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Pilot Study on Fingerprinting the Isotopic Signatures of Fossiliferous Limestones as a Tool for Forensic Investigation of Fossil Trafficking (Cretaceous Crato Formation, Brazil)

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Abstract

Unauthorized fossil trafficking violates national legislation and deprives cultural and natural heritage. This study proposes a pilot method to fingerprint the origin of fossils by characterizing the carbon and oxygen stable isotope signatures of fossil-bearing limestones to provide a non-destructive quantitative tool against illegal fossil trade. This promising approach has been applied to the Crato Formation (NE Brazil), which is a renowned Lower Cretaceous fossiliferous lacustrine limestone. This study aims at establishing the range of isotopic oxygen ($\delta^{18}O$) and carbon ($\delta^{13}C$) values of the Crato Formation's laminated calci-mudstone by compiling isotopic data from previous studies, conducting new analyses, and comparing with other fossiliferous lacustrine limestones from Brazil (Cretaceous Codó Formation) and the USA (Eocene Green River Formation). This preliminary evaluation determined a distinctive isotopic signature of the Crato Formation fossil-bearing "sete cortes" ("seven cuts") ethnostratum, with VPDB $\delta^{18}O$ and $\delta^{13}C$ arithmetic means of −5.94‰ and +0.90‰, standard deviations 0.76‰ and 0.61‰, medians of −5.89‰ and +0.73‰, and interquartile ranges of 1.47‰ and 1.24‰, respectively. This pilot investigation establishes a methodological groundwork for the development of a global database integrating lithofacies and geochemical parameters of fossil-bearing limestones to expedite the identification and restitution of illegally extracted paleontological heritage.

Keywords: fossil lagerstätte; fossil trafficking; limestone; stable oxygen and carbon isotopes; Crato Formation; Brazil

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1. Introduction

Illegal trading of geological material (e.g., minerals, metals, and fossils) is an increasing global crime, and expectantly, the role played by forensic geologists in disrupting this criminal activity will also increase [1,2]. The legal fossil trade is a worldwide phenomenon with high supply and demand from public research centers, educational institutions, and private collectors [2]. Despite active legislation in most countries, it might be difficult to control illegal fossil smuggling in remote areas, with the resulting loss of invaluable cultural heritage and scientific paleontological material [2]. Geosciences have proven to provide numerous useful and effective approaches for forensic investigation of crimes [3–9], integrating various disciplines and analytical techniques, traditionally serving to decipher Earth processes and the interactions among the lithosphere, atmosphere, hydrosphere, and biosphere, such as sedimentology, pedology, petrography, paleontology, geochemistry, and geophysics [4,8–18]. Specifically, geochemistry has been applied in forensic investigation both for tracing and dating records of crime evidence [19–23]. This preliminary study proposes to fingerprint the origin of smuggled fossils using a non-destructive geochemical technique based on the stable oxygen and carbon isotope and trace element concentration signatures of the limestone rocks containing the targeted fossils. Analyses of stable carbon and oxygen isotope ratios of marine and terrestrial carbonate rocks, sediment, and fossil shells are widely used approaches in sedimentary geology and paleontology for paleoenvironmental, paleo-climatic, paleo-oceanographic and diagenetic reconstructions [24,25], but they are increasingly used in forensic investigations [26], geographical provenance identification, and the adulteration of food and beverage products [27–30]. The identification of the oxygen and carbon isotope signature of limestones embedding valuable fossils, subjected to illegal trafficking, has been applied for the first time, to our knowledge, in this pilot investigation to the fossil-bearing rock slabs belonging to the Crato Formation extracted from active quarries in northeastern Brazil (Figure 1).

The Crato Formation represents a remarkable stratigraphic unit housing one of the world's paramount geological and paleontological sites from the Cretaceous Period. The Crato Formation crops out in the Araripe Basin, which is located in NE Brazil (Figure 1), bordering the states of Ceará, Pernambuco, and Piauí. The Crato Formation consists of a mixed carbonate–siliciclastic succession, featuring intercalated beds of finely laminated limestones (Figure 2). Deposited during the Aptian age, approximately 115 million years ago, the Crato Formation primarily formed within a lacustrine depositional system, eventually influenced by marginal marine sedimentation [31–33]. The laminated limestone beds harbor a wealth of exceptionally well-preserved fossils (Figures 2 and 3A), occasionally including soft tissues, thus designating this sedimentary succession as a Konservat-Lagerstätte [33].

Paleontological exploration in the Araripe Basin commenced in 1823 with the researchers J.B. von Spix and C.F.P. von Martius, who led a scientific expedition dispatched by King Maximilian I Joseph of Bavaria to investigate the treasures of the "New World". During their expedition, they documented the presence of fossils in the region, notably reporting fish fossils found within limestone layers, which are now attributed to the Crato and Romualdo Formations [34]. Since then, numerous researchers have undertaken studies to improve the understanding of the origin and composition of fossils within the basin, with particular focus on the Crato Formation. The extensive outcrop exposures within the Araripe Basin, the abundance of well-preserved fossils, and the exceptional degree of preservation have attracted, over the years, both curious individuals and dedicated scientists, alongside illicit fossil traffickers. Consequently, this valuable geological heritage has been subjected to predatory exploitation through uncontrolled mining and the illegal collection and smuggling of fossil specimens over the past two centuries [34].

Recently, there have been reports indicating that both national and foreign researchers and scientists, lacking the proper authorizations, engage local "peixeiros" (literally meaning "fishermen" or "fish mongers" but referring to unauthorized fossil fish collectors), who extract fossils in the region, to obtain specimens that are then transported to laboratories and collections outside Brazil. Moreover, fossil traffickers have negotiated these specimens online through commercial platforms such as eBay [35]. Among noteworthy recent cases, there is the one involving a pterosaur fossil, *Tupandactylus imperator*, which was listed on an online auction platform with a starting price of EUR 23,400 [35,36]. Another case that drew significant attention in the press and the worldwide academic community was the discovery of a new dinosaur species, named *Ubirajara jubatus*, identified through the examination of specimens exported from the laminated limestone of the Crato Formation by a team of international researchers. Their findings were originally published in the scientific journal Cretaceous Research. Described as a primitive bird, the specimen was chicken-sized, bipedal, and adorned with rudimentary feathers. This discovery alarmed Brazilian researchers, who reported the illegal extraction of the fossil, prompting a social media campaign for its repatriation. Subsequently, the Federal Public Ministry of Juazeiro do Norte (State of Ceará) launched an investigation into the removal of the fossil and requested the authorities in the country where it was located to seize and return the material [37]. Consequently, the publication was retracted, and the fossil was recently repatriated to Brazil and entrusted to the care of the Plácido Cidade Nuvens Paleontological Museum in Santana do Cariri, in the Brazilian State of Ceará.

According to Brazilian laws, fossils found and collected within the national territory belong to the nation, as per Decree-Law n. 4146/42 [38] and Article 20, paragraph X of the Federal Constitution of 1988 [39], classifying them as cultural assets. Their extraction requires prior authorization from the National Mining Agency (ANM), formerly known as the National Department of Mineral Production (DNPM). Exporting federal property without legal authorization or in violation of authorizing titles constitutes a crime against public property, specifically usurpation, as defined in Article 2 of Law n. 8176/91 [40]. Violators of the law are subject to penalties ranging from one to five years of imprisonment and a fine. According to Decree n. 98,830 of January 15, 1990 [41], the Ministry of Science and Technology is responsible for assessing and authorizing activities involving the collection and study of biological and mineral specimens within the national territory by foreigners for research purposes. These activities are contingent on the co-participation and co-responsibility of Brazilian institutions with a high and recognized technical–scientific expertise in the field of research related to the work to be carried out.

The Brazilian Federal Police is the responsible authority for combating illegal practices involving natural heritage in Brazil. In the last decade, up to 23,677 fossil specimens were seized, and 44 fossil examination reports were conducted, according to the data collected from the Superintendence of the Federal Police in the State of Ceará (information from Ministério da Justiça, Polícia Federal of State of Ceará 2021). Nevertheless, in the USA and some European countries, the sale of fossils is legal. Therefore, for the Brazilian Public Ministry to request the repatriation of specimens allegedly smuggled from Brazil, their origin must be unequivocally proven. Many illegally extracted fossils, seized and smuggled, exhibit specific and lithological characteristics of the host rocks similar to those of the Crato Formation of the Araripe Basin. Moreover, similar fossiliferous limestones exist in other regions of Brazil and worldwide, making their distinction requiring meticulous, potentially expensive, and time-consuming studies.

Given the facts described, the aim of this research is to determine the stable oxygen and carbon isotopic signatures distinctive of the most prized fossiliferous laminated limestone bed of the Crato Formation. These signatures, which can be obtained with a non-destructive and easily accessible technique [24], will be used as an additional identifier of the geographical origin of specimens seized by the Brazilian Federal Police. Thus, this pilot study has the potential to contribute to the recovery of illegally smuggled fossil specimens through a relatively quick and cost-effective method, providing quantitative, easily measurable, and concrete evidence of the crime of usurpation of public property. It may also result in a proof of concept, so far constrained to lacustrine laminated fossiliferous limestone from Brazil, for the development of a lithofacies type and geochemical database of worldwide vertebrate fossil-bearing carbonate deposits that can serve as a resource for scientific researchers and forensic investigators.

