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**TAPHONOMY OF FOSSIL GROUPS FROM THE CRATO MEMBER  
(SANTANA FORMATION, ARARIPE BASIN, EARLY CRETACEOUS,  
NORTH-EAST BRAZIL): GEOBIOLOGICAL, PALAEOECOLOGICAL, AND  
PALAEOENVIRONMENTAL IMPLICATIONS**

***TAFONOMIA DE GRUPOS FÓSSEIS DO MEMBRO CRATO (FORMAÇÃO  
SANTANA, BACIA DO ARARIPE, EOCRETÁCEO, NE DO BRASIL):  
IMPLICAÇÕES GEOBIOLÓGICAS, PALEOECOLÓGICAS E  
PALEOAMBIENTAIS***

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## **DEDICATION**

I dedicate this dissertation to each person who helped me in my research, especially my family and advisors, who never spared efforts to give me freedom of thought, and my friends, who have always encouraged me to question myself, doubt my conclusions, and develop creativity.

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*Equipped with his five senses, man explores the universe  
around him and calls the adventure Science.*

Edwin Powell Hubble

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## Abstract

Over the past decades, the so-called Fossil *Lagerstätten* have been in the core of discussions concerning the palaeobiological and geological history of the Earth. In particular, the Crato Member from the Santana Formation (Araripe Basin, north-east Brazil) is one of the most significant Cretaceous *Lagerstätten* since it records exceptionally well-preserved fossil invertebrates, vertebrates, and plants, deposited in palaeolake carbonate beds. The main aim of this dissertation is to shed light on the preservational processes responsible for the fossilization of insects and fishes still retaining 3D soft tissues. Petrographic thin sections and several palaeometric techniques – micro-Raman Spectroscopy, conventional X-ray Fluorescence (XRF), Synchrotron micro-XRF (SR- $\mu$ XRF), Particle-Induced X-ray Emission (PIXE), Scanning Electron Microscopy (SEM), and Energy-Dispersive X-ray Spectroscopy (EDS) – have been employed to characterize the host rock, soft-tissue morphology and preservational fidelity, and fossil elemental and molecular compositions in centimetre to micrometre scale. The results reveal that while insects and fish soft-tissues found in the so called beige limestones (BL) are replaced by framboidal pyrite pseudomorphs (after pyrite oxidation) occurring together with putative bacterially-secreted extracellular polymeric substances (EPS), labile tissues of fishes from the grey limestones (GL) are kerogenized. In insects, there is a variation of framboid size inward the fossils, which is here interpreted as a product of the balance between diffusion and mineral nucleation rates. Moreover, the preferential distribution of Zn and Cu in pyritized insect/fish labile structures in comparison to their rock matrix is here considered as being the result of element fixation in bacterial biofilms. Zn concentrated in bones of kerogenized fishes and Fe/Cu occurring in their soft tissues are considered to have been incorporated during fish life. In the particular case of fishes, a model originally proposed for metazoan preservation in the Precambrian is here applied to explain the variation of fossilization between the BL and GL facies. Petrographic thin sections reveal that GL have generally higher clay/organic matter contents than BL, thus implying that burial rates might have been more intense in the former. This could have placed decaying carcasses more quickly in the methanogenesis sedimentary zone, in that way being kerogenized. On the other hand, carcasses deposited in the BL facies could have spent a longer period in the sulphate-reduction zone, which would have accounted for pervasive



pyritization. Additionally, microspar low porosity, cement and clay could have diminished both downward migrations of electron acceptors for anaerobic bacterial respiration processes – particularly sulphate-reduction (SR) and methanogenesis – and of their biogeochemical products, narrowing the SR zone, which would have lowered the impact of pyritization in some levels. While pyritization has recorded 3D muscle fibres, sarcolemma, putative cell nuclei, tendons and eyes, kerogenization has yielded connective tissues, integument and compressed/distorted muscle fibres. In conclusion, it is here proposed that palaeoenvironmental/geobiological-influenced facies have yielded fossils with a variable preservational-fidelity gradient, accordingly to each taphonomic pathway followed.

**Key words:** taphonomy, palaeometry, Early Cretaceous, Araripe Basin, Crato Member, exceptional preservation, taphonomic window, taphonomic model, preservational fidelity, fossil insects, fossil fishes, pyrite, kerogen.

## Resumo

Nas últimas décadas, os *Lagerstätten* estiveram no centro das discussões relativas à história paleobiológica e geológica da Terra. Em particular, o Membro Crato da Formação Santana (Bacia do Araripe, Nordeste do Brasil) é um dos mais significantes *Lagerstätten* do Cretáceo já que registra invertebrados, vertebrados e plantas excepcionalmente preservados em sedimentos carbonáticos de um palaeolago. O principal objetivo desta dissertação é lançar luz sobre os processos de preservação responsáveis pela fossilização de insetos e peixes com tecidos moles em 3D. Lâminas petrográficas e diversas técnicas paleométricas – micro-Espectroscopia Raman, Fluorescência de raios-X (FRX) convencional, micro-FRX com fonte de luz síncrotron (RS- $\mu$ FRX), emissão de Raios-X induzida por partículas (PIXE), microscopia eletrônica de varredura (MEV) e espectroscopia de energia dispersiva de Raios-X (EDS) – foram empregadas para caracterizar a rocha matriz, a morfologia e fidelidade de preservação dos tecidos moles e as composições elementares e moleculares dos fósseis em escala de centímetros e micron. Os resultados revelam que, enquanto insetos e tecidos moles de peixes encontrados nos denominados calcários beges (BL) são substituídos por

