



THROWING LIGHT ON AN UNCOMMON PRESERVATION OF BLATTODEA FROM THE CRATO FORMATION (ARARIPE BASIN, CRETACEOUS), BRAZIL

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ABSTRACT – Fossilization results from several physical-chemical-geological processes. Original labile and non-bioclastic structures rarely survive throughout this process. In particular, the Crato Formation (Araripe Basin) is one of the most significant Cretaceous *Konservat-Lagerstätten* due to its well-preserved invertebrates, mainly three-dimensional insects. In general, Crato insects exhibit brown-orange color, constituted by goethite or hematite replacements. In this context, we used the scanning electron microscopy coupled to energy dispersive spectrometer and Raman spectroscopy to analyze *Araripeblattia dornellesae*, a 115 million-years-old fossil from Crato Formation, Araripe Basin. Our results show that a dark-color material rather than the brown-orange pattern preserve this specimen. The carbon is restricted to the fossil imprint, indicating some retention of the original organic remains. In addition, the presence of original organic components allowed to approach the biochemical aspects further than simple morphology, as well as to elucidate the taphonomical complexity involved in this preservation style.

Keywords: taphonomy, Lower Cretaceous, Crato Formation, *Lagerstätten*, organic components, Scanning Electron Microscopy.

RESUMO – O processo de fossilização é resultado de uma série de processos físico-químicos ao longo do tempo geológico, onde dificilmente estruturas orgânicas são preservadas. Particularmente, a Formação Crato (Bacia do Araripe) é um dos mais importantes *Konservat-Lagerstätten* do Cretáceo, devido à presença de invertebrados bem preservados, inclusive fósseis de insetos em três dimensões. De maneira geral, os insetos da Formação Crato apresentam coloração marrom ou laranja, substituídos por goethita ou hematita. Neste contexto, utilizou-se microscopia eletrônica de varredura, espectroscopia de energia dispersiva e espectroscopia Raman para analisar *Araripeblattia dornellesae*, um fóssil de 115 milhões de anos. Este espécime apresenta coloração escura, em vez de padrão marrom/alaranjado. O elemento carbono aparece restrito a morfologia do fóssil indicando retenção de constituintes orgânicos. A presença de componentes orgânicos preservados pode, além da abordagem morfológica, facilitar nosso entendimento sobre os aspectos bioquímicos de organismos pretéritos, bem como elucidar sobre os complexos processos tafonômicos envolvidos neste tipo de preservação.

Palavras-chaves: tafonomia, Cretáceo Inferior, Formação Crato, *Lagerstätten*, componentes orgânicos, Microscopia Eletrônica de Varredura.

INTRODUCTION

It is generally assumed that the laminated limestones of the Crato Formation were deposited in a lacustrine environment during Early Cretaceous. This unit outcrops in the northeast side of the Araripe Basin and stands out for being an important paleontological site in northeastern Brazil (Figure 1). The fossils are relatively abundant and diverse, including trace fossils, invertebrates, vertebrates, plants, and palynomorphs (Viana & Neumann, 2000). Due to the exceptionally preserved biota (Davis & Martill, 1999; Fielding *et al.*, 2005; Heads *et al.*, 2005; Menon & Martill, 2007; Silva *et al.*, 2013; Delgado *et al.*, 2014; Barling *et al.*, 2015; Osés *et al.*, 2016, 2017) the Crato Formation is considered a *Konservat-Lagerstätte*. The Crato Formation represents one of the largest Cretaceous insect deposits in the world, considering both taxonomic diversity and number of individuals (Grimaldi, 1990). In this unit, the following orders are represented: Orthoptera, Isoptera, Homoptera, Hymenoptera, Blattodea, Ephemeroptera, Odonata, Dermaptera, Raphidioptera, Diptera, Trichoptera, Coleoptera, Neuroptera and Heteroptera.

The fossil entomofauna from the Crato Formation is in an exceptional state of preservation, with most of the specimens displaying head, thorax and abdomen including fragile morphological structures such as appendages and wings. Commonly, the insects are three-dimensionally preserved demonstrating indisputably similarities to modern species. Such preservation is fundamental to provide data about the evolution of the insects. Almost all of paleoentomofauna from Araripe Basin occurs within the laminated limestones of Crato Formation. The majority of the fossils have a brown tone due to iron oxides/hydroxides (hematite, goethite, limonite), which replaces the original organic matter, as observed by Menon & Martill (2007), Delgado *et al.* (2014), Barling *et al.* (2015) and Osés *et al.* (2016). Wang *et al.* (2012) also documented similar preservation features in Juhol biota insects, northeastern China.