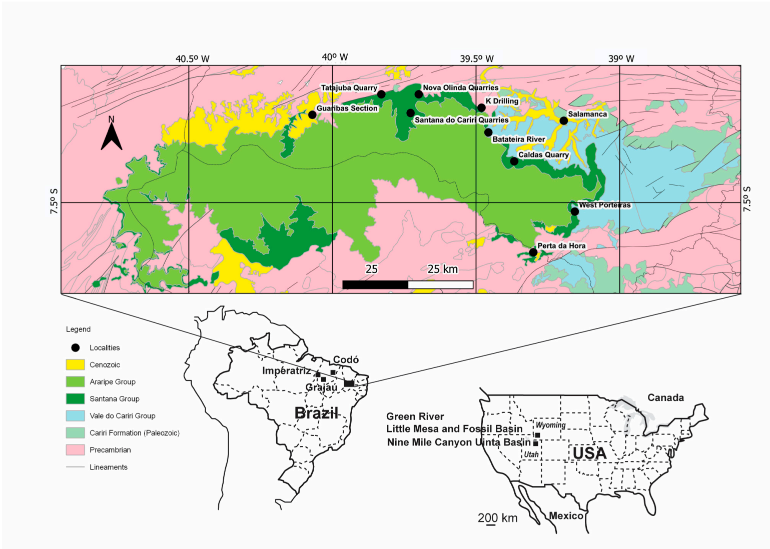

Figure 1. Simplified geological map [42] of the Araripe Basin and its location in northeastern Brazil. In the lower left, the simplified geographic map of Brazil showing the location of the samples from the Lower Cretaceous Codó Formation [43]. In the lower right corner, the simplified geographical map of the USA with the location of the samples from the Eocene Green River Formation in Utah and Wyoming [44].

Figure 2. (**A**) Mining front of laminated limestone from the Crato Formation in the Nova Olinda Quarry (see location reported in Table 1). (**B**) Appearance of the “sete cortes” bed on the mining front in the Nova Olinda Quarry. Note the cut lines separating 10 cm thick slabs.

Table 1. Location, stratigraphic units, and references of the compiled publications and this study. The C1 to C6 units and "sete cortes" ("7 Cuts") ethnostratum refer to the Lower Cretaceous Crato Formation samples.

Locality	**City (State)**	**Sampled Interval (Formation)**	**Latitude**	**Longitude**	**References**
Nine Mile Canyon, Little Mesa, and Fossil Basin	Duchesne (Utah); La Barge and Kemmerer (Wyoming) (USA)	Green River Fm. (Eocene)	39° 47.1′ N 38° 38.3′ N 41° 47.3′ N	110° 23.8′ W 108° 21.1′ W 110° 32.2′ W	[44] and this work
Codó, Grajaú and Imperatriz	Codó, Grajaú and Imperatriz (Maranhão)	Codó Fm. (Lower Cretaceous)	4° 33.6′ S 5° 44.7′ S 5° 31.6′ S	43° 57.7′ W 46° 6.6′ W 47° 29.4′ W	[43]
Guaribas Section	Araripe (Ceará)	C6 unit (except 7 Cuts)	7° 11.7′ S	40° 4.2′ W	[32]
Salamanca	Missão Velha (Ceará)	C6 unit (except 7 Cuts)	7° 12.9′ S	39° 11.7′ W	[32]
Perta da Hora	Serrita (Pernambuco)	C6 unit (except 7 Cuts)	7° 40.4′ S	39° 18.0′ W	[45]
West Porteiras	Porteiras (Ceará)	C6 unit (except 7 Cuts)	7° 31.9′ S	39° 9.4′ W	[45,46]
K-Drilling	Crato (Ceará)	C1-C5 units	7° 10.2′ S	39° 28.9′ W	[45]
Caldas Quarry	Barbalha (Ceará)	C3 and C6 units (except 7 Cuts)	7° 21.4′ S	39° 22.0′ W	[46]
Batateira River	Crato (Ceará)	C3-C6 units (except 7 Cuts)	7° 15.3′ S	39° 27.4′ W	[32,45,46]
Santana do Cariri Quarries	Santana do Cariri (Ceará)	C6 unit (except 7 Cuts)	7° 11.3′ S	39° 43.7′ W	[46]
Nova Olinda Quarries	Nova Olinda (Ceará)	C6 unit (including 7 Cuts)	7° 7.4′ S	39° 42.0′ W	[32,47]
Tatajuba Quarry	Santana do Cariri (Ceará)	7 Cuts	7° 7.4′ S	39° 49.8′ W	[47]

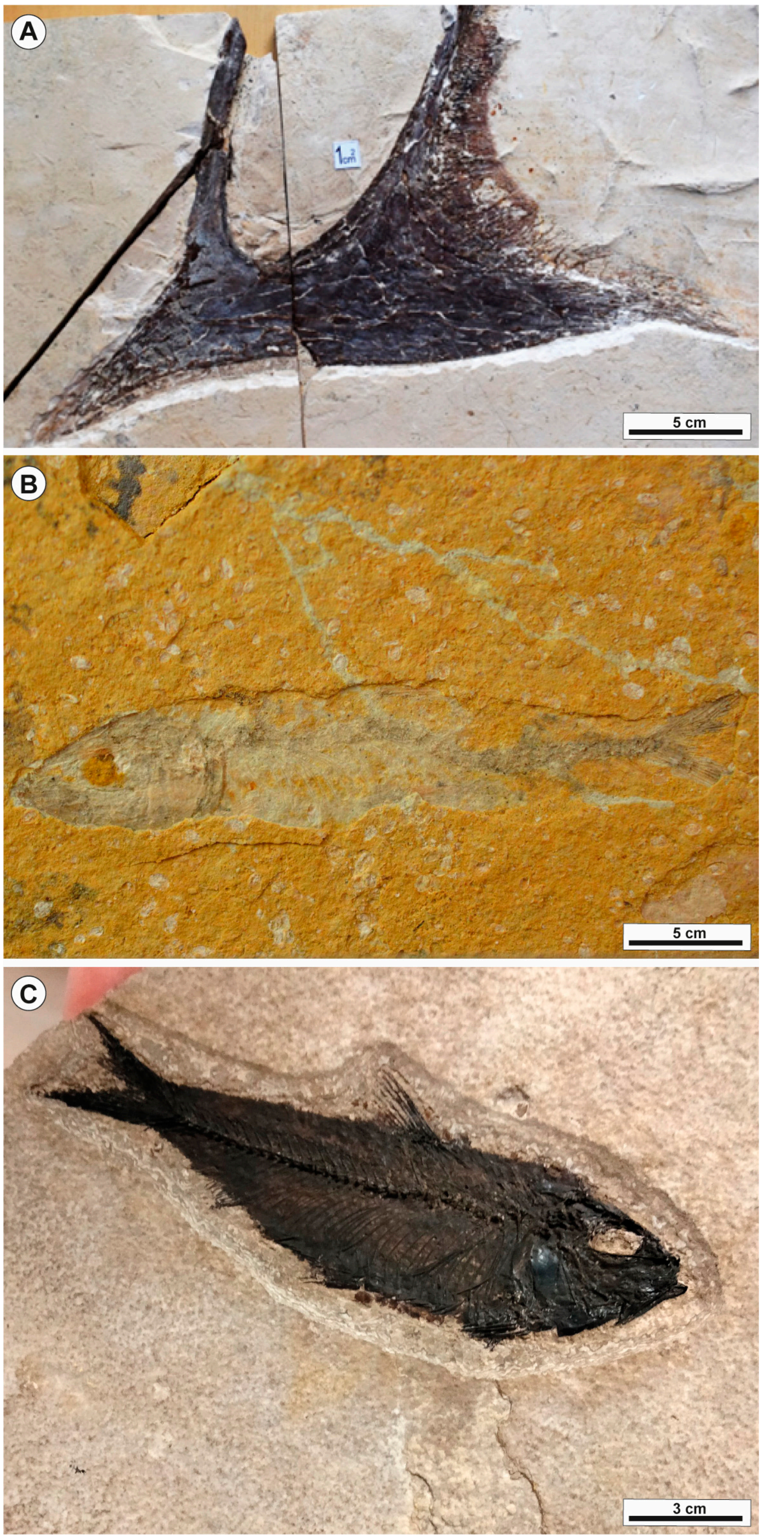

Figure 3. (**A**) Fossil pterosaur crest of an unidentified species, in the laminated limestone of the Crato Formation. It was seized by the Federal Police in the *Santanaraptor* operation. The specimen is now housed at the Plácido Cidade Nuvens Paleontology Museum in Santana do Cariri, State of Ceará, Brazil. (**B**) Fossil fish specimen from the Codó Formation (Maranhão, Brazil), showing vertebral segmentation, used with the Rafael Matos Lindoso author's permission [48]. (**C**) Green River Formation (Eocene, WY, USA) laminated calci-mudstone with fish fossil analyzed for stable oxygen and carbon isotopes at the University of Milan. The fossil fish (approximate length 12 cm) represents a specimen of the genus *Knightia* and belongs to the paleontological collection of the Department of Earth Sciences of the University of Milan, Italy.

2. Geological Background

The Araripe Sedimentary Basin in northeastern Brazil (Figure 1) covers an area of approximately 9000 km^2 and extends across the states of Piauí, Pernambuco, and Ceará, between longitudes 38° 30′ W and 40° 50′ W and between latitudes 7° 05′ S and 7° 50′ S. Elongated in an east–west direction, the basin measures 160 km in length and 50 km in width, reaching elevations of up to 900 m. Classified as a plateau, this geomorphological feature stands prominently out in the regional landscape due to its elevated position, steep escarpments, and gentle westward slope [49]. The Araripe Basin is situated within Precambrian terrains of the Borborema Province, bounded by the Patos Lineament to the north and the Pernambuco Lineament to the south. It has been interpreted as an aborted rift [50,51], with its development attributed to the extensional and strike-slip tectonics [52] associated with the opening of the South Atlantic Ocean, during the Wealdenian Reactivation [53]. The opening initiated in the Late Jurassic and persisted into the Early Cretaceous. The Araripe Basin deposits encompass fluvial and lacustrine environments subdivided into sequences [49]. Adopting the tectonostratigraphic subdivision scheme proposed by Ponte and Ponte Filho [54], and integrated by Assine [49], the sequences are classified as Paleozoic, represented by the Cariri Formation; Pre-Rift, encompassing the first two units of the Vale do Cariri Group (Brejo Santo and Missão Velha Formations); Rift, represented by the Abaiara Formation; Post-Rift I, spanning the entire Santana Group (Barbalha, Crato, Ipubi, and Romualdo Formations); and Post-Rift II, represented by the Araripe Group (Araripina and Exu Formations).