pseudomorfos de pirita framboidal (após oxidação da pirita), os quais ocorrem juntamente com possíveis substâncias poliméricas extracelulares secretadas por bactérias (EPS), tecidos de peixes dos calcários cinza (GL) são querogenizados. Em insetos, existe variação de tamanho dos framboides para dentro dos fósseis, que é aqui interpretada como produto do equilíbrio entre as taxas de difusão e de nucleação dos minerais. Além disso, a distribuição preferencial de Zn e Cu em estruturas piritizadas de insetos e peixes em comparação com a sua matriz é aqui considerada como sendo o resultado da fixação de elementos químicos em biofilmes bacterianos. Zn concentrado nos ossos de peixes com querogenizados e Fe/Cu observados em seus tecidos moles são considerados como tendo sido incorporados durante a vida dos peixes. No caso particular de peixes, modelo originalmente proposto para a preservação de metazoários do Pré-cambriano é aqui aplicado para explicar a variação de fossilização entre as fácies BL e GL. Lâminas petrográficas revelam que os GL têm geralmente teor de argila/matéria orgânica maior do que os BL, implicando que as taxas de soterramento poderiam ter sido mais intensas nos GL. Isto teria colocado carcaças em decomposição mais rapidamente na zona sedimentar de metanogênese, sendo formado o querogênio. Por outro lado, carcaças depositadas na fácies BL poderiam ter passado período mais longo na zona de redução de sulfato, o que teria levado à piritização generalizada. Além disso, a baixa porosidade do microespaço, o cimento e a argila poderiam ter diminuído a migração de aceptores de elétrons dos processos de respiração bacteriana anaeróbia – particularmente redução de sulfato (RS) e metanogênese – e de seus produtos biogeoquímicos, estreitando a zona de RS, o que teria reduzido a influência da piritização em alguns níveis. Enquanto que a piritização resultou na preservação de fibras musculares em 3D, sarcolema, possíveis núcleos celulares, tendões e olhos, a querogenização preservou tecidos conjuntivos, tegumento e fibras musculares distorcidas e compactadas. Em conclusão, é aqui proposto que fácies influenciadas por processos paleoambientais e geobiológicos produziram fósseis com gradiente diferencial de fidelidade de preservação de acordo com cada via tafonômica seguida.

**Palavras-chave:** tafonomia, paleometria, Eocretáceo, Bacia do Araripe, Membro Crato, preservação excepcional, janela tafonômica, modelo tafonômico, fidelidade de preservação, insetos fósseis, peixes fósseis, pirita, querogênio.

## 1. Introduction

The term Fossil *Lagerstätten* (or taphonomic windows) was coined by the German palaeontologist Adolf Seilacher 30 years ago and embraces rock deposits which are particularly rich in qualitative and quantitative fossil content (Seilacher et al., 1985). Therefore, according to these authors, Fossil *Lagerstätten* yield palaeobiological and taphonomic data which provide a broad and complete view of sedimentary facies genesis. Seilacher et al. (1985) also stressed that *Lagerstätten* rely on unique palaeoenvironmental, sedimentary, and diagenetic processes.

Several examples of fossil *Lagerstätten* record evidence of trends and patterns that shaped life on Earth: Archaean – Apex Chert (Schopf, 1993) and Strelley Pool Formation (Wacey et al., 2011), both from Australia, shed light on the very early steps of microbial life; Proterozoic – Mistaken Point, Canada (e.g. Narbonne, 2005), improved studies on the Ediacara biota; Cambrian – Burgess Shale, Canada (Briggs et al., 1994), whose fossils point out to biogeochemical marine transitions which took place in the Precambrian/Cambrian boundary (e.g. Callow and Brasier, 2009); Devonian – Rhynie Chert, Scotland, enabled palaeontologists to have a view of the very early stages of terrestrial plant evolution (e.g. Trewin, 2003); Jurassic – Solnhofen Limestone, Germany (e.g. Seilacher et al., 1985), on which the earliest bird *Archaeopteryx* was found; Eocene – Messel Shale, Germany (e.g. Schaal and Ziegler, 1992), whose high abundance of extraordinarily preserved fossil mammals, birds, insects, among others, enabled scientists to understand an early Cenozoic ecosystem.

Besides evolutionary and palaeoecological information, exceptionally well-preserved fossils also provide data for palaeobiogeographical inferences since they enable scientists to assess the temporal range of events of origin and extinction in different regions, contributing to the identification of radiations and extinctions (e.g. Martínez-Delclòs et al., 2004). Remarkable fossil preservation also yields evidence of soft tissues, enabling detailed morphologic research, and also evidence of the colour of extinct life forms, which raises palaeoecological research to a new stage of sophistication (McNamara, 2013). Furthermore, the study of fossil preservation modes is of paramount relevance since it provides information on the fidelity of preservation, and chemical and mineralogical compositions of fossil samples, leading to innovative

approaches to palaeoenvironmental, sedimentary, and geobiological research (e.g. Laflamme et al., 2011).

The fossil record is biased by palaeoenvironmental, biological, physical, geochemical, geobiological, and diagenetic factors, which affect palaeontological data interpretation (Allison and Bottjer, 2011). The area of Earth Sciences which deals with fossilization processes and fossil alteration is Taphonomy. Its importance relies on the capacity of yielding palaeoenvironmental, palaeoclimatic, palaeoecological, and sedimentary information, as well as of identifying modifications of original morphological features of organisms, improving taxonomic research, among other aspects (Allison and Bottjer, 2011).

Over the last few years, scientists have applied a broad range of cutting-edge non-destructive high-resolution imaging and geochemical techniques to unlock data kept in rare and/or exceptionally well-preserved fossils therefore raising novel questions to be answered. This branch of palaeontology is currently named Palaeometry (Riquelme et al., 2009; Delgado et al., 2014 – Appendix 1). For instance, Raman spectroscopy, Confocal Laser Scanning Microscopy (CSLM), Focused Ion Beam (FIB), Transmission Electron Microscopy (TEM), Nanoscale Secondary Ion Mass Spectrometry (nano-SIMS), among other approaches have been employed to assess the biogenicity, phylogeny, palaeoecology, and microtaphonomy of Precambrian microfossils (Schopf & Kudryavtsev 2009; Brasier et al., 2015); Time-of-Flight Secondary Ion Mass Spectrometry (ToF-SIMS) has been used to examine the presence of protein and blood traces (among other biomolecules, defining the field of molecular palaeontology; Briggs and Summons, 2014) on fossil vertebrates and invertebrates (Greenwalt et al., 2013; Bertazzo et al., 2015); X-ray Fluorescence (XRF) and Particle-Induced X-ray Emission (PIXE) have been extensively applied to taphonomic research (Bergmann et al., 2010; Riquelme et al., 2013; Gueriau et al., 2014); Synchrotron X-ray Microtomography (SR- $\mu$ CT) has contributed to solving longstanding concerns on the taxonomic placement and palaeoecology of the Ediacaran fossil *Corumbella werneri* (Pacheco et al., 2015). Brazilian pioneers have been also conducting palaeometric work particularly regarding the taphonomy of the Santana Formation *Lagerstätte* (Filho et al., 2011; Buck, 2013; Osés, 2013; Delgado et al., 2014; Oliveira et al., 2015). In this taphonomic window, the Crato Member (Early Cretaceous) records exceptionally well-preserved fossil invertebrates, vertebrates, and plants. Some issues regarding

preservational processes of 3D insects and fish soft-tissues still remain open and circumscribe the aims of this dissertation.