Among the different groups of fossil insects, the Blattodea (cockroaches) are one of the most abundant both in number of described species and number of individuals. Most of cockroach fossils display brown coloration. However, some specimens show a darker hue, as a consequence of certain concentrations of carbon, sulfur and phosphate (Menon & Martill, 2007). Such substances may be interpreted as the original organic remnants. Mainly hydrocarbons, proteins and lipids (Lockey & Dularay, 1986) compose the cuticle of modern cockroaches. Over the last decades, attention has been focused on various aspects of the arthropods taphonomy, particularly in our understanding of the environmental circumstances under which exceptional preservation is likely. Nevertheless, taphonomic processes influencing to exceptional preservation are still largely unresolved, although oxygen-deficient bottom water and hypersalinity seem to be important factors in preservation of organic parts of arthropods (Allison, 1988; Briggs, 1991; Briggs & Kear, 1993; Briggs *et al.*, 1998; Stankiewicz *et al.*, 2000; Babcock *et al.*, 2001; Oliveira *et al.*, 2015). The goal of the present study is to evaluate a 115 million years-old fossil of *Araripeblatta dornellesae* (Mendes & Coelho, 2007) (LP/UFC CRT 847) from Crato Formation. Here, we used a large-field scanning electron microscopy (LF-SEM) coupled to energy dispersive spectroscopy (EDS) and Raman spectroscopy to characterize compositional aspects of the fossil at multiple length scales. We chose LP/UFC CRT 847 because it is the only clearly articulated specimen (including antennae, articulated wings and veins) preserved into carbonaceous material. The articulated specimens with open wings and without abdominal deformity indicate that they underwent little transport *post-mortem* (Mendes, 1998; Duncan *et al.*, 2003).

GEOLOGICAL SETTING

The Araripe Basin is an elongated basin, with a west to east trending, being one of the largest basins in the interior of the Brazilian Northeast (Figure 1). Assine (2007) identified

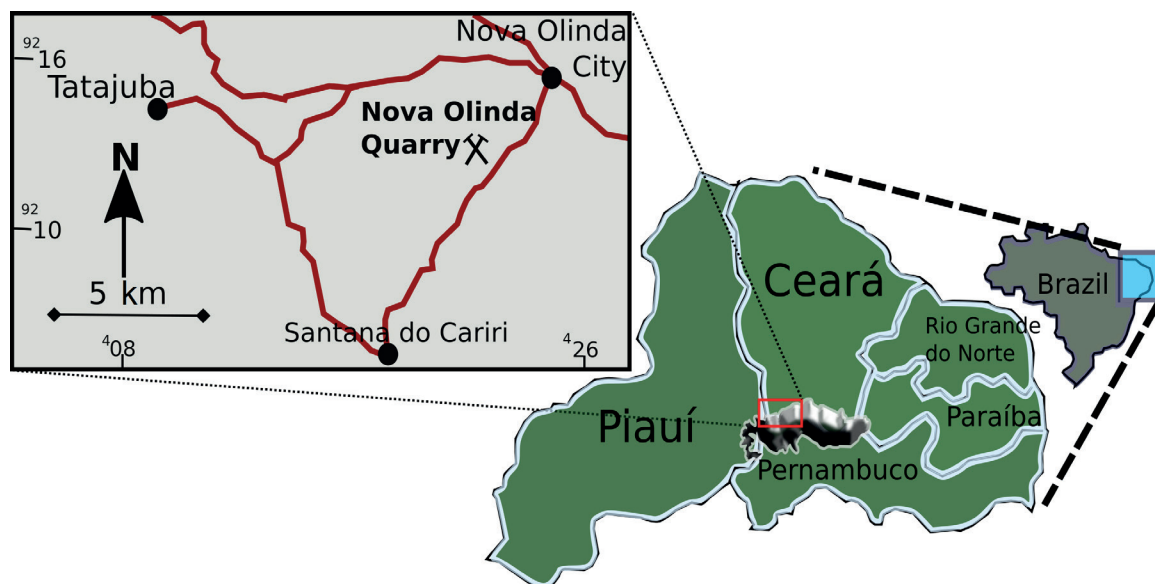


Figure 1. Location of the Araripe Basin in Northeast Brazil and the studied quarry in the Nova Olinda city. Red lines correspond to roads.

five depositional tectonic-sedimentary sequences limited at the top and bottom by regional discordance: the Paleozoic Sequence; Pre-Rift; Rift; Post-Rift I and Post-Rift II.

The Crato Formation is embedded in the post-Rift I sequence concerning the second lacustrine phase of the Araripe Basin (Assine *et al.*, 2014). This unit comprises sediments around 60 m thick and consists of carbonates interbedded with a series of claystones, siltstones and sandstones. The laminated carbonate facies represents the worldwide recognized Crato *Fossil-Lagerstätte*. A restricted lacustrine or lagoonal environment with a stratified water column represents the depositional system. The upper part of the water column was likely well oxygenated, whereas the lake/lagoon bottom was anoxic (Heimhofer *et al.*, 2010). Neumann (1999) suggests that carbonate units represent cyclic pulses controlled by climatic conditions, especially rainfall. The presence of halite pseudomorphs (NaCl) (Martill *et al.*, 2007) is an unequivocal evidence of hypersalinity levels. The Crato Formation is part of the *Sergipea variverrucata* palynozone, which implies an Aptian age for this unit (Coimbra *et al.*, 2002).

MATERIAL AND METHODS

Fossil material

The specimen studied in this work is an adult Blattodea belonging to the species *Araripeblatta dornellesae* (Figure 2). The fossil is originated from the limestone layers of the Crato Formation, found on Pedra Cariri Quarry in Nova Olinda, State of Ceará, Brazil. The material is deposited in the paleontological collection of the Universidade Federal do Ceará under the code LP/UFC CRT 847.