Recent studies have proposed revisions of the sequences of the Pre-Rift and Rift phases of the Araripe Basin [55]. In this framework, Fambrini et al. [56] reclassified the basal units (Vale do Cariri Group) as follows: i. Rift Initiation (Late Jurassic)—including Brejo Santo Formation and the lower half of the Missão Velha Formation, representing the initial subsidence phase; ii. Rift Climax (Early Cretaceous)—encompassing the upper half of the Missão Velha Formation and the Abaiara Formation, thus corresponding to the period of maximum extension of the basin. The other sequences were maintained as in previous classifications [55,56].

Stratigraphy of the Crato Formation

The Crato Formation (Figure 2A), part of the Santana Group [42], comprises laminated limestone beds deposited in a lacustrine setting [57]. However, alternating siliciclastic lithofacies, such as marlstones, shales, siltstones, and sandstones, are also present, especially at transitional boundaries [45]. These lithologies are interpreted as shallow lacustrine deposits, potentially linked to associated alluvial systems, deltas, or fan deltas [45,58]. The presence of specific microfossils [59,60] and sedimentary structures within some siliciclastic beds suggests sporadic marine incursions into the lacustrine system. Nevertheless, these marine incursions were likely limited in both duration and extent [32,61].

The pioneer work by Neumann [45] identified six major carbonate units (C1–C6) within the Crato Formation based on studies in quarries, on outcrops, and from drilled borehole cores. These units represent cycles of lacustrine expansion and retreat driven by climatic variations over timescales of 200–500 kyrs [62]. Among these, the uppermost unit, C6, hosts the fossil-bearing lagerstätte bed and exhibits the widest geographical extent within the Cariri Valley [34,45].

Most of the paleontological studies, however, lacked stratigraphic precision of the identified specimens, often solely referring to "laminated limestone" or the "lagerstätte" of the Crato Formation, without specifying the carbonate unit or specific level under study within the formation [33]. To address this, Corecco et al. [63] adopted the informal nomenclature used by the quarry workers for specific levels within the commercially exploited limestone of the C6 unit ("Pedra Cariri"), an approach known as ethnostratigraphy. This nomenclature considers not only physical characteristics, such as cutting quality and color, but also the fossil content. Through field interviews and laboratory analysis, Corecco et al. [63] defined 17 ethnostrata within the C6 limestone. Among these ethnostrata, the one locally labeled as "sete cortes" ("seven cuts") is particularly significant as it harbors diverse vertebrate fossils, such as anurans, lizards, dinosaurs, and pterosaurs. Its name stems from the standard thickness of quarried slabs (10 cm) due to its seven commercially valuable "cuts" at the base of C6. Located just above the local water table at Nova Olinda Quarries (location reported in Table 1), the "sete cortes" extends at least 1 m stratigraphically below, for a total thickness of 1.7 m (Figure 2B). Additionally, it is characterized by rhythmic lamination ranging from a gray to a beige color [63,64].

Informal stratigraphic designations, such as those by Neumann [45] carbonate units and Corecco et al. [63] ethnostrata, hold value and recognition within both the *International Stratigraphic Guide* [65] and local codes [66]. The U.S. Stratigraphic Nomenclature Committee [67] categorizes them as parastratigraphic units. These informal units are typically described using lower-case common nouns, adjectives, or geographic terms [68].

3. Previous Stable Oxygen and Carbon Isotope Studies on the Crato Formation Limestone

Neumann [45] spearheaded the first stable oxygen and carbon isotope investigation within the Crato Formation, marking the most comprehensive endeavor in terms of sample quantity, geographical distribution, and stratigraphic coverage to date. Neumann's study [45] was remarkable for extending the isotopic analyses to the carbonate units from C1 to C5, differently from the predominant focus on the fossiliferous unit C6 limestone beds [33]. A total of 106 isotope analyses were conducted on 72 carbonate rock samples deriving from quarries, outcrops, and borehole cores, primarily comprising the identified depositional facies of the laminated limestones (LLs) and claystone–carbonate rhythmites (CCRs), alongside marlstone and diagenetic features such as concretions and cone-in-cone carbonate crystal structures. As a result, Neumann [45] determined the oxygen $\delta^{18}O$ values ranging from −8.4‰ to −1.6‰ (VPDB) and carbon $\delta^{13}C$ values ranging from +3.2‰ to +11.3‰. Drawing from the isotopic range delineated by Scoffin [69], Neumann [45] inferred deposition in a hydrologically closed lacustrine basin for the Crato Formation limestone, as evidenced by the positive and relatively high covariance indices observed between carbon and oxygen isotope ratios, particularly notable in carbonate units C3 and C6 [45].

Heimhofer et al. [47] undertook a study investigating the sedimentary and paleoenvironmental conditions prevalent during the deposition of the fossil-rich strata of the Crato Formation. They highlighted variations in thickness and color observed in the laminated limestone and claystone–carbonate facies [47]. Heimhofer et al. [47] analyzed samples from three stratigraphic sections located in quarries in Caldas (Barbalha, Ceará), south of Nova Olinda, and Tatajuba (Santana do Cariri, Ceará), specifically focusing on the primary fossil-bearing interval of carbonate unit C6 (later identified as the ethnostratum "sete cortes" by Corecco et al. [62]). Through the integration of isotopic analyses with other petrographic and geochemical methods, Heimhofer et al. [47] corroborated Neumann's [45] interpretation of a closed lake system, while also remarking on the possible influx of meteoric water and a low contribution of carbon from soils. Heimhofer et al. [47] reported VPDB $\delta^{18}O$ values ranging from −7.1‰ to −5.1‰, and $\delta^{13}C$ from −0.1‰ to +1.9‰, exhibiting positive and relatively high covariance consistent with Neumann [45].

Silveira [46] conducted stratigraphic, sedimentological, and geochemical analyses of the limestone beds overlying the "sete cortes" ethnostratum, within the carbonate unit C6, with the aim of investigating the mechanism of carbonate precipitation in lacustrine settings during the Early Cretaceous, whether abiotic physico-chemical, biogenic, or both, and the possibility of diagenetic and pedogenic effects under shallow-water conditions. Silveira [46] analyzed 38 samples collected from quarries and outcrops at seven locations, obtaining stable isotope values ranging from −8.3‰ to +0.2‰ for $\delta^{18}O$ (VPDB) and from −18.2‰ to +2‰ for $\delta^{13}C$. Silveira [46] interpreted the low negative $\delta^{13}C$ values as resulting from subaerial exposure, incipient soil development, and reworking, possibly influenced by meteoric freshwaters.

Varejão et al. [32] undertook an integrated examination encompassing stratigraphic, sedimentological, paleontological, and geochemical analyses within the Santana Group, extending it to stratigraphic units overlying and underlying the Crato Formation. The investigation of the Santana Group by Varejão et al. [32] was complemented with data from basins situated farther south (Tucano and Jatobá), enabling a comprehensive assessment of the collective evolutionary trajectory over time. Focusing specifically on the Crato Formation, Varejão et al. [32] scrutinized the laminated limestone (LL), claystone–carbonate rhythmite (CCR) facies, and the laminated microbial carbonate (stromatolite) deposits. However, their analysis was confined to levels positioned above the "sete cortes" ethnostratum within carbonate unit C6, conducted at four locations in the State of Ceará (Nova Olinda, Guaribas, Batateira, and Salamanca). These authors [32] suggested hypersaline lake conditions, inferred from the carbonate stable isotope values akin to those observed in modern and ancient lacustrine limestones [44]. Nonetheless, unlike the characteristic covariance typically indicative of closed lake systems, as identified by Neumann [45] and Heimhofer et al. [47], the $\delta^{18}O$ and $\delta^{13}C$ values by Varejão et al. [32] did not exhibit such traits. In fact, Varejão et al. [32] proposed that the diagenetic influence of meteoric water on the marginal lacustrine system during deposition could have impacted the isotopic signature, albeit they deemed it sufficiently minor not to challenge the proposed paleoenvironmental interpretations. The isotopic values reported by Varejão et al. [32] ranged from −8.6‰ to −0.2‰ for $\delta^{18}O$ (VPDB) and from −3.1‰ to +1.5‰ for $\delta^{13}C$. Similar to Silveira [46], Varejão et al. [32] measured negative $\delta^{13}C$ values, reflecting the influence of meteoric freshwater.

4. Materials and Methods

This research collected oxygen and carbon isotopic ratio data from studies previously published by various authors [32,43–47]. The analyzed samples primarily originate from lacustrine limestones across various localities and stratigraphic ages. These lacustrine carbonates and associated microbialites were studied for diverse purposes, including establishing relationships with other depositional systems, determining salinity conditions, correlating across different lake stratigraphic evolution, assessing diagenetic effects, and investigating modes of carbonate precipitation, whether physico-chemical or biogenic, associated with microbial carbonates. Some of these studies [32,45–47] identified facies, texture and sedimentary structure features distinctive of the fossiliferous laminated limestones of the Crato Formation at visual examination. Considering that initial approximations in forensic investigation often rely on visual similarity, only data from finely millimeter-laminated limestones, marlstones, calcilutites, or calci-mudstones were selected. Depositional facies within the Crato Formation, specifically the laminated limestones (LLs) and claystone–carbonate rhythmites (CCRs), as classified by some authors [32,45], were individually considered in the analysis, despite lacking noticeable differences at the macroscopic level.