## **2. The goals of the present research**

The laminated limestones from the Crato Member, Santana Formation, Araripe Basin, north-eastern Brazil (Assine, 2007), is a worldwide famous *Lagerstätte*. It is known for its remarkably well-preserved fossil invertebrates (particularly insects), vertebrates (e.g. fishes, turtles, dinosaurs, and pterosaurs), and flora (spores, pollen, gymnosperms, basal flowering plants) (Martill et al., 2007a). Several cases of soft-tissue preservation have been hitherto reported on vertebrates (Davis and Martill, 1999; Fielding et al., 2005; Pinheiro et al., 2012) and insects (Delgado et al., 2014; Barling et al., 2015) recorded in the Crato Member limestones. This biota was preserved within an Aptian (Early Cretaceous) palaeolake during the last stages of the South America-Africa breakup and consists of the most complete window into the palaeobiology of a continental depositional palaeoenvironment from the Cretaceous (Martill et al., 2007a). However, the fossil record of this outstanding *Lagerstätte* still keeps unsolved geological and palaeobiological enigmas, such as the diversification of social insects, the early evolution of flowering plant, and the coevolution of insects and angiosperms (Labandeira and Eble, 2000; Soares et al., 2013).

Regarding the global relevance of the Crato Member, taphonomic research encompassing microscale morphological characterization and geochemical mapping is needed to resolve the very interesting issues concerning the processes and patterns of preservation which yielded fossils with such fine detailed morphological features. Following this rationale, the present masters dissertation has the following aims:

- 1- by means of palaeometric techniques on macro to microscale resolution, characterize and understand the fossildiagenetic mechanisms and processes, which led to the exceptional preservation of fossil insects and fishes from the Crato Member;
- 2- assess preservational patterns and propose taphonomic models;
- 3- discuss the possible role of microorganisms in the fossilization processes;

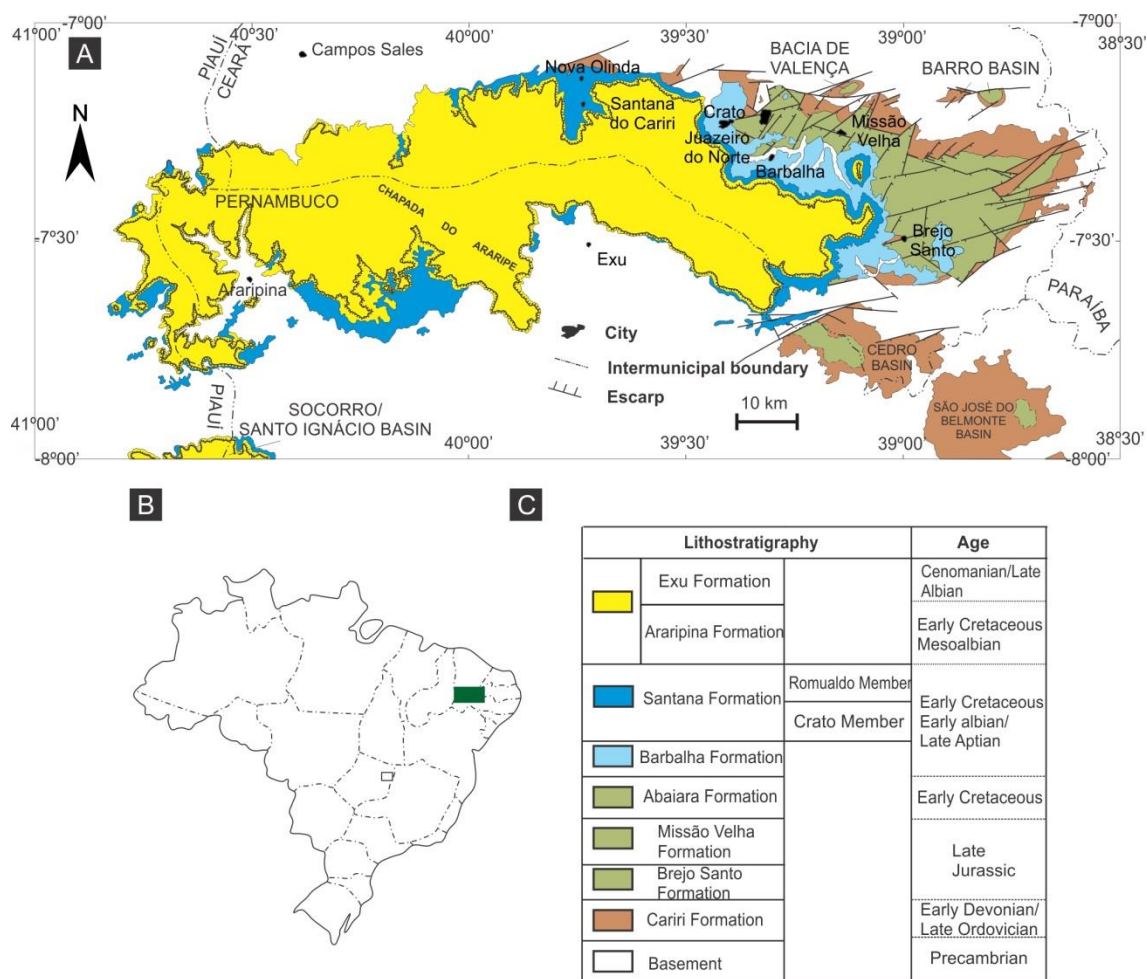
- 4- understand the palaeoenvironmental dynamics of the geological unit under study;
- 5- understand the role of palaeoenvironmental, palaeoecological, geobiological, and sedimentary interconnections in the Crato Member depositional environment, in the process of fossil preservation.

