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Syngenetic and diagenetic features of evaporite-lutite successions of the Ipubi Formation, Araripe Basin, Santana do Cariri, NE Brazil

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ABSTRACT

The Ipubi Formation in the Araripe Basin (Northeast Brazil) has evaporite-lutite successions rich in gypsum, a mineral of great regional economic relevance, a highlighted stratigraphic mark, and also a natural boundary for underlying successions potentially analogous to "Pre-Salt" hydrocarbon reservoirs of the Brazilian coastal basins. In this study, syngenetic and diagenetic aspects of the Ipubi Formation at Santana do Cariri (Ceará State) were investigated by means of facies analysis, petrography, and mineralogical/chemical analyses of evaporites and shales.

The results show that the contact relationship between evaporites and marly shales, without signs of subaerial exposure and laterally adjacent, was associated with shallow, calm and somewhat anoxic waterbodies, locally salt-supersaturated (brines) but under seasonal variations of water levels. This scenario could have shared place with hydrothermal phenomena in a *playa* lake depositional system. Regarding diagenesis, although there is evidence supporting pseudomorphic replacement of gypsum by anhydrite, the burial of the Ipubi Formation would have been limited due to the frequent occurrence of gypsum without any trace of chemical replacement.

1. Introduction

Evaporites are saline deposits precipitated originally from superficial or nearly superficial ion-saturated waters (brines) due to the effect of evaporation (Collinson and Thompson, 1982; Warren, 2006). On the scope of sedimentary geology, this definition would cover both authoctonous chemical sediments and their diagenetic equivalents in sedimentary rocks (Giannini and Riccomini, 2000). For this reason, some works restrain the term evaporite to chemical deposits superficially-formed by direct action of evaporation of waters under hypersaline conditions (e.g. Krumbein and Sloss, 1958; Pettijohn, 1975; Suguio, 1980; Boggs, 2009). In effect, by its highly reactive nature, few saline deposits older than Neogene would exhibit completely primary textures, i.e. without diagenetic modifications (Warren, 2006). Such modifications commonly include deformation and physical disrupting (halokinesis) by sedimentary overload, chemical solution and replacement, eventually preserving the original crystalline habit (pseudomorphism). In an interpretative viewpoint, the origin of evaporites should always be linked to arid conditions, where the climate evaporation rates overcome precipitation (Garrels and Mackenzie, 1971).

On the context of evaporites, the ones of sulphate composition are mainly formed by gypsum ($CaSO_4 \cdot 2H_2O$). Gypsum is a saline mineral, monoclinic, commonly prismatic, white in colour and streak if pure, of low hardness (2) and relative density (2.32 g/cm^3), and with vitreous to nacarous lustre (Leinz and Campos, 1978). When heated and powdered, gypsum becomes plaster material able to absorb a third of its volume in water, forming a plastic mass that hardens quickly. The most ancient related use of gypsum was 8000 years BC, as drywalls and plasterboards on buildings of the now Syria and Turkey regions (Costa, 2007). Modern applications of gypsum include manufacturing of industrial cement, chalk, soil pH correctives, enamel, acids, fondants, dehydrating, binders, smelting slag and even beer.

The aim of this study is to analyse gypsum-rich, evaporite-lutite successions of the called Ipubi Formation at Nova Olinda (Ceará

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State, Northeast Brazil), in the scope of its sedimentary history regarding syngenetic and diagenetic aspects. In the southern Ceará State, Brazil, the Araripe sedimentary basin presents gypsum intervals of the Ipubi Formation that are today the most important onshore reserve of this quality known in the country (Departamento Nacional de Produção Mineral, 2014a). The gypsum from Araripe Basin has been exploited since the first half of the 20th century, historically counting for more than 90% of the Brazilian national production. Scientifically, the gypsum from Araripe Basin records a seldom situation on that evaporites of that composition outcrops apparently without any sign of strong pseudomorphic replacement during diagenesis. In consequence, and coupled with its wide occurrence on the Araripe Basin, the Ipubi Formation is among the most important stratigraphy markers of the Northeastern Brazil (Assine, 2007; Neumann and Assine, 2015). Moreover, the stratigraphic context of these evaporites stimulate research on potential models for hydrocarbon reservoirs from the so-called “Pre-Salt Layers” of the Brazilian coastal basins, a subject of great economic interest today.

2. Geological setting

2.1. The Araripe Basin

The Araripe Basin is located in the region of confluence between the Brazilian states of Ceará, Pernambuco, Piauí and Paraíba, with an area of about 8000 km^2 (Ponte and Ponte Filho, 1996), of which more than 50% are over the Ceará territory (Fig. 1). The relief is remarked in the landscape by the presence of the *Chapada do Araripe*, a tableland feature E-W long, smoothly inclined westwards and bordered by steep cliffs (Assine, 2007). In the surrounding plains, the sedimentary deposits of the basin are distributed mainly in the east and northeast flanks of the *chapada*, region called Cariri Valley. The maximum estimated thickness of the Araripe Basin is about 1500 m (Ponte et al., 1991).

Structurally, the Araripe Basin lies over Precambrian terrains of the Transversal Fold Zone of Borborema Province, southwards of Patos lineament and northwards of Pernambuco lineament (Brito Neves et al., 2000). These lineaments bound the latitudes of occurrence of the Transversal Fold Zone itself, which correspond to ancient (Brasiliano) dextral shear zones, which in turn would have conditioned both the installation of the basin and its fragmentation in places controlled by faults striking NE and WNW. This fragmentation would have evolved at the agency of events from a Cretaceous taphrogenesis (Wealdenian Reactivation) spreading inland (Almeida et al., 1967), which culminated with the opening of the Brazilian Atlantic margin (Assine, 1990; Matos, 1992; Ponte and Ponte Filho, 1996).

Pioneer gravimetric (isopachs) and magnetometric (structures) assessments (Rand and Manso, 1984; Castro and Branco, 1999) show that the Araripe Basin is divided into several grabens and half-grabens, grouped in two main sets of the sub-basins Feira Nova and Cariri (Matos, 1992). These sub-basins are separated by the Dom Leme Horst (Ponte and Ponte Filho, 1996), a basement high limited by normal faults striking NE in contact with the sedimentary deposits.

2.2. Stratigraphy and sedimentary evolution

The current state of knowledge about the formation and evolution of the Araripe Basin deals with implantation of a rift-type basin: initiated by extensional tectonic subsidence in a narrow (up to 100 km) and elongated (up to 1000 km) depression, with relative margin uplifts, and high-angled normal faults limited in one or both sides (Prosser, 1993). The Araripe Basin is believed to be part of a preterit basinal system that connects it to other rift basins southwards in the states of Bahia and Pernambuco (Assine, 2007; Kuchle et al., 2011). This idea has also been suggested by palaeocurrent studies of several alluvial units of the Araripe Basin which are direction, alignment and age-coincident with the same type units from the southward basins during the “pre-rift, rift and post-rift I” sequences (Assine, 1994, 2007; Kuchle et al., 2011). Similarly, this model reinforces suspicion that the main/master border fault(s) of the Araripe rift basin could correspond to normal faults limiting the Dom Leme High itself (horst), since an axial palaeoflux is expected according to the presently more accepted models for rift basins during the rift initiation phase (Leeder and Gawthorpe, 1987; Prosser, 1993).