In this study, data from the Crato Formation [32,45–47] were subjected to statistical analysis to delineate ranges of $\delta^{18}O$ and $\delta^{13}C$ isotopic ratio values characteristic of this formation. The aim was to refine, through successive and increasingly stringent approximations, the distinctive isotopic range of the "sete cortes" ethnostratum hosting the most coveted vertebrate fossils, which are prime targets for traffickers and collectors. Additionally, isotopic data from the Codó Formation (Lower Cretaceous, Parnaíba Basin [43]; Figures 1 and 3B) and the Green River Formation (Eocene, UT and WY, USA [44,70]; Figures 1 and 3C) lacustrine deposits were utilized for comparisons with the Crato Formation. The Codó and Green River Formations were selected for comparison with the Crato Formation oxygen and carbon isotope values because these sedimentary units similarly consist of carbonates accumulated in closed lacustrine basins with fluctuating salinity under semi-arid climatic conditions and comprise laminated limestones and shales [43–45].

Furthermore, their contrasting geological ages and geographic location were also considered relevant criteria to inspect a wide spectrum of stable isotope values. The Brazilian Codó Formation is fairly coeval with the Crato Formation (Aptian–Albian) [43,45], whereas the USA Green River Formation is significantly younger (Eocene) [70]. The Green River Formation comprises laminated calci-mudstones rich in vertebrate fossils that are globally traded and might be mistaken for the Crato Formation fossiliferous laminated limestone during visual inspection by a non-expert in vertebrate paleontology. These lithological, depositional environment, and fossiliferous similarities, paralleled by distinct stratigraphic age and geographic locations, provide a wide dataset of fossiliferous limestone isotopic signatures to help discriminate the Crato Formation under study.

These comparisons were based on visually similar sedimentary units in terms of lithology and depositional textures, both nationally and internationally (Table 1). Specifically, data from the Green River Formation in the Uinta Basin (Nine Mile Canyon) in Utah and the Green River Basin in Wyoming, as published in Della Porta [44], were integrated with three new analyses conducted for this study on a laminated calci-mudstone slab containing a fish fossil (specimen belonging to the paleontological collection of the Department of Earth Sciences of the University of Milan, Figure 3C) from the Fossil Basin (Kemmerer) in Wyoming, with the isotopic oxygen and carbon averages being referenced accordingly.

The new stable oxygen and carbon isotope analyses of three samples of the Green River Formation fossil-bearing laminated calci-mudstone (Figure 3C) were performed using an automated carbonate preparation device (Gasbench II) and a Thermo Fisher Scientific Delta V Advantage continuous flow mass spectrometer at the Department of Earth Sciences, University of Milan. Carbonate powder samples were reacted with >99% orthophosphoric acid at 70 °C. The carbon and oxygen isotope compositions are expressed in the conventional delta notation calibrated to the Vienna Pee-Dee Belemnite (VPDB) scale by the international standards IAEA 603 and NBS-18. Analytical reproducibility for these analyses was better than ±0.1‰ for both $\delta^{18}O$ and $\delta^{13}C$ values.

The data were organized according to both geographical and stratigraphic criteria. Geographically, the initial grouping involved comparing the Crato Formation with the Codó Formation [43] and the Green River Formation [44]. Subsequently, the data from the Crato Formation were stratigraphically categorized, with a separation between those from the carbonate unit C6 and those from units C1-C5 [45]. The subsequent grouping employed a mixed approach, focusing solely on data from the carbonate unit C6, which has the broadest geographic distribution, organized by occurrence across ten localities. However, in two of these locations, only data from the ethnostratum "sete cortes" (base of the unit C6 [62]) were included. Following this, a stratigraphic criterion was applied, grouping data exclusively from unit C6 and comparing samples from the "sete cortes" ethnostratum with those from the other levels, stratigraphically overlying it [46]. Finally, data from the Crato Formation were grouped once again based on their depositional facies within the carbonate unit C6, distinguishing between those from the laminated limestone (LL) facies and claystone–carbonate rhythmite (CCR) facies (Figure 4) as recognized from previous studies [32,45,47].

A statistical summary was compiled for all groupings of isotopic data, outlining their central tendency measures and variability. For discrete values within the groups, correlation graphs depicting the relationship between the isotopes and boxplots were generated for comparison. Regarding correlation analysis, the linear correlation coefficient (r) was assessed for significance level or Type I error (*p*), representing the probability of erroneously rejecting a true null hypothesis (H_0). In this context, H_0 posits the absence of a significant correlation (r≈0), while p indicates a nonexistent correlation [71]. As for the boxplots, they were segregated into oxygen and carbon isotope datasets, incorporating whiskers and identifying potential outliers, which were values above and below up to 1.5 times the interquartile range (IQR). All statistical analyses and graphical representations were conducted using the Minitab 15 software.

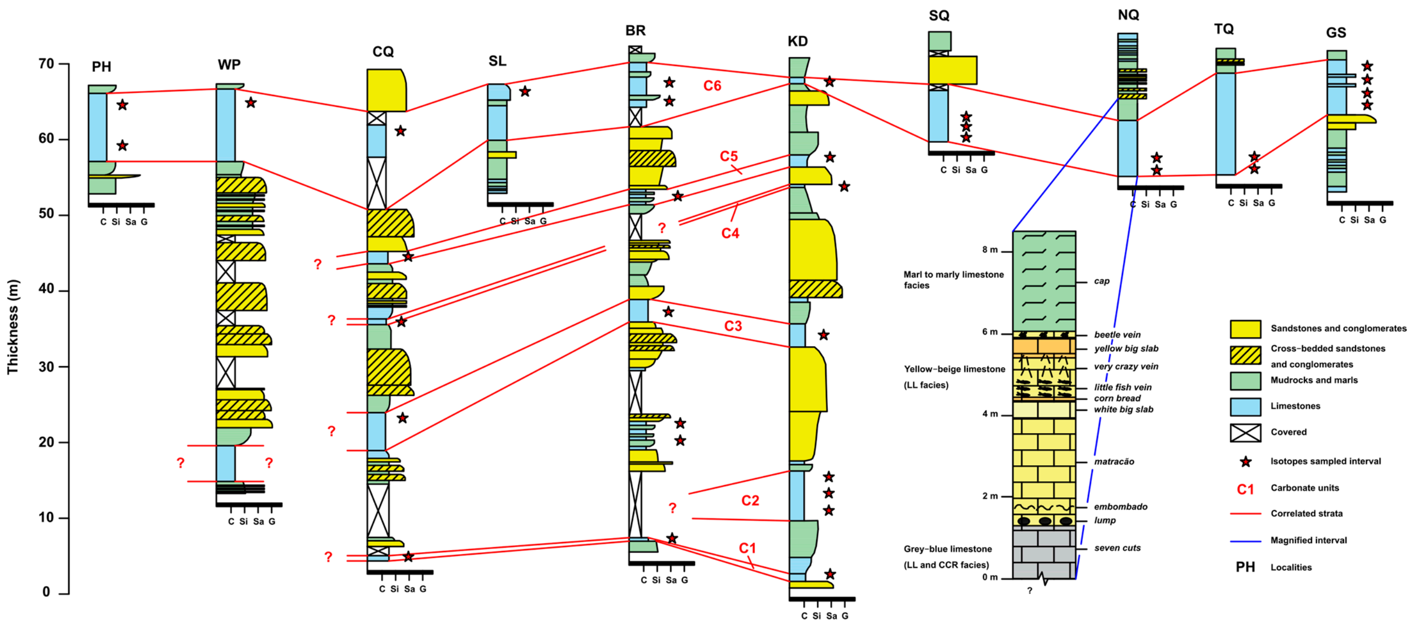

Figure 4. Simplified stratigraphic columns and vertical location of isotopic samples of the Crato Formation from ten different localities in the Cariri Valley, Araripe Basin, Brazil [32,45–47]. Question mark "?" marks uncertain correlations. See Table 1 for the geographic coordinates. Locations: PH—Perta da Hora; WP—West Porteiras; CQ—Caldas Quarry; SL—Salamanca; BR—Batateira River; KD—K-Drilling; SQ—Santana do Cariri Quarries; NQ—Nova Olinda Quarries; TQ—Tatajuba Quarry; GS—Guaritas Section. Correlation and carbonate units are based on [45], and the highlighted, enlarged section of unit C6 in NQ Nova Olinda, with the interpretation of ethnostrata, is based on [63].

5. Results and Interpretation

General Description of Groupings

The limestone samples from the Crato Formation (Table 1; Figure 4) display considerable variability in oxygen and carbon isotopic compositions across the various studies compiled (Table 2; Figure 5). The overall range of values extends from 17.1‰ for $\delta^{18}O$ to 29.2‰ for $\delta^{13}C$. Even when accounting for more representative values within the unit, with an average of −5.0‰ for $\delta^{18}O$ and −1.3‰ for $\delta^{13}C$, accompanied by a standard deviation of 3.5‰ for oxygen and 4.3‰ for carbon, these findings suggest a significant potential for overlap between these values and those observed in discrete analyses of randomly selected samples from the Codó and Green River Formations. This likelihood of overlap is further accentuated by the Crato Formation's broader range of isotopic values compared with those of the Codó and Green River Formations, which is attributable to both its larger sample size (n = 213) and the varying geographical and stratigraphic collection conditions (Tables 1 and 2).