The results herein presented should contribute to improve the understanding of the fossilization processes that have operated in the Crato palaeolake. Beside this, they should help to interpret comparable preservational modes in other palaeoenvironments over the geologic time. This contribution sheds light on how the evolution of palaeoenvironmental conditions throughout time is related to exceptional fossil preservation, which records key changes in the history of life (e.g. Allison and Bottjer, 2011).

### **3. Geologic context**

#### **3.1. Basin evolution, stratigraphy and age**

The Araripe Basin is located in northeastern Brazil (Fig. 1), consisting of part of the states of Ceará, Pernambuco, and Piauí. The Araripe Basin is a continental rift basin, bounded by NE and WNW faults, and formed during the opening of the South Atlantic Ocean by the reactivation of Precambrian faults (Fig. 1; Ponte and Ponte Filho, 1996; Assine, 2007).



**Figure 1.** **A**, Geologic map of the Araripe Basin. **B**, Location of the Araripe Basin in the Brazilian territory. **C**, Simplified stratigraphic chart of the stratigraphy and age of the Araripe Basin. Modified after Assine (2007).

In the present contribution, the stratigraphic proposal for the Araripe Basin of Assine (2007) will be considered. Nevertheless, at some points of the text, other stratigraphic proposals may appear.

The base of the Araripe Basin is comprised by the Cariri Formation (Beurlen, 1962) (Late Ordovician/Early Devonian) (Assine, 2007). The tectonic-sedimentary evolution of the Araripe Basin is divided into four supersequences, from the base to the top (after Assine, 2007):

1- Pre-rift Supersequence: siliciclastic fluvial-lacustrine sediments from the Brejo Santo and the Missão Velha formations (Vale do Cariri Group), dated as Late Jurassic through ostracods and palynomorphs (Coimbra et al., 2002);

2- Rift Supersequence: deltaic, fluvial and lacustrine siliciclastic sediments from the Abaiara Formation (Vale do Cariri Group), which is attributed to the Early Cretaceous, based on ostracod biozonation (Coimbra et al., 2002);

3- Post-rift Supersequence: the Barbalha Formation, fluvial (siliciclasts)/lacustrine (pelites and carbonates) cycles and the Santana Formation, both units occurring within the Aptian-Albian Araripe Group (Coimbra et al., 2002). The lower succession of the Barbalha Formation comprises the “Batateira Beds”, which represent a succession of fluvial, deltaic, and lacustrine palaeoenvironments.

The Santana Formation, with the Crato Member at its base, outcrops above the Barbalha Formation. The former unit is attributed to the Late Aptian based mainly on palynomorph biostratigraphy (Coimbra et al., 2002). At the top of the Crato Member, supratidal gypsum layers and shales, known as “Ipubi Beds” occur (Assine, 2007). Transgressive events led to the deposition of siliciclastic marine sediments, in which there are occurrences of shales with carbonate fossiliferous nodules of the Romualdo Member (Kellner, 2002), the top unit of the Santana Formation (Assine, 2007). Both “Ipubi Beds” and Romualdo Member are related to the Late Aptian-Early Albian interval, according to palynozones (Coimbra et al., 2002). Above the Romualdo Member, a marine coquina level occurs, which is covered by regressive freshwater facies (Beurlen, 1971), at the upper part of the Santana Formation. Mesoalbian Araripina Formation, located above the Santana Formation is a heterolithic facies of alluvial fan plains. This unit is overlaid by fluvial sediments of the Exu Formation (Araripe Group), located at the top of the Araripe Basin. The absence of microfossils makes it impossible to assign a specific age to this unit (Coimbra et al., 2002), although stratigraphic correlation suggests a possible Albian-Cenomanian age (Coimbra et al., 2002; Assine, 2007).

Neumann and Cabrera (2002) and Neumann et al. (2003) argued that the Aptian–Albian lacustrine sequences of the Araripe Basin have been deposited in a shallowing upward sequence (transgressive-regressive cycle), based on evidence for recurrent cycles of six carbonate-siliciclastic facies comprising the “Batateira Beds”, the



Santana Formation (Crato Member), and the “Ipubi Beds”. The latter two units are discussed below. The interplay of the lacustrine facies would have been controlled by the precipitation/evaporation balance and by the dynamics of fluvial drainage (Neumann et al., 2003).

Furthermore, Neumann et al. (2002) assumed that the Aptian-Albian lacustrine evolution of the Araripe Basin comprises four facies associations of the “Batateira Beds” and the Santana Formation (Crato Member only): marginal deltaic, internal terrigene, internal mixed and internal carbonate (C1 to C6), yielding interdigitated/interbedded siliciclastic and carbonate beds. These six carbonate beds evolved from the north to the south (unit C6 yielding the wider carbonate covering), while the palaeolake itself filled the basin from the opposite direction (Neumann et al., 2002). According to these authors, the carbonate units C1 to C6 correspond to the end of five minor transgressive cycles (C1, C2, C3/C4, C5, and C6), within a wider transgressive lacustrine sequence (“Batateira Beds” and Crato Member).

Castro et al. (2006) identified nine cycles of facies distributed among six depositional sequences, also comprising the “Batateira Beds” and the Santana Formation. Four cycles of facies were identified in the Crato Member: lacustrine limestone and bituminous shale (CF); deltaic shale, sandstone, and sigmoidal sandstone (FA-As); lacustrine marginal marl and sandy siltite (MA); and, fluvio-estuarine sandstone with cross-stratification and bioturbation (Axb). The top of sequence S2 has lacustrine limestones (CF), which represent a transgressive event. This sequence is overlaid by shales, sandstones, marls, siltites, and limestones (FA-AS, MA, CF) of sequence S3.1, deposited in deltaic and lacustrine settings. Above, sequence S3.2 is characterized by sandstones, shales, and limestones (Axb, FA, CF), which correspond to fluvio-estuarine, deltaic, and lacustrine facies. Sequence S3.3 is comprised by sandstones, limestones, and shales (As, CF) of deltaic and lacustrine origin. Castro et al. (2006) generally correlated their sequences (upper S2 to S3.3) to the Neumann et al. (2002) transgressive cycles C1 to C6, as shown in Fig. 2.

