesii (Sommer, 1956) Brito,1967

Maranhites stockmansii (Martin) Martin, 1984

Mediocorpore conspicuus Burjack and Oliveira *apud* Oliveira, 1997, *nomen nudum*

Multiplicisphaeridium ramusculosum (Deflandre) Lister, 1970

Naevisphaeridium desiderata (Burjack and Oliveira *apud* Oliveira, 1997) *nomen nudum* nov. comb

Naevisphaeridium sprucegrovense (Staplin, 1961) nov. comb.

Navifusa bacilla (Deunff) Playford, 1977

Navifusa bacilla (Deunff) Playford, 1977 forma *crescentis*

Navifusa bacilla var. *procera* (Deunff, 1966) nov.comb.

Palacanthus ledanoisii Deunff, 1957, emend Playford, 1977

Petrovina connata Oliveira and Burjack, 1996

Polyedryxium embudum Cramer, 1964

Polyedryxium fragosulum Playford, 1977

Polyedryxium pharaone Deunff, 1961

Polyedryxium sp. or *Muraticavea munifica* Wicander and Wood, 1981

Pseudolunulidia micropunctata Brito and Quadros, 1984

Pseudomaranhites densus Quadros, 1996

Pterospermopsis crassimarginata Burjack and Oliveira *apud* Oliveira, 2007, *nomen nudum*

Pterospermopsis pernambucensis (Brito) Eisenack et al., 1973

Puteoscortum limai Burjack and Oliveira *apud* Oliveira, 1997

Puteoscortum williereae Martin, 1981

Pyloferites sp.

Stellinium comptum Wicander and Loeblich, 1977

Stellinium divisum Le Hérissé, 2013, *nomen nudum*

Stellinium micropolygonale (Stockmans and Willière) Playford 1977

Stellinium oppidum Deunff, 1980

Tasmanites spp.

Tinacula simplex Quadros, 1999

Tunisphaeridium tentaculaferum (Martin) Cramer, 1971

Tyligmasoma sp. A of Playford in Playford and Dring, 1981

Umbellasphaeridium deflandrei (Moreau-Benoit) Jardine et al., 1972

Umbellasphaeridium saharicum Jardiné, Combaz, Magloire, Peniguel and Vachey, 1972

Unellium ampullium Wicander, 1974

Unellium lunatum (Stockmans and Willière, 1966) Eisenack et al., 1979

Unellium piriforme Rauscher, 1969

Veryhachium arctatum Deunff, 1980

Veryhachium capitata Le Hérissé, 2001, *nomen nudum*

Veryhachium insanum Le Hérissé, 2001, *nomen nudum*

Veryhachium pannuceum Wicander and Loeblich Jr., 1977

Veryhachium roscidum Wicander, 1974

Veryhachium trispinosum (Eisenack) Deunff, 1966

Veryhachium trispinosum/roscidum complex

Winwaloeusia distracta (Deunff) Deunff, 1977

Chitinozoans

Angochitina avelinoi (Lange, 1952)

Angochitina mourai (Lange, 1952)

Angochitina sp. C ? Grahn and Melo, 2005

Lagenochitina sp. A Grahn and Melo, 2005

Fungochitina microspinosa Grahn and Melo, 2005

Fungochitina pilosa Collinson and Scott, 1958

Lagenochitina sommeri (Lange, 1952)

Miospores

Auroraspora macra Sullivan, 1968

Camarozonotriletes concavus Loboziak and Streel, 1988

Chelinospora concinna Allen, 1965

Cyrtospora tumida Breuer and Steemans 2013

Diducites plicabilis Van Veen 1981

Geminospora lemurata Balme emend. Playford, 1983

Geminospora piliformis Loboziak et al., 1983

Grandispora gabesensis Loboziak and Streel, 1989

Grandispora gracilis (Kedo) Streel in Becker et al. 1974

Grandispora libyensis Moreau-Benoit, 1980

Grandispora tabulata Loboziak and Streel, 1988

Lophozonotriletes media Taugourdeau-Lantz, 1967

Rugospora bricei Loboziak and Streel, 1988

Rugospora flexuosa (Jushko) Streel in Becker et al., 1974

Rugospora radiata (Kedo) Byvscheva, 1985

Samarisporites sp. C in Loboziak et al., 1983

Samarisporites triangulatus Allen, 1965

Verruciretusispora magnifica (McGregor) Owens, 1971

Verrucosisporites bulliferus Richardson and Mc Gregor, 1986

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Conflict of interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

P. Steemans

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Fig. 1: Location map of the studied sites.

Fig. 2: Sedimentological compound section of sites 10, 12 and 11, and the correspondent TOC contents of the Pimenteiras Formation in the sites.

Fig. 3: Sedimentological section of site 35, and the correspondent TOC contents of the Pimenteiras Formation in the site.

Fig. 4: Sedimentological section of site 37, and the correspondent TOC contents of the Pimenteiras Formation in the site.

Fig. 5: Miospore biostratigraphic scale from the Old Red Sandstone Continent (A), correlated with Brazil (B). Most probable age of the analysed samples (C). Extension of the most diagnostic species of Acritarchs and other microalgal remains recognised in the material studied, age and correlation of the interval represented.

Fig. 6: Miospore biostratigraphic scale from the Old Red Sandstone Continent (A), correlated with Brazil (B). Based on the biostratigraphic range observed in the Boulonais sections, France (Streel et al., in preparation) of some important species. Most probable age of the analysed samples (C)

Tbl. 1: Pyrolysis and Total Organic Carbon content (TOC) data from Sites 10, 11, 12 and 35 using Rock-Eval Pyrolysis method.

Profundidade	**Barquinha**	**Barq.+Amost.**	**Descar.**	**Peso**	**R.I**	**COT %**	**S %**		**Massa (mg)**	**S1 (mg/g)**	**S2(mg/g)**	**S3 (mg/g)**	**Tmax(° C)**	**COT %**	**IH**	**IO**
AMT 01	**37.049**	**37.302**	**37.297**	**0.253**	**98**	**1.40**	**0.17**	AM-1	63.02	0.05	4.75	0.2	425	1.40	339	14

AMT 01 A	**38.237**	**38.485**	**38.481**	**0.248**	**98**	**1.29**	**0.10**	1-TOA	54.69	0.07	4.46	0.16	431	1.29	346	12
AMT 02	**37.907**	**38.161**	**38.158**	**0.254**	**99**	**1.47**	**0.05**	2-TO	58.99	0.07	5.11	0.4	426	1.47	348	27
AMT 03	**36.953**	**37.206**	**37.202**	**0.253**	**98**	**1.22**	**0.05**	3-TO	56.68	0.07	3.79	0.24	426	1.22	311	20
AMT 04	**38.079**	**38.331**	**38.329**	**0.252**	**99**	**1.24**	**0.05**	4 TO	51.7	0.05	4.41	0.14	428	1.24	356	11
AMT 05	**38.421**	**38.670**	**38.656**	**0.249**	**94**	**1.16**	**0.11**	5 TO	73.5	0.04	1.65	0.15	430	1.16	142	13
AMT 05 A	**37.116**	**37.370**	**37.361**	**0.254**	**96**	**1.10**	**0.07**	5A TO	56.74	0.02	0.91	0.29	430	1.1	83	26
AMT 06	**37.736**	**37.984**	**37.973**	**0.248**	**96**	**0.77**	**0.11**									
AMT 07	**37.297**	**37.547**	**37.526**	**0.250**	**92**	**0.55**	**0.05**									
AMT 07 REP	**37.328**	**37.578**	**37.562**	**0.250**	**94**	**0.54**	**0.07**									
AMT 08	**37.706**	**37.957**	**37.945**	**0.251**	**95**	**0.98**	**0.10**	8TO	56.28	0.03	0.69	0.22	429	0.98	70	22
AMT 09	**38.005**	**38.256**	**38.247**	**0.251**	**96**	**2.21**	**2.02**	9 TO	67.1	0.05	3.3	0.17	422	2.21	149	8
AMT 10	**38.567**	**38.817**	**38.810**	**0.250**	**97**	**2.01**	**0.46**	10 TO	48.24	0.06	4.64	0.41	427	2.01	231	20
AMT 11	**37.753**	**38.002**	**37.993**	**0.249**	**96**	**1.34**	**0.20**	11 TO	49.5	0.06	2.21	0.36	423	1.34	165	27
AMT 12	**37.425**	**37.679**	**37.673**	**0.254**	**98**	**1.31**	**0.10**	12 TO	61.1	0.08	2.09	0.54	425	1.31	160	41

AMT 13	**38.896**	**39.149**	**39.137**	**0.253**	**95**	**6.57**	**2.16**	13 TO	48.41	0.17	9.55	0.92	417	6.57	145	14
AMT 14	**37.809**	**38.059**	**38.054**	**0.250**	**98**	**3.38**	**0.45**	14 TO	60.5	0.11	7.99	0.73	427	3.38	236	22
AMT 15	**37.231**	**37.482**	**37.466**	**0.251**	**94**	**3.09**	**0.40**	15 TO	51.2	0.1	3.14	1.22	418	3.09	102	39
AMT 16	**37.394**	**37.644**	**37.637**	**0.250**	**97**	**2.51**	**0.40**	16 TO	53	0.07	3.54	0.71	424	2.51	141	28
AMT 17	**38.948**	**39.201**	**39.195**	**0.253**	**98**	**3.19**	**0.28**	17 TO	51.7	0.11	7.78	1.06	427	3.19	244	33
AMT 18	**36.971**	**37.220**	**37.216**	**0.249**	**98**	**2.68**	**0.24**	18 TO	73.1	0.07	7.31	0.44	428	2.68	273	16
AMT 19	**38.099**	**38.349**	**38.200**	**0.250**	**40**	**1.00**	**0.07**	19 TO	62.6	0.02	0.47	2.47	433	1	47	247
AMT 20	**37.465**	**37.714**	**37.584**	**0.249**	**48**	**0.76**	**0.17**									
AMT 21	**38.130**	**38.381**	**38.260**	**0.251**	**52**	**1.68**	**0.09**	21TO	51.7	0.02	0.93	1.04	435	1.68	55	62
AMT 22	**37.932**	**38.182**	**38.046**	**0.250**	**46**	**0.75**	**0.18**									
AMT 23	**38.606**	**38.856**	**38.847**	**0.250**	**96**	**1.18**	**0.07**	23 TO	55.3	0.06	2.31	0.32	427	1.18	196	27
AMT 24	**37.981**	**38.232**	**38.229**	**0.251**	**99**	**2.01**	**0.10**	24 TO	62.4	0.07	4.31	0.74	426	2.01	214	37
AMT 25	**37.994**	**38.246**	**38.243**	**0.252**	**99**	**0.88**	**0.08**									
AMT 25 REP	**39.173**	**39.423**	**39.418**	**0.250**	**98**	**0.89**	**0.08**									

AMT 26	**37.961**	**38.214**	**38.212**	**0.253**	**99**	**1.97**	**0.26**	26 TO	51.3	0.06	4.06	0.27	427	1.97	206	14
AMT 27	**37.356**	**37.605**	**37.601**	**0.249**	**98**	**2.35**	**0.37**	27 TO	51.6	0.08	4.14	0.62	424	2.35	176	26
AMT 28	**37.105**	**37.357**	**37.332**	**0.252**	**90**	**1.59**	**0.28**	28 TO	50.2	0.05	0.9	1.03	419	1.59	57	65
AMT 29	**38.741**	**38.993**	**38.984**	**0.252**	**96**	**0.89**	**0.08**									
AMT 30	**36.572**	**36.821**	**36.812**	**0.249**	**96**	**0.90**	**0.11**	30 TO	66	0.03	0.55	0.33	428	0.9	61	37
AMT 31	**38.380**	**38.628**	**38.615**	**0.248**	**95**	**0.97**	**0.14**	31 TO	47.6	0.03	0.44	0.4	422	0.97	45	41
AMT 32	**38.480**	**38.731**	**38.727**	**0.251**	**98**	**1.48**	**0.13**	32 TO	51.7	0.06	2.48	0.56	425	1.48	168	38
AMT 33	**38.240**	**38.487**	**38.482**	**0.247**	**98**	**1.34**	**0.31**	33 TO	53.6	0.05	1.6	0.23	421	1.34	119	17
AMT 34	**37.565**	**37.816**	**37.809**	**0.251**	**97**	**1.14**	**0.13**	34 TO	54.2	0.04	1.66	0.28	422	1.14	146	25
AMT 35	**37.702**	**37.952**	**37.929**	**0.250**	**91**	**0.28**	**0.07**									
AMT 36	**39.198**	**39.449**	**39.431**	**0.251**	**93**	**0.19**	**0.08**									
AMT 37	**38.857**	**39.110**	**39.070**	**0.253**	**84**	**0.39**	**0.11**									
AMT 37 ARG BRC	**37.783**	**38.035**	**38.012**	**0.252**	**91**	**1.00**	**0.18**	37 ARG BRC	67.77	0.02	0.82	0.22	426	1.00	82	22
AMT 38	**38.417**	**38.665**	**38.638**	**0.248**	**89**	**0.32**	**0.11**									