Nevertheless, the Crato Formation exhibits a distinct linear correlation pattern between the $\delta^{18}O$ and $\delta^{13}C$ values, which is notably weaker (r = +0.108, p = 0.115) when compared with that of the Codó Formation (r = −0.713, p = 0.014) and the Green River Formation (r = +0.487, p = 0.066). Moreover, the Crato Formation's positive correlation contrasts with that of the Codó Formation. The disparate dataset from the Crato Formation must result in the weak linear correlation between $\delta^{18}O$ and $\delta^{13}C$. Nonetheless, some previous studies have reported robust linear correlation indices and corresponding covariance between oxygen and carbon isotopic data from the Crato Formation [45,47]. Such covariance would suggest endorheic lacustrine and arid hydrological conditions during the Early Cretaceous in the Araripe Basin.

The Crato Formation stands out for its significantly broader range and greater variability of isotopic data compared to other formations, which is particularly evident in its abundance of outliers. Despite its larger sample size and varying geographical and stratigraphic conditions, the Crato Formation exhibits smaller IQR values in three out of four instances, measuring only 1.6‰ for $\delta^{18}O$ and 2.5‰ for $\delta^{13}C$. This indicates a more statistically cohesive grouping within the overall sample set of the Crato Formation, as illustrated in Figure 5.

Upon reorganizing the data from the Crato Formation, there is a marginal enhancement in the linear correlation indices. The significance level slightly decreases for the carbonate unit C6 in contrast with the aggregated result for the carbonate units C1-C5 (p = 0.091 vs. 0.270), while the overall linear correlation slightly increases in the latter group (r = +0.172 vs. +0.130). The positions and IQRs of unit C6 closely resemble those of the entire Crato Formation, likely because most of the data (80%) in prior studies are concentrated on the unit housing the fossil-rich lagerstätte [34]. However, the prevalence of extreme values in the oxygen isotopic ratio persists notably within unit C6. In contrast, despite its smaller sample size, the group comprising the other carbonate units exhibits substantially larger IQRs, surpassing 10‰ for $\delta^{18}O$ values (Figure 6).

When comparing seven different sites where the C6 carbonate unit of the Crato Formation occurs in the Cariri Valley, a significant heterogeneity in the linear correlation between oxygen and carbon isotope data becomes apparent. This heterogeneity is reflected in the correlation index (r), which ranges from +0.135 at the Batateira River to +0.915 in the West Porteiras, and displays negative values at the Perta da Hora and Nova Olinda Quarries. Similarly, the significance index (p) varies from 0.709 at the Batateira River to 0.00 in the West Porteiras and Tatajuba Quarries. In terms of boxplots, the lowest IQRs are observed for the datasets from the Nova Olinda and Tatajuba Quarries. There, in all instances, a notable improvement in restricting the variability of isotopic values compared to the C6 unit occurs, particularly through the elimination of extreme values and the reduction in the IQR in the Nova Olinda samples, departing from over 1‰ to approximately 0.2‰. It is worth noticing, however, that the data from the C6 unit of the Nova Olinda and Tatajuba localities are limited to those of the "sete cortes" ethnostratum. Additionally, the number of Tatajuba samples (n = 60) is higher than that in the other localities (Figure 7).

Table 2. Statistical summary of the referenced samples and their groupings. Values are in ‰ VPDB and were assembled from published studies and analyses from the present study. Locations: BR—

Batateira River; WP—West Porteiras; PH—Perta da Hora; SQ—Santana do Cariri Quarries; CQ—Caldas Quarry; NQ—Nova Olinda Quarries; TQ—Tatajuba Quarry; 7C—"sete cortes" ("seven cuts") ethnostratum; LL—laminated limestone; CCR—claystone–carbonate rhythmite.

Group	n	Max	Min	Mean	SD	Q1	Median	Q3	IQR
$\delta^{18}O$ Crato	213	8.70	−8.40	−4.98	3.54	−6.78	−6.03	−5.21	1.58
$\delta^{13}C$ Crato	213	11.00	−18.22	−1.30	4.36	−1.58	0.34	0.90	2.48
$\delta^{18}O$ Codó	11	−1.04	−7.76	−5.29	2.69	−7.51	−6.86	−2.25	5.26
$\delta^{13}C$ Codó	11	2.52	−13.40	−4.70	4.96	−7.35	−6.77	0.01	7.36
$\delta^{18}O$ Green River	15	−2.22	−8.27	−4.80	1.69	−5.66	−4.46	−3.56	2.10
$\delta^{13}C$ Green River	15	3.69	−4.45	−0.05	1.82	−0.90	0.20	0.49	1.40
$\delta^{18}O$ C6	170	0.23	−8.25	−5.72	1.47	−6.78	−5.99	−5.25	1.53
$\delta^{13}C$ C6	170	11.00	−18.22	−1.47	4.77	−1.87	0.37	1.01	2.88
$\delta^{18}O$ C1-5	43	8.70	−8.40	−2.07	6.61	−6.90	−6.10	6.10	13.00
$\delta^{13}C$ C1-5	43	1.10	−7.20	−0.61	1.94	−1.20	0.00	0.70	1.90
$\delta^{18}O$ C6 BR	10	−3.40	−7.79	−6.29	1.22	−6.95	−6.47	−5.95	1.01
$\delta^{13}C$ C6 BR	10	1.10	−13.94	−9.49	5.67	−13.79	−13.42	−4.65	9.14
$\delta^{18}O$ C6 WP	12	−1.60	−7.21	−4.00	2.08	−6.14	−3.90	−2.10	4.04
$\delta^{13}C$ C6 WP	12	3.20	−18.22	−4.15	6.73	−7.20	−3.60	2.02	9.23
$\delta^{18}O$ C6 PH	8	−0.50	−7.20	−4.04	2.58	−6.90	−3.60	−1.65	5.25
$\delta^{13}C$ C6 PH	8	11.00	−10.00	−2.45	6.13	−5.53	−3.55	−0.85	4.68
$\delta^{18}O$ C6 SQ	13	0.23	−8.25	−6.14	2.01	−6.87	−6.68	−6.11	0.76
$\delta^{13}C$ C6 SQ	13	−1.80	−14.45	−11.06	4.16	−13.67	−12.72	−8.38	5.29
$\delta^{18}O$ C6 CQ	4	−2.38	−3.73	−2.88	0.64	−3.56	−2.71	−2.38	1.18
$\delta^{13}C$ C6 CQ	4	−0.39	−3.32	−1.79	1.20	−2.94	−1.73	−0.71	2.24
$\delta^{18}O$ 7C NQ	**10**	**−5.34**	**−5.85**	**−5.55**	**0.15**	**−5.63**	**−5.54**	**−5.43**	**0.20**
$\delta^{13}C$ 7C NQ	**10**	**1.32**	**0.84**	**1.13**	**0.14**	**1.04**	**1.16**	**1.24**	**0.20**
$\delta^{18}O$ 7C TQ	**60**	**−5.10**	**−7.06**	**−6.06**	**0.68**	**−6.81**	**−6.14**	**−5.30**	**1.51**
$\delta^{13}C$ 7C TQ	**60**	**1.93**	**−0.14**	**0.86**	**0.66**	**0.34**	**0.60**	**1.71**	**1.37**
$\delta^{18}O$ 7C	**74**	**−3.47**	**−7.41**	**−5.94**	**0.76**	**−6.78**	**−5.89**	**−5.30**	**1.47**
$\delta^{13}C$ 7C	**74**	**1.93**	**−0.14**	**0.90**	**0.61**	**0.41**	**0.73**	**1.66**	**1.24**
$\delta^{18}O$ above 7C	58	0.23	−8.25	−5.67	1.75	−6.83	−6.15	−4.80	2.03
$\delta^{13}C$ above 7C	58	3.20	−18.22	−5.09	6.24	−12.84	−3.11	0.36	13.20
$\delta^{18}O$ LL	113	0.23	−8.25	−5.92	1.38	−6.82	−6.19	−5.54	1.29
$\delta^{13}C$ LL	113	3.20	−18.22	−2.32	5.29	−3.34	0.30	0.72	4.05
$\delta^{18}O$ CCR	28	−2.10	−8.19	−5.44	1.33	−5.55	−5.27	−5.17	0.38
$\delta^{13}C$ CCR	28	2.40	−2.03	1.25	1.02	0.50	1.75	1.83	1.33

