

# DATING PRE-HISTORIC PAINTED FIGURES FROM THE SERRA DA CAPIVARA NATIONAL PARK, PIAUÍ, BRAZIL

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**Abstract.** Painted rock art figures from the Boqueirão da Pedra Furada and Toca do Paraguaio shelters (Serra da Capivara, Piauí, Brazil) have been recently analysed using various recognised scientific techniques. After the geochemical results, the samples that revealed carbonate were used for the dating process. Uranium-series disequilibrium dating (U/Th) methods were applied to two of those samples: sample 4 from Toca do Paraguaio (TP04) (a dark red geometric figure), and sample 2 from Toca do Boqueirão da Pedra Furada (BPF2) (a white circular figure). The dates obtained from the U/Th dating provided maximum ages of the paintings. These dates also allowed indirect association of the painting events with the data retrieved from the archaeological excavation. In this paper, we consider, albeit briefly, the archaeological history of the sites and their context into a broader landscape and suggest that geometric figures belong to the early Holocene.

#### Introduction

One of the largest concentrations of painted rock art regions globally is found within the Brazilian state of Piaui, an area known as the Serra da Capybara National Park (SCNP) (Fig. 1). The hundreds of thousands of figures known here appear divided between various figurative styles and various broad chronological phases (Guidon 1989; Nascimento 2009; Nash 2009; Pessis et al. 2010). Many rock art shelters have been studied since the 1970s, when researchers began to work in the field systematically (Guidon 1975; Arnaud et al. 1984; Guidon and Delibrias 1986; Pessis 1987).

Archaeological excavations were initially carried out with the primary objective of contextualising the accompanying graphic images that were present on many of the cave and rockshelter walls. The development of excavations has led to several new research topics involving traditional and innovative techniques to record the archaeology and rock art (Lahaye et al. 2013; Boëda et al. 2014; Lourdeau 2019).

The Serra da Capivara covers around 129 100 hectares and has a minimum of 1300 archaeological sites in its landscape, and of those over 600 painted rock art sites, with an extra thirty sites that contain petroglyphs. Additionally, 87 more sites have both paintings and petroglyphs.

The setting for this rock art tradition includes various floras and faunas that inhabit a dramatic geomorphological backdrop (Nascimento 2009; Buco 2012). The peripheral areas of the Serra da Capivara comprise a flat, partly cultivated brushwood plateau called *Sertão* (Caputo and Lima 1984). This semi-arid landscape encompasses an upland massif of exposed, mostly unwooded and weather-beaten Silurian sandstone, which has been eroded, first by water, then by wind and rain over time (Caputo and Lima 1984). The region occupied by the SCNP is found at the eastern edge of the Parnaíba Sedimentary Basin and the Peripheral Depression of São Francisco. A slope with a 200–250 m drop forms the dominant relief of the region (Pellerin 2014). Within the hinterland, between the sandstone massif and the plateau, is the *caatinga* scrub forest (Guérin and Faure 2014).

In geological terms, the sandstone massif was formed some 430 million years ago when the region was a warm tropical sea (Nash 2009). Hidden and dispersed along the base of this giant massif is a series of rockshelters where archaeological material and rock art is present. The rock art of the Serra da Capivara National Park is considered one of the richest testimonies of symbolic behaviour of pre-Historic communities that inhabited the landscape around the Serra da Capivara massif (Nash 2009).

#### **Contextual issues**

Absolute dating of rock art is not without its difficulties. Currently, chronometric dating methods can extract (rare) organic material from pigments if present. However, organic residues from pigments must be large enough to date. The size of such residues can

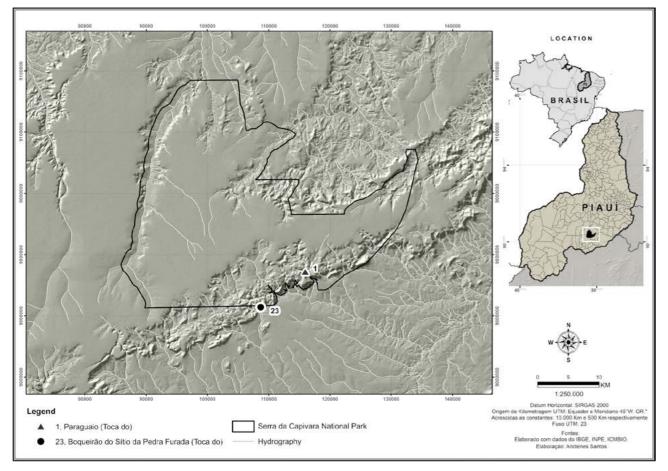


Figure 1. Location of the Serra da Capybara National Park.

create conservation issues, especially in terms preservation of rock surfaces where samples have been taken. The age of many paintings remains difficult to substantiate. In the absence of direct dating, archaeologists have usually assigned an age to paintings based upon excavations near the rock wall or below it (Fontugne et al. 2013; Lourdeau 2019).

The ability to accurately date rock art has important implications in understanding the origins of this unique artistic endeavour, the symbolism it may have projected, and the cognitive abilities of communities that inhabited this and other ancient landscapes (Pearce and Bonneau 2018).