AMT 39	**38.055**	**38.306**	**38.292**	**0.251**	**94**	**3.46**	**0.14**	39 TO	53.93	0.11	4.62	1.64	422	3.46	134	47
AMT 40	**38.579**	**38.829**	**38.821**	**0.250**	**97**	**2.78**	**0.18**	40 TO	61.65	0.08	3.21	1.48	419	2.78	115	53
AMT 41	**38.995**	**39.242**	**39.224**	**0.247**	**93**	**3.14**	**0.19**	41 TO	56.13	0.11	5.05	2.36	425	3.14	161	75
AMT 42	**37.907**	**38.156**	**38.150**	**0.249**	**98**	**3.96**	**1.06**	42 TO	59.47	0.11	8.6	0.5	425	3.96	217	13
AMT 42 REP	**37.296**	**37.545**	**37.535**	**0.249**	**96**	**3.93**	**1.06**									
AMT 43	**38.720**	**38.972**	**38.970**	**0.252**	**99**	**0.00**	**0.04**									
AMT 44	**37.606**	**37.857**	**37.828**	**0.251**	**88**	**0.62**	**0.07**									
AMT 45	**37.752**	**38.002**	**37.995**	**0.250**	**97**	**0.35**	**0.05**									
AMT 46	**38.001**	**38.249**	**38.229**	**0.248**	**92**	**0.28**	**0.04**									
AMT 47	**38.987**	**39.237**	**39.225**	**0.250**	**95**	**0.26**	**0.05**									
AMT 48	**37.161**	**37.411**	**37.385**	**0.250**	**90**	**4.24**	**0.29**	48 TO	82.47	0.13	4.99	1.64	420	4.24	118	39
AMT 49	**37.803**	**38.053**	**38.045**	**0.250**	**97**	**1.94**	**1.31**	49 TO	83.31	0.03	2.86	0.2	430	1.94	147	10
AMT 50	**38.894**	**39.140**	**39.120**	**0.246**	**92**	**6.95**	**2.01**	50 TO	45.56	0.26	11.28	1.95	420	6.95	162	28
AMT 51	**38.847**	**39.097**	**39.087**	**0.250**	**96**	**2.43**	**0.00**	51 TO	46.81	0.05	4.47	0.11	427	2.43	184	5

AMT 52	**37.475**	**37.727**	**37.712**	**0.252**	**94**	**1.53**	**0.19**	52 TO	38.3	0.06	1.86	0.94	424	1.53	122	61
AMT 53	**37.365**	**37.618**	**37.613**	**0.253**	**98**	**0.84**	**0.41**									
AMT 54	**37.726**	**37.978**	**37.970**	**0.252**	**97**	**1.00**	**0.10**	54 TO	62.7	0.04	2.82	0.38	432	1	282	38
AMT 55	**38.542**	**38.795**	**38.793**	**0.253**	**99**	**3.05**	**0.31**	55 TO	51.1	0.05	9.26	0.76	429	3.05	304	25
AMT 58	**37.930**	**38.183**	**38.180**	**0.253**	**99**	**2.37**	**0.78**	58 TO	38.8	0.05	7.36	0.3	429	2.37	311	13
AMT 59	**37.178**	**37.429**	**37.425**	**0.251**	**98**	**2.75**	**0.11**	59 TO	42.1	0.08	9.87	0.82	431	2.75	359	30
AMT 60	**39.030**	**39.281**	**39.276**	**0.251**	**98**	**2.30**	**0.71**	60 TO	48.2	0.04	6.15	0.49	426	2.3	267	21
AMT 61	**38.214**	**38.465**	**38.461**	**0.251**	**98**	**2.76**	**0.85**	61 TO	58.7	0.06	7.9	0.31	427	2.76	286	11
AMT 62	**36.806**	**37.059**	**37.053**	**0.253**	**98**	**3.03**	**1.05**	62 TO	44.9	0.07	10.18	0.25	428	3.03	336	8
AMT 62 REP	**39.236**	**39.489**	**39.485**	**0.253**	**98**	**3.06**	**1.05**									
AMT 63	**38.887**	**39.138**	**39.135**	**0.251**	**99**	**2.70**	**0.31**	63 TO	50.2	0.07	8.6	0.47	429	2.7	319	17
AMT 64	**39.396**	**39.645**	**39.641**	**0.249**	**98**	**2.38**	**0.13**	64 TO	62.9	0.06	6.18	0.97	427	2.38	260	41
AMT 65	**37.615**	**37.865**	**37.862**	**0.250**	**99**	**2.31**	**0.44**	65 TO	46.5	0.05	7.2	0.52	426	2.31	312	23
AMT 66	**37.219**	**37.471**	**37.468**	**0.252**	**99**	**2.32**	**0.13**	66 TO	57.9	0.07	7.61	0.79	431	2.32	328	34

AMT 67	**38.217**	**38.471**	**38.465**	**0.254**	**98**	**3.05**	**1.45**	67 TO	51.2	0.09	8.94	0.24	427	3.05	293	8
AMT 68	**37.502**	**37.753**	**37.746**	**0.251**	**97**	**3.49**	**0.43**	68 TO	41.2	0.12	9.27	0.9	427	3.49	266	26
AMT 69	**39.032**	**39.282**	**39.274**	**0.250**	**97**	**1.72**	**0.28**	69 TO	59.7	0.06	2.69	0.75	421	1.72	156	44
AMT 70	**38.590**	**38.841**	**38.840**	**0.251**	**100**	**0.99**	**0.12**	70 TO	50.7	0.03	1.52	0.39	429	0.99	154	39
AMT 71	**38.054**	**38.307**	**38.301**	**0.253**	**98**	**1.24**	**0.46**	71 TO	55.3	0.04	1.83	0.18	427	1.24	148	15
AMT 72	**38.912**	**39.158**	**39.148**	**0.246**	**96**	**0.92**	**0.32**									
AMT 73	**37.025**	**37.278**	**37.272**	**0.253**	**98**	**1.01**	**0.16**	73 TO	47.9	0.03	1.17	0.28	427	1.01	116	28
AMT 74	**37.726**	**37.975**	**37.968**	**0.249**	**97**	**1.22**	**0.54**	74TO	61	0.03	0.87	0.24	420	1.22	71	20
AMT 75	**37.450**	**37.701**	**37.694**	**0.251**	**97**	**2.80**	**0.27**	75 TO	45.4	0.13	8.99	1.73	424	2.8	321	62
AMT 76	**38.900**	**39.150**	**39.145**	**0.250**	**98**	**4.20**	**1.24**	76 TO	47.4	0.03	5.06	0.31	433	4.2	120	7
AMT 77	**38.194**	**38.444**	**38.441**	**0.250**	**99**	**3.82**	**0.56**	77 TO	40.4	0.04	3.93	0.45	432	3.82	103	12
AMT 78	**38.330**	**38.580**	**38.575**	**0.250**	**98**	**3.73**	**0.32**	78 TO	49.8	0.03	3.72	0.21	432	3.73	100	6
AMT 80	**37.050**	**37.302**	**37.294**	**0.252**	**97**	**0.43**	**0.12**									
AMT 81	**37.592**	**37.842**	**37.824**	**0.250**	**93**	**0.91**	**0.16**									

AMT 81 REP	**38.820**	**39.070**	**39.053**	**0.250**	**93**	**0.91**	**0.16**									
AMT 82	**37.531**	**37.782**	**37.765**	**0.251**	**93**	**1.27**	**0.19**	82 TO	65.3	0.08	4.31	0.24	424	1.27	339	19
AMT 83	**39.256**	**39.509**	**39.504**	**0.253**	**98**	**1.00**	**0.12**	83 TO	75.7	0.06	2.07	0.31	421	1.00	207	31
AMT 84	**36.700**	**36.952**	**36.950**	**0.252**	**99**	**1.04**	**0.12**	84 TO	62.8	0.08	2.12	0.23	422	1.04	204	22
AMT 85	**37.733**	**37.981**	**37.977**	**0.248**	**98**	**2.69**	**1.03**	85 TO	53.4	0.07	3.02	0.35	422	2.69	112	13
AMT 86	**38.273**	**38.522**	**38.502**	**0.249**	**92**	**2.46**	**3.85**	86 TO	64.7	0.05	2.99	0.27	421	2.46	122	11
AMT 87	**37.968**	**38.217**	**38.204**	**0.249**	**95**	**2.50**	**2.89**	87 TO	45.3	0.22	5.89	1.15	428	2.50	236	46
AMT 88	**38.769**	**39.017**	**39.008**	**0.248**	**96**	**2.84**	**4.15**	88 TO	47.8	0.06	3.27	0.65	425	2.84	115	23
AMT 89	**38.549**	**38.802**	**38.800**	**0.253**	**99**	**2.05**	**0.10**	89 TO	48.8	0.04	1.16	0.56	426	2.05	57	27
AMT 90	**37.435**	**37.687**	**37.685**	**0.252**	**99**	**1.83**	**0.07**	90 TO	42.6	0.04	1.63	0.7	425	1.83	89	38
AMT 91	**37.112**	**37.365**	**37.365**	**0.253**	**100**	**1.69**	**0.24**	91 TO	53.9	0.08	4.24	1.47	423	1.69	251	87
AMT 92	**38.289**	**38.541**	**38.540**	**0.252**	**100**	**1.00**	**0.10**	92 TO	53.9	0.03	1.05	0.48	425	1.00	105	48
AMT 93	**36.732**	**36.983**	**36.982**	**0.251**	**100**	**0.98**	**0.05**	93 TO	40.3	0.04	1.21	0.44	428	0.98	123	45
AMT 94	**37.730**	**37.984**	**37.980**	**0.254**	**98**	**0.85**	**0.06**									

AMT 95	**38.691**	**38.943**	**38.940**	**0.252**	**99**	**1.44**	**0.09**	95 TO	60.5	0.06	2.58	0.85	427	1.44	179	59
AMT 51	**38.319**	**38.444**	**38.438**	**0.125**	**95**	**2.21**	**11.60**									

Site	Samples	S1 (mg HC/g rock)	S2 (mg HC/g rock)	S3 (mg CO2/g rock)	Tmax(°C)	TOC (%)	HI (mg HC/g TOC)
11	**12**	0.08	2.09	0.54	425	1.31	160
	11	0.06	2.21	0.36	423	1.34	165
	10	0.06	4.64	0.41	427	2.01	231
	9	0.05	3.30	0.17	422	2.21	149
12	**18**	0.07	7.31	0.44	428	2.68	273
	17	0.11	7.78	1.06	427	3.19	244
	16	0.07	3.54	0.71	424	2.51	141
	15	0.10	3.14	1.22	418	3.09	102
	14	0.11	7.99	0.73	427	3.38	236
	13	0.17	9.55	0.92	417	6.57	145
10	**8**	0.03	0.69	0.22	429	0.98	70
	7						
	6						
	5	0.04	1.65	0.15	430	1.16	142
	48	0.13	4.99	1.64	420	4.24	118
	49	0.03	2.86	0.20	430	1.94	147
	50	0.26	11.28	1.95	420	6.95	162
	51	0.05	4.47	0.11	427	2.43	184
	52	0.06	1.86	0.94	424	1.53	122
	54	0.04	2.82	0.38	432	1.00	282
	55	0.05	9.26	0.76	429	3.05	304
	58	0.06	7.36	0.30	429	2.37	311
	59	0.08	9.87	0.82	431	2.75	359
	60	0.04	6.15	0.49	426	2.30	267
	61	0.06	7.90	0.31	427	2.76	286
	62	0.07	10.18	0.25	428	3.03	336
35							
	63	0.07	8.60	0.47	429	2.70	319
	64	0.06	6.18	0.97	427	2.38	260
	65	0.05	7.20	0.52	426	2.31	312

	66	0.07	7.61	0.79	431	2.32	328
	67	0.09	8.94	0.24	427	3.05	293
	68	0.12	9.27	0.90	427	3.49	266
	69	0.06	2.69	0.75	421	1.72	156
	70	0.03	1.52	0.39	429	0.99	154
	71	0.04	1.83	0.18	427	1.24	148
	73	0.03	1.17	0.28	427	1.01	116
	74	0.03	0.87	0.24	420	1.22	71
	75	0.13	8.99	1.73	424	2.80	321
	76	0.03	5.06	0.31	433	4.20	120
	77	0.04	3.93	0.45	432	3.82	103
	78	0.03	3.72	0.21	432	3.73	100

	82	0.08	4.31	0.24	424	1.27	339
	83	0.06	2.07	0.31	421	1.00	207
	84	0.08	2.12	0.23	422	1.04	204
	85	0.07	3.02	0.35	422	2.69	112
	86	0.05	2.99	0.27	421	2.46	122
37	87	0.22	5.89	1.15	428	2.50	236
	88	0.06	3.27	0.65	425	2.84	115
	89	0.04	1.16	0.56	426	2.05	57
	90	0.04	1.63	0.7	425	1.83	89
	91	0.08	4.24	1.41	423	1.69	251

samples	**TOC (%)**
1	1.31
2	1.34
3	2.01
4	2.21
5	2.68
6	3.19
7	2.51
8	3.09
9	3.38
10	6.57
11	0.98
12	1.16

Plate I

Acritarchs: Specimens are located according the sample number and the England finders coordinates. The diameter size is provided in µm.

1. *Maranhites brasiliensis* Brito, 1965 emend Burjack and Oliveira 1989. Site 11, AM 12, 18K 224, D24/2, total diameter 125 µm

2. *Maranhites lobulatus* Burjack and Oliveira 1989, Site 11, AM 12, 18K 224, Q26, total diameter 145 µm

3. *Maranhites stockmansii* (Martin) Martin, 1984, Site 11, AM 12, 18K 224, Q26, total diameter 145 µm

4. *Maranhites mosesii* (= *M. insulatus* Burjack and Oliveira, 1989) (Sommer, 1956) Brito,1967. Site 11, AM 12, 18K 224, E35/4, total diameter 120 µm

5. *Petrovina connata* Oliveira and Burjack, 1996. Site 10, AM 7, 18K 220, K13/4, total diameter 145 µm, cells 45 µm.

6. *Pterospermopsis crassimarginata* Burjack and Oliveira *apud* Oliveira, 2007, *nomen nudum*. Site 11, AM 12, 18K 224, D19/2, total diameter 45-50 µm

7. *Crucidia camirense* (Lobo Boneta) Ottone, 1996, Site 10, AM 7, 18K 220, F16/3, total total lenght of the processes 80-85 µm.

8. *Pseudolunulidia micropunctata* Brito and Quadros, 1984. Site 35, AM 68, 18L30, 77131, J44/2, central body width 41 µm.