The first mentions to the deposits of the Araripe Basin would be from the 19th century (Mabesoone and Tinoco, 1973), where limestones with abundant remains of fish fossils are described. The first stratigraphic subdivision cites “basal conglomeratic arenite”,

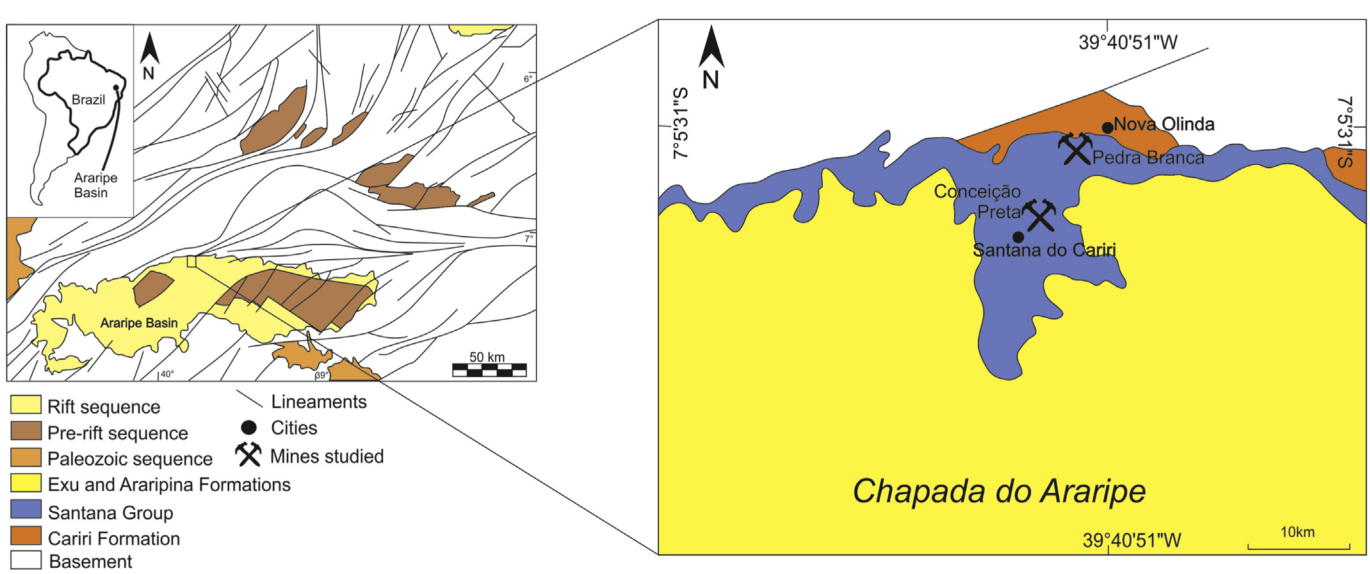

Fig. 1. Location of the Araripe Basin and the mines studied here. Modified from Matos (1992) and Assine (2007).

"lower arenite", "Sant'Anna limestone" and "upper arenite" (Small, 1913). According to Assine (2007), the lithostratigraphic basis of study for the Araripe basin was given by Beurlen (1962, 1963) who pioneered the recognition of fluvial and lacustrine stratigraphic units.

2.3. The Santana Group and the Ipubi Formation

The Santana Group comprehends the "Post-rift I Tectonosedimentary Sequence" of the Araripe basin (Neumann and Assine, 2015) which records an almost complete stratigraphic depositional sequence. This group represents the apex of the thermal subsidence of the basin during the Cretaceous (Neoaptian-Eoalbian), when its rate of sinking would be smaller than in the predecessor phase, but encompassing a broader area. Evidence of this would come from places in the basin where deposits of the Santana Gr. lie directly on the basement, mainly in its west side (Assine, 1994, 2007).

The initial interval of the Post-rift I Sequence would have started by the deposition of two major fluvial, fining upward cycles finalised by terrigenous to marly deposits on the top (Chagas, 2006; Chagas et al. 2007; Assine, 2007), in the Barbalha Fm. The end of the fluvial cycles and subsequent start of the predominance of marly-to-calcareous lacustrine deposits would mark the beginning of the Crato Fm., whose basal contact with the Barbalha Fm. is not well-established due to scarcity of identifiable and/or correlative distems along basin. In part, this fact could occur by a variable palaeogeographic transition of the basin during that time interval where, additionally, the depositional systems would have expanded and altered its prior position due to a wandering depocentre under thermal subsidence. Even in apparently more complete successions (up until dozens of metres), as in Nova Olinda city, the transition between the two units seems to be complicated. In Nova Olinda, there are oucrops of green shales of variable composition (terrigenous to marly), among which appear decimetre-thick tabular layers, alternating fine sandstones and laminated calcilutites by up to 10 m (Santos et al., 2015).

In the Crato Fm., predominates millimetre-thick laminated calcilutites in discontinuous banks of up to 20 m (Assine, 2007), whose interfingered contact with marly green shales is not always observable in the outcrop scale. Abundant fossils occur in the calcareous layers, such as small fishes, insects, crustaceous, arachnids, chelonians, lizards and pterosaurs (Mabesoone and Tinoco, 1973; Assine, 2007). In the shales, the fossils are limited to ostracods. This mentioned faunal association should be evidence of lacustrine depositional systems without marine influence for the Crato Fm. (Neumann, 1999).

Macroscopically, excepting planar laminations of the limestones, there are few primary sedimentary structures in the Crato Fm. In most cases, these structures are centimetre-sized halite pseudomorphs, partially replaced (later) by siliceous cement during the diagenesis (Martill et al., 2007). More often, asymmetrically-folded local layers occur, decimetre thick, sometimes associated with small (centimetre) reverse faults attributed to slumping phenomena (Martill et al., 2008) with lost of fluidity at the end of the flux. Microscopically, the presence of millimetre fenestral structures following the lamination alignment is common, sometimes with pendant mud on the ceilings of cavities as way-up (geopetal) indicators (Santos et al., 2013; Santos and Azevedo, 2014). In some levels, centimetre-sized mushroom shaped carbonate nodules appear (Martill et al., 2008); its composition is similar to the host rock, and lack of deformation in the surrounding laminations suggests an eodiagenetic origin. Regarding secondary sedimentary structures, the major highlights are siliceous concretions, generally elliptic ("eggy"), and centimetre to decimetre sized among the layers of laminated limestone. There, although the siliceous concretions had produced some deformation in the vicinity, they present internal continuity of the lamination through their body, as evidence of diagenetic origin. Extreme, another evidence of silicification is given by entirely replaced layers where the high hardness and low reactivity to hydrochloric acid are easily verified in the field, notwithstanding the apparent preservation of all primary structures of the limestone. The origin for the mentioned diagenetic evidence of silicification (pseudomorphs, concretions, replaced layers) in the Crato Fm. is still controversial, as regarding its time of formation (eodiagenesis, mesodiagenesis) or involving replacement phenomena (conventional diagenesis? *Sensu-strictu* hydrothermalism?) (Martill et al., 2008).

Despite several occurrences in the Cariri Valley, the Ipubi Formation (Santana Gr.) would be more conspicuous in the west side of the Araripe Basin, from Ipubi to Araripina cities (Pernambuco State), the main gypsum-producing region in Brazil. There, the deposits sometimes lie directly on the crystalline basement, newly showing the spatial enlargement of the basin during the thermal subsidence phase. In the Ceará State, north side of the basin, there are several small mining plants for exploitation of gypsum, the largest of them operating informally (Departamento Nacional de Produção Mineral, 2014b; 2014c). Among the large formal minings, however, two are noteworthy in the Ceará State: the ones of the Pedra Branca and Conceição Preta mines, both in Santana do Cariri, where the dismount and production of the gypsum are open-pit mined in countertops.

The Ipubi Formation lays over the Crato Fm. as lensoidal (locally tabular) and discontinuous (both vertically and laterally) deposits of largely gypsitic evaporites, with variable thickness from few decimetres to 30 m, interbedded with green and black shales (Menor et al., 1993). The gypsum in the Ipubi Formation presents primary texture of palisades, internally formed by grouped columnar crystals. The other enterolithic structures would be secondary, represented by alabastrine, selenite ("rose") and nodular textures (Assine, 2007). According to Silva (1988), fibrous varieties of gypsum in the Ipubi Formation would constitute the late saline generation, formed in "diagenetic conditions". Assuming the diagenetic evolution proposed by Silva (1988) and Assine (2007), all the mentioned secondary textures would be premature/shallow (up to few dozens of metres, eodiagenetic), except the fibrous varieties, where the conditions would be late/deep (at least hundreds of metres, mesodiagenetic).