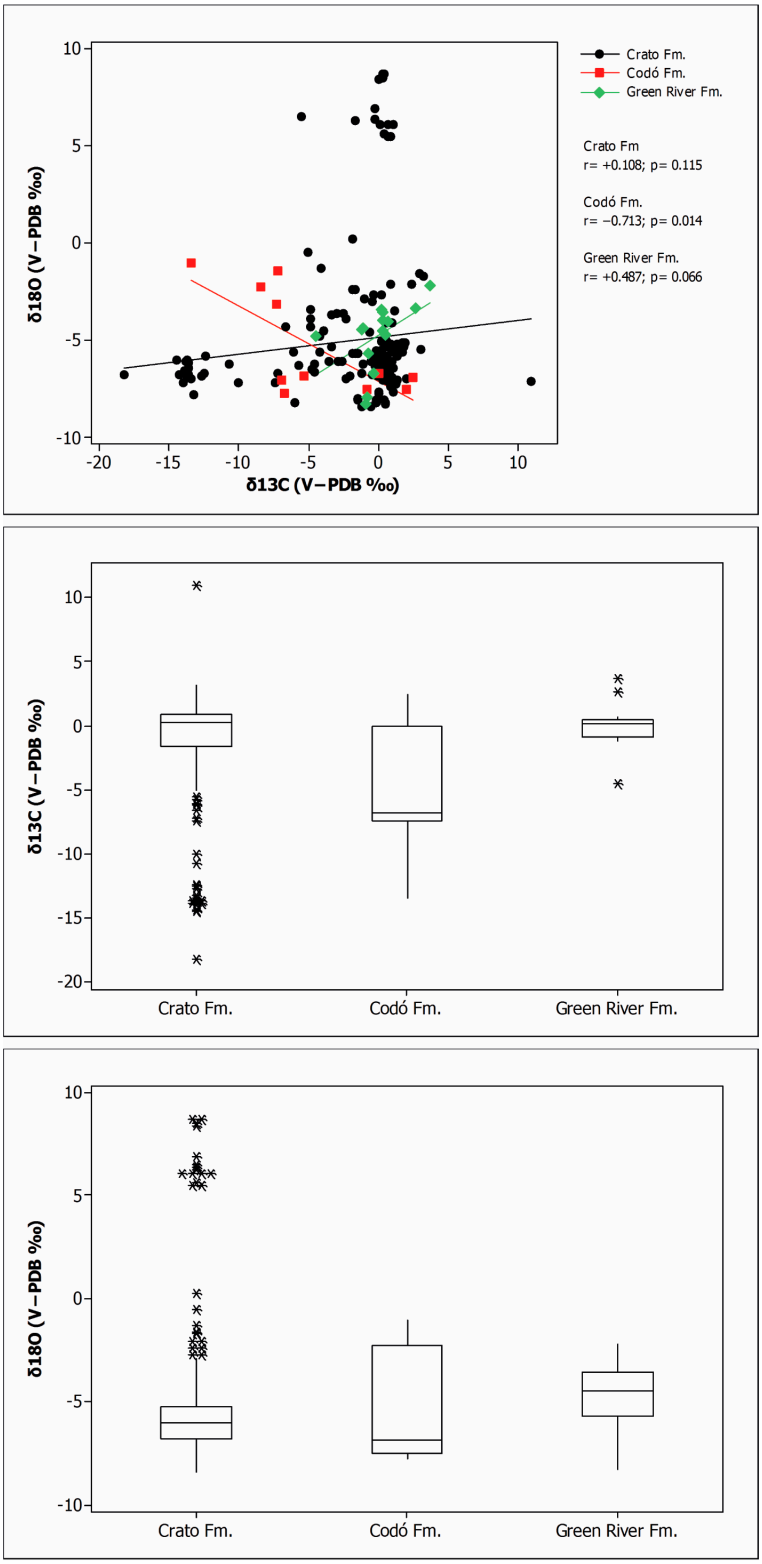

Figure 5. Scatterplot and boxplots for $\delta^{18}O$ and $\delta^{13}C$ values in ‰ VPDB of the Crato, Codó, and Green River Formations, showing the large spread of the Crato Formation stable isotope data when no geographic or stratigraphic grouping of the sample is performed.

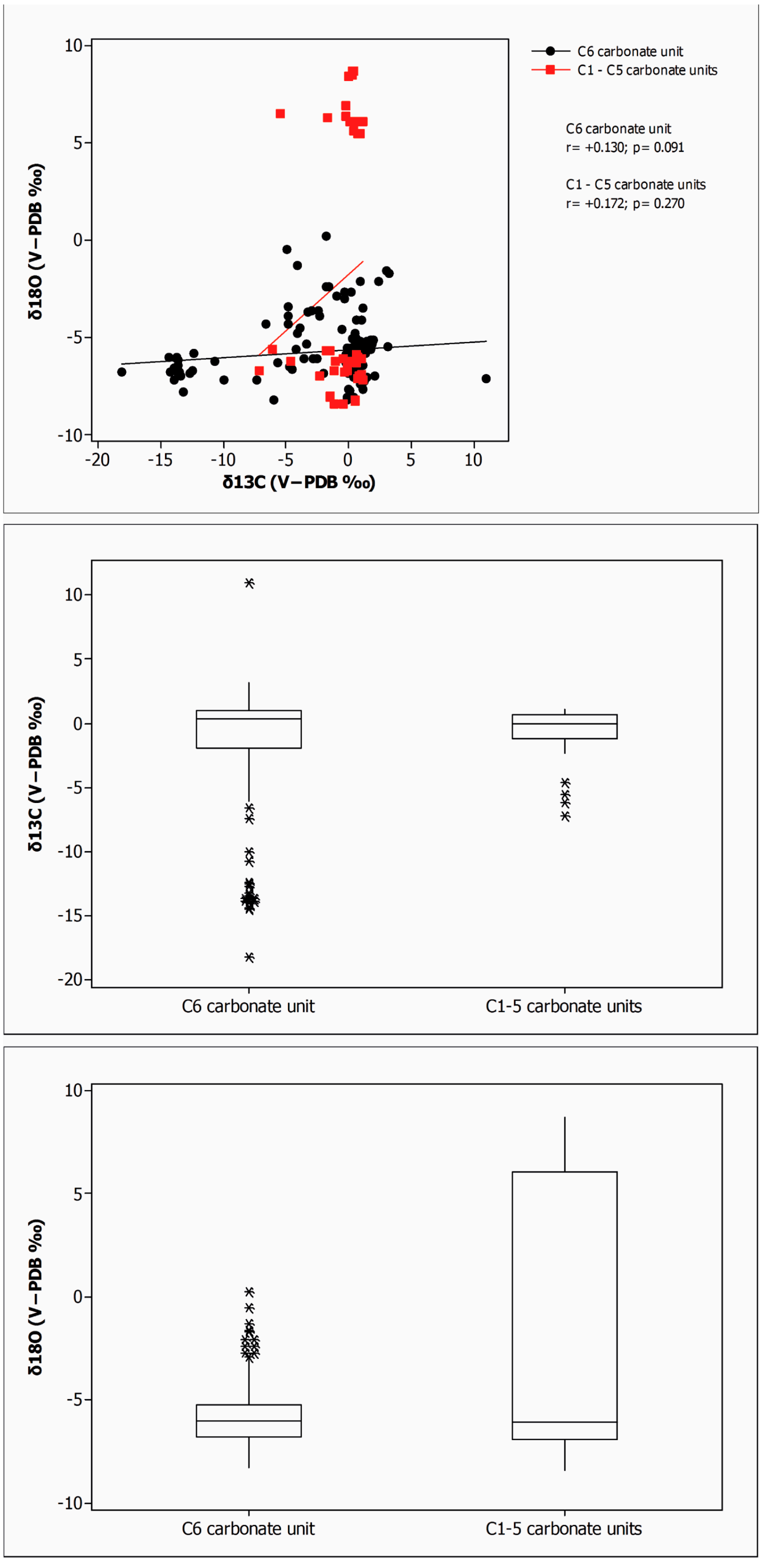

Figure 6. Scatterplot and boxplots for δ^{18}O and δ^{13}C values of the Crato Formation, comparing the fossiliferous carbonate unit C6 with the other carbonate units from C1 to C5, showing a wide scatter of isotopic data when no grouping for location is performed.