Analytical approaches

Raman spectroscopy. This is a very precise technique to determine carbon in several kinds of samples. Carbon may present as graphite, amorphous or disordered structures, among many other possibilities. The Raman spectrum was obtained by a LabRAM HR (Horiba) spectrometer equipped with a liquid N₂-cooled CCD detector behind a 600 g/mm grating, using 785 nm laser radiation for excitation (~2 mW at the sample surface). The final power at the end of the objective lens (Leica 50x, NA = 0.80) used to focus

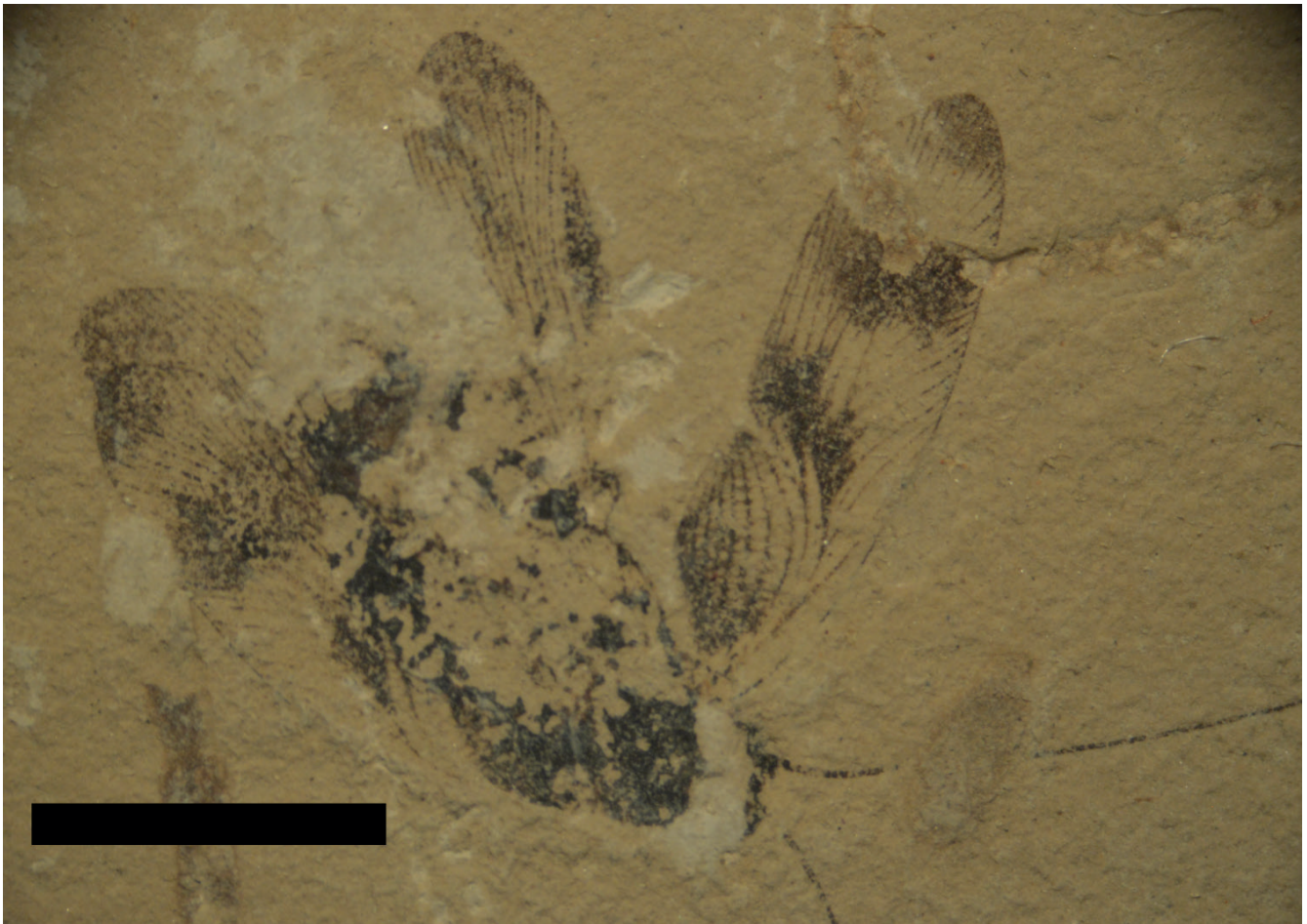


Figure 2. Optical photomicrograph of *Araripeblatta dornellesae* showing the wings open. Scale bar = 1 cm.

on the sample surface was 1 mW with an integration time of 20 s. Raman spectroscopic analyses were performed at the Laboratório de Espectroscopia Vibracional (Physics Department) of the Universidade Federal do Ceará.

Large-field energy dispersive spectroscopy (EDS) and scanning electronic microscopy (SEM) analysis. Large-field scans were performed in an electron microscope Quanta-450FEG (FEI) equipped with a gaseous analytical detector (GAD). In addition, an X-ray detector model 150 (Oxford) was used in X-ray energy dispersive spectroscopy (EDS). The specimen was inserted into the SEM without any preparation. Analyses were performed under low vacuum (approximately 100 Pa with water vapor), by capturing both BSE and X-ray in order to generate the elemental maps. The specimen was scanned at a beam acceleration voltage of 20 kV, with a working distance of 15 mm. SEM/EDS analyses was performed at the Central Analítica of the Universidade Federal do Ceará.

RESULTS

The cockroach fossil studied here shows well-preserved morphology, with the three tagmata (head, thorax and abdomen) and appendices. According to Duncan *et al.* (2003), after cockroach death, in 21 days the internal abdominal organs liquefy giving the carcass a swollen appearance. The head separates from thorax after 30 weeks. In addition, the presence of antennae and wings, both anterior and posterior (note all veins), indicate that cockroach underwent little *post-mortem* alteration. Otherwise, the forewing should displays breakage along the CuP (cubitus posterior) vein (Duncan *et al.*, 2003) (Figure 2).