9. *Navifusa bacilla* (Deunff) Playford, 1977 forma *crescentis*. Site 10, AM 7, 18K220, T42/4, width of central body 25 µm.

10. *Disparifusa urariaensis* Le Hérissé, 2013, *nomen nudum*, Site 11, AM 12, 18K224, N15/2, length 52 µm, width 22 µm.

11. *Umbellasphaeridium deflandrei* (Moreau-Benoit) Jardiné et al., 1972. Site 11, AM 10, 18K 223, L31/2, total diameter 38 µm

12, 13. *Hologinella quadrispina* Jardiné, et al., 1972. 12. Site 11, AM 9, 18K 222, H17/4, central body 45x50 µm, process length 6-10 µm. 13, Site 35, AM 78, 18L33, 77146, G29, central body 42x24 µm, process length 14.5 µm

14-16. *Duvernaysphaera cruciformis* Le Hérissé, 2001, *nomen nudum sp. nov.sp.*14. Paratype, site 35, AM 58, 18L27, 77116, S36, central body 30 µm, flange 5,6 µm; 15. Paratype, site 35, AM 58, 18L27, 77116, P32, central body 36 µm, flange 3 µm; 16. Holotype, site 35, AM 76, 18L32, 77141, Q28, central body 36 µm, flange 6,7 µm

17. *Duvernaysphaera angelae (=D. tessella)* Deunff, 1964. Site 11, AM 12, 18K224, Q38/3, Total diameter 45 µm.

Plate II

Acritarchs: Specimens are located according the sample number and the England finders coordinates. The diameter size is provided in µm.

1. *Mediocorpore conspicuus* Burjack and Oliveira *apud* Oliveira, 1997, *nomen nudum.* Site 11, AM 12, 18K 224, E16/2, total length 1-à µm, width, 40 µm, length of the central body 85 µm.

2. *Duvernaysphaera radiata* Brito, 1967a. Site 11, AM 12, 18K 224, M43/3, total diameter 140 µm.

3. *Puteoscortum williereae* Martin, 1981. Site 35, AM 78, 77148, C40/3, CC 42 µm, LP 21 µm

4. *Advenasphaeridium australis* Burjack and Oliveira 1997, *nomen nudum.* Site 11, AM 11, 18K 223, H43/2, central body 39 µm, Lp 33 µm

5. *Puteoscortum williereae* Martin, 1981. Site 37, AM 89, 77163, S41, CC 33 µm, Lp 22 µm

6. *Leiofusa bisubulata* Brito and Quadros, 1984. Site 35, AM 68, 77133, Q 29/4, central body 35x156 µm, Lp 56 µm.

7-9. ?*Chomotriletes* sp. A. 7. Site 11, AM 9, 18K 222, J13/4, total diameter 56 µm. 8. Site 37, AM 85, 77151, P15, total diameter 31 µm; 9. Site 37, AM 85, 77151, N32 Total diameter 50 µm.

10. *Unellium ampullium* Wicander, 1974. Site 11, AM 12, 18K 224, V28/2, central body 18 µm, process length 20 µm.

11. *Diaphorochroa gracile* Burjack and Oliveira *apud* Oliveira, 1997, *nomen nudum* . Site 35, AM 65, 77126, M43, CC 40 µm, LP 18 µm

12. *Naevisphaeridium sprucegrovense* (Staplin, 1961) nov. comb. Site 35, AM 65, 77126, 039/4, CC 28 µm, LP 12 µm

13. *Naevisphaeridium desiderata* (Burjack and Oliveira *apud* Oliveira, 1997) *nomen nudum nov comb.* Site 37, AM 85, 77151, H36/1. Central body 31 µm, Lp 11 µm

14. *Puteoscortum limai* Burjack and Oliveira *apud* Oliveira, 1997. Site 11, AM 11, 18K 223, P25/4, central body 48 µm, Lp 22 µm

15. *Stellinium micropolygonale* (Stockmans and Willière) Playford 1977. Site 10, AM 7, 18K220, F14/4, central body 33, Lp 48 µm

16. *Estiastra spinireticulata* Oliveira and Burjack, 1997. Site 35, AM 78, 77146, U33/4, CC 33 µm, LP 10 µm

17. *Veryhachium capitata* Le Hérissé, 2001 *nomen nudum* . Site 35, AM 75, 77136, J42/3, CC 22 µm, LP 9 µm.

18. *Veryhachium insanum* Le Hérissé, 2001, *nomen nudum*. Site 35, AM 78, 77146, U33/4, CC 24.5 µm, LP 9 µm

19. *Tinacula simplex* Quadros, 1999 Site 35, AM 58, 18L27, 77118, R39, length 89 µm, maximum width 54 µm

20. *Bipolarisvelata accreta* Burjack and Oliveira *apud* Oliveira, 1997, *nomen nudum*.Site 11, AM 12, 18K 224, E26, central body 55x35 µm, polar filamentous projections 50-65 µm long.

Plate III

Chitinozoans: Specimens are located according the sample number and the England finders coordinates. The scale bar is annoted in µm.

1, 2. *Angocitina avelinoi* (Lange 1952). Site 11, AM11 15, O22/0; AM11 15, R30/3.

3, 4. *Angochitina mourai* (Lange 1952). Site 35, Am49 40, C32/0; Am58 50, M23/0.

5, 6. *Angochitina mourai* long neck variant *sensu* Grahn and Melo 2005. Site 37, Am85 85, H14/4; AM 58 50 U23/4.

7, 8. *Angochitina* sp.C? Grahn and Melo 2005. Site 37, AM89 95, X14/4; AM89 95, M17/3.

9, 10. *Fungochitina microspinosa* Grahn and Melo 2005. Site 37, AM89 95, M17/2; AM65 60, W24/0.

Plate IV

Chitinozoans: Specimens are located according the sample number and the England finders coordinates. The scale bar is annoted in µm.

1, 2. *Fungochitina pilosa*? (Collinson and Scott 1958). Site 35, AM65 60, C29/0; AM82 55, K17/2.

3, 4. *Lagenochitina sommeri* (Lange 1952). Site 11, AM9 11, J25/3; AM89 95, U29/0.

5, 6. *Lagenochitina* sp.A Grahn and Melo 2005. Site 37 AM89 95, O24/0; AM11 15, K27/1.

Plate V

Miospores: Specimens are located according the sample number and the England finders coordinates. The scale bar is annoted in µm.

1-3. *Acinosporites acanthomammillatus* Richardson, 1965. 1 : Site 10 Am7 2, K19/2 ; 2 : Site 10 Am7 2, M21/0 ; Site 11 Am9 10, D17/3.

4-6. *Acinosporites eumammillatus* Loboziak et al., 1988. 4 : Site 10 Am7 2, Q17/3 ; 5 : Site 10 Am7 2, D17/1/3 ; Site 10 Am7 2, K23/0.

7-9. *Acinosporites lindlarensis* Riegel, 1968. 7 : Site 35 Am89 96 O15/3 ; 8 : Site 35 Am89 96 A, Q20/2 ; Site 35 Am89 96 B Q20/2.

10. *Ancyrospora langii* (Taugourdeau-Lantz) Allen, 1965. Site 35 Am65 60, G23/1.

11. *Ancyrospora simplex* Guennel, 1963. Site 35 Am76 75, W28/2.

Plate VI

Miospores: Specimens are located according the sample number and the England finders coordinates. The scale bar is annoted in µm.

1-3. *Ancyrospora* sp. 1. 1 : Site 35 Am 65 60, V18/2 ; 2 : Site 35 Am75 70, F34/1 ; 3 : Site 35 Am75 70, F34/1.

4-5. *Aneurospora gregsii Aneurospora greggsii* (McGregor) Streel, 1974. 4 : Site 10 Am8 6, N15/2 ; 5 : Site 11 Am9 10, T17/2.

6-7. *Apiculiretusispora* sp. 6 : Site 10 Am7 2, K18/1 ; 7 : Site 10 Am7 2, N16/1-3.

8. *Archaeozonotriletes* sp. Am 68 65, U23/1.

9-11. *Archaeozonotriletes variabilis* Naumova, 1953. 9: Site 11 Am7 2, E17/0 ; 10 : Site 11 Am9 10, V19/4 ; 11 : site 11 Am9 10, G22/0.

12. *Auroraspora* sp. Site 11 Am8 6, T18/0.

Plate VII

Miospores: Specimens are located according the sample number and the England finders coordinates. The scale bar is annoted in µm.

1-3. *Auroraspora macra* Sullivan, 1968. 1 : Site 10 Am7 2, V17/2 ; 2 : Site 10 Am7 2, R19/3-4 ; 3 : Site 10 Am7 2, C23/2.

4-5. *Camarozonotriletes concavus* Loboziak et Streel, 1988. 4 : Site 10 Am7 2, O24/4 ; 5 : Site 35 Am89 95, M19/0.

6, 9. *Chelinospora concinna* Allen, 1965. Two different foci on the same specimen ; Site 10 Am7 2, V19/3B.

7-9. *Chelinospora paravermiculata* Loboziak et al., 1988. 8 : site 11 Am9 10, N18/0 ; 9 : site 35 Am 89 96, V20/0.

10. *Chelinospora* sp. Site 10 Am8 6, D16/3.

11-12. *Corystisporites corystus* Richardson, 1965. 11 : site 35 Am68 65, T26/2 ; 12 : site 35 Am85 85, 732-895.

Plate VIII

Miospores: Specimens are located according the sample number and the England finders coordinates. The scale bar is annoted in µm.

1. *Cymbosporites catillus* Allen, 1965. Site 10, Am8 6, K27/3.

2. *Cymbosporites cyathus* Allen, 1965 . Site 10, Am8 6, U19/3.

3-6. *Cymbosporites magnificus* (McGregor) McGregor and Camfield 1982. 3 : Site 10 Am7 2, R16/0 ; 4 : Site 10, Am 7 2, T16/3 ; 5 : Site 10, Am8 6 , W23/0 ; 6 : Site 11, Am9 10, J16/3.

7. *Cyrtospora* sp. Site 10, Am 7 K18/1-3.

8-9. *Cyrtospora tumida* Breuer and Steemans 2013. Site 10, Am8 6, M22/1; Site 10 Am8 6, M26/0.

10. *Diatomozononotriletes franfklinii* McGregor and Camfield 1982. Site 10 Am 7 2, 017/2.

11-12. *Dibolisporites* sp. 1. 11 : Site 10 Am7 2, R22/3 ; 12 : Site 11, Am9 10, H17/0.

Plate IX

Miospores: Specimens are located according the sample number and the England finders coordinates. The scale bar is annoted in µm.

1. *Dibolisporites* sp. 1. Am78 80, W32-0.

2. *Emphanisporites rotatus* McGregor emend. McGregor, 1973. Site 10, Am7 2, N21/3.

3-4. *Geminospora convoluta* Breuer and Steemans, 2013. 3: Site 10 Am7 2, M23/4; 4 : site 37, Am89 95, F24/4.

5-6. *Geminospora lemurata* Balme emend. Playford, 1983. 5 : Site 10, Am7 2, X16/2 ; 6 : Site 10, Am 7 2, B17/0.

7-8. *Geminospora macromanifesta* (Naumova) Arkhangelska 1953. 7 : Site 35, Am76 75, N28/3 ; 8 : Site 37 Am 88 90, O28/0.

Plate X

Miospores: Specimens are located according the sample number and the England finders coordinates. The scale bar is annoted in µm.

1-3. *Geminospora punctata* Owens, 1971. 1 : Site 10, Am8 6, K16/0 ; 2 : Site 10, Am8 6, Q20/3 ; 3 : Site 10, Am7 2, F16/3.

4-5. *Grandispora gabesensis* Loboziak and Streel, 1989. 4 : Site 37, Am89 95, X22/1-3 ; 5 : Site 37 Am89 95, Q19/0.

6-7. *Grandispora libyensis* Moreau-Benoit, 1980. 6 : Site 35, Am58 50, M26/0 ; 7: Site 35, Am62 55, U24/0.

Plate XI

Miospores: Specimens are located according the sample number and the England finders coordinates. The scale bar is annoted in µm.

1-2. *Grandispora naumovae* (Kedo) McGregor, 1973. 1: Site 37 Am85 85, Q29/0 ; 2 : Site 12, Am17 35, V20/0.

3-4. *Grandispora permulta* (Daemon) Loboziak et al. 1999. 3 : Site 10, Am8 6, M19/0 ; 4 : Site 37, Am89 96, V17/0.

5-6. cf. *Grandispora* sp . 1. 5 : Site 35 Am68 65, T18/0 ; 6 : Site 37, Am89 95, L27/4.

Pl. XII

Miospores: Specimens are located according the sample number and the England finders coordinates. The scale bar is annoted in µm.

1-2. *Grandispora* sp. 2. 1 : Site 37, Am89 95, N14/0 ; 2 : Site 35, Am65 60, Q15/3.

3. *Grandispora* sp.3. Site 35, Am76 75 ; P20/0.

4-5. *Grandispora* sp. 4. 4 : Site 35, Am 76 75, P20/0 ; Site 35, Am75 70, L35/3.

6. *Grandispora* sp. A in Loboziak et al., 1981. Site 3, Am88 90, V20-3.

Pl. XIII

Miospores: Specimens are located according the sample number and the England finders coordinates. The scale bar is annoted in µm.

1-2. *Grandispora tabulata* Loboziak and Streel, 1988. 1 : Site 37, Am89 96, X17/2 ; 2 : Site 10, Am7 2, B21/3.

3. *Incertae sedis*. Site 35, Am78 80 ; P19/0**.**

4. *Grandispora velata* (Richardson) McGregor, 1973. Site 35, Am85 85, X31/3.

5. *Hystricosporites sp.* Site 35, Am58 50, V25/0.