Transgressive Cycles	Depositional Sequences	Cycles of Facies	Lithology	Palaeoenvironment
C6 (Lower S3.3)	S3.3	AS, CF	Sandstones, Shales, Limestones	Deltaic, Lacustrine
C5	S3.2	Axb, FA, CF	Sandstones, Shales, Limestones	Fluvio- estuarine, Deltaic, Lacustrine
C3/C4	S3.1	FA-AS, MA, CF	Shales, Sandstones, Marls, Siltites, Limestones	Deltaic, Lacustrine
C1/C2	S2 (Top)	CF	Limestones	Lacustrine

**Figure 2.** Correspondence of the transgressive cycles of Neumann et al. (2002) and the depositional sequences of Castro et al. (2006) for the Crato Member.

At this point, attention should be drawn to the Crato Member (Santana Formation). This unit outcrops only in the eastern portion of the Araripe Basin (Viana and Neumann, 2000), consisting of carbonates forming intermittent banks, which are up to 15 m thick, interbedded and interdigitated with marls, shales, sandstones, and siltstones (Viana and Neumann, 2000; Assine, 2007), as discussed above. In each carbonate unit (C1 to C6), basal clay-carbonate rhythmites (submicrofacies sm1) are overlaid by micritic laminated limestones (submicrofacies sm1-6) (Viana and Neumann, 2000; Silva et al., 2002; Neumann et al., 2003). Sm2 has undulations and loop bedding structure; sm3 is characterized by peloids; sm4 has undulations, micro-slumps, and micro-ripples; sm5 consists of fine parallel lamination; sm6 exhibits parallel/wave lamination and ostracods (Silva et al., 2002). Each carbonate unit has an average duration of 200.000-500.000 years (Neumann and Cabrera, 2002).

The laminated clay-carbonate rhythmite facies is characterized by couplets of light micritic laminae and dark laminae, the latter rich in terrigenous components (clay) and organic matter (including plant debris and ostracods; Neumann et al., 2003;

Heimhofer et al., 2010) associated to pyrite crystals. The content of  $C_{org}$  is less than 4% (Neumann et al., 2003). The calcite crystals (5-10  $\mu\text{m}$ ) are idiomorphic and include rhombohedral and polyhedral forms, with dissolution features (Heimhofer et al., 2010). Heimhofer et al. (2010) also reported framboidal pyrite and phyllosilicates and noticed high inter-grain porosity and low cement development. This polymorph-poor facies, with salt pseudomorphs was deposited at a distal location under anoxic conditions (Neumann et al., 2002; 2003) since it is typical of inner lacustrine settings (Neumann et al., 2008). In turn, the fine laminated limestone facies is composed of pairs of light micritic laminae (low magnesium calcite; Silva et al., 2002) and dark laminae with disseminated pyrite (Heimhofer et al., 2010). The calcite crystals are 5-15  $\mu\text{m}$  in length and, therefore, the laminated limestones are considered microspar (Heimhofer et al., 2010). In contrast with the clay-carbonate rhythmite facies, the laminated limestones show less inter-grain porosity due to well-developed spar cement and skeletal grains (Heimhofer et al., 2010). This facies has a lower contribution of detrital material and organic matter ( $C_{org} < 1\%$ ) than the clay-carbonate rhythmites (Neumann et al., 2003) and it includes ostracods and plant remains (Heimhofer et al., 2010). The laminated limestones were also deposited under the same conditions of the clay-carbonate facies (Neumann et al., 2003). In both facies, organic matter is either comprised by amorphous dispersed elements associated to the calcite or by pyritized branchlets of higher land plants (Neumann et al., 2003). These authors argued that regarding its composition and poor cell preservation, these branchlets might have undergone bacterial decay. Detrital quartz may also occur in both facies.

Non-laminated levels also occur in the Crato Member (Dias-Brito and Tibana, 2015). The laminated limestones are underlain by ostracod-rich organo-limestone laminites, which has mudstone/wackestone laminae. These are characterized by ostracods and peloids interlaminated with organic-clay laminae, which have ostracods and limestone nodules infilled with microbial spheres and ostracods (Dias-Brito et al., 2015).

### **3.2. Palaeoenvironment and diagenesis of the Crato Member carbonate units**

The preserved area of the Crato palaeolake is about 7.500  $\text{km}^2$  wide. The biota thrived in a warm tropical–subtropical palaeoclimate, seasonally controlled by humid

and dry cycles, leading to permanent density stratification owing to thermal heterogeneities along the water column (Neumann et al., 2003). The semiarid-arid conditions are supported by the predominance of the conifer family Cheirolepidiaceae. Still regarding the Crato palaeoflora, Ferns, Bennettitales, Gnetales, angiosperms, and large conifers covered areas around the waterbody (Neumann et al., 2003).

The deltaic body and the smooth inclination of the bottom relief have resulted in both interdigitation between siliciclastic and carbonate successions, and small scale sedimentary structures (micro-faulting and loop bedding; Neumann et al., 2002). The presence of faults, loop bedding, and slumps owns to minor syndepositional seismic activity (Silva et al., 2002). The lacustrine sequence thickness varies from 50 to 70 m (Silva et al., 2002).

Isotopic analyses of oxygen ( $\delta^{18}\text{O}$  values between -5 ‰ and -7 ‰) performed in the Crato Member lowermost laminated limestones revealed that the depositional palaeoenvironment received meteoric  $^{18}\text{O}$ -depleted waters and pointed to a freshwater waterbody (Heimhofer et al., 2010). Carbonate carbon isotopic data ( $\delta^{13}\text{C}$  within -0.2 ‰ and 1.9 ‰) support that the palaeoenvironment was a stratified lake, poorly connected with external water sources, with stagnant bottom waters, reflecting the uptake of  $^{12}\text{C}$  by microorganisms in the water column. Positive  $\delta^{13}\text{C}$  values are due to limited mixing of  $^{12}\text{C}$  in the water, although equilibrium with the atmosphere might also have played a role (Heimhofer et al., 2010). Alternatively, Heimhofer et al. (2010) also suggested the dissolution of marine carbonates and/or the isolation of the palaeolake as explanations for such  $\delta^{13}\text{C}$  high positive values. Furthermore, these authors also alleged that the lack of fine cyclic patterns on isotope variation could be explained by the palaeoenvironmental stratification, although post burial homogenization cannot be ruled out. On the other hand, they claimed that overall differences do exist between clay-carbonate rhythmite and laminated limestone facies. Isotopic excursion to more negative  $\delta^{18}\text{O}$  values from the former to the latter facies means an opening of the palaeoenvironment during highstand water levels simultaneously with laminated limestone deposition.