Elemental mapping of LP/UFC CRT 847 acquired through LF-EDS shows that the fossil is preserved as dark-colored material composed predominantly of carbon (Table 1). The SEM analyses of LP/UFC CRT 847 reveal that the soft-tissue consists of a carbonaceous material without a well-defined microfabric often associated with calcitic filaments (Figure 3).

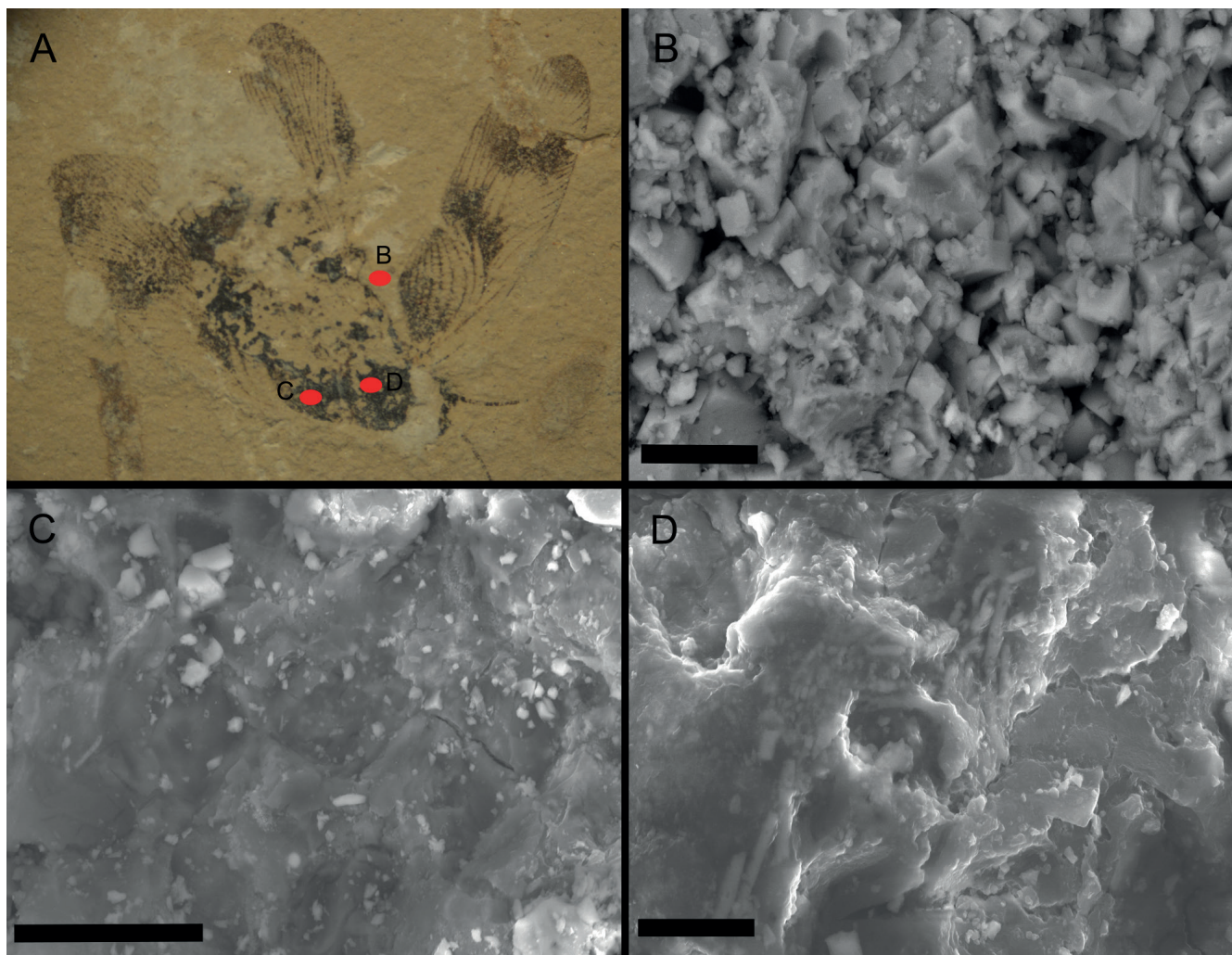


Figure 3. Micrographs of LP/UFC CRT 847. A, specimen with points (B–D) analyzed. B, carbonate matrix with abundant euhedral to subhedral crystallites of calcite. C, scanning electron microscopy secondary electron image of amorphous material occurring consistently along the specimen body. D, micrographs of calcitic filaments associated with LP/UFC CRT 847. Scale bars: B = 10 μm ; C–D = 5 μm .

Elements such as carbon, sulfur and phosphorus are more concentrated on the fossil outline than in the surrounding matrix.

The largest concentrations of carbon (Table 1) occur almost strictly at the body outline. It is present in the fossil in three areas analyzed: wing, antenna and body (Figure 4). EDS indicates carbon concentrations in LP/UFC CRT 847 range from 11 % to 15.2 %, the average value is 13.4 %. This value is lower than the carbon value of modern Blattodea cuticles, such as *Blattella germanica* (Kaya *et al.*, 2015). However, this value is higher than average organic carbon for Crato Formation, which is generally less than 1 % according to Catto *et al.* (2016).