6. *Knoxisporites dedaleus* (Naumova) Streel, 1977. Site 10, Am7 2, G16/4.

7-8. *Lophozonotriletes dentatus* Hughes and Playford, 1961. 7: Site 10 Am7 2, S22/3 ; 8 : Site 10, Am8 6, T16/1.

9-10. *Lophozonotriletes media* Taugourdeau-Lantz, 1967. 9; Site 10 Am7 2, L16/0 ; 10 : H16/2.

11. *Lopozonotriletes* sp. Site 10, Am 8 6 ; Q15/4.

Pl. XIV

Miospores: Specimens are located according the sample number and the England finders coordinates. The scale bar is annoted in µm.

1-4 n. g. n. sp. 1: site 35, Am19 4, V20/0 ; 2 : Site 11, Am9 10, V20/0 ; 3 : Site 11, Am9 10, X22/3 ; 4 : Site 11, Am9 10 , P16/0.

5. *Raistirickia* sp. A in Richardson 1965: Site 11, Am9 10, Q18/2.

6. *Retusotriletes* sp. Site 10, Am7 2, J21/2.

7-8. *Rugospora* cf. *Rugospora bricei* in Loboziak and Streel, 1988: Site 10, Am7 2, B19/0 ; 8 : Site 10, Am7 2, O18/36.

9. *Samarisporites* sp. , Site 10, Am7 2, F19/3.

10-12. *Samarisporites* sp. C in Loboziak et al. 1983. 10 : Site 10, Am7 2, T16/3 ; 11 : Site 10, Am 7 2, 12, V24/1 ; Site 11, Am9 10, O17/2.

Pl. XV

Miospores: Specimens are located according the sample number and the England finders coordinates. The scale bar is annoted in µm.

1-3. *Samarisporites triangulatus* Allen, 1965. 1 : Site 10, Am8 6, X21/2 ; 2 : Site 11, Site 11, Am9 10, Q22/1 ; 3 : Site 10, Am7 2 ; G21/0.

4-6. *Verrucosisporites bulliferus* Richardson et Mc Gregor, 1986.4 : Site 10, Am7 2, V16/4 ; 5 : Site 37, Am89 96 ; V17/1 ; 6 : Site 7, Am 8 6, G27/4.

7-9. *Verrucosisporites premnus-scurrus* Morphon Breuer and Steemans, 2013. 7 : Site 10, Am7 2, B17/1-3 ; 8 : Site 11, Am9 10, N18/3 ; Site 10, Am7 2, S18/3.

Highlights

- Miospores, acritarchs and chitinozoans are relatively abundant and well preserved
- Palynomorphs provide a concordant age corresponding to the mid-Frasnian
- Miospores are correlated with the conodont biostratigraphic scale from the Boulonnais
- Palynomorphs are characteristic of the Gondwana and more precisely from Brazil
- Effective generation of hydrocarbon is dependent of the igneous effect

Figure 1

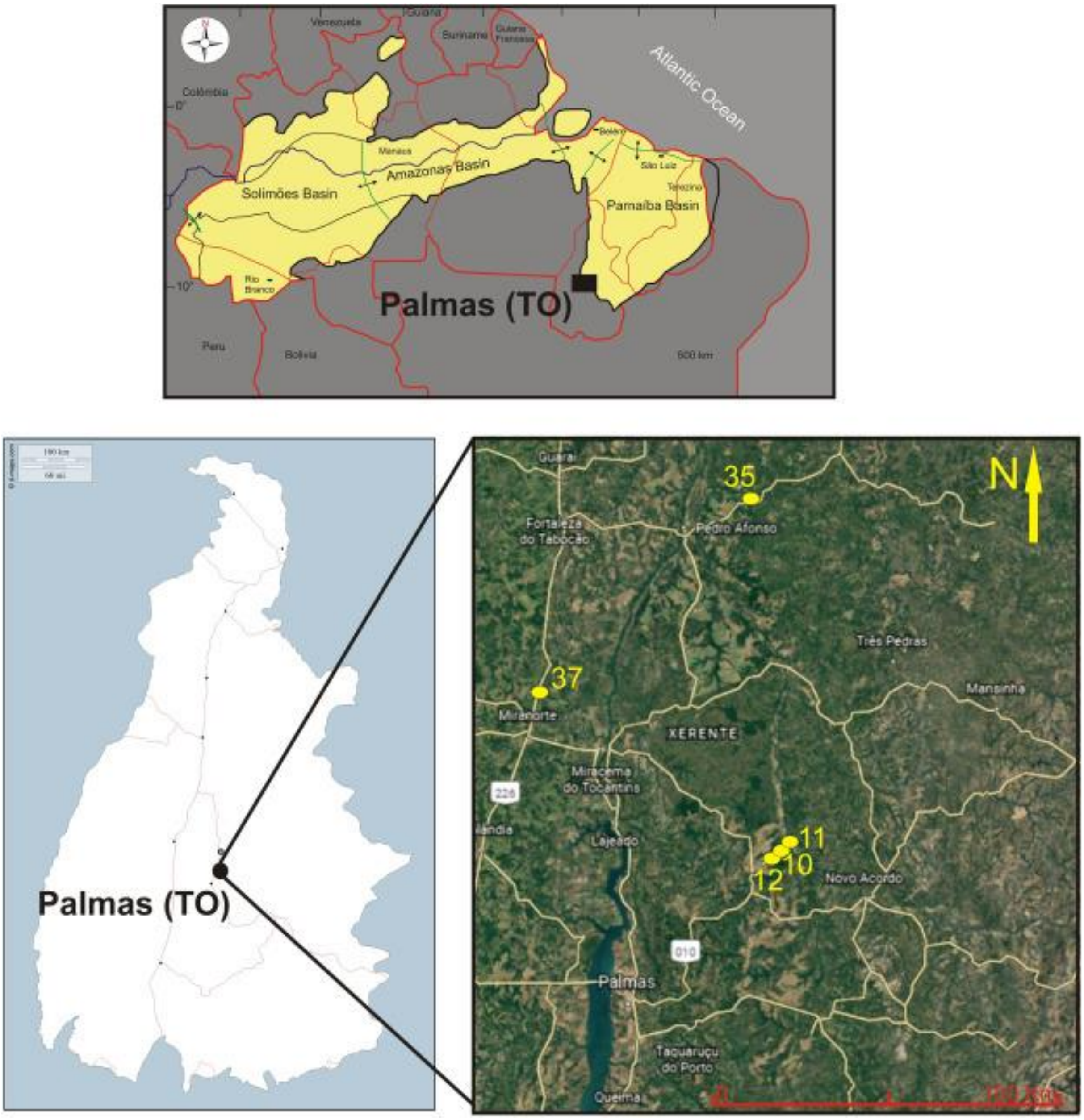
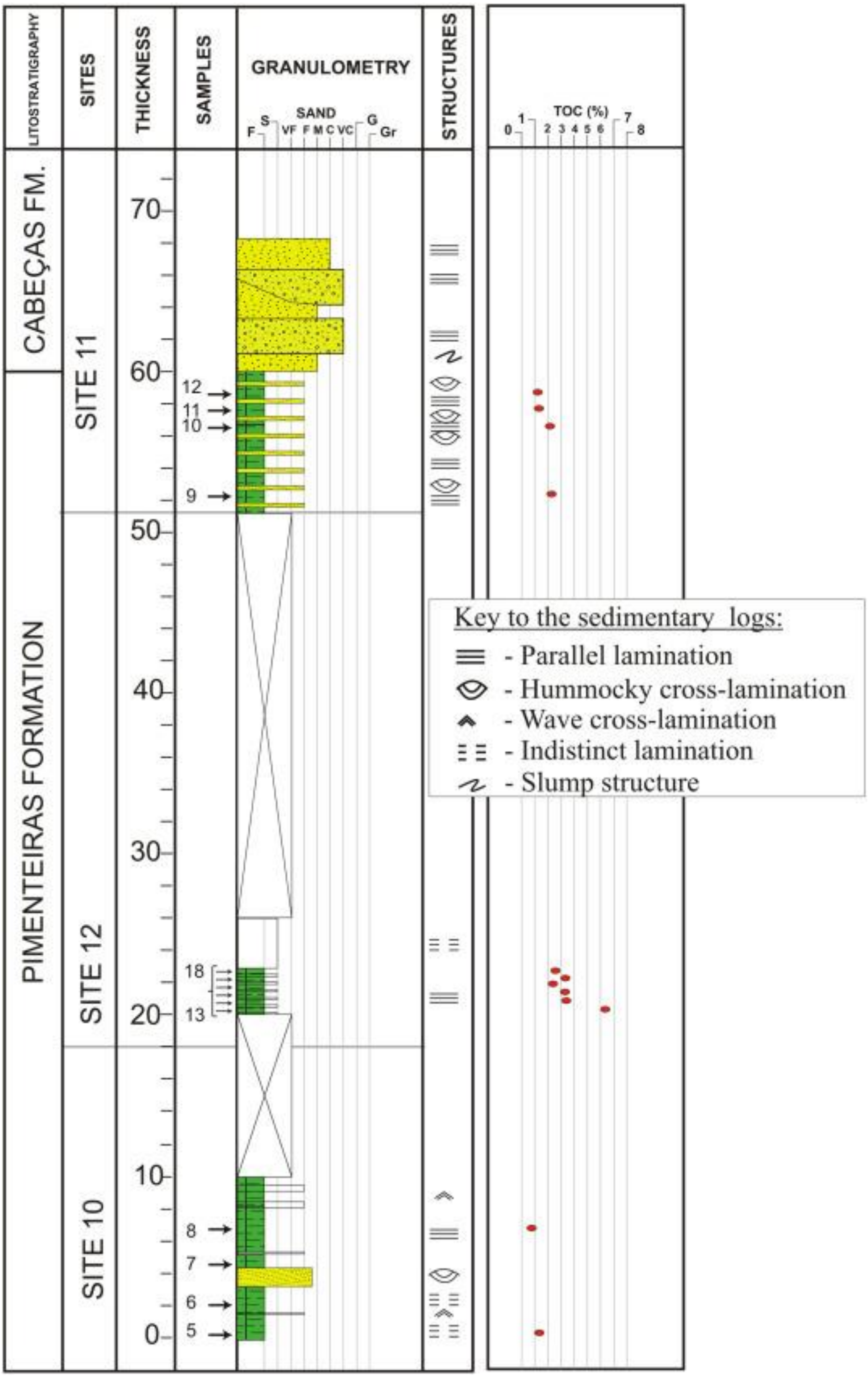


Figure 2


LITOSTRATIGRAPHY
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TOC (%)
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CABEÇAS FM.
PIMENTEIRAS FORMATION
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SITE 12
SITE 10
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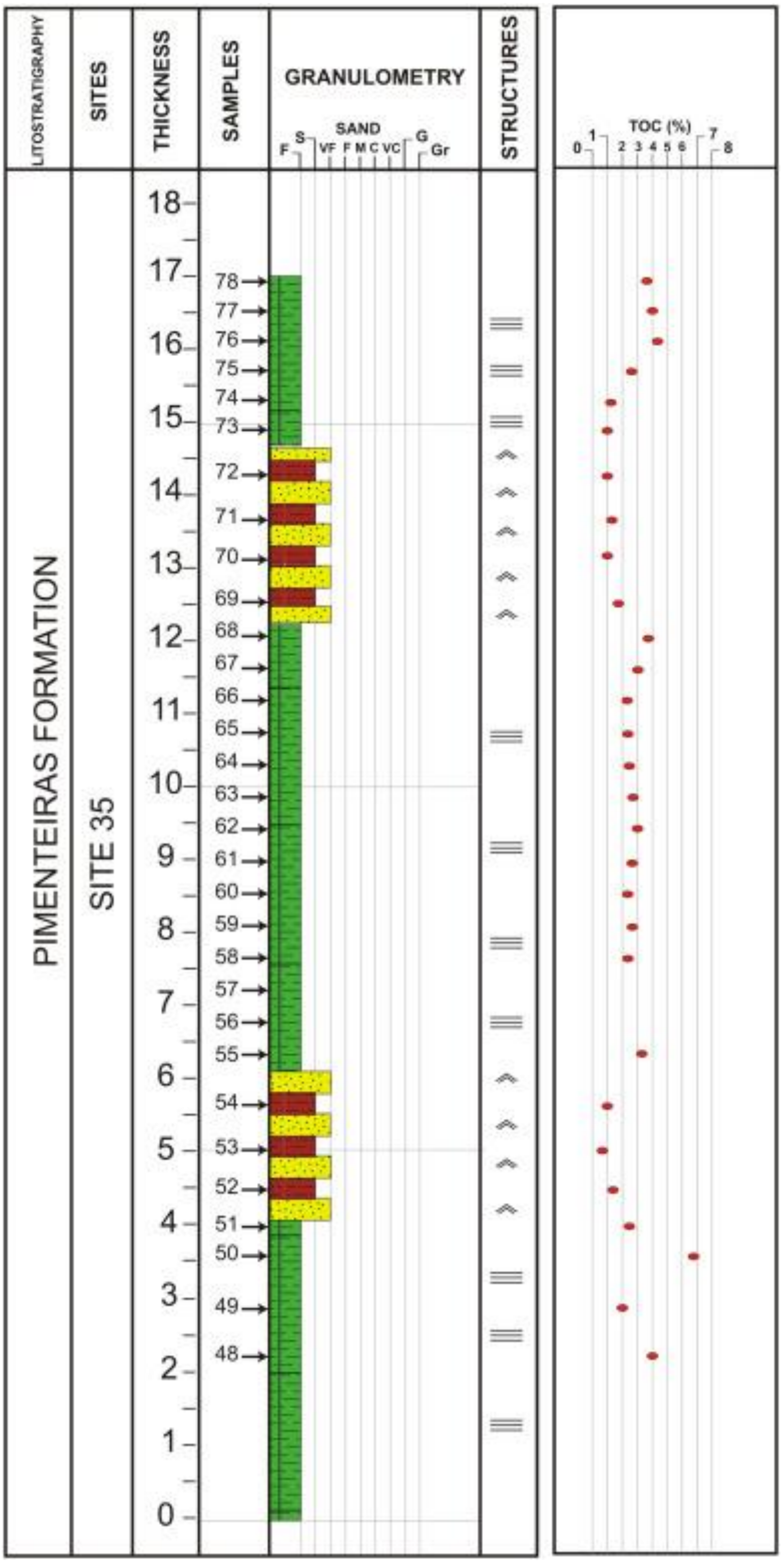
Key to the sedimentary logs:
- Parallel lamination
- Hummocky cross-lamination
- Wave cross-lamination
- Indistinct lamination
- Slump structure