The green and black shales intercalating the Ipubi Formation are eventually clam shrimp (Conchostraca) bearing (Assine, 2007). In the specific case of black shales, regarded as bituminous, some authors assert the microfossil content would include dynoflagellates supposedly marine (e.g. Lima, 1978), bringing an interpretation of oceanic water ingression at that time. For others, however, only non-marine ostracods and carbonised plant remains would occur (beyond the mentioned clam shrimps), under today's more accepted interpretative view for the deposition of the Ipubi Formation (Assine, 2007). The more evoked analogue model for the palaeogeography of the Ipubi Fm. is based on the modern salt pans of Southern Australia (i.e. Menor et al., 1993; Assine, 2007).

The contact between Ipubi (bottom) and Romualdo (top) formations is made through diastems difficult to identify when the evaporites are not present (Assine, 2007) since the Romualdo Fm. is composed mainly by shales. When present, these diastems would be recognised on the top of the evaporites as a thin conglomerate level (as in the Araripina city) or arenites and conglomerates (fining upwards lensoidal bodies) (at Santana do Cariri) (Menor et al., 1993; Assine, 2007). In the latter case, the truncation of the diastem would be direct on the gypsum or indirectly on dark shales associated to them, and would also include extraclasts from the basement (Freire, 2013). Alternatively, when the gypsum is absent, these diastems could sometimes be identified by palaeocaliche surface (Silva, 1986).

3. Materials and methods

Eleven samples were collected from the Ipubi Formation in the Pedra Branca and Conceição Preta mines, Santana do Cariri, Northeast Brazil, of which seven are from evaporitic intervals and the remaining from lutites (Fig. 5). The samples were analysed by polarised and reflected light petrography microscope, X-ray difratometry (XRD) and fluorescence (XRF).

The petrography analysis involved confection and description of thin sections from evaporites of the Pedra Branca (two samples) and Conceição Preta (five samples) mines, aiming to describe textures and mineralisations related to its sedimentary history. The samples were analysed in an Olympus BX41 microscope on the Microscopy Laboratory (Department of Geology, UFC). Evaporite samples were also chemically analysed by X-ray difratometry (XRD) and fluorescence (FRX), seeking to identify small mineralogical fractions and major elements involved in the formation of the rocks. In these last two analyses, the four samples from lutites intervals were also included, and equally divided between the investigated mines. The equipments used to XRD and XRF analyses were an XPert Pro MPD (PANalytical) diffractometer and a ZSX Mini II (Rigaku) sequential X-ray spectrometer, both from the X-Ray Laboratory (Department of Physics, UFC).

4. Results and discussion

4.1. Geometry and depositional facies of the mineralised bodies

The gypsum evaporite in the Pedra Branca mine exhibits a lensoidal shape with more than 12 m in thickness (Fig. 2). Its colour is mainly white (eventually grey), coarse-grained by the presence of saturated selenite and palisade textures, and fibrous gypsum veins associated to pelitic films and laminas. Green shales overlap the gypsum on an incomplete exposition of about 2 m thick, attributed to Romualdo Fm.

The Conceição Preta mine shows the most complete evaporite succession outcropping the Ipubi Formation on the Ceará State, with two metric lenses of gypsum separated by a lutite interval of mixed and variable composition among terrigenous and carbonate, occurring since marly shales to marls (carbonates >35%) (Fig. 2). The gypsum presents variable colours (white, grey, brown), and massive to fibrous textures. The upper gypsum lens is the more white and crystalline, and would be correlated to the one outcropping in the Pedra Branca mine. The marly interval also exhibits variable colours (green, yellow, brown) in thin layers and horizontal laminas.

Based on the description of the outcrops in Pedra Branca and Conceição Preta mines, three depositional facies are described, two of which are pelitic and the remaining, gypsitic (dominantly), with recurrence. The lutite separating gypsitic layers (evaporite) is distinct by its more calcareous composition and low fissility when compared to the shales (S) from the top of the Pedra Branca mine (Menor et al., 1993), and therefore denominated laminated marl facies (Ml). In the studied mines, the layers in the three facies have metric thickness and abrupt contacts with each other. The layers of the evaporite facies (E) are laterally discontinuous (pinch out), where they present interfingered contacts with the marly facies. This fact results on a tabular to lensoidal geometry for both facies, whereas the marly facies have only a tabular geometry. All facies would have been formed under calm and alkaline aqueous environments, eventually supersaturated in salts as shallow brines (Table 1; Fig. 4).

Locally, the horizontal geometry of the three facies can be irregular, presenting smooth symmetrical folds of decametre length (Fig. 3). The origin of this type of ductile deformation could be depositional by overloading on saline and/or muddy layers (halokinesis, lutokinesis), especially if the sediments were soft during burial. In this context, Mabesoone and Tinoco (1973) mentioned occurrence of enterolithic folds among evaporite layers of the Santana Fm., which could be associated with a similar mechanism of deformation (Almeida, 2010). Lastly, evidence of brittle deformation also occurs as high angle normal faults (domino

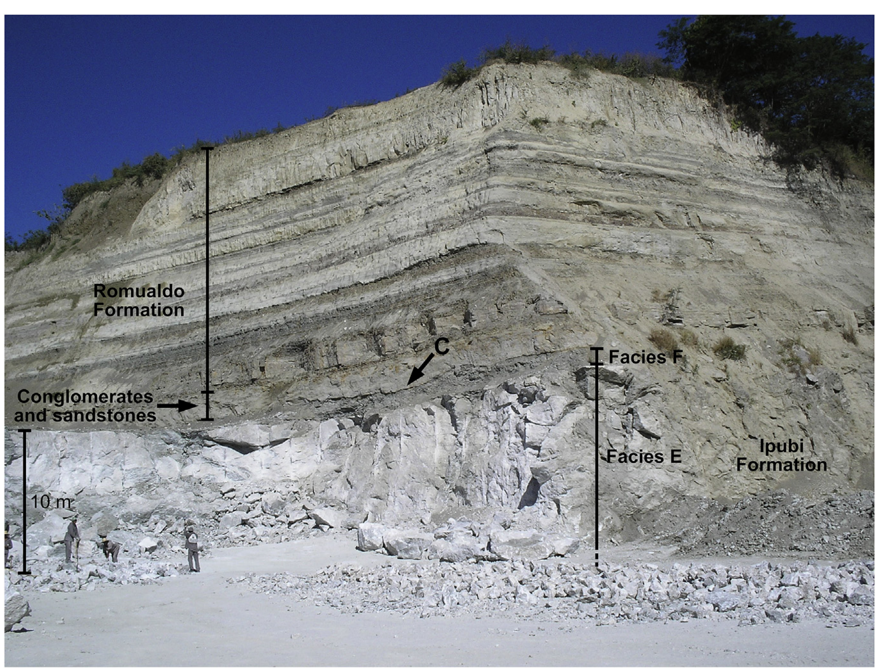

Fig. 2. Aspect of the Pedra Branca mine in Santana do Cariri, Ceará State.

Table 1
Depositional facies of the Ipubi Formation in the Pedra Branca and Conceição Preta mines.

Facies	Description	Interpretation
E	Tabular to lensoidal layers, metric to decametric, of gypsitic to anhydritic evaporite, white to grey colour, coarse-grained crystalline. There is some internal plane-parallel bedding, centimetre-sized, irregular, of mainly vertical (columns) alabastrine crystals, grouped above more massive and impure saline levels.	Chemical precipitation of salts on shallow endorheic brines of arid climate, bottom-nucleated at the waterbody and/or crystallised and sunk from its surface.
S	Metric, tabular layers of green shale, slightly calcareous, with millimetric lamination (fissility) visible to indistinct.	Decantation of slightly calcareous mud under calm and alkaline waters.
Ml	Metric, tabular layers of impure marl, greenish-yellow to brown colour, highly calcareous, with indistinct to visible millimetric lamination.	Decantation of tightly calcareous mud under calm and alkaline waters.