The elemental map for sulfur and phosphorus (Figure 4) shows that both concentrations are higher on the fossil surface than in the surrounding matrix. Sulfur is substantially reduced in the matrix except for a portion near the extended forewing, where the host rock is fractured. The fracture possibly enabled the lateral transfer of organic sulfur from the fossil tissue. The phosphorus was found to be restricted to the interior of the specimen and it was not detected on the wing or on the antennae.

The EDS data shows that calcium is the main element present in the matrix, making clear the contrast between the fossil and the lithological component. The determination of some elements can provide important information about the post-diagenetic changes in the matrix (Heimhofer *et al.*, 2010; Forchielli *et al.*, 2014). The presence of iron and manganese (Figure 5) certainly indicates precipitation of these metals under oxidizing conditions. During the post-diagenetic phase, the precipitation of these metals can promote certain taphonomic distortions contributing to reduction in the quality of preservation of fossils, or even causing their destruction. In this case, Fe and Mn distributions are more related to the matrix than to the fossil itself. The high concentrations of these elements (Fe, Mg and Mn) can be verified along a small fracture in the rock (Figure 5).

The presence of carbon is also attested by Raman spectroscopy. The Raman spectrum of the Blattodea fossil (Figure 6) reports two characteristics first order peaks in the region identified as the D and G bands, observed respectively at 1331 and 1555 cm^{-1} . The superposition of an intense luminescence background would make it very difficult to obtain the Raman scattering along the sample. Probably,

Table 1. Average weight (%) of elements contained in the analyzed areas of LP/UFC CRT 847 fossil.

Scanning Areas	Element (wt%)									
	Ca	C	F	Mg	Si	S	Fe	Al	Mn	P
Wing	40.3	14.0	1.4	1.0	0.5	0.4	0.3	0.2	0.2	0.0
Antenna	40.1	11.0	1.6	1.1	0.8	0.3	0.5	0.4	0.0	0.0
Body	38.2	15.2	1.4	1.0	0.8	0.5	0.5	0.4	0.2	0.1

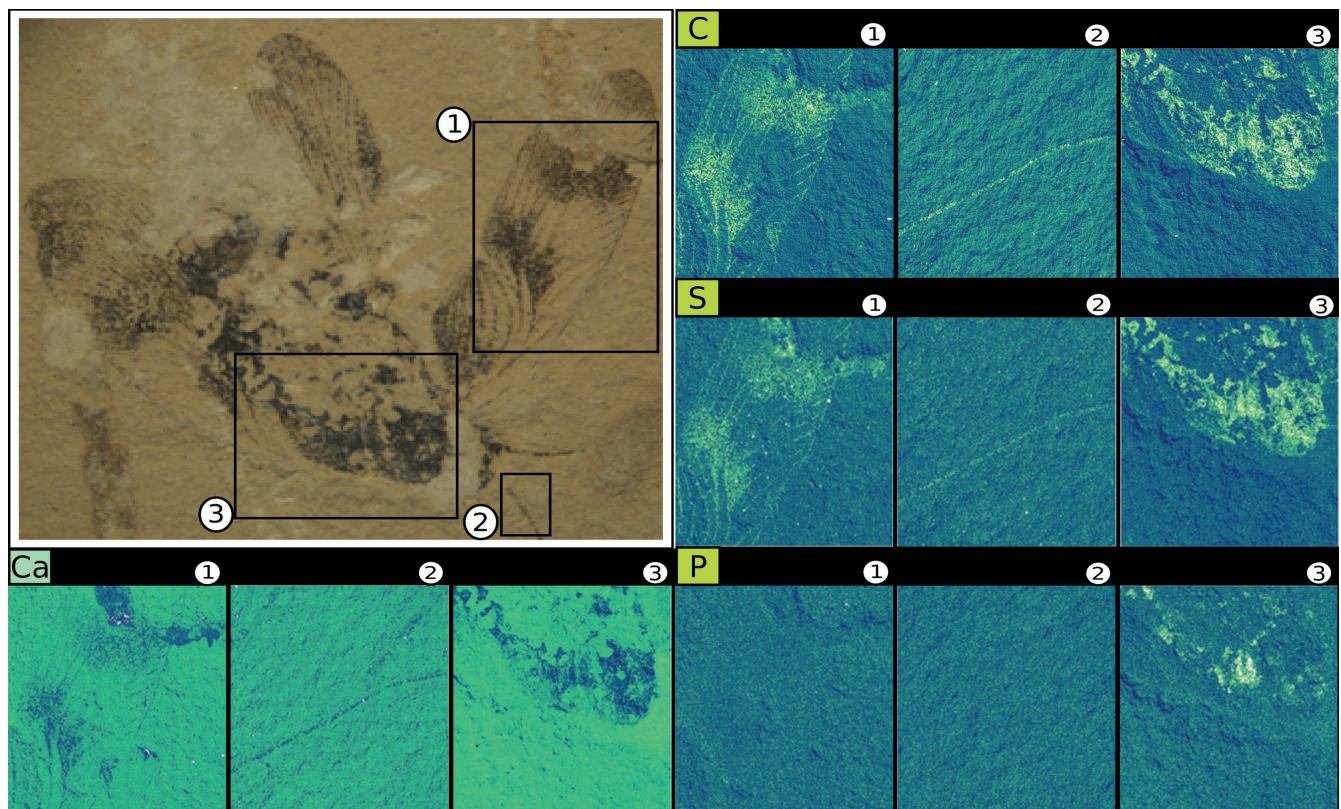


Figure 4. Elemental maps from Blattodea, Crato Formation. Large-Field analyses were performed in three delimited areas 1, 2 and 3 for carbon (C); sulphur (S); phosphorus (P) and calcium (Ca). In the map, greenish tones mean presence, whereas blue-dark tones mean absence of element.