Figure 3


LITOSTRATIGRAPHY
SITES
THICKNESS
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GRANULOMETRY
STRUCTURES
F
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TOC (%)
0 1 2 3 4 5 6 7 8
PIMENTEIRAS FORMATION
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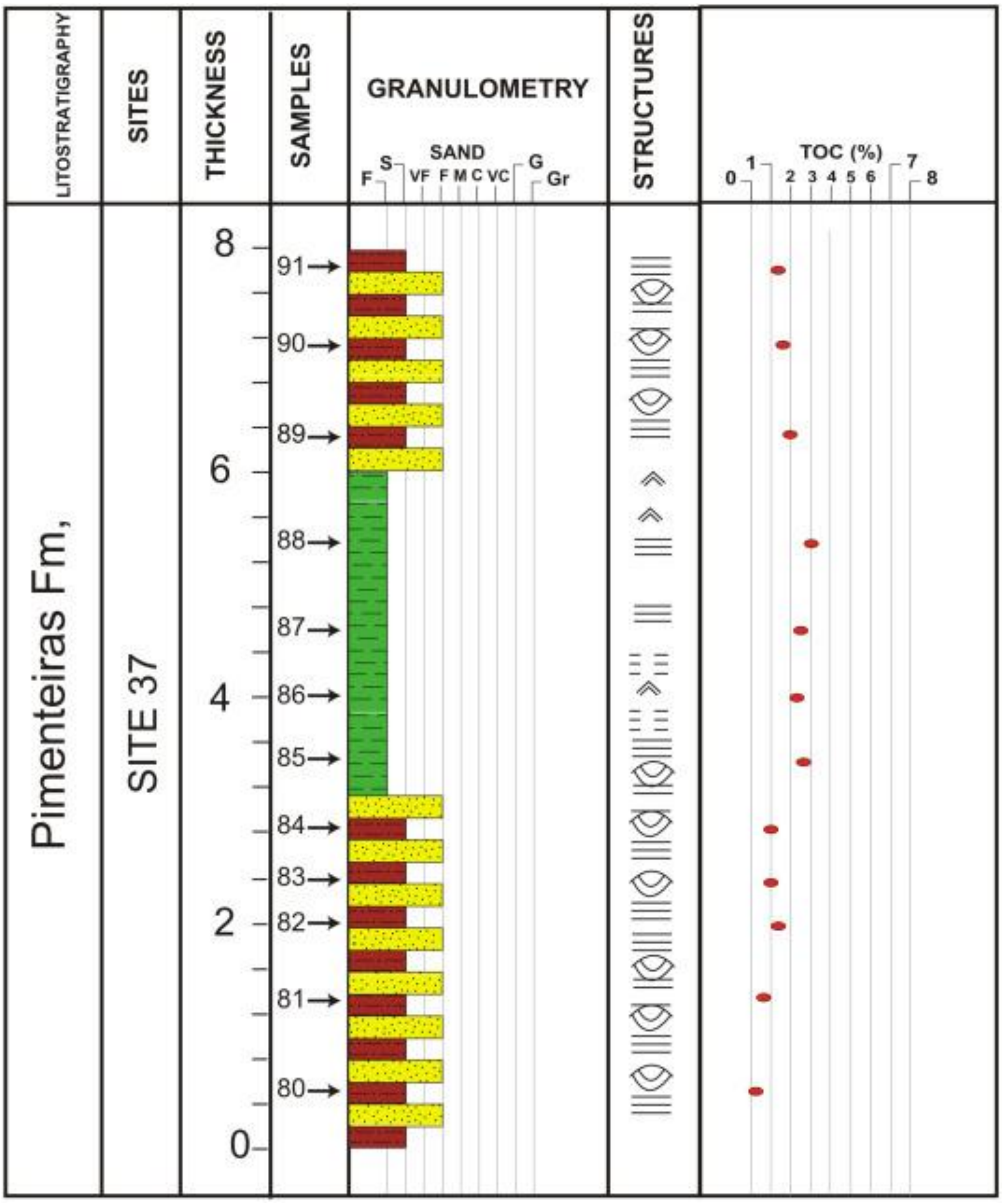
Figure 4


LITOSTRATIGRAPHY
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STRUCTURES
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Pimenteiras Fm,
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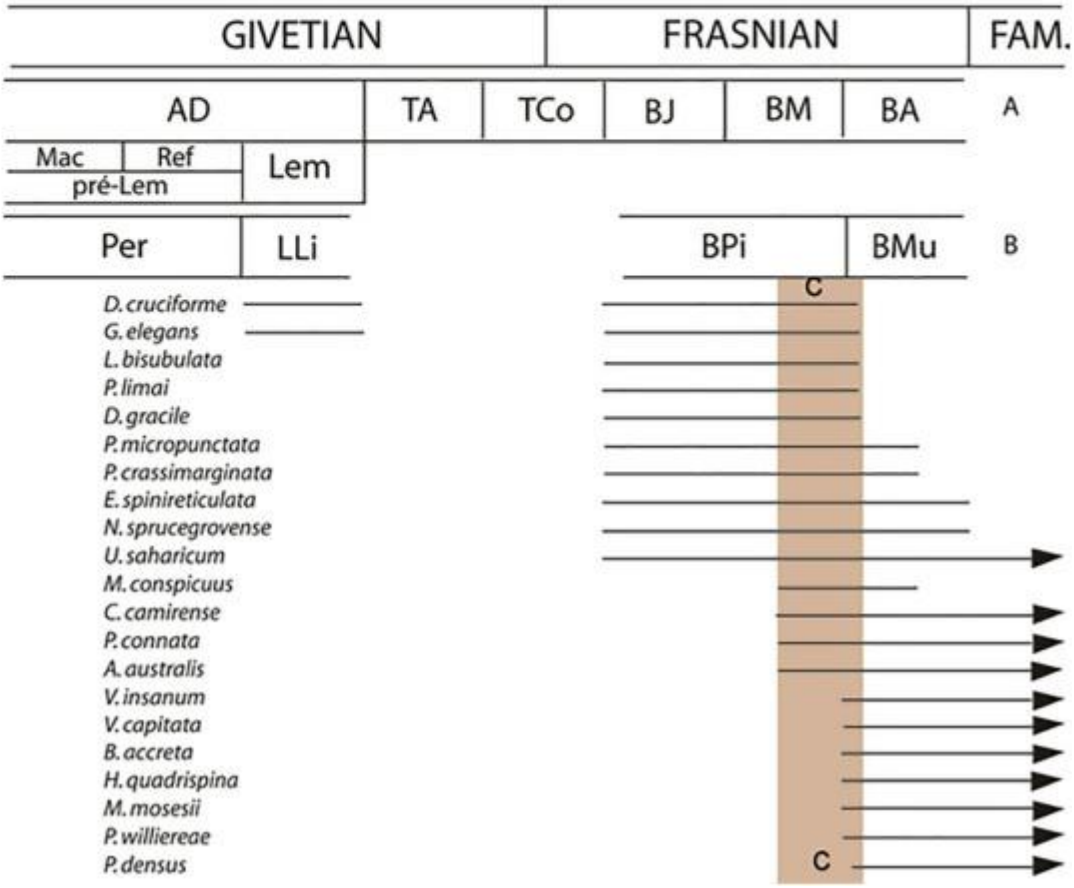
Figure 5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D.cruciforme
G.elegans
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P.limai
D.gracile
P.micropunctata
P.crassimarginata
E.spinireticulata
N.sprucegrovense
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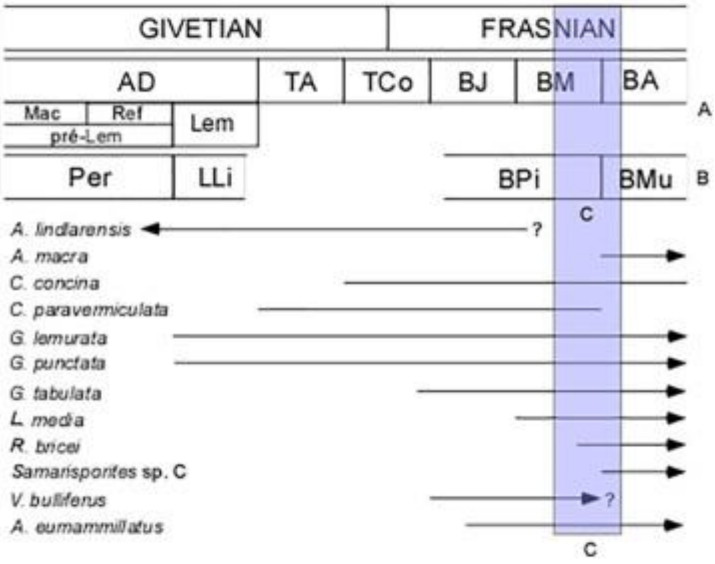
P.williereae
P.densus
C

Figure 6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BMu
B
C
A. lindlarensis
?
A. macra
C. concina
C. paravermiculata
G. lemurata
G. punctata
G. tabulata
L. media
R. bricei
Samarisporites sp. C
V. bulliferus
?
A. eumammillatus
C

Plate I.

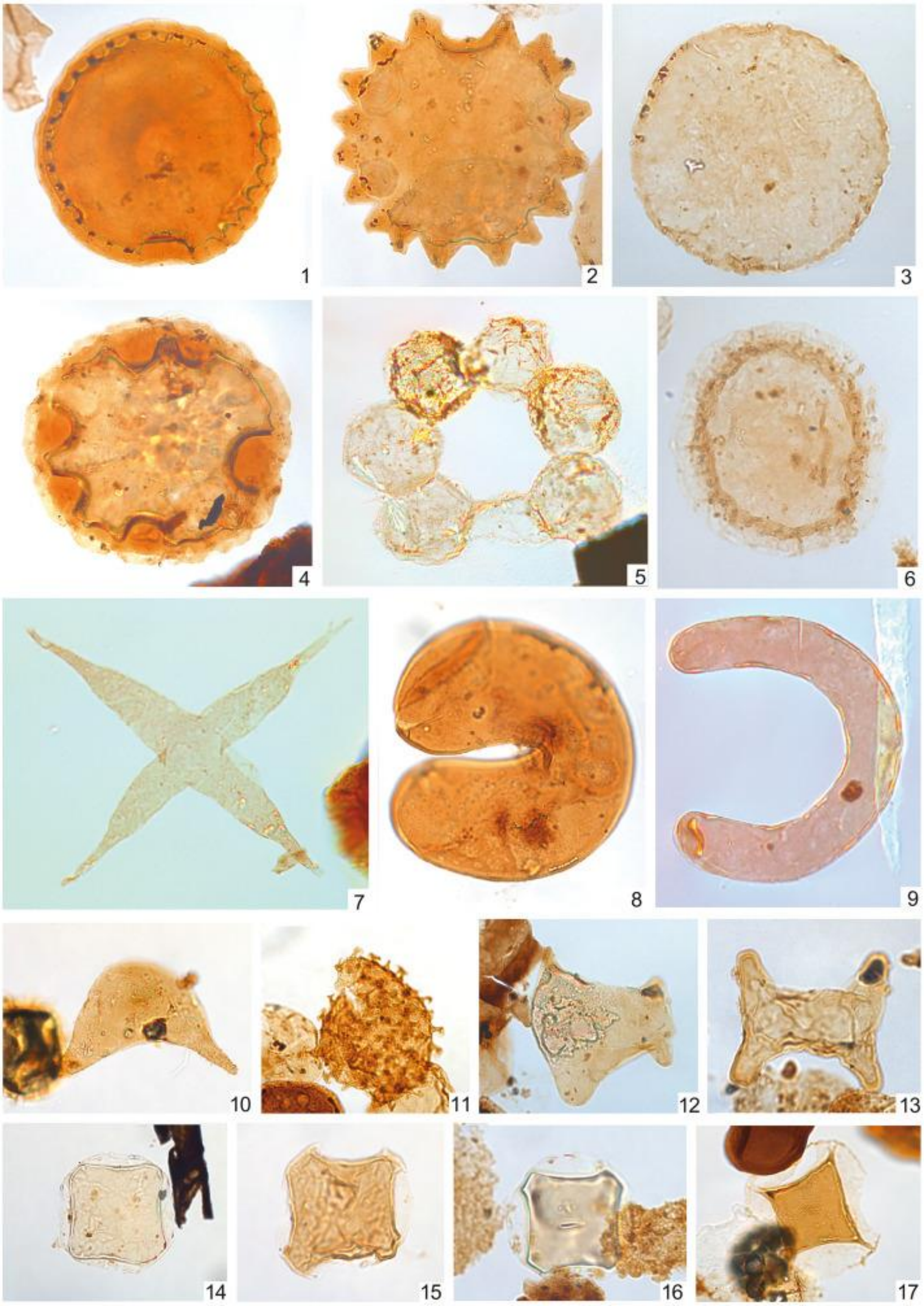

Plate II.