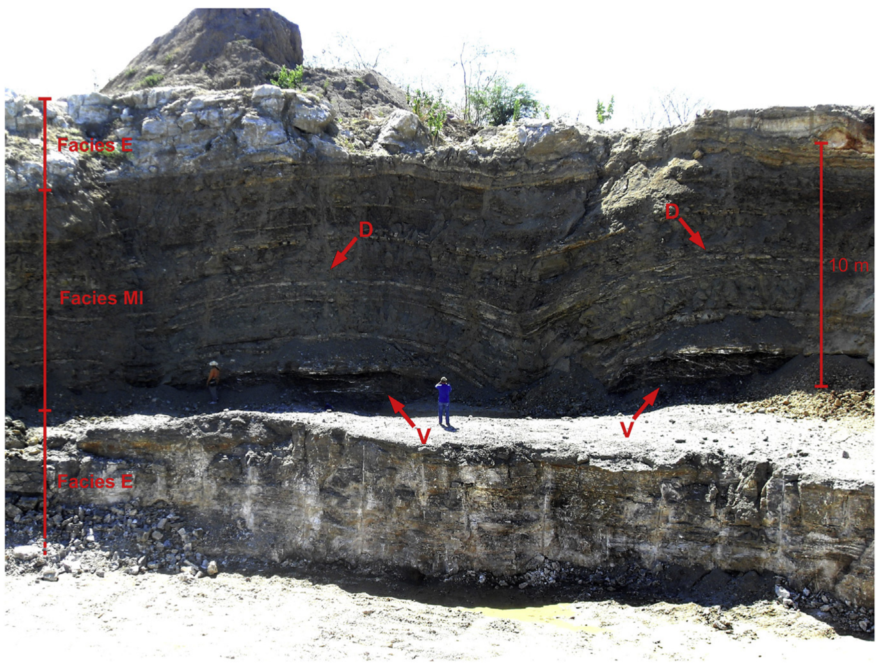

Fig. 3. Detail of the enterolithic folds (D) and veins of satin spar (V) in the Conceição Preta mine.

and listric types) of centimetric displacement, mainly in the contact among gypsum and the superimposed green shales (S). In the Pedra Branca mine, fault-opened spaces are filled by veins of microfibrous gypsum ("satin spar") and by terrigenous lutite (Fig. 3).

4.2. Petrography

Due to its authoctonous nature, the evaporites are essentially made of authigen cement. In the Ipubi Formation, the saline composition is mainly gypsitic (>95%), but also includes salts of anhydrite, sparse crystals of calcite and an opaque, dark cementing substance.

The gypsum exhibits mainly millimetric groupings of fibrous crystals with less than 100 μm each crystal, where the groups, individually, have minerals more or less juxtaposed and/or aligned to the same point of saline growth or nucleation. In each group, the crystals irradiate from different nucleation points, with mineral growth directions slightly variable (at angles of up to 45° in the thin sections) (Figs. 5C and 6B). As a result, different groups of fibrous crystals have intertruncate endings, generating a complex entanglement. Major crystals of gypsum (columns; palisades macroscopically) occur as more organised groupings, chiefly with syntaxial growth along levels perpendicular to the rock bedding (visible to the naked eye) (Fig. 5A). The majority of the contacts are point or long type, but there is occurrence of concave-convex type denoting a greater degree of compactation in some portions of the evaporite.

Some ungrouped portions of fibrous crystals of gypsum can occur, among which there are crystals even larger (up to 200 μm) and disorganised, but presenting a typical "lath shaped" enterolithic structure (Hallsworth and Knox, 2003) (Fig. 6D). In the same places, euhedral spatic crystals also occur, isolate among the fibrous gypsum. Additionally, more equidimensional relicts of gypsum appear non fibrous and greater than 100 μm, in open/porous pockets without any apparent organisation (Figs. 6C and 7C). Even amid the ungrouped portions of the gypsum, there are sparse calcite crystals of up to 50 μm, sometimes euhedral.

Zones of low crystalline gypsum appear in places more or less aligned to the bedding, in portions where brown opaque cement arises among the matrix, and denotes a major impurity (Figs. 5A, B, 6A, 7). Such portions are distributed in bundles several millimetres thick, with different crystallinity grades and brownish shades. Dissolution features occur either among within or at the contact with the brownish bundles, giving secondary porosity of the not fabric selective "channel" type (Choquette and Pray, 1970) (Fig. 5A). In other places, the same brown cement emerges as discontinuous bundles separating groups of gypsum with differentiated fabric (mainly columnar and fibrous).

Strongly birefringent anhydrite crystals ($CaSO_4$) arise in some places of the evaporite, usually as all-directions, fibrous-radiate

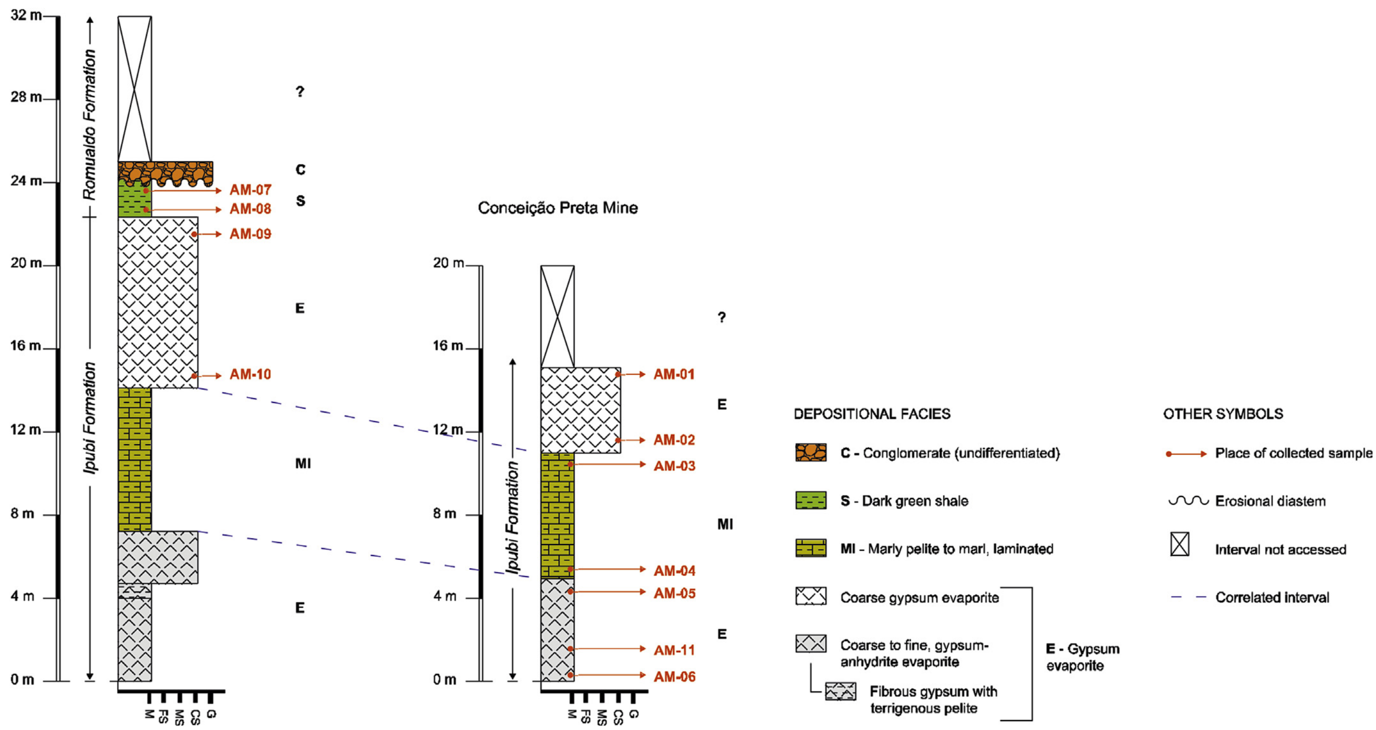

Fig. 4. Stratigraphy and depositional facies of the Pedra Branca and Conceição Preta mines (reinterpreted from Freire, 2013).

groupings of displacive growth ("chicken wire"), and up to 500 μm in diameter (Fig. 7). Occasionally, the anhydrite occur as groups of more or less euhedral spatic portions, and with diameters similar to the "chicken wired" groupings. There is an apparent proximity relationship between the fibrous-radiated anhydrite crystals and the brownish zones (or more rarely, in the midst of them), including disruption of this last one, whereas spatic crystals appear mainly isolated among the fibrous-gypsum matrix (Fig. 5C, D, 6C, 7).