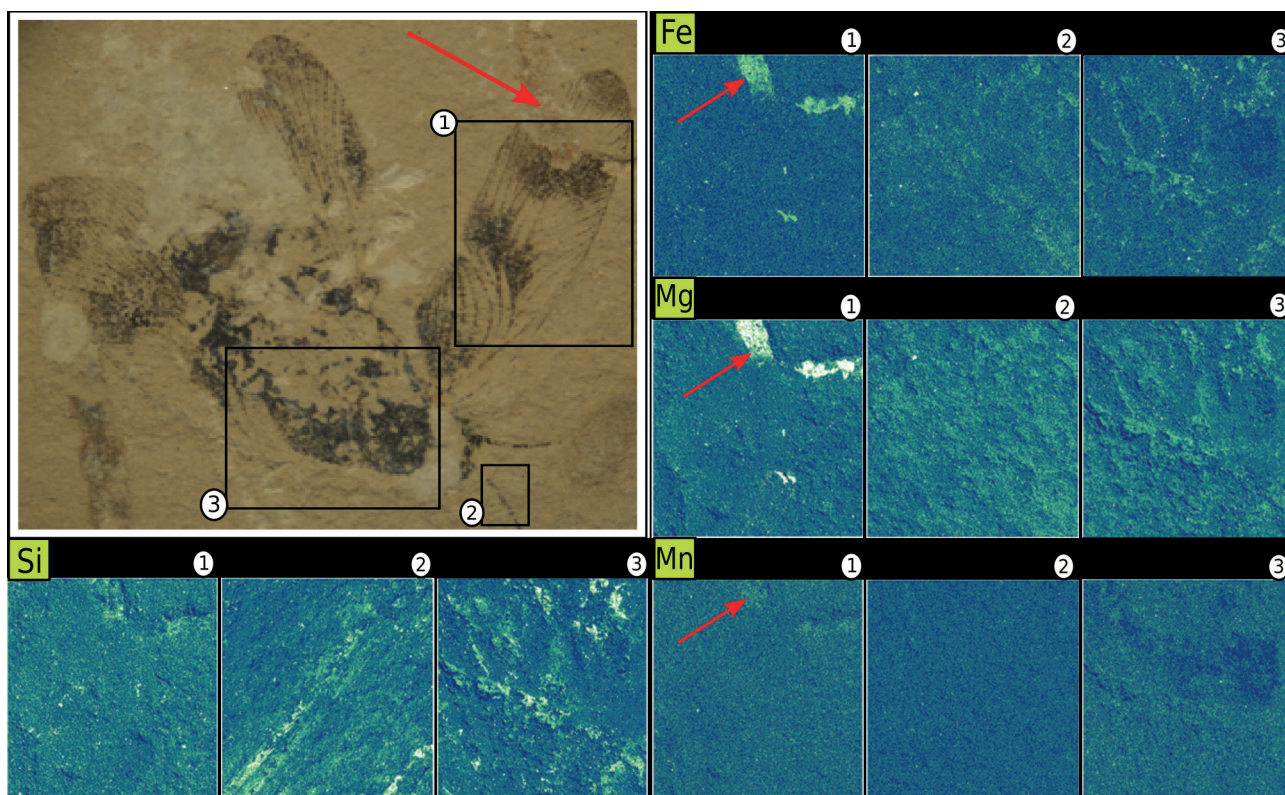


Figure 5. Elemental mapping revealing low inorganic content, Fe, Mg, Mn and Si, in *Araripeblatta dornellesae*. The greatest concentrations occur along a small fracture near the wing body (as indicated by the red arrow). Greenish tones mean presence, whereas blue-dark tones mean absence of element.

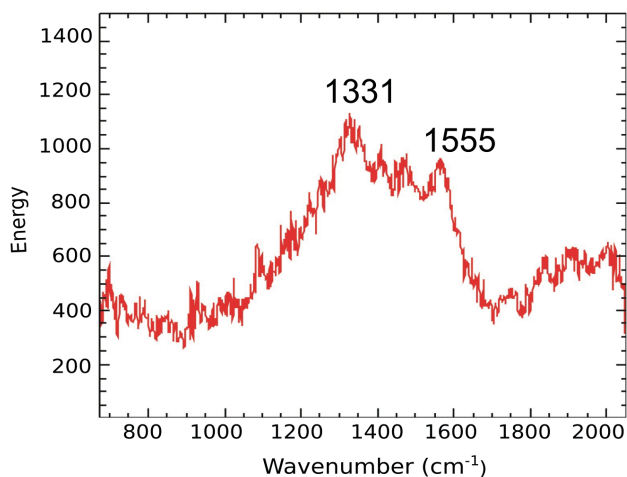


Figure 6. Raman spectrum from LP/UFC CRT 847. The peaks 1331 and 1555 cm^{-1} are associated with amorphous carbon.

the sample contains excess hydrogen molecules, possibly hydroxyls, which hide the carbon bonds. These two broad peaks confirm the carbon presence in amorphous structure. As pointed out these bands are expected to appear in disordered systems and amorphous carbon.