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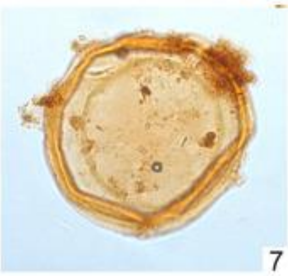
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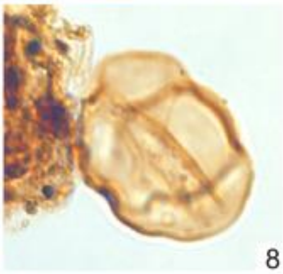
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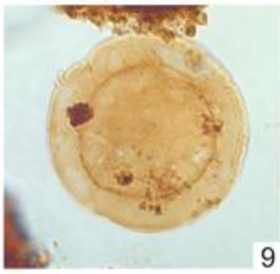
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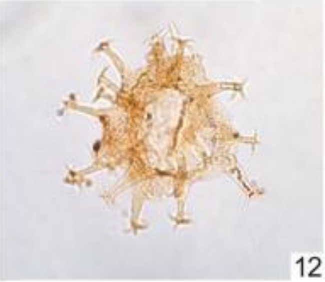
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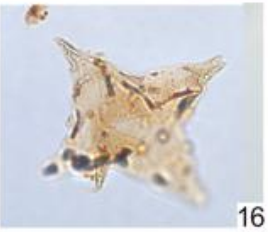
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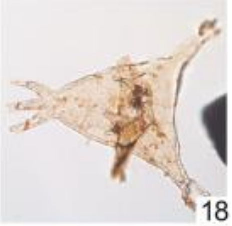
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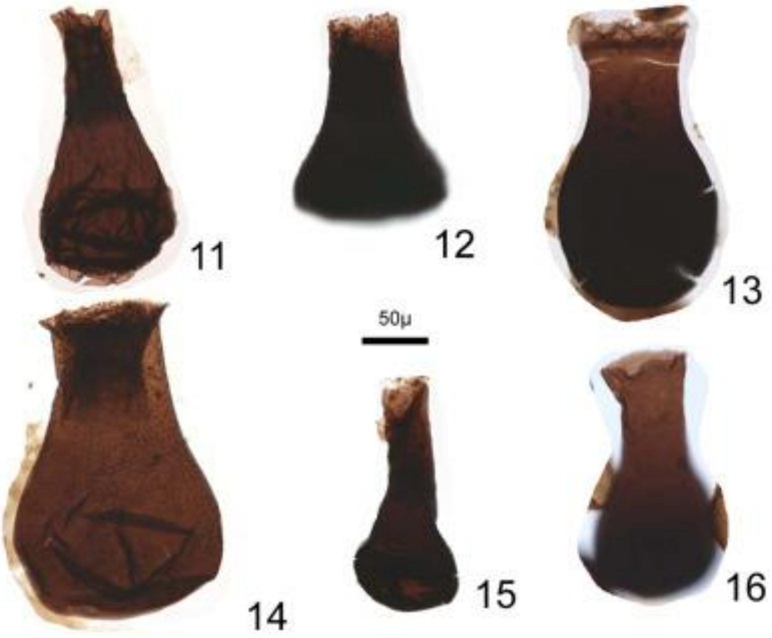
Plate III.

Plate IV.



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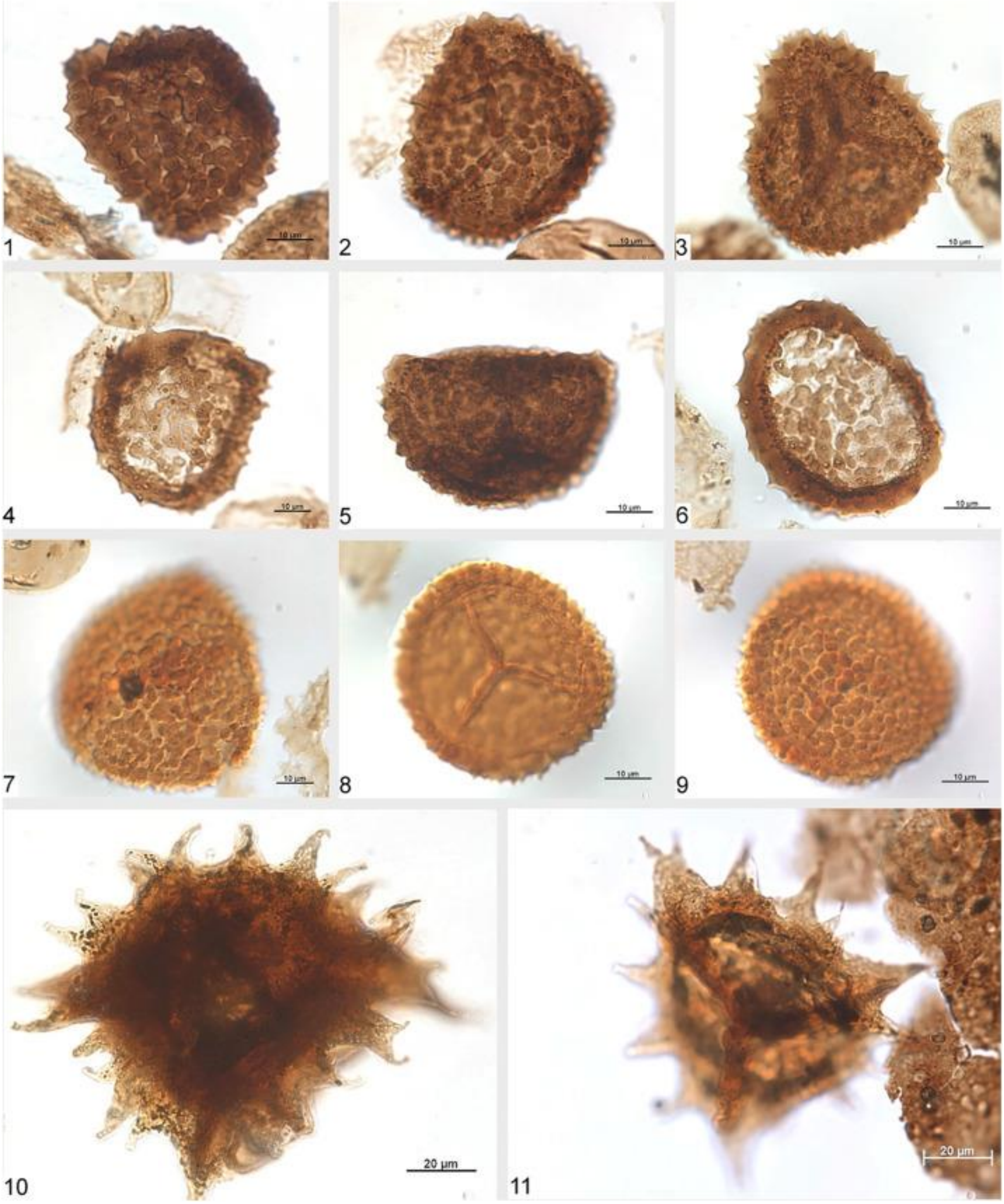
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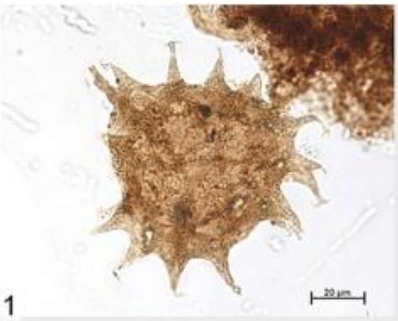
Plate VI.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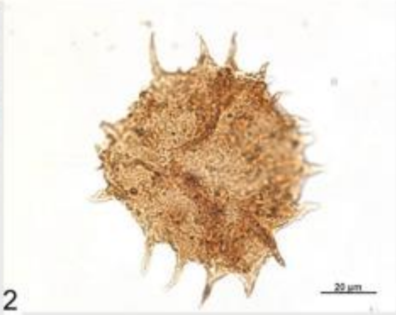
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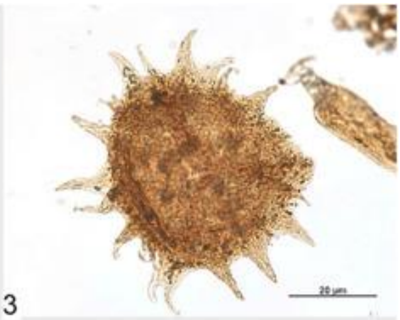
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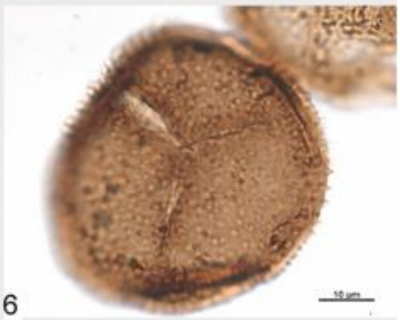
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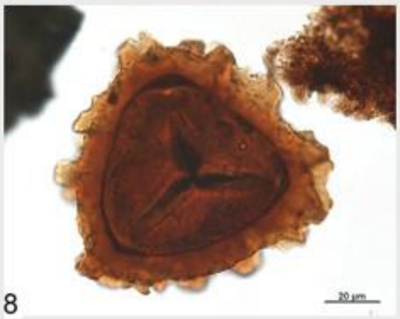
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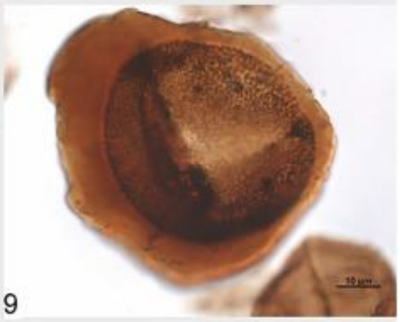
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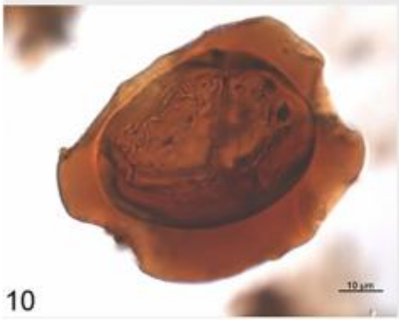
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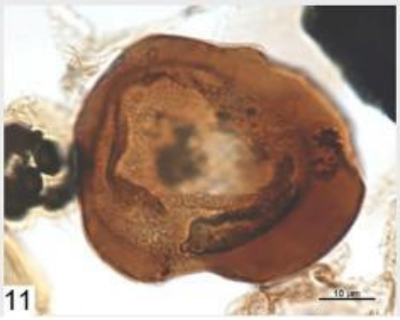
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Plate VII.


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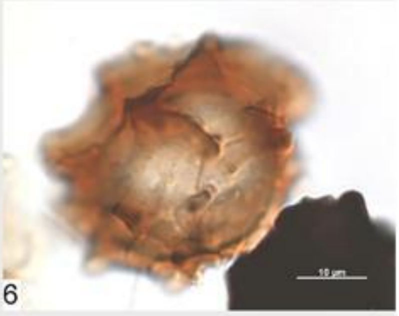
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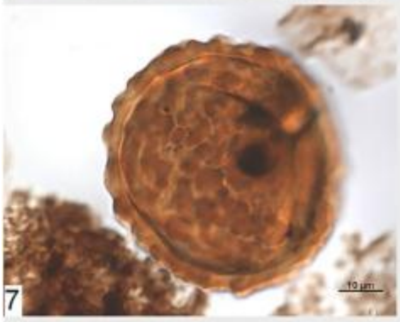
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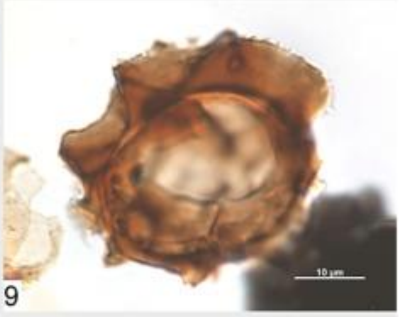
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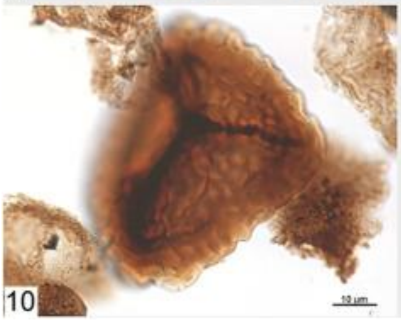
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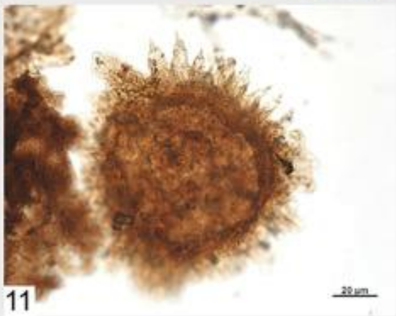
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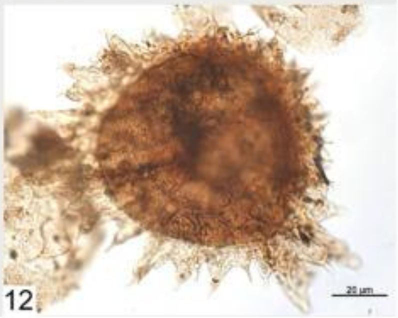
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Plate VIII.