An opaque cementing material, black-coloured and low crystallinity, occurs mainly scattered among the irradiated crystals of gypsum, sometimes as more concentrated bands; in many cases, it follows the mineral orientation of the different bands and groupings of gypsum (Fig. 5C, D, 6B). More rarely, this same cement subdivides the crystals of gypsum in millimetre-sized blocks, where it appears with irregular contours, sometimes staggered. In the latter case, the black cement fills predominantly horizontal pores, parallel to the rock bedding (Fig. 6A). These blocks subdivided by the black cement occur both with the same poor-crystallinity of the cement and in brown-coloured bands (terrigenous impurities?) of slightly major crystallinity. Under reflected light, the black cement is frosted and brightless, suggesting the presence of organic matter (perhaps diagenetically modified) rather than sulphides or oxides. In this context, Silva (1988) mentioned occurrence of algae tissue remains and pellets among the layers of the Ipubi Formation, especially in more impure terrigenous zones of the gypsum.

The result of petrography analysis for the evaporites of the Ipubi Formation suggests a more complex depositional history than that of other lithologies of the Santana Gr., here evoked in five phases. The first phase, syndepositional, would start with the nucleation and crystalline growth of gypsum (along its crystallographic C-axis) in a shallow (less than 10 m deep), hypersaline (brine) waterbody (Fig. 8). Initially, with enough available space, the crystalline growth would be randomly oriented both in the surface of the waterbody and upon its bottom roughnesses (Adams et al., 1992). On the water surface, at first, the crystals are balanced by the effect of surface tension, until they reach weight and/or dimension, becoming unbalanced and finally they begin to sink (i.e. Collinson and Thompson, 1982). At the bottom, sunken crystals ("laths") are randomly distributed but slightly layered, becoming fresh irregularities that allow the nucleation of new crystals. The eventual excess of crystals at the bottom level limits its lateral growth, so that only vertically-oriented nucleations (palisades, columnar gypsum) stand out from the bottom (Schreiber and Schreiber, 1977; Collinson and Thompson, 1982; Silva, 1988).

The second phase is penecontemporaneous, related to brief cycles of hydric supply greater than the loss of water by evaporation in the waterbody (Fig. 8). As a response, the water level rises and becomes unsaturated in Ca^{+2} and $SO4^{-2}$ ions, suspending the production of new crystals and also partially dissolving the ones (columnar crystals) located on the bed surface. This generates a low-crystallinity level in the evaporite, accompanied by a mixture with terrigenous (muddy) sediment whose transport is favoured by the aforementioned augment of the hydric supply (Collinson and Thompson, 1982; Silva, 1988). Parallely, the supply of organic matter associated to the mud also increases with the terrigenous influx on the flooded basin.

The third phase is eodiagenetic, where chemical transformations in the some-buried (few metres) evaporites would occur. Despite the little sedimentary load, the soft salts suffer compactation and enterolithic phenoms of solution and recrystallisation, where there is loss of water, and nucleation and growth of dehydrated sulphates of fibrous-radiated anhydrite (macroscopic nodules). However, when among zones of terrigenous impurity, the intraestratal solution of gypsum eventually formed mud-supported cavities that allowed entry of salt-saturated water with enough space for nucleation and growth of alabastrine gypsum. This last argument is based on occurrence of alabastrine gypsum in the muddy zone, adjoining the contact with the purer zone, where an extension of gypsum crystal joins a fibrous-radiated anhydrite (Fig. 7C). Finally, displacive growth of fibrous-radiated anhydrite

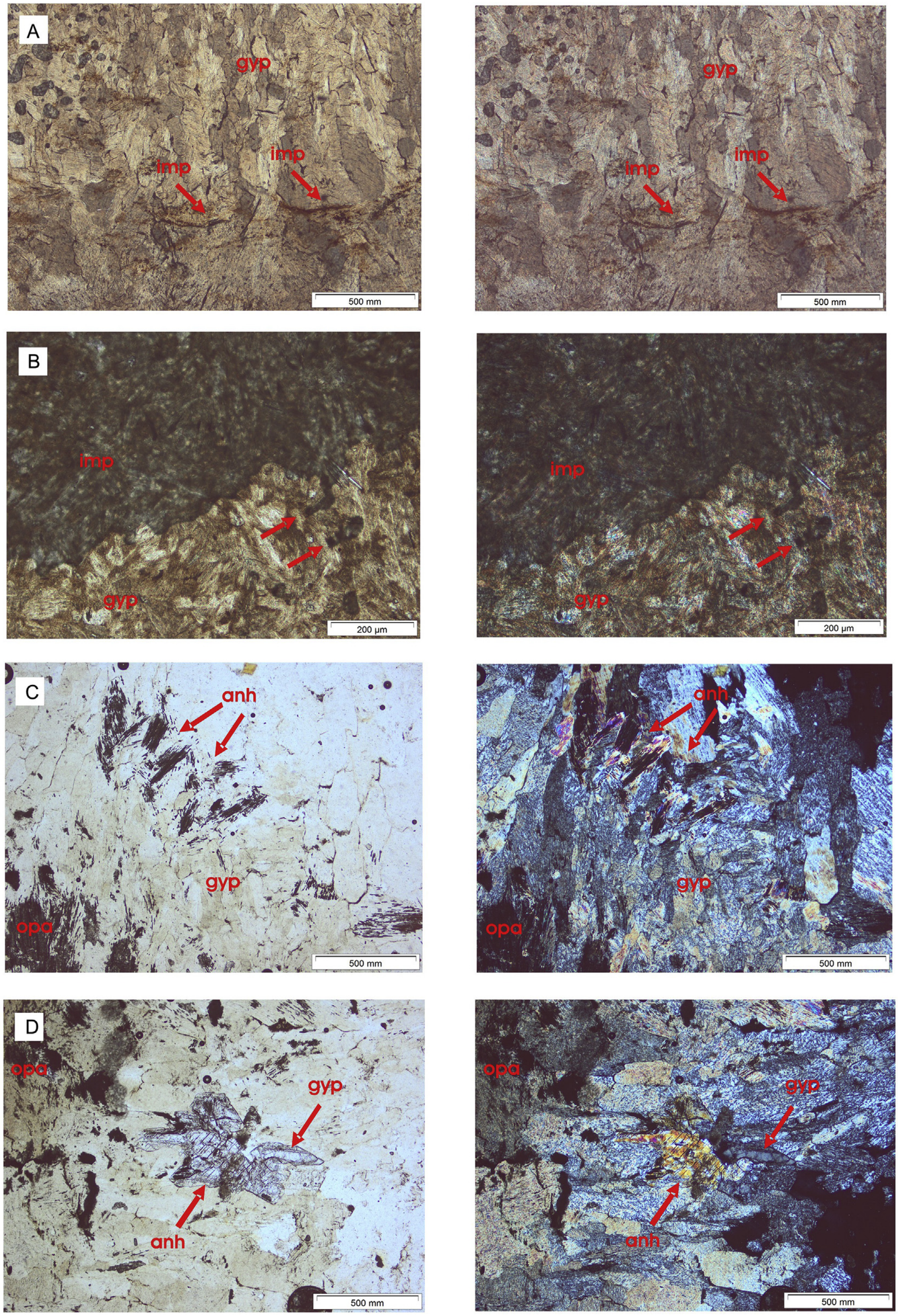

Fig. 5. Photomicrography plates under polarised light of the samples AM-01 (A), AM-02 (B) and AM-05 (C and D). Symbols – gyp: gypsum; anh: anhydrite; imp: impure sulphate (with terrigenous and/or organic matter); opa: opaque cement. Parallel polarisers to the left, crossed polarisers to the right. See text for explanation.

would correspond to nodules developed by reactions between the host sediment and intrastratal (brine) waters at the advanced eodiagenesis.