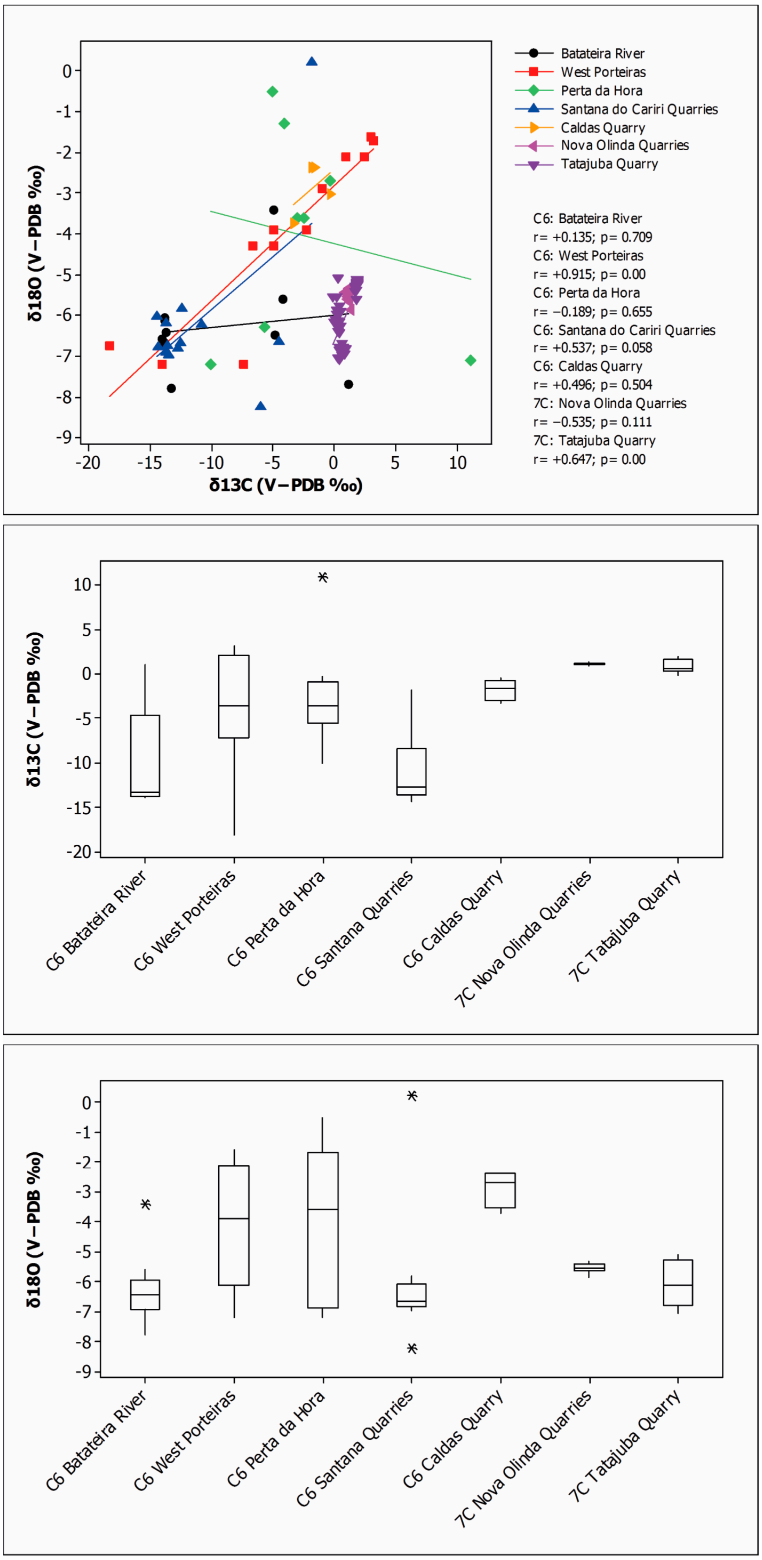

Figure 7. Scatterplot and boxplots for $\delta^{18}O$ and $\delta^{13}C$ values of the Crato Formation carbonate unit C6 in different locations showing the differences in isotopic data due to geographic location, possible different depositional environment, and diagenesis. For the Nova Olinda and Tatajuba Quarry locations, the data for unit C6 are restricted to the "sete cortes" ethnostratum (7C).

When comparing the $\delta^{18}O$ and $\delta^{13}C$ values of the carbonate unit C6 limited to the "sete cortes" ethnostratum with those from the same C6 unit but positioned above the ethnostratum, it becomes evident that the latter exhibits a lower correlation level, with r = +0.340 and p = 0.009, in contrast to r = +0.547 and p = 0.00. Furthermore, the IQRs of the "sete cortes" ethnostratum are notably smaller for both oxygen and carbon isotopic composition, suggesting reduced data variability and greater homogeneity (Figure 8). When comparing samples attributed to the depositional facies of unit C6 (LL and CCR), they exhibit lower overall correlation levels and greater data variability compared with the previous analysis between the "sete cortes" ethnostratum and the remaining C6 unit overlying the "sete cortes". The $\delta^{18}O$ and $\delta^{13}C$ values of the two facies indicate that the CCR has a higher correlation level (r = +0.501, p = 0.007) than the LL (r = +0.166, p = 0.08), suggesting less dispersion in the former despite the disparity in the number of samples (LL: n = 113; CCR: n = 28). The notable increase in the number of outliers in the boxplots further underscores the overall greater data dispersion of the two facies compared to the diagrams from the previous analyses (Figure 9).

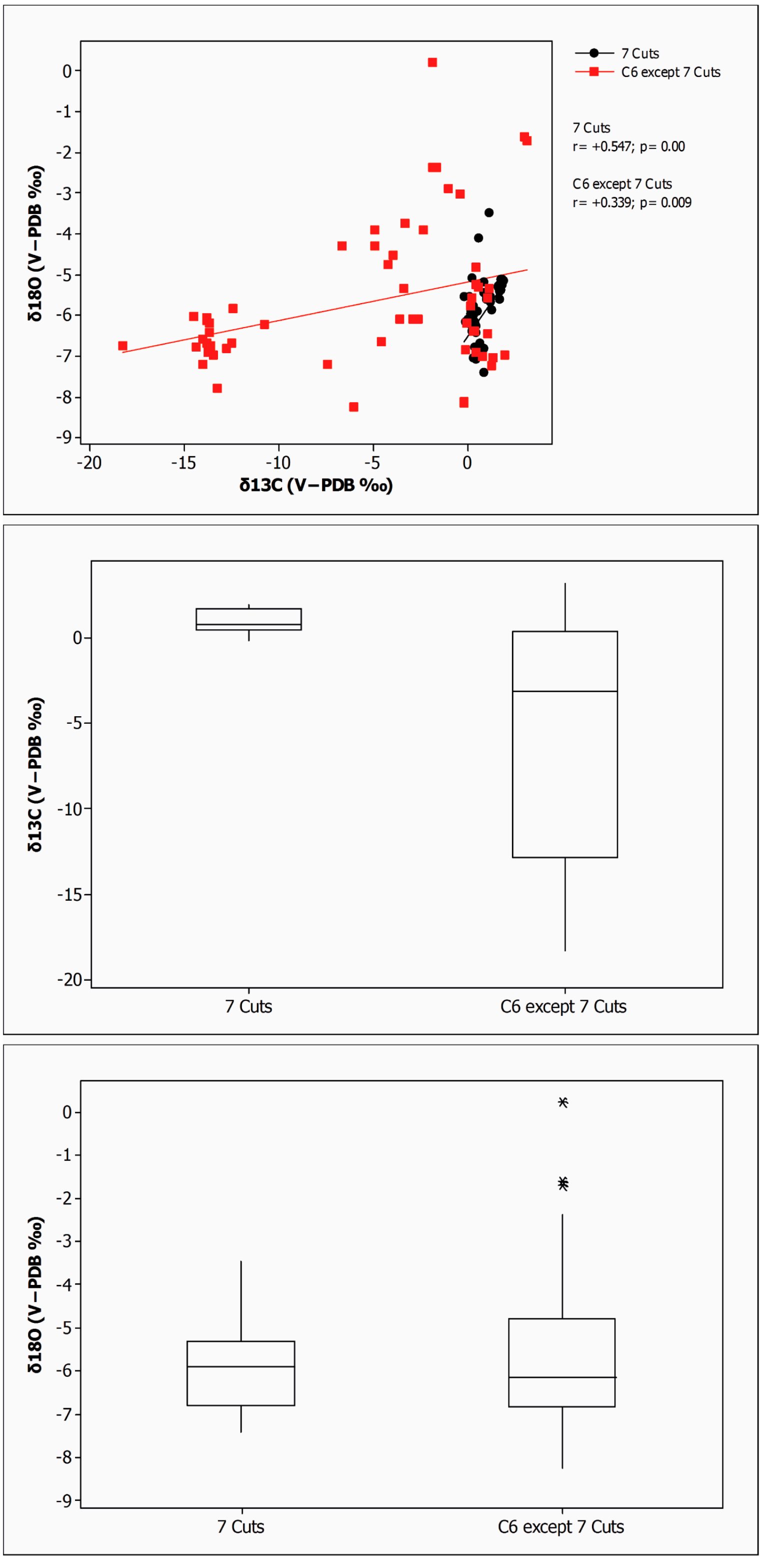

Figure 8. Scatterplot and boxplots for $\delta^{18}O$ and $\delta^{13}C$ values of the carbonate unit C6 of the Crato Formation, comparing data from the "sete cortes" (7 Cuts) ethnostratum with the overlying C6 beds, showing how the range of stable isotope values is better constrained within the 7 Cuts.