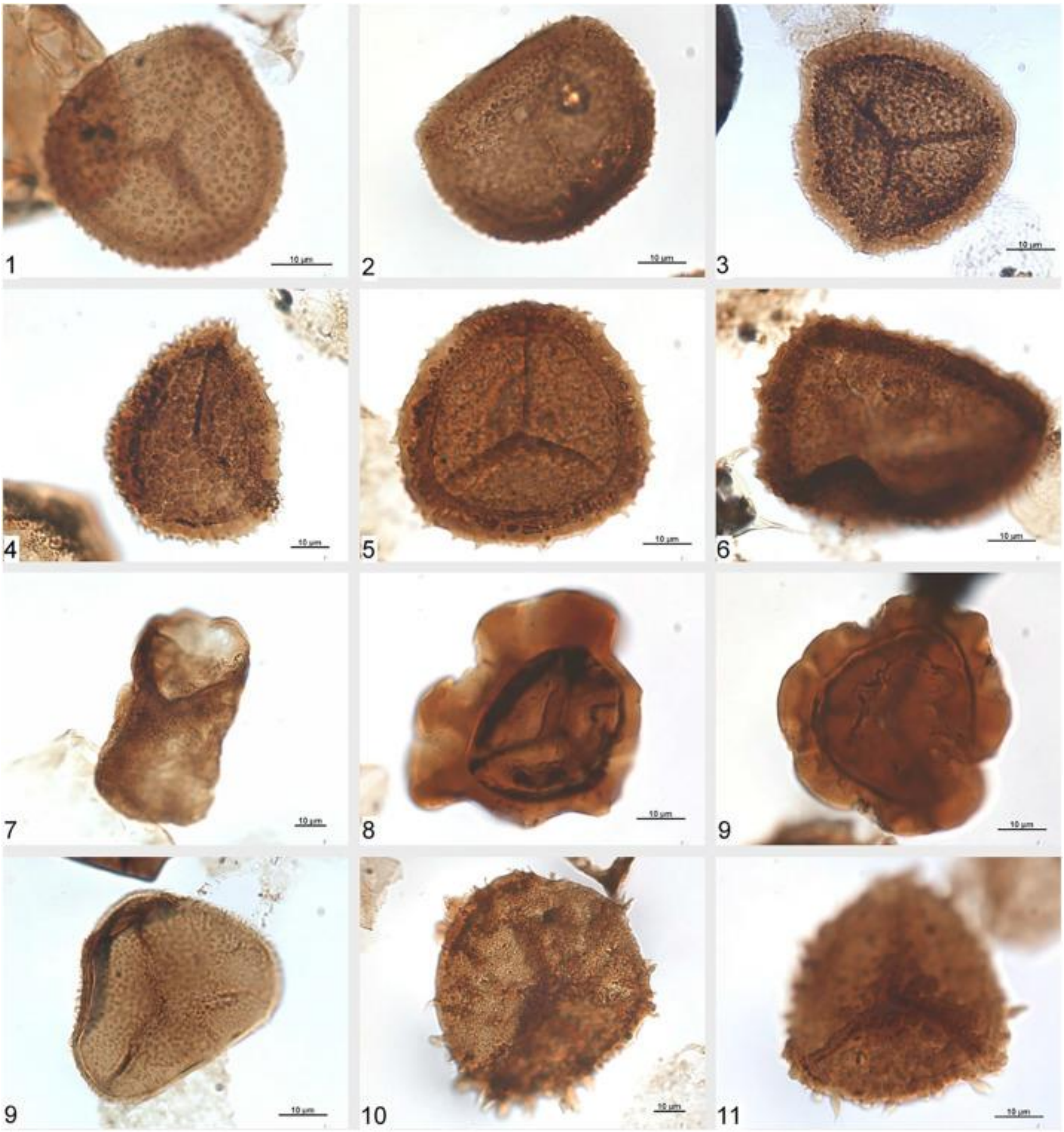
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Plate IX.

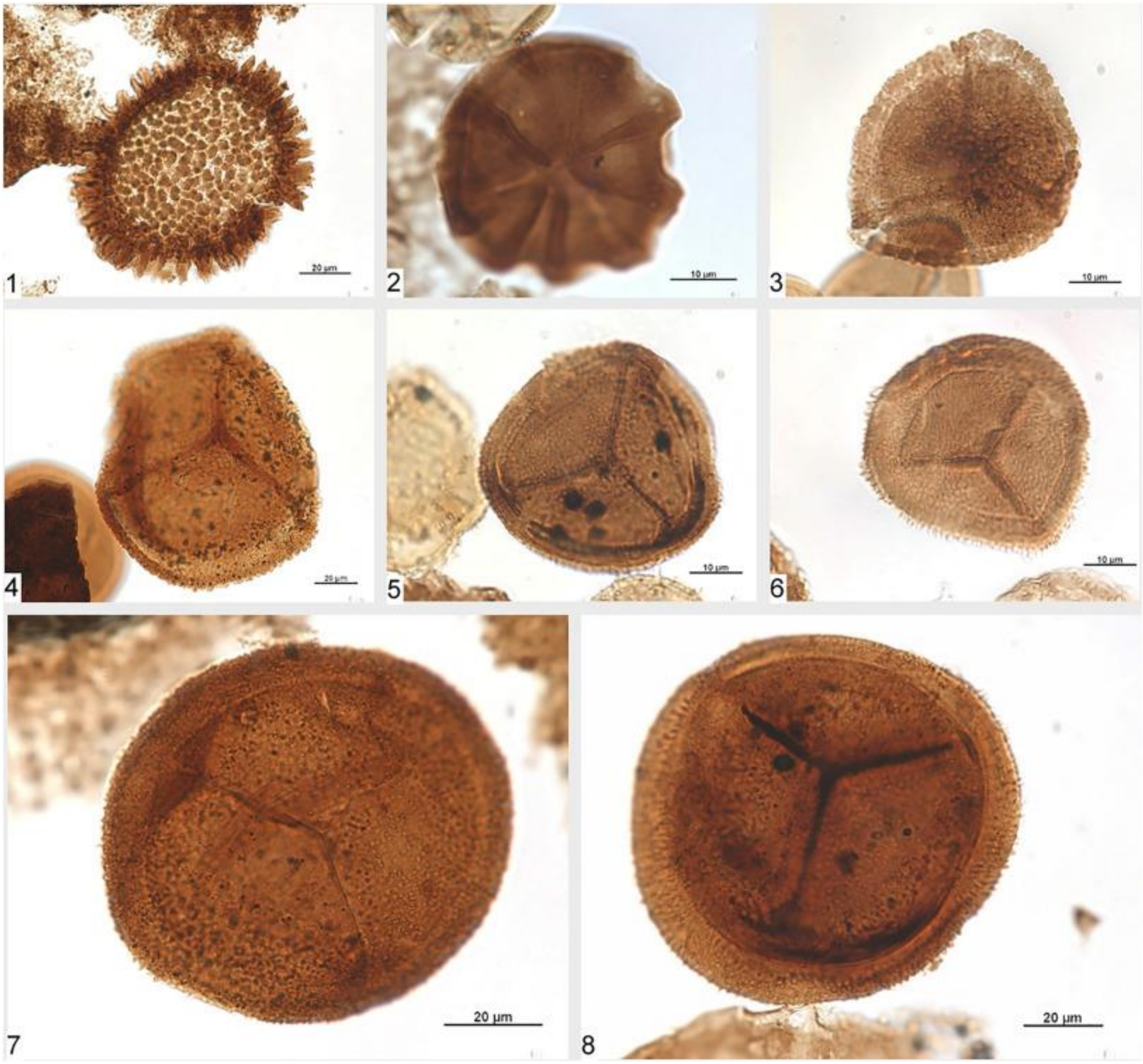

Plate X.


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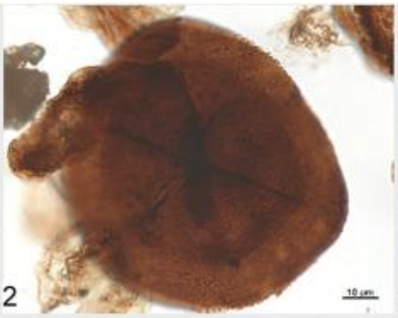
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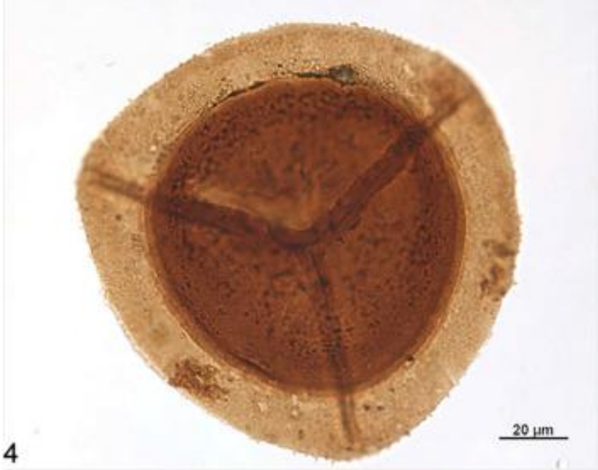
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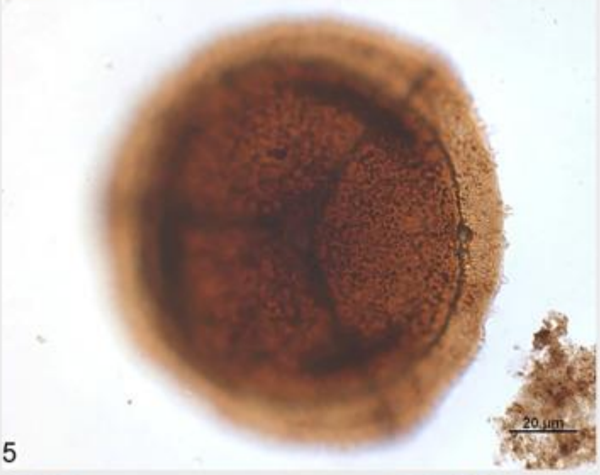
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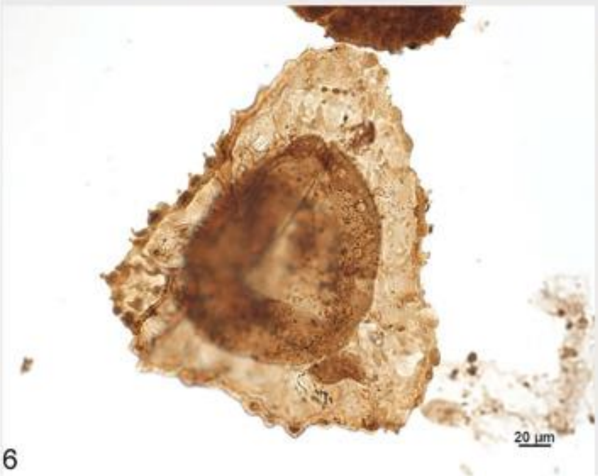
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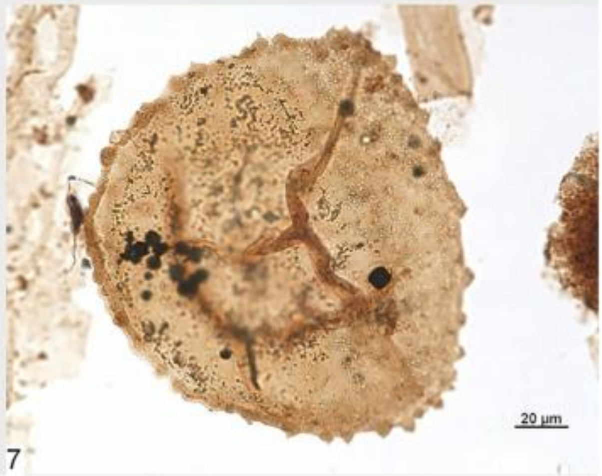
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Plate XI.

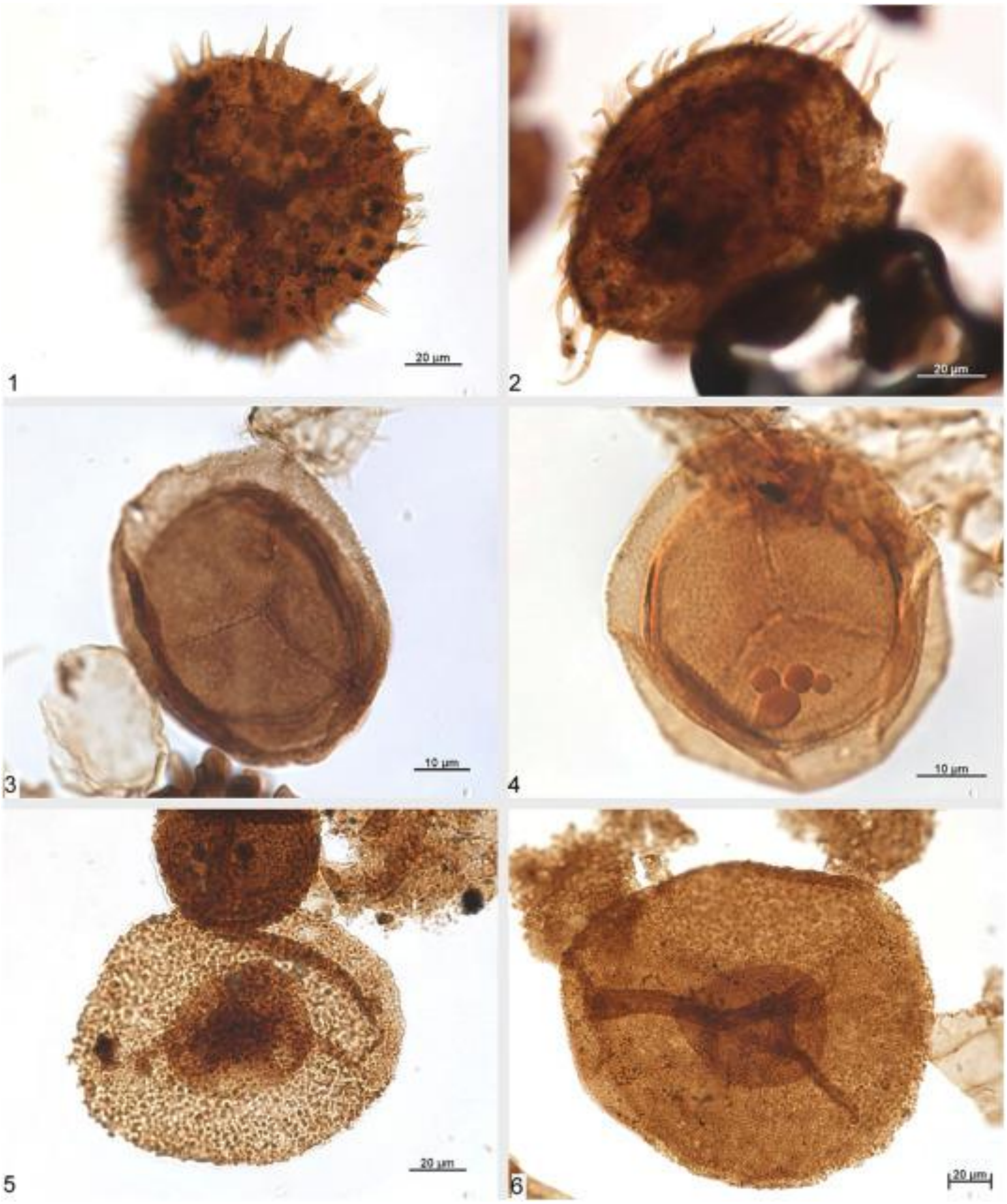


Plate XII.