The fourth phase, mesodiagenetic, would correspond to transformations occurred at greater depths, at least dozens to hundreds of metres. In the Ipubi Formation, evidence of mesodiagenesis is due to the pervasive presence of fibrous anhydrite formed at the expense of gypsum, yet maintaining the original crystalline shape of gypsum. This same occurrence was mentioned by Silva (1988) regarding the Ipubi Formation, who called it "sparse laths of

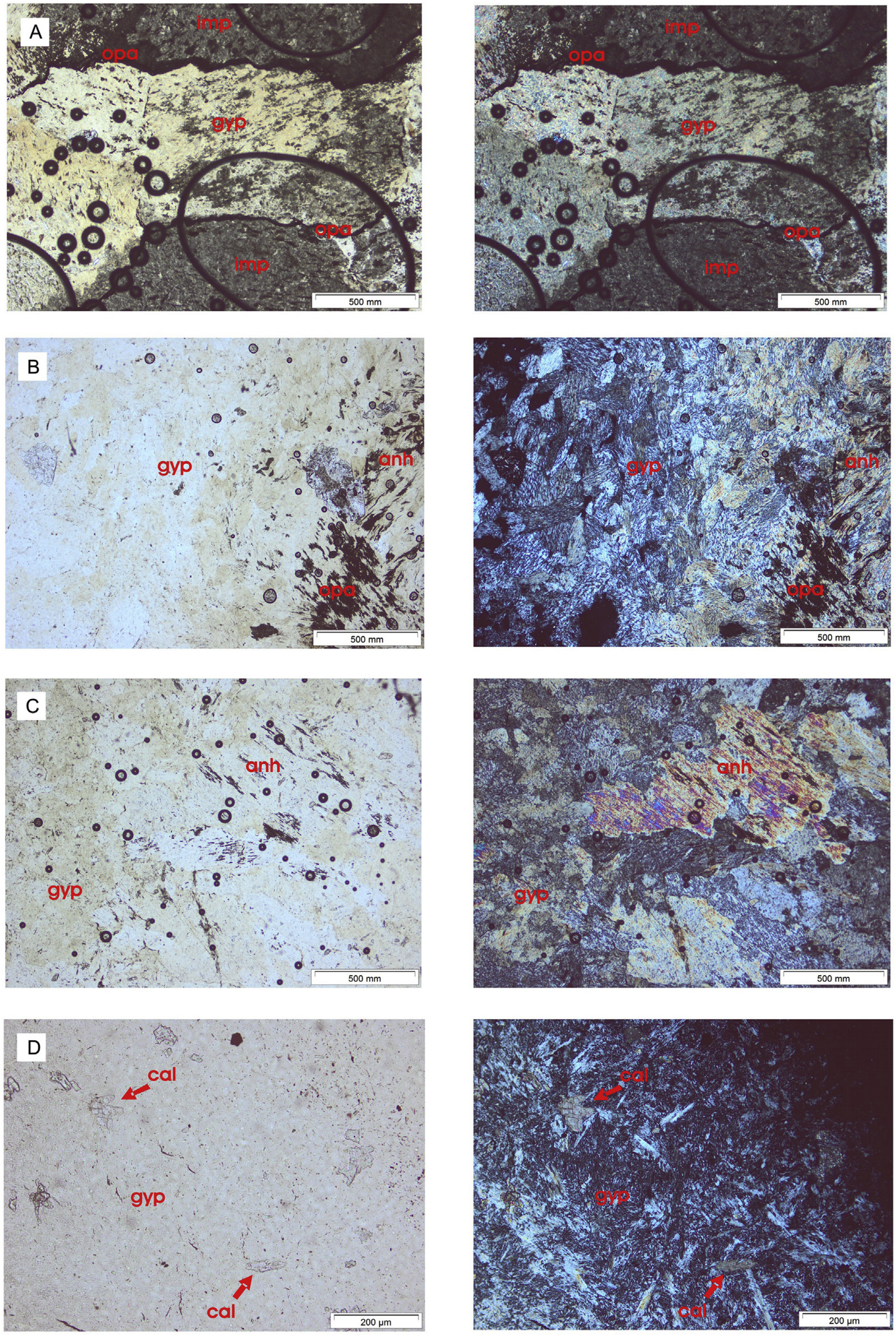

Fig. 6. Photomicrography plates under polarised light of the samples AM-06 (A and B), AM-09 (C) and AM-10 (D). Symbols – gyp: gypsum; anh: anhydrite; imp: impure sulphate (with terrigenous and/or organic matter); opa: opaque cement; cal: calcite. Parallel polarisers to the left, crossed polarisers to the right. See text for explanation.

anhydrite" among the gypsum. According to the author, the formation of fibrous anhydrite in the Ipubi Formation was incipient because the burial would have reached a mere 350–380 m, under pressures that still allow existence of primary (depositional) gypsum, with no sign of transformation. Murray (1964) and Silva (1988) affirm that would be necessary at least 900 m of burial to transform the whole primary gypsum into anhydrite.

The fifth phase would be telodiagenetic, with return of the more

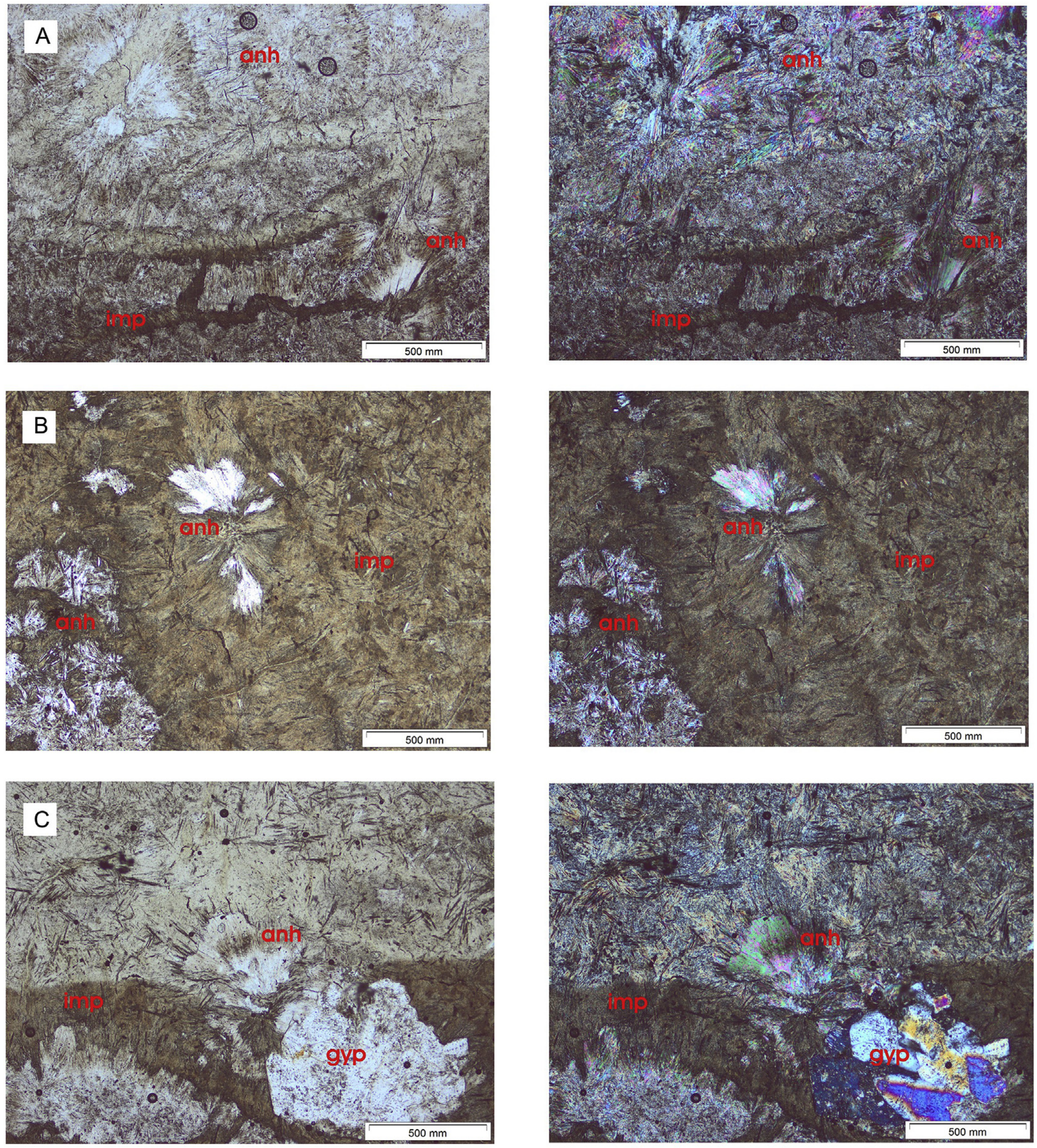

Fig. 7. Photomicrography plates under polarised light of the sample AM-11. Symbols – gyp: gypsum; anh: anhydrite; imp: impure sulphate (with terrigenous and/or organic matter); opa: opaque cement. Parallel polarisers to the left, crossed polarisers to the right. See text for explanation.