Neumann et al. (2003) suggested permanent water stratification in relation to  $\text{O}_2$  (i.e. meromictic conditions; Fig. 3). Anoxic bottom conditions are supported by the occurrence of disseminated galena and sphalerite in the limestones as well as by the lack

of bioturbators (Heimhofer and Martill, 2007). Water column stratification (yielding a bottom water monimolimnion and an upper water column mixolimnion, inhabited by aquatic insects, turtles, and fishes) could be explained by stagnation and/or high rates of surface water primary productivity providing a high flux of organic matter to the lake floor, the decay of which by aerobic bacteria reduced bottom water oxygen and, eventually, led to anaerobic conditions in deep waters (Heimhofer and Martill, 2007). This recycling of organic matter would have yielded low production of autochthonous organic matter in the lake bottom, further enhanced by stratification (Neumann et al., 2003). Alternatively, Catto et al. (2016) suggested that low organic matter preservation in some levels could be explained by bottom spells of freshwater, which could have oxidized organic matter. Additionally, a photic zone euxinia (PZE) could also have been seasonally established in the waterbody (Fig. 3), as evidenced by the presence of isorenieratene, a biomarker of Chlorobiacea, which thrives below the oxic-anoxic water column interface and performs photosynthesis using hydrogen sulphide instead of oxygen (Heimhofer and Martill, 2007).

The occurrence of salt pseudomorphs and xerophytic vegetation pollen support a semi-arid to arid palaeoclimate (Heimhofer and Martill, 2007), which is further evidenced by the low diversity of the palaeolake biota and by the low amount of organic matter recorded in the rocks, possibly owing to the chemical stratification (Neumann et al., 2003). The events of hypersalinity would have been possibly triggered by the dissolution of former precipitated evaporites, the evidence of which is supported by the presence of five types of halite pseudomorphs (Martill et al. 2007b). The high salinity could have been increased by the occasionally endorheic nature of the basin, while freshwater conditions would have been established near fluvio-deltaic river mouths (Neumann and Cabrera, 2002). Endorheic lacustrine basins have dynamic shores, with retrogradational and progradational cycles and enhanced chemical mineral precipitation owing to high evaporation rates plus low water input (Neumann and Cabrera, 2002; Neumann et al., 2008). These salinity fluctuations can be explained on palaeogeographical and palaeoclimatic grounds since, in the Early Cretaceous, the area was subjected to monsoon influence (Neumann and Cabrera, 2002). Martill et al. (2007b) have pointed out that fluctuation in salinity levels could account for anoxia in the palaeolake.

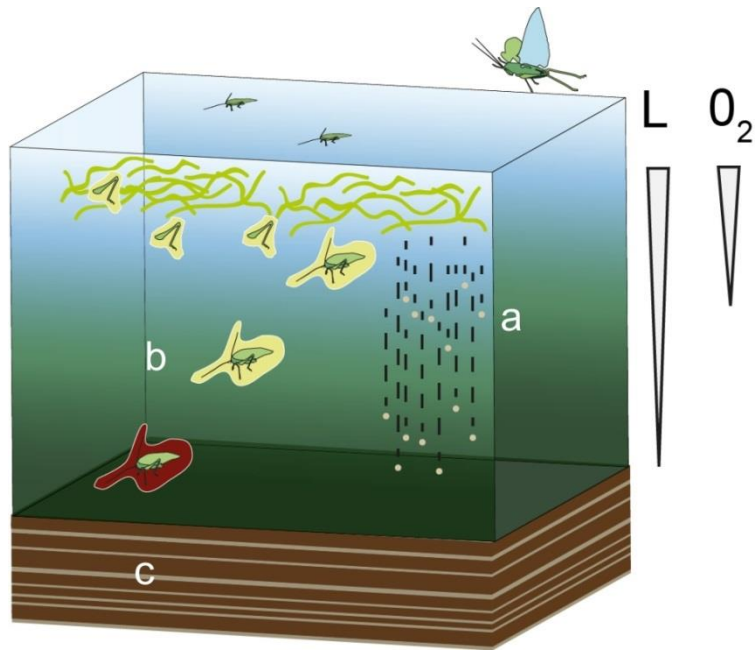
Terrigenous sediments might have been restricted to marginal settings during carbonate deposition (low Al, K, Ti, and Si; Heimhofer et al., 2010). Heimhofer et al. (2010) enumerated factors which account for the precipitation of carbonate at lacustrine settings: terrigenous input, carbonate bioclasts, benthonic microbial influence, and microbially induced and/or mediated authigenic precipitation. The evidence of mass flow deposits in the Crato Member is scarce, except in one location, cited by Heimhofer and Martill (2007). Moreover, the lack of reworked calcite crystals further rules out the hypothesis of a terrigenous origin for the carbonate (Heimhofer et al., 2010). Additionally, the laminite is not predominantly built up by bioclastic particles, though ostracods do occur at some levels, particularly near the lake edge (Heimhofer et al., 2010; Dias-Brito and Tibana, 2015).