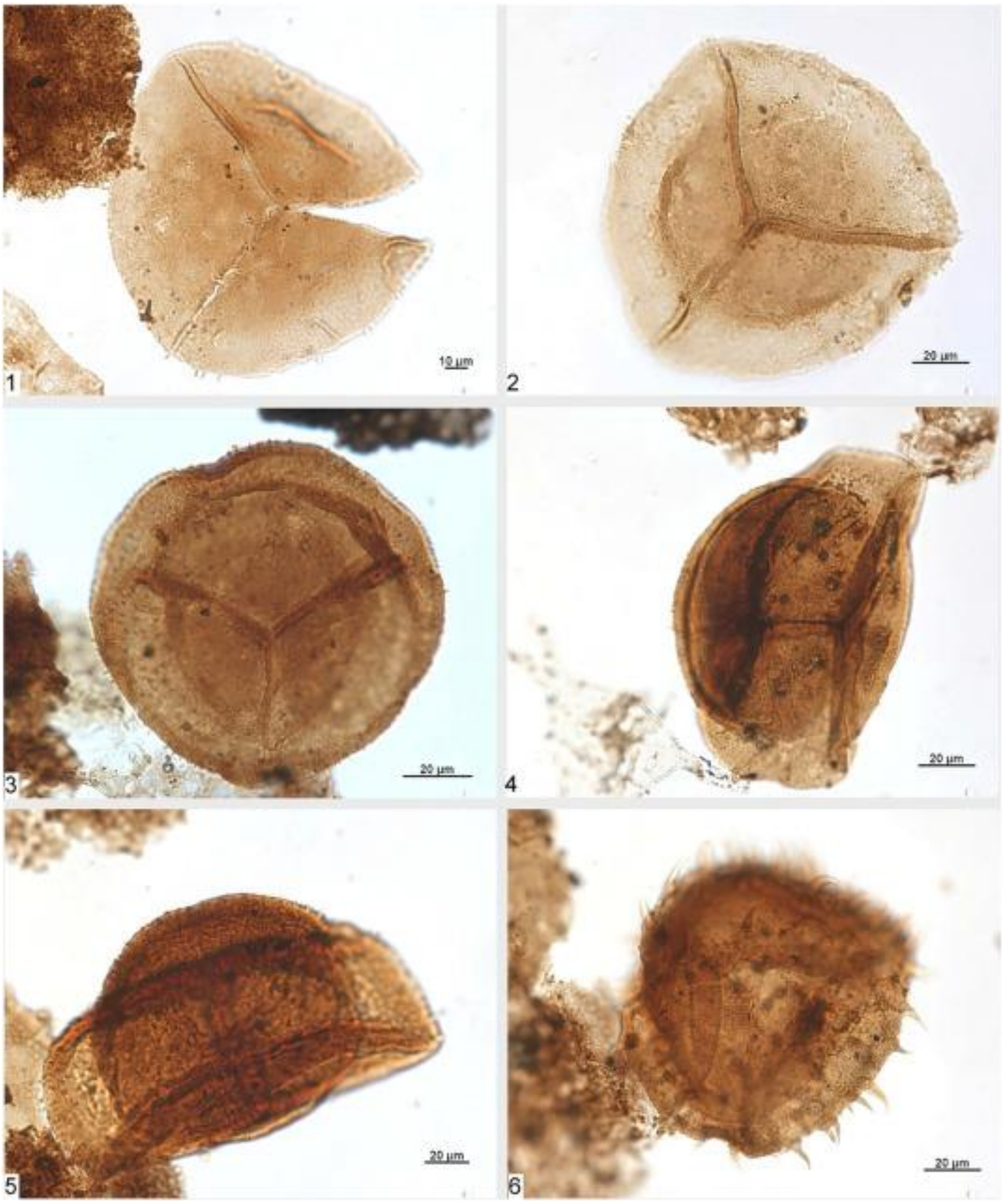
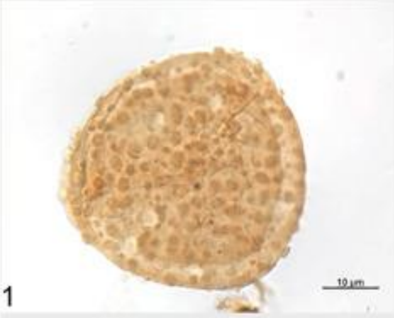


Plate XIII.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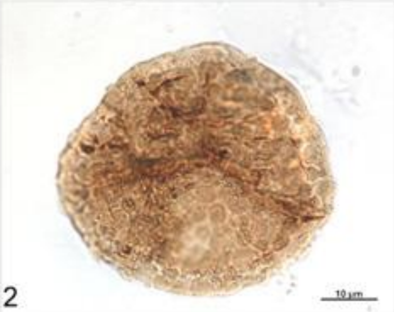
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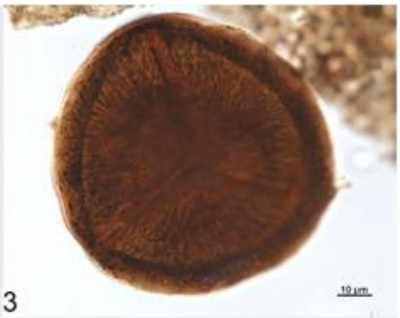
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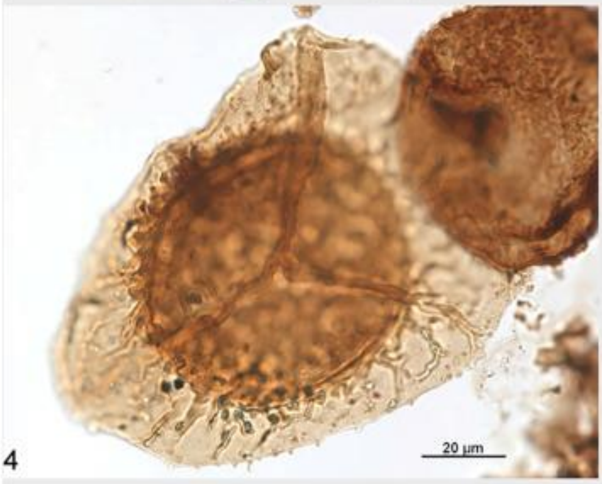
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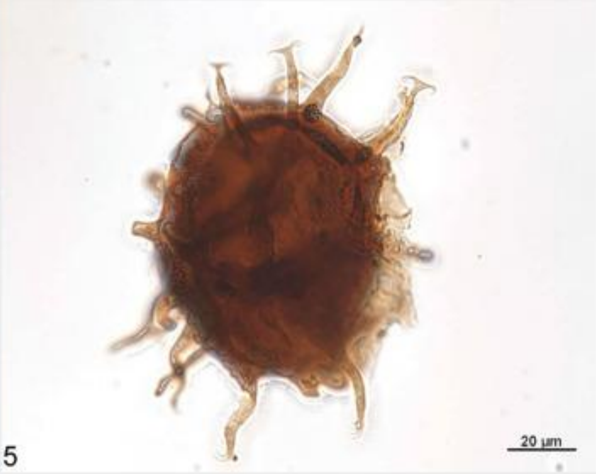
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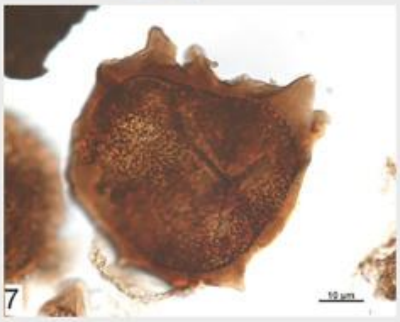
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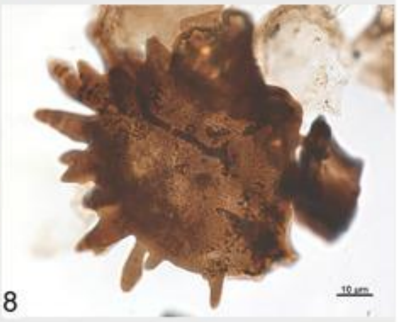
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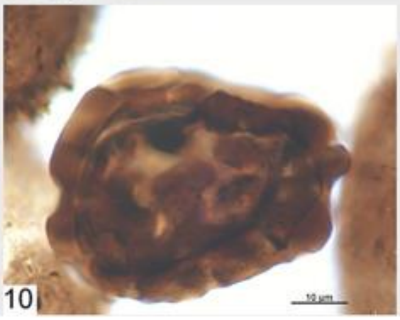
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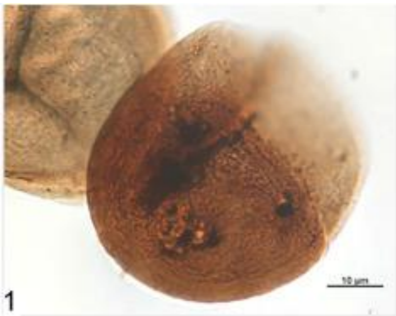
Plate XIV.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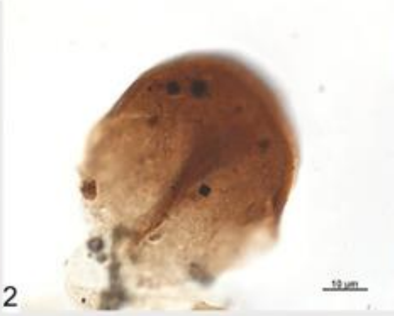
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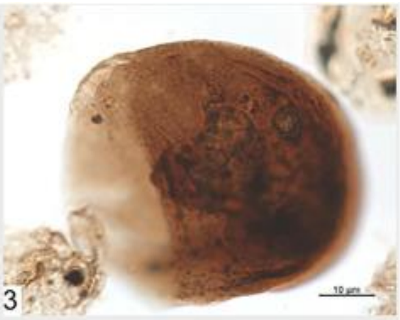
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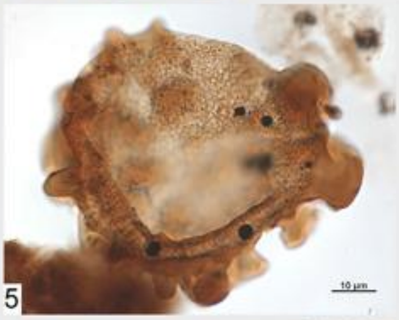
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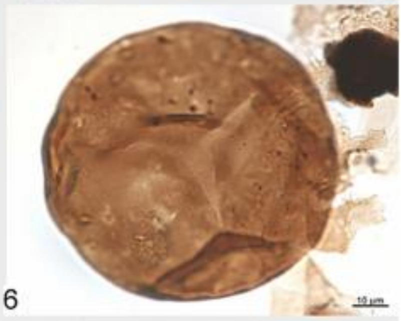
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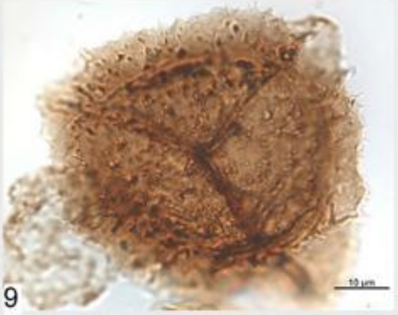
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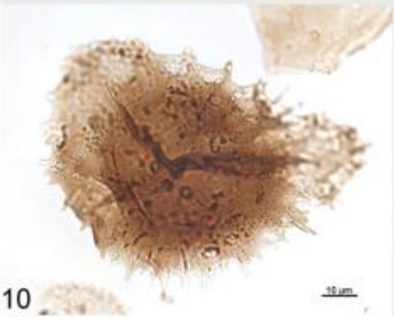
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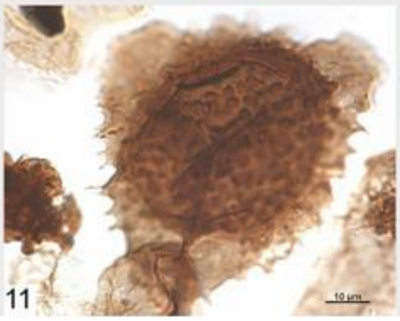
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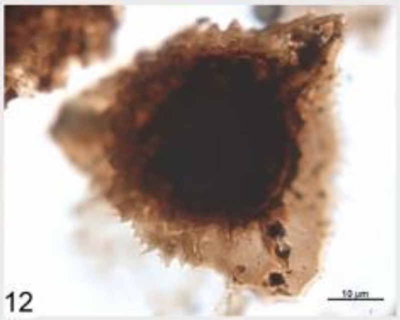
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Plate XV.