favourable conditions to the stability of the hydrated saline forms (gypsification) in the evaporite, thanks to the exhumation and/or nearly exhumation (backstripping) of the evaporites after the previous phase. As the falling pressure proceeds, some anhydrite crystals destabilises and form gypsum once more, now with enough intrastratal space to grow in an almost-drusy way (alabastrine gypsum). According to Silva (1988), the macroscopy-telodiagenetic equivalent of the alabastrine gypsum would be the "porphyroblasts" and "rosette" forms of gypsum. The telodiagenetic transformation of anhydrite back to gypsum still releases excessive calcium sulphate, that goes into solution but hydrates quickly and fills any available space as microfibrous gypsum – the macroscopically-called "satin spar" (Holliday, 1970; Silva, 1988). Apart from these facts, other syndepositional forms, as palisades, can present evidence of telodiagenetic recrystallisation formed by a high internal fibrousness.

In this study, sparse calcite crystals were observed among the disorganised gypsum palisades, but they could not be securely placed in the depositional history of the evaporites. A probable palaeogeographic association of the evaporites with the laminated limestones in the Crato Fm. is highlighted here, including a possible intrabasinal transport of fragments between these two environments. Within the context of the Ipubi Formation, Silva (1988) mentioned the occurrence of "dolomite lumps in connection with algae filaments" (in the impure zone of the evaporite) and "millimetre rhythmic anhydrite-micrite couplets", none observed in the present study.

4.3. Mineralogical and chemical analyses

Altogether, the result of the XRD analyses performed in the evaporitic intervals of the Ipubi Formation are in agreement with the petrography analysis, where it shows a predominance of gypsum followed by an important content of anhydrite in the basal strata of the Conceição Preta Mine. There, the content of anhydrite reaches 33% in the lower half of the basal strata exposed at the

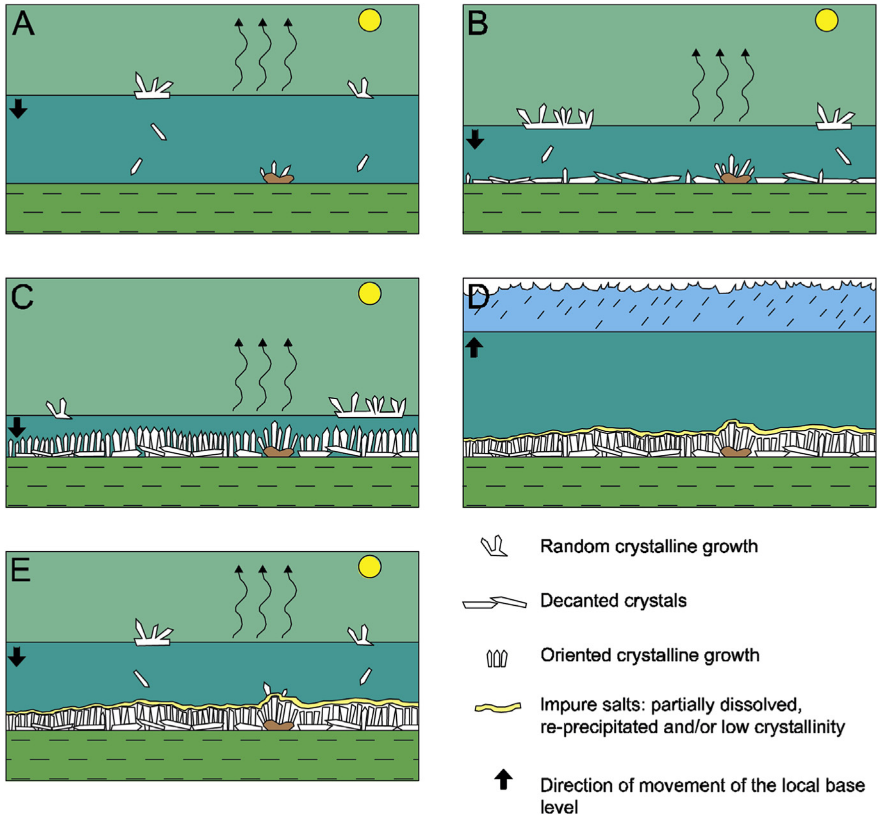

Fig. 8. Syngenetic (syndepositional and penecontemporaneous) cyclic model for the deposition of evaporites on the Ipubi Formation (not for scale). See text for explanation.

mine. Additionally, the XRD points to the presence of celestite ($SrSO_4$), mineral unidentified by petrography, at levels up to 7% in the same place.

The lutite layers were also investigated by XRD, which identified quartz, orthoclase, calcite and goethite, along with the clay minerals nontronite and saponite. In sediments and sedimentary rocks, nontronite and saponite are clay minerals considered to be proxies for the action of hydrothermalism in *sensu strictu*: superficial or nearly-superficial hot waters derived from acid magmatism or heated by igneous intrusions or leakages (Bischoff, 1972; Keeling et al., 2000; Warren, 2006; Anthony et al., 2013). In particular, nontronite is associate with hydrothermal fluids enriched in dissolved iron and silica (Bischoff, 1972), substances whose origin in the studied lutites could be related to the same source of goethite and quartz, both found in the same sample. The fact that nontronite and saponite occur both in the marly intervals and in the shale reinforces this idea, and by extension, the hypothesis of a hydrothermal origin for the pervasive silicification found in the laminated limestones of the Crato Fm. (Martill et al., 2008).

Regarding XFR, the results presented are in agreement with the mineralogy identified by XRD (Tables 2 and 3). The main quantified oxides of the major elements (>1%, abundance order) were CaO, SO_3 and SrO, in the saline intervals, and SiO_2, CaO, Al_2O_3, Fe_2O_3, MgO and K_2O, in the lutite intervals. As seen in XRD, the high

Table 2
Percentage of major elements in the samples from evaporite intervals of the Ipubi Formation. Mine symbols: PB (Pedra Branca) and CP (Conceição Preta).

Sample	Mine	CaO	SO_3	SrO	Fe_2O_3	SiO_2	Cl_2O	K_2O	Al_2O_3	P_2O_5
AM-09	PB	72.86	26.14	–	0.27	0.31	–	–	0.18	–
AM-10	PB	73.53	25.73	0.19	0.21	0.17	–	–	0.15	–
AM-01	CP	71.76	27.19	0.5	0.54	–	–	–	–	–
AM-02	CP	72.2	26.49	0.49	–	0.3	–	–	–	–
AM-05	CP	72.53	26.89	0.27	0.18	–	–	–	–	0.11
AM-06	CP	57.57	27.38	14.44	0.19	0.36	0.02	–	–	–
AM-11	CP	75.01	27.39	0.31	–	–	0.03	0.08	0.15	–

Table 3
Percentage of major elements in the samples from lutite intervals of the Ipubi Formation. Mine symbols: PB (Pedra Branca) and CP (Conceição Preta).