The presence of wrinkle and ripple like structures (Heimhofer and Martill, 2007; Martill and Wilby, 1993), similar to Microbially Induced Sedimentary Structures (MISS) (Noffke et al., 2001), suggests that the depositional surface was, at least, covered by patches of microbial mats or biofilms. However, only isolated slabs without precise stratigraphic location have been recovered (Heimhofer and Martill, 2007). Moreover, putative gas domes have also been reported (Martill et al., 2007b). However, owing to the fragmentary distribution of such evidence plus the lack of typical petrographic evidence, Heimhofer et al. (2010) have ruled out the role of a benthonic microbiota as the origin of the carbonate. Alternatively, these authors have suggested microbially induced and/or mediated authigenic precipitation of calcite crystals in the water column, which could have been seasonally controlled by temperature fluctuations and by blooms of autotrophic microorganisms (i.e. 'whitings'; Fig. 3) (Heimhofer and Martill, 2007; Gierlowski-Kordesch, 2010). The carbonate deposition due to 'whitings' is controlled by rising water temperature during summer and autumn (Gierlowski-Kordesch, 2010) and by autotrophic microorganism proliferation due to a rise of nutrient supply (Heimhofer et al., 2007). The former factor leads to decreasing carbonate solubility, while the latter enhances carbonate nucleation rates and the uptake of CO<sub>2</sub> from the water, therefore contributing to calcite precipitation (Heimhofer et al., 2007). However, different authors have recently shown evidence for the activity of benthonic microbial mats or biofilms at the edge of the palaeolake, where non-laminated clayey mudstones/wackestones rich in fibro-radiated spherules, microbial spheres, ostracods, gastropods, bivalves, and angular-shaped terrigenous quartz do

occur (Dias-Brito and Tibana, 2015). Moreover, Catto et al. (2016) have further shown that microfossils and EPS are ubiquitous throughout the Crato Member beds, thus challenging Heimhofer et al. (2010) previous interpretation. Dolomitic structures, as observed in a Crato Member locality (Martill et al., 2008), might have been originated by sedimentation controls of carbonate at lacustrine settings particularly by water input and circulation, including groundwater (Gierlowski-Kordesch, 2010).

The preservation of the undisturbed fine lamination (Fig. 3) seems to have occurred by the following factors: deposition below normal wave base level, limited bottom current action, and lack of benthonic fauna (e.g. bioturbators plus absence of trace fossils; Heimhofer and Martill, 2007), though submicrofacies with undulations and ripples do occur in some levels (Silva et al., 2002).

The absence of thermal action diagenetic features, such as degraded organic matter (e.g. palynomorphs display original colour), the lack of recrystallization of calcite crystals, cement-filled fractures, stylolites, pressure-solution features, and fractures, besides the occurrence of exceptional soft-tissue preservation, fit a low depth burial idea (Heimhofer and Martill, 2007; Heimhofer et al., 2010). This proposition is consistent with the low degree of compaction of the rocks, further supported by high porosity, particularly in the clay-carbonate rhythmites. This latter characteristic is explained by organic matter infilling the pores, which leads to low nucleation cement rates (Heimhofer et al., 2010). Furthermore, these authors pointed out that original stable mineralogy has possibly inhibited cement growth and recrystallization.



**Figure 3.** Diagram showing an inner setting of the Crato palaeolake. The palaeoenvironment was stratified with regard to  $O_2$  (right side of the figure). Light intensity (L) is shown on the right. The calcite could have been precipitated during ‘whitings’ (a). A photic zone euxinia (PZE) was at least seasonally established below the oxic-anoxic interface of the water column (b). The lamination of the laminated limestone is undisturbed (c). Drawing by Bruno Becker Kerber.

#### 4. Material and methods

The fossil specimens used in this research belong to the Scientific Palaeontological Collection of the Institute of Geosciences of the University of São Paulo (IGc-USP). The fossil insects are identified by the code **GP/1E** (invertebrate collection), while the fossil fishes are recognized by the prefix **GP/2E** (vertebrate collection). The following samples have been analyzed: GP/2E 9666, GP/2E 9005, GP/2E 9006, GP/2E 9014, GP/2E 7781g, GP/2E 7786f, GP/2E 7913e, GP/2E 7782j, GP/2E 7780e, GP/1E 9435, GP/1E 7105, GP/1E 8440, GP/1E 8397, GP/1E 8827, GP/1E 6820, GP/1E 10368, and GP/1E 9137. The complete list of samples and their correspondent thin sections can be found in Appendix 2.



The specimens were photographed in a Zeiss Stemi 2000-C stereomicroscope coupled to a Zeiss AxioCam ICc3 camera. The image acquisition was made in the software AxionVision 4.8.

In this research, different techniques were employed for studying external or internal parts of insects, both fish bones and soft tissues, and the host rock. The geochemical analyzes were performed systematically, being repeated in different points in a representative set of samples, when available. Here follow the techniques and methods employed in this research:

### **Petrographic thin sections**

Thin sections perpendicular to the rock lamination and cross-sections of fish vertebral column, along with soft-tissues, were made. The aims were: (1) observe and characterize soft-tissue and bone morphology in cross-section, and (2) describe host carbonate petrography. Furthermore, since thin sections provide flat surfaces, they enabled accurate EDS and synchrotron micro-XRF mapping (discussed below). Therefore, two types of thin sections were made: (1) 30  $\mu\text{m}$ -thick with cover slips used chiefly for mineral, texture, and structure description/identification, and (2)  $>30$   $\mu\text{m}$  (around 50  $\mu\text{m}$ )-thick without cover slips used for geochemical analyses. For these, thicker thin sections (i.e. more rock volume) enhanced the measured signal (Davis and Martill, 1999), yielding high-quality geochemical maps.

### **Raman spectroscopy**

When materials are illuminated by a monochromatic visible light source, incident photons interact with the molecular vibrations of the material (Neuville et al., 2014). As a consequence, scattered photons may have different energy in comparison to the incident photons, defining the Raman scattering phenomenon. This energy shift is known as Raman shift, which is measured by a spectrometer, yielding a characteristic spectrum (i.e. graphic) of the material molecular vibrations. Raman spectroscopy can be used for the chemical and mineralogical characterization of samples, being widely applied in mineral identification (Faria and Lopes, 2007; White, 2009) since it avoids most of the destructive procedures usually associated with conventional geochemical methods. In-situ analyzes were performed to investigate the minerals that have replaced original organic compounds during fossilization, with the advantage of having sub-

micron resolution and mapping capacity, as the spectrometer is coupled to a confocal microscope. The equipment setup included both a micro-Raman inVia Renishaw, coupled to a laser of 785 nm wavelength and another similar equipment coupled to a 532 nm wavelength laser. A CCD detector was used in both setups. The measurements were performed both at the Research Unity in Astrobiology, Laboratory of Astrobiology (NAP/Astrobio-USP, Astrolab) and at the Laboratory of Molecular Spectroscopy (LME) of the Institute of Chemistry of USP. The Raman spectra were then analysed in the software Origin<sup>®</sup> 8 and data were interpreted using the RRUFF database.