DISCUSSION

According to Neumann & Cabrera (2002), the Crato paleolake had endoreic drainage and was characterized as a meromictic water body containing epilimnion and hypolimnion separated by a pronounced chemocline. Heimhofer & Martill (2007), suggest that the decomposition of organic matter combined with a permanently stagnant hypolimnion could have caused a reduction in the oxygen available turning the bottom of the water column anoxic. The bacterial decay of organic matter under anaerobic conditions is significantly less efficient and slower than aerobic decomposition. The presence of halite pseudomorphs (NaCl) in the carbonates from Crato Formation (Martill *et al.*, 2007) are further evidence of a depositional system in shallow water under arid conditions. A saline condition caused by evaporation may also have contributed to reduction of oxygen, because the solubility of O_2 decreases when salinity increases. Such scenario is has suggested an environment of high salinity and high evaporation rates associated with long dry periods (Martill *et al.*, 2007; Heimhofer *et al.*, 2010; Silva *et al.*, 2013).

These environmental conditions (anoxic and hypersaline water column) associated with a low energy depositional system and gradual carbonate precipitation, created ideal conditions to the preservation of non mineralized tissues

(Martínez-Delclòs *et al.*, 2004). Thus, insects and other organisms deposited on this site were not severely attacked by bacteria being virtually “frozen”.

The Raman spectrum is evidence for the two characteristic peaks already known in the literature as the G and D bands. The D band is originated by resonance processes (being commonly attributed to disorder because of the network disorder). The G peak is due to stretching vibrations of all pairs of sp² atoms (Ferrari & Robertson, 2001). Therefore, the peaks 1331 and 1555 cm⁻¹ (Figure 6) indicate that amorphous carbon is present in the *Araripeblatta dornellesae*. Amorphous carbon was previously observed in a fossil trunk from the Crato Formation (Silva *et al.*, 2013), reinforcing the hypothesis of remaining carbon content. Baas *et al.* (1995) demonstrated that the aliphatic component encountered in fossil insect tissues is not necessarily the result of migration

from an external source. Stankiewicz *et al.* (2000) and Gupta *et al.* (2006) interpreted the presence of aliphatic polymer as a product of the polymerization of cuticular lipids. According to Stankiewicz *et al.* (1997), amorphous character in fossil arthropod cuticles is consistent with polymerization of epicuticular lipids within the cuticle. The CH₄ is the unique hydrocarbon generated during diagenesis process. Thus, our results resemble the vertebrate (fish) pyritization-kerogenization model proposed by Osés *et al.* (2017). In turn, Osés and his colleagues were based on the Ediacaran-Cambrian taphonomic model of Schiffbauer *et al.*, (2014). According to the study of Osés *et al.* (2017), we considered that LP/UFC CRT 847 undergone preservation by kerogenization (Figure 7).

In addition to carbon, sulfur and phosphorus occurrences are also indicative of original organic remains. The sulfur