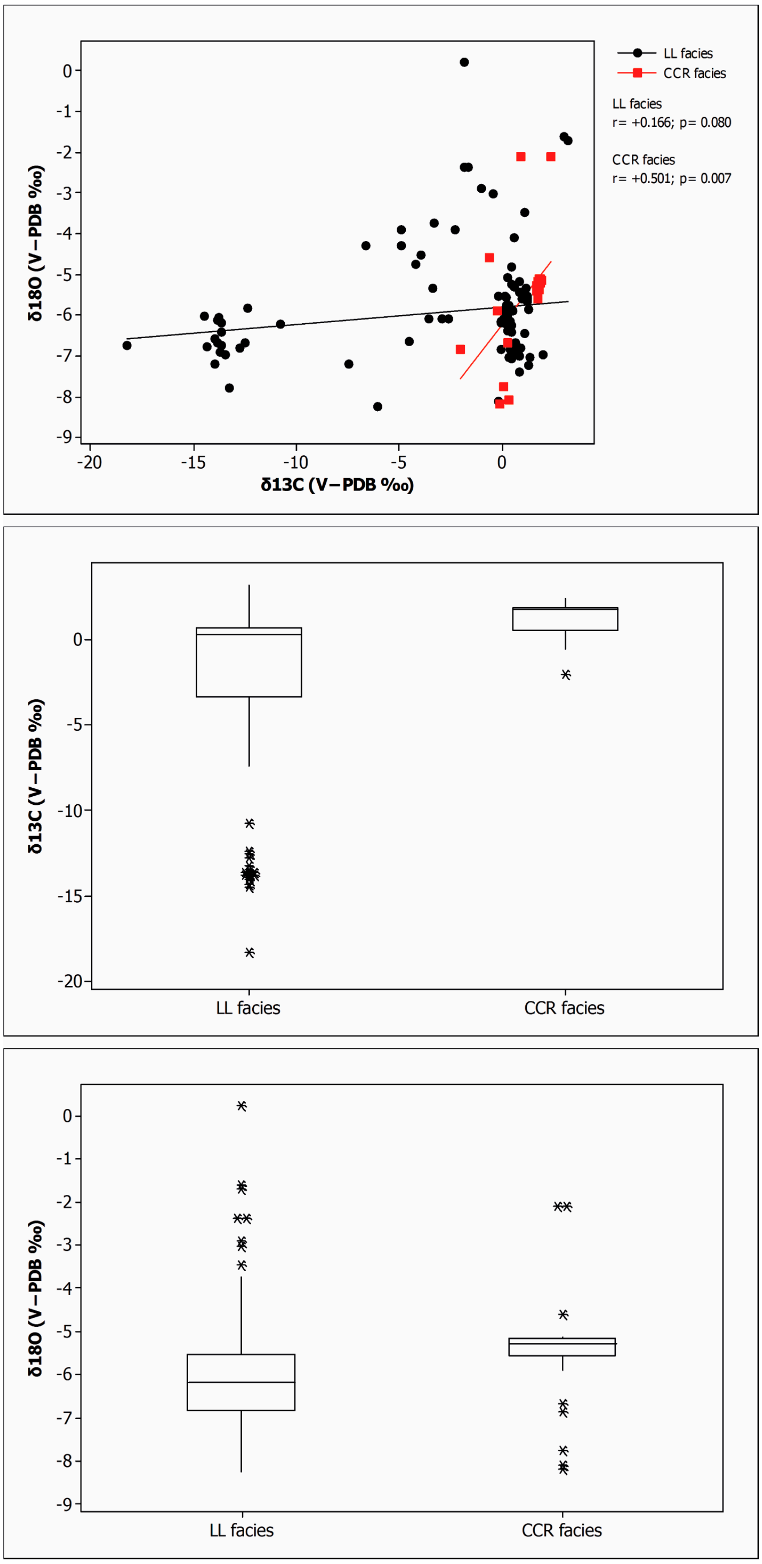

Figure 9. Scatterplot and boxplots for δ[18]O and δ[13]C values of the two main depositional facies of the carbonate unit C6 of the Crato Formation: laminated limestone facies (LL) and claystone–carbonate rhythmite facies (CCR), showing that sample discrimination per facies is not suitable for the wide spectrum of laminated limestones.

6. Discussion—Implications for Forensic Investigation of Fossil-Bearing Limestone

The stable oxygen and carbon isotope data from the Crato Formation limestone showcased in this study highlight the importance of accurately grouping these measurements based on geographical location and stratigraphic level (Figures 5–8), focusing on the limestone beds harboring the most prized fossils ("sete cortes" reported as 7 Cuts in Figure 8).

Historically, the fossils of the Crato Formation have been collected with minimal (when any) control over their geographical and stratigraphic origins [33], leading to limitations in scientific discussions regarding their spatial distribution, habitat, age, stratigraphic correlation, and their relevance in forensic investigations. Notwithstanding, recent studies have made strides in addressing this issue by pinpointing their most probable occurrence locations, primarily through field research and ethnostratigraphic surveys [32,63]. Corecco et al. [63] compiled location data for specimens through interviews with quarry workers engaged in the commercial extraction of the Crato Formation limestone for cladding materials, known as "Pedra Cariri", which are also instrumental in discovering new fossils. These efforts have revealed that the majority of valuable specimens are unearthed from the basal layer of carbonate unit C6 ("sete cortes" ethnostratum) in quarries south of Nova Olinda city [45,57,72]. Therefore, the isotopic characterization of this ethnostratum emerges as a crucial tool for forensic investigation, as statistical analysis reveals substantial isotopic variability when data from the literature are not meticulously grouped based on strict geographical and stratigraphic criteria (Figures 5–7). To the untrained eye, fossiliferous limestones from the Crato Formation can easily be mistaken with those from other regions, such as the Green River Formation (USA; Figure 3C), as well as with fossils from formations within Brazil, like the Codó Formation (Parnaíba Basin; Figure 3B), and even from carbonate units C1 to C5 within the Crato Formation itself. Moreover, it has been established that grouping by depositional facies within the Crato Formation, specifically in LL laminated limestone and CCR claystone–carbonate rhythmite facies, as recognized from previous studies, results in more scattered isotopic data compared with those confined to the "sete cortes" ethnostratum (Figure 9). Additionally, relying solely on depositional facies for grouping proves to be relatively unreliable, undermining the credibility and practicality of the forensic investigation, unless supplemented by microscopic petrographic examination of thin sections.

Based on the aforementioned assumptions and the examined data extracted from prior literature, particularly sourced from Nova Olinda and the Tatajuba Quarries, the "sete cortes" ethnostratum (Figure 8, Table 2) displays the following statistical patterns in its oxygen $\delta^{18}O$ and carbon $\delta^{13}C$ isotopic composition, respectively: arithmetic means of −5.94‰ and +0.90‰; standard deviations of 0.76‰ and 0.61‰; medians of −5.89‰ and +0.73‰; and IQRs of 1.47‰ and 1.24‰.

This preliminary attempt of using the oxygen and carbon isotopic signature of fossil-bearing limestones can prove to be useful in tracing illegal fossil trafficking and could be applied to other valuable fossils subjected to smuggling to develop an international database made available to public research and police institutions. This pilot study should be considered an initial and foundational proof of concept towards the development of a reliable, non-destructive, and relatively rapid and low-cost tool for determining the geographic origin of fossil specimens from the Crato Formation and other fossiliferous carbonate units. The $\delta^{18}O$ and $\delta^{13}C$ values, appropriately grouped for geographic location and stratigraphic levels, should be integrated with other geochemical parameters of the fossil-bearing limestone beds, such as trace element concentrations combined with microfacies and diagenetic investigation through petrographic analysis of thin sections. The integration of additional geochemical and petrographic screening techniques may better constrain the geographic attribution of the fossils, but it reduces the efficiency of the proposed approach based solely on oxygen and carbon isotope signatures by increasing the costs and timing of the identification process.

The limited dataset presented here, collecting $\delta^{18}O$ and $\delta^{13}C$ measurements from the Crato, Codó, and Green River Formations, could be enriched with additional geochemical data from laminated limestones harboring vertebrate fossils from the Crato Formation and other lacustrine fossiliferous strata stored in public institution collections and museums worldwide. The expansion of this preliminary $\delta^{18}O$ and $\delta^{13}C$ dataset, integrated with diagenetic and other geochemical parameters, will be mandatory to validate the proposed method and to fulfill the goal of contrasting illegal fossil trades through the development of a globally accessible database. This database should provide paleontological, lithofacies, petrographic, diagenetic, and geochemical (e.g., oxygen and carbon isotopes and trace element concentrations) data along with essential supporting information, including detailed descriptions of the analytical methods used, the geological and stratigraphic context of each sample, and accurate georeferencing of the collection sites.

According to Brazilian legislation, the paleontological heritage within Brazilian territory is regarded as property of the Federal Government, and the Federal Police is the institution responsible for investigating fossil trafficking and illegal extraction [38,39]. Hence, the population and management of such a database of fossiliferous carbonates might be entrusted to the National Institute of Criminalistics, which serves as the central forensic body in Brazil, as prescribed by Article n. 158-E of Federal Decree-Law 3689 of 3 October 1941 [73]. To ensure the chain of custody of the data, powder samples of the host limestones may be stored for counter-evidence, as per Article 158-A of Federal Decree-Law no. 3689 of 3 October 1941 [73]. National and foreign researchers affiliated with Brazilian research institutions may be granted access to this centralized database in accordance with Decree No. 98.830, of 15 January 1990 [41]. Scientific research institutions will be invited to contribute any new data of the same nature obtained in the course of their research, thereby ensuring the continuous expansion and updating of the database.

7. Conclusions

This pilot study presents statistical parameters derived from the published literature concerning the oxygen and carbon stable isotope values extracted from the basal section of the carbonate unit C6 within the Aptian (Lower Cretaceous) lacustrine limestone succession of the Crato Formation. This distinctive interval, known as the "sete cortes" ethnostratum, comprises a fossiliferous laminated limestone bed (fossil lagerstätte) and serves as a repository for vertebrate fossils, highly sought after in illicit trade. Despite the heterogeneity observed in the $\delta^{13}C$ and $\delta^{18}O$ values across the entire Crato Formation, the calculated statistical parameters pertaining to the C6 fossiliferous "sete cortes" ethnostratum are deemed robust. This assessment also accounts for potential overlaps in isotopic values with other fossiliferous lacustrine laminated carbonates as the Codó Formation (Lower Cretaceous, Parnaíba Basin, Brazil) and the Green River Formation (Eocene, UT and WY, USA). Various data groupings within the Crato Formation, categorized by geographical and stratigraphic criteria, underscore the presence of a statistically distinct core of isotopic values, notably characterized by relatively narrow standard deviations and interquartile ranges in measures of central tendency and data variability.

Originally, published isotopic analyses of the database utilized in this study were primarily directed towards non-paleontological and non-forensic objectives, often utilizing samples from the Crato Formation that lacked fossils. Consequently, the established patterns ideally warrant future validation through isotopic analyses, adding other geochemical parameters such as trace elements integrated with petrographic and diagenetic analyses, on limestone slabs harboring vertebrate fossil specimens, including those documented and housed in public institutions. This validation seeks to ascertain whether these patterns align with reliable isotopic signatures that can serve as a supplementary tool in the geographical identification of confiscated fossils, thereby bolstering forensic investigations.

Author Contributions: Conceptualization: M.A.P.d.S., D.R.d.N.J. and A.M.B.; methodology: D.R.d.N.J. and G.D.P.; software: M.A.P.d.S. and D.R.d.N.J.; validation: M.A.P.d.S., D.R.d.N.J. and G.D.P.; formal analysis: M.A.P.d.S.; investigation: M.A.P.d.S.; resources: D.R.d.N.J., A.M.B. and G.D.P.; data curation: M.A.P.d.S. and D.R.d.N.J.; writing—original draft preparation: M.A.P.d.S. and D.R.d.N.J.; writing—review and editing: D.R.d.N.J., A.M.B. and G.D.P.; visualization: D.R.d.N.J. and G.D.P.; supervision: D.R.d.N.J. and G.D.P.; project administration: D.R.d.N.J.; funding acquisition: D.R.d.N.J., A.M.B. and G.D.P. All authors have read and agreed to the published version of the manuscript.

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Conflicts of Interest: The authors declare no conflicts of interest.

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