### **X-ray fluorescence (XRF)**

XRF consists of the following principle: when materials are excited by a high-energy X-ray photon source, electrons move from inner to outer atom orbitals and, consequently, external electrons occupy the vacancy left inside (Verma, 2007). In response to this process, X-ray photons with the same energy of that lost (to occupy the internal orbital) by the outer electron are emitted. This energy value is the energetic difference between the two orbitals involved. This X-ray emission is called fluorescence, being the energy value characteristic of the excited atomic element. XRF is used to characterize the elemental composition of samples. This is a very useful technique since it enables measuring several elements simultaneously, in a non-destructive way, not requiring high amounts of sample (Verma, 2007). XRF was employed in fossils and their host rock to characterize elemental composition and its distribution, aiming to understand the meaning of the latter. In this way, it was possible to test whether elements are representative of the original composition of the organisms or the result of fossilization processes. The analyses were performed at the Institute of Physics of USP (IF-USP), using a portable mini Amptek X-ray tube of Ag anode and a Silicon Drift Detector (SDD - X-ray semiconductor detector). Data processing was performed using the software WinQXAS, RUMP, and Microsoft Excel<sup>®</sup>.

Synchrotron micro-XRF (SR- $\mu$ XRF) point analysis and mapping were performed with the same objectives as conventional XRF (discussed above). Moreover, specific characterization of phosphorus was made to quantify this element in the rock matrix and in fossil insects, aiming to assess preservational processes. In comparison to the employed conventional XRF, SR- $\mu$ XRF has the advantage of  $\mu$ m-scale spectra collection, plus mapping mode, enabling the detailing of very fine structures and the

characterization of elemental distribution among them. In addition, SR- $\mu$ XRF enables the measurement of elements with concentrations below the detection limit of conventional XRF. Phosphorus analysis was performed in the soft X-ray spectroscopy (SXS) beamline, and point/map characterization of several elements were made in the XRF beamline of the Brazilian Synchrotron Light Laboratory (LNLS).

### **Particle-induced X-ray emission (PIXE)**

The physical principles behind PIXE are very similar to XRF, but in the former, the excitation is made by protons or other particles with charge (Verma, 2007). The PIXE technique is employed in qualitative and quantitative elemental analysis of materials and was used for the same purpose as the XRF. However, PIXE is different from XRF in regard to the following aspects (Verma, 2007): it is a surficial technique, that is, protons interact with atoms in a shallow sample area (ca. tens of microns); the efficiency for detecting elements with X-ray energies higher than Ca is better than XRF; and, compared to XRF, PIXE has a greater detection sensitivity. Furthermore, the equipment available allows sample mapping and has a smaller diameter beam in comparison with the portable (conventional) XRF equipment, allowing investigation of smaller structures. The analysis was performed in the external beam setup of the 1.7 MV-tandem accelerator of the Laboratory of Materials and Ionic Beams (LAMFI) of the IF-USP.

### **Scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDS)**

A SEM is basically composed of an electron gun and electromagnetic lenses (Leng, 2008). The electron probe scans lines along the sample surface, which emits electrons that are captured by a detector. Then, an image is built based on the correlation between scanned points and points in a screen. SEM was used to yield very detailed images at micrometric scales of fossil ultrastructures, including the morphology of both preserved soft tissues and minerals replacing the specimens.

EDS is based on the same physical principles of XRF, but X-ray emission is triggered by a high energy electron beam (Leng, 2008). EDS microanalysis was employed to evaluate the elemental composition of the samples – particularly light elements, such as oxygen and carbon – yielding point spectra and maps, the latter

revealing a correlation between structures and composition. The analyzes, including secondary (yielding topographic contrast) and backscattered (yielding elemental contrast) electron micrographs, were performed at the Brazilian Nanotechnology National Laboratory (LNNano) and at the Laboratory of Technological Plasmas (LaPTec) of the São Paulo State University, using a JEOL JSM-6010 LA microscope and also a FEI Quanta 650 FEG microscope, both coupled to EDS equipment. In the former SEM, an X-ray Dry SD Hyper (EX-94410T1L11) detector with resolution of 129 to 133 eV for the Mn K $\alpha$  line at 3000 cps was used. In order to avoid surface charging, some samples were coated with a thin layer of gold-palladium using a DESK-V HP Cold Etch/Sputter system. Whenever possible, EDS analysis was not used in coated samples, and EDS mapping was performed in thin sections, to avoid misinterpretation of elemental composition.

**Obs.:** Details on experimental setup are found in the following chapters (articles).

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**Appendix 2** – List of specimens and thin sections.

<b>Sample</b>	<b>Brief description</b>	<b>Facies (GL - grey limestones; BL - beige limestones)</b>
GP/2E 9666	<i>Dastilbe crandalli</i>	GL
GP/2E 9005	<i>Dastilbe crandalli</i>	GL
GP/2E 9006	<i>Dastilbe crandalli</i>	GL
GP/2E 9014	<i>Dastilbe crandalli</i>	BL
GP/2E 7781g	<i>Dastilbe crandalli</i>	BL
GP/2E 7786f	<i>Dastilbe crandalli</i>	BL
GP/2E 7913e	<i>Dastilbe crandalli</i>	BL
GP/2E 7782j	<i>Dastilbe crandalli</i>	BL
GP/2E 7780e	<i>Dastilbe crandalli</i>	BL
GP/1E 9435	Insect	BL
GP/1E 7105	Insect - Orthoptera	BL
GP/1E 8440	Insect - Hemiptera	BL
GP/1E 8397	Insect - Orthoptera	BL
GP/1E 8827	Insect - Orthoptera	BL
GP/1E 6820	Insect - Indetermined exoskeleton	BL
GP/1E 10368	Insect - Orthoptera	BL
GP/1E 9137	Insect - Blattodea	BL

<b>Sample</b>	<b>Thin section</b>	<b>Brief description</b>
GP/2E 9666	GP/L 16, GP/L 17	Thin section cross-cutting rock lamination and fish
GP/2E 9006	GP/L 18, GP/L 19	Thin section cross-cutting rock lamination and fish
GP/2E 7781g	GP/L 20, GP/L 21	Thin section cross-cutting rock lamination and fish
GP/1E 9435	GP/L 172	Thin section cross-cutting rock lamination and insect