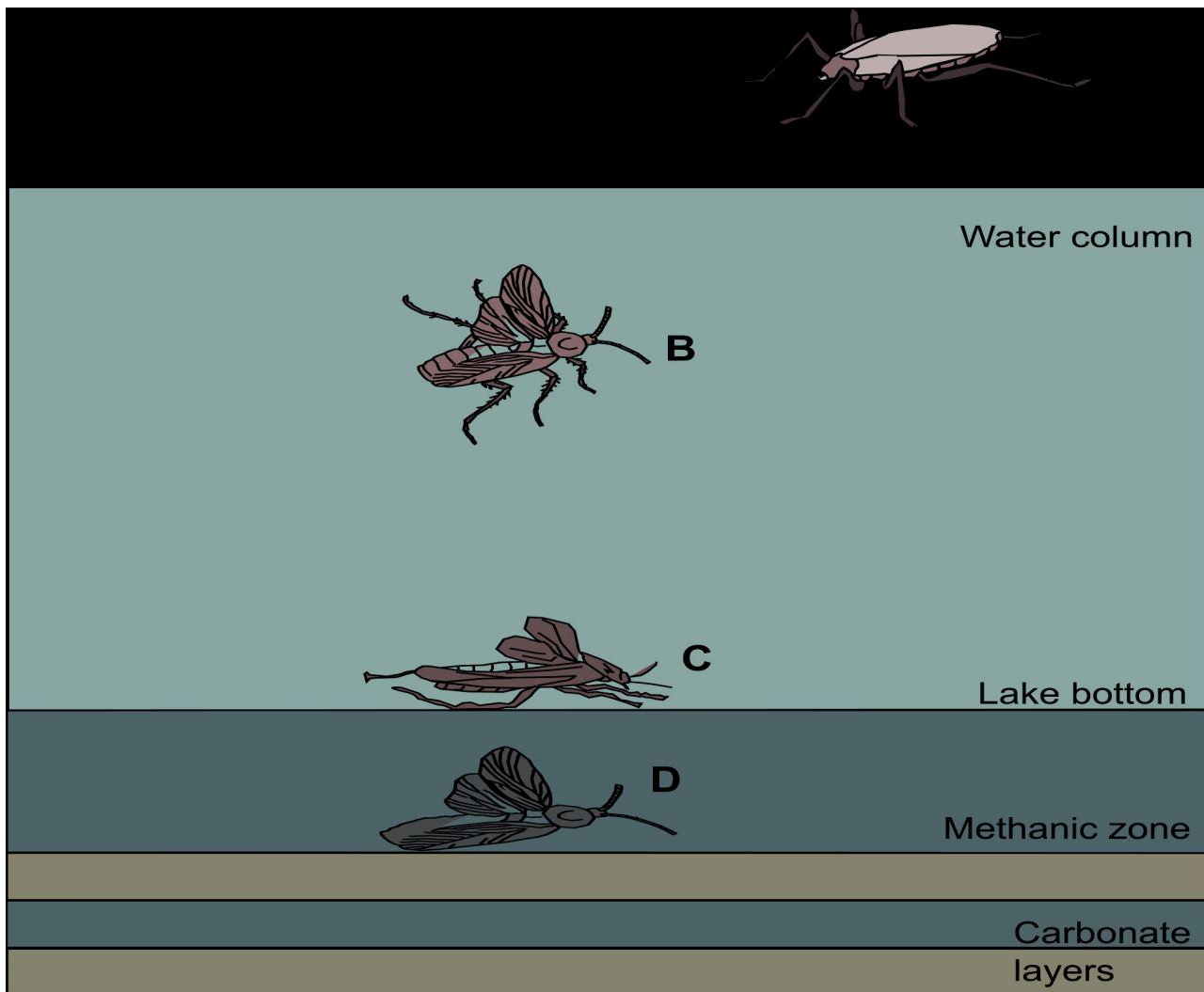


Figure 7. Diagram showing probable LP/UFC CRT 847 taphonomic pathways. **A**, the specimen must have been dragged into the lake by a number of factors such as flooding, lakefront fires, or even exposure to toxic gases. **B**, the LP/UFC CRT 847 reached the lake surface still alive; the wings opened attest this (Mendes, 1998). Cockroaches can spend up to 36 h on the water surface (Martínez-Delclòs *et al.*, 2004). It must have been trapped by microbial mats which living in the water column and pulled down. **C**, at the bottom lake, such a hostile environment caused by oxygen depletion and excessive concentration of salt (it can explain the lack of sediment mixing by benthic organisms) can have prevented the carcass retained the integrity for prolonged periods. **D**, once in the methanic zone, where decomposition is limited to labile tissues (Konhauser, 2007; Schiffbauer *et al.*, 2014) LP/UFC CRT 847 underwent transformation of organic matter in situ within cuticle.

concentration is higher along a discontinuity in the host rock. The porosity in the carbonate matrix is millimeter sized making it difficult to percolation of fluids inside. Thus, the fracture behaves as a microzone of lower pressure facilitating the passage of fluids and the precipitation of ions. The phosphorus concentration is low, but previous studies, such as Bergmann *et al.* (2010), reported a tendency to a higher phosphorus removal in carbonate environments. The co-occurrence of these elements into Blattodea fossil imprint may imply preservation of the original organic components.

This preservation process seems to have occurred through transformation of biopolymers that were transformed into more stable forms during diagenesis (Stankiewicz *et al.*, 1997; Briggs 1999; Wang *et al.*, 2009). Recently, different types of biopolymers have been attributed to different groups of fossil insects. The preservation of organic carbon in LP/UFC CRT 847 implies previous requirements such as rapid burial to avoid disarticulation; the cockroach fossil must have remained for a long period within methanogenic zone, lower sulfate percolation (see Cai *et al.*, 2012, Schiffbauer *et al.*, 2014, Osés *et al.*, 2017). In the methanogenic zone, the decomposition is limited by labile tissues due to inability of decomposing heavy organic molecules (Canfield & Thamdrup, 2009; Schiffbauer *et al.*, 2014). Thus, the carbonaceous preservation is not the product of random repolymerization of organic matter in the sediment, but rather polymerization *in situ*, within the cuticle during mesodiagenetic stage (Stankiewicz *et al.*, 1998).

The elemental maps also indicate the presence of inorganic compounds related to the host matrix. Silicon probably comes from early diagenesis related to the increase of evaporation and/or decreasing of pH, facilitating the precipitation of silica (SiO₂). High environmental CaCO₃ may explain the high concentration of calcium. Fluor and aluminum are also assigned to the matrix. Manganese, iron, and magnesium are considered geochemically mobile elements. In this case, such elements act as indicators of the degree of post-diagenetic alteration undergone by the host rock and, consequently, of the specimen studied.

CONCLUSIONS

The preservation of organic remains in fossil results from a complex interaction among biological and geochemical processes that occur sequentially since the death of the organism. The specimen of the Blattodea species *Araripeblatta dornellesae* analyzed in the present paper displayed the full three tagmata (head, thorax and abdomen) and appendices. The good preservation of the specimen provided important information about the taphonomy of Blattodea in the Crato Formation.

The LF-EDS analysis showed that the presence of amorphous carbon contrasts almost exactly with the distribution of calcium (related to the carbonate matrix). This aspect supports the hypothesis that LP/UFC CRT 847 is preserved into carbonaceous components. Therefore, our results indicate that the vertebrate pyritization-kerogenization

model proposed by Osés *et al.* (2017) can be extended to the insects of the Crato strata. These carbonaceous components potentially provide insights into ancient biology of insects in this unit. Furthermore, the LF elemental maps displayed a series of geochemically derived components that do not correlate with biological structures, in particular, Mg, Fe and Mn, which were interpreted as oxides resulting from post-diagenetic percolation fluids.

Here, we worked with only one specimen. Therefore, it is insufficient to propose a general taphonomic model for kerogenized insects. Finally, this study may be considered as a starting point in the investigation of this different preservational mode of insects in the Crato Formation.

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