Sample	Mine	SiO_2	CaO	Al_2O_3	Fe_2O_3	MgO	K_2O	TiO_2	SO_3	P_2O_5	MnO	Cl_2O	Na_2O	ZnO
AM-07	PB	23.77	10.44	7.04	5.81	2.89	1.72	0.79	0.48	0.44	0.59	0.01	–	–
AM-08	PB	53.0	3.32	18.99	10.42	5.95	4.36	1.31	0.37	0.38	0.45	13.03	0.14	–
AM-03	CP	42.35	10.30	15.25	11.63	9.55	3.65	1.21	5.19	0.32	0.22	–	0.29	0.04
AM-04	CP	40.81	28.38	15.12	7.46	3.24	2.88	1.12	0.30	0.51	0.17	–	–	–

content in strontium is related to places rich in celestite at the basal saline strata. In turn, high contents in alumina and ferric iron in the lutite intervals are due to the presence of orthoclase and clay minerals (Al_2O_3) as well as goethite and nontronite (Fe_2O_3).

5. Genetic model

Several observations of calcium sulphate evaporites in ancient deposits and recent depositional systems led Warren (2006) to formulate a general syngenetic model for these types of deposits. In this model, the typical syndepositional features of gypsum from shallow brines (<10 m deep) are crystals bottom-nucleated and/or reworked (sometimes including the presence of ripple marks). In deep water (>10 m), it was observed in recent systems that there was an intense stratification of waters in function of the salinity, where the denser and deeper haloclines support the less dense and shallower ones. This avoids a break in the water stratification even in seasonal periods of rainfalls and/or hydric incoming of fresh waters. The extremely saline and dense waterbody bottom blockades haline gradients that could generate an *in situ* growth, which is why primary evaporites from deep waters are scarce in nucleated crystals, being almost always formed by accumulation of salts passively sunk from the shallower haloclines. Despite this, a reworking of saline crystals eventually would be possible by episodic processes of transport in the basin (i.e. turbidity currents). Regarding penecontemporaneous features, Warren (2006) mentions nodules disrupting layers (haloturbation) and microcavities (composed either by gypsum or anhydrite), both in evaporitic mudflats exhumed by a falling local base level.

As seen, the findings in the Ipubi Formation – nucleated and/or sparse crystals of gypsum, without significant evidence of dissolution – corroborates for a generation in shallow brines (<10 m deep), with little or no reworking of the decanted gypsum crystals. The apparent absence of structures indicative of subaerial exposition, both in the evaporites and shales of the Ipubi Fm. also suggests the idea of a perennial saline waterbody, although shallow and subject to water (and saline) levels variations. Evaporites are considered to be unequivocal evidence of aridity conditions for the time interval of its depositional systems. However, their mere presence as genuine salts, substitutions or pseudomorphs, does not necessarily mean that there has been subaerial exposition, but only that a hypersaline waterbody once existed (Collinson and Thompson, 1982). Other structures should be investigated, none of which have been so far described for the Ipubi Fm. within the localities of the present study.

A succession relationship between the limestones and evaporites of the Santana Gr. would be possible in the evolutionary context of the units. In this case, the limestones would correspond to “evaporitic carbonates” (cf. Warren, 2006), typically formed in the initial stages of the brine concentration. Nevertheless, this idealised succession is based on the ‘Usiglio sequence of precipitation’ of the recent oceanic waters, whose chemical proportion between the different dissolved ions is more or less the same around the world. In non-marine brines for example, the ionic concentration is strongly dependent on the original chemistry brought from the source areas to the endorheic basin. Even in situations known to be marine, the possibility of ionic variation in the global oceans through time should be considered (Pettijohn, 1975; White, 2001; Warren, 2006).

The major complexity involved in the formation of Ipubi Fm.s’ evaporites is also patent by the discontinuous stratigraphic relationship of the evaporites with the “impure” lutite intervals (marly shales and marls). This fact has led the majority of the authors to propose the palaeogeographic context of the Ipubi Fm.s’ evaporites as naturally isolated salines, in area under anoxia but only locally

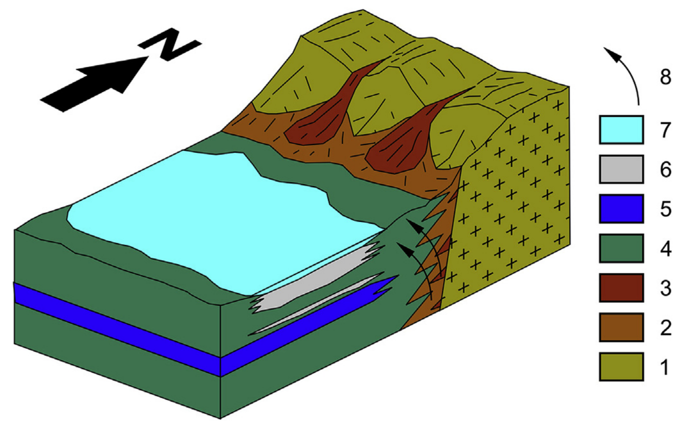

Fig. 9. Block diagram of the palaeogeographic model for the Ipubi Formation (Santana Gr.) during the Eocretaceous. Legend: 1-basement horst; 2-sandstones from distal alluvial fans; 3-conglomerates from proximal alluvial fans; 4-calcareous shales and marls from shallow and marginal lakes; 5-laminated limestones; 6-evaporites; 7-shallow alkaline (endorheic) lake; 8-hydrothermal fluids migrating from the border fault.

saturated as brine (Silva, 1988; Menor et al., 1993; Assine, 2007), in depositional systems of *playa*, *playa* lake and/or *sabkha* (Silva, 1988). In the modern conception of arid depositional systems, these last three terms are separated according to its yearly ratio of evaporation/precipitation and presence/absence of marine connection, so that the evaporitic intervals of the Ipubi Fm. would have been in a *playa* lake system: under fluctuations of the waterbody level but with no marine connections (Briere, 2000) (Fig. 9). The more evoked modern analogues for the evaporite successions of the Ipubi Fm. are the salines from Southern Australia (i.e. Menor et al., 1993; Assine, 2007). Taking into account the possibility of hydrothermalism contemporaneously to the deposition of that formation (Martill et al., 2008 and the present work), other models as the shallow hydrothermal brines from the Danakil Depression (Afar, West Africa Rift Valley) could be likewise considered. A possible source for the hydrothermalism in the studied area is the Patos Lineament, located few kilometres northwards. This bounding structure has been reactivated along its history in the Transversal Zone (Almeida et al., 1967; Corsini et al., 1998), and moreover could serve as a conduit for heated fluids.

Several works in the Santana Gr. regard the vertical appearing of the layers of gypsum as being the end of the downright carbonate successions in this stratigraphic unit. On an “almost complete” depositional sequence scale for the Santana Gr. as a whole (Assine, 2007), it would be indicative of increasing in the salinity, although probably related to the beginning of a transgressive period of the base level, eustatic, and culminating with a marine ingression on the topmost Romualdo Fm. This view does not discard the possibility of high frequency variations in the local base level, since there is occurrence of vertically alternate layers of limestone and sandstone, and of evaporite and lutite (Neumann, 1999), as seen for example in a continuous section obtained in Nova Olinda region (Santos et al., 2015).

6. Conclusions

The study of evaporite-lutite successions of the Ipubi Formation allowed the identification of three depositional facies (E, S, Ml) related to variable aridity events in a lacustrine system during the Eocretaceous. Such climate fluctuations would also be triggered in high frequency events, as attested in less crystalline, impurer laminas inside the gypsum intervals. The absence of structures linked to subaerial exposition suggests that the evaporite-forming waterbody was perennial, even if subject to variations on its levels. On the other hand, the lateral and vertical contacts of the evaporite with dark coloured lutites also presuppose calm environments under reducing Eh conditions, despite its limited depth.

Eodiagenetic transformations are attested mainly by enterolithic phenoms of dissolution (of gypsum) and recrystallisation (in anhydrite form) of calcium sulphate, as seen principally by petrography. Deeper transformations (mesodiagenetic) are given by gypsum pseudomorphs in fibrous anhydrite; however, the frequent occurrence of gypsum without any sign of transformation suggests that this phase was not markedly widespread, and also that the burial of the Ipubi Formation must not have been too deep (a few hundred metres at most).

The mineralogy and chemism of the studied successions reinforce the impure behaviour (marly) of the lutite intervals; there, the presence of nontronite and saponite raises suspicion of hydrothermal activity in a *sensu strictu* (contemporaneous) during the deposition of the Ipubi Formation, in a *playa* lake depositional system.

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