When researchers lack opportunities for direct dating by implementing analytical methods, attempts at dating the rock art can broadly be described as falling into the following categories (all of which yield questionable results):

- Iconographic determination
- Stylistic claims
- Presumed technique of execution •
- Association with archaeological finds through ٠ excavation
- Topographic proximity
- Weathering and patination studies ٠
- Superimposition of motifs
- A combination of two or more of these approaches •

Using a combination of these and recently introduced scientific methods, rock art researchers, in general, are in a favourable position to gain a better understanding of how and when rock art was created.

The uranium-series disequilibrium (U/Th) dating method has proven its efficacy and reliability for measuring the dating of marine corals and continental speleothems (flowstones), secondary carbonate deposits (Pons-Branchu et al. 2014). Recently, improvements in analytical techniques, including TIMS (thermo-ionisation mass spectrometry) and then MC-ICPMS (multicollector-inductively coupled plasma mass spectroscopy) and laser ablation, have permitted the dating of minute samples and augmented the potential of this method (Pons-Branchu et al. 2014). 'Absolute' dating methods have challenged some of our ideas about the chronology of rock art in Europe, but the application of indirect methods (including the use of U/Th dating) is not without its problems (White et al. 2020).

In terms of dating oxalate material, some examples have been previously reported (Steelman et al. 2002; Watchman et al. 2005; Ruiz et al. 2012; Jones et al. 2017; Di Maida et al. 2020; Brumm et al. 2021) and in different contexts (Watchman 1991, 2004; Watchman and Campbell 1996; Mazel and Watchman 1997, 2003; Watchman et al. 1997, 2000). Despite the proximity of dates obtained from finely laminated crusts, criticism concerning the origins of carbon, and the contemporaneity of oxalate formation with the associated painted image, have led to uncertainties about the dating of rock surface coatings (Gillespie 1997; Rosenfeld and Smith 1997). In the uranium-thorium series dating method, the age of calcite formation is assumed to provide a minimum age (terminus ante quem) for the underlying paintings or engravings or a maximum age (terminus *post quem*) when it is the surface material that is dated. However, many issues have been raised about this method (Plagnes et al. 2003; Bednarik 2012; Clottes 2012; Fontugne et al. 2013; Pons-Branchu et al. 2014; Tang and Bednarik 2021). According to some authors (Pons-Branchu et al. 2014), these issues are linked in part with the development of calcite deposits within the caves and rockshelters concerning the effects of moisture that may distort results by leaching the uranium, for example (Plagnes et al. 2003). Difficulties are also linked to a possible initial <sup>230</sup>Th content from detrital material such

as clay that must be considered when correcting the results. To these methodological difficulties, we must add those associated with sampling the re-precipitated calcite, which must be free of any contamination by the surrounding limestone, any inclusion of which would distort the data obtained (Fontugne et al. 2013; Pons-Branchu et al. 2014).



#### The focus of research

The Toca do Paraguaio (Fig. 2) is a rockshelter placed among a deep and Figure 3. Toca do Boqueirão da Pedra Furada. © SG, 2019. narrow valley on the top of the plateau

of Bom Jesus do Gurguéia (Arnaud et al. 1984), near to the municipality of Coronel José Dias, and inside the Serra da Capivara National Park. This site is around 70 m in length, oriented north-south, and opens out towards the east. The site stands around 420 m above sea level and is surrounded by scrub vegetation (Guidon 1975; Gomes et al. 2019). The site is divided into two sections based on the local geology, described as upper and lower floor levels. The upper level forms a sheltered area that has been naturally cut into a conglomerate deposit. This deposit overlies a sandstone layer that forms the lower section of the site (Guidon 1975). In terms of rock art assemblage, painted figures are the most numerous, with anthropomorphous figures dominating the assemblage (Santos 2013).

Toca do Boqueirão da Pedra Furada shelter (Fig. 3), one of the best-known sites in the archaeological area, is a rockshelter occupied by hunter-gatherers from the Late Pleistocene to Middle Holocene times (Boëda et al. 2013, 2014). It stands out because of its impressive



Figure 2. Toca do Paraguaio, front view. © FUMDHAM Archive, 1978.

rock art (Fig. 4), of the important archaeological research already done and of the alleged antiquity of the occupation. This shelter has always been the focus of attention for potential pre-Clovis occupation layers (Guidon and Delibrias 1986; Parenti 2001; Santos et al. 2003). Archaeological remains retrieved from this site were the object of several research projects, most concerning lithic artefacts, combustion structures, stratigraphic context and rock art (Guidon 1989; Guidon et al. 1994; Chaves 2000; Parenti 2001; Felice 2002; Santos et al. 2003; Boëda et al. 2013; 2014; Mota and Scheel-Ybert 2019).

The Toca do Paraguaio shelter is a site that represents the Serra da Capivara style. This distinct style is set within the 'Nordeste tradition' and presents a figurative set of over 900 abstract and figurative painted images (Santos 2013). Most of these images were initially sketched as outlines (a feature that is possible to see using DStretch) and were later infilled with red pigment made from local haematite sources.



Figure 4. Panels from Toca do Boqueirão da Pedra Furada. © SG, 2019.

The artists creating these images were concerned with two- and three-dimensional images as different shades or hues of red were used to highlight certain parts of the image. The tools used in each production event included large and small brushes. Smaller brushes were employed to create smaller images, while larger figures, such as cervids, 'rheas' and 'people' were produced using larger brushes (Santos 2013). The rock art panel sections, especially within the upper part of the rockshelter, comprise abstract or geometric motifs that include colour stains, handprints, lines, and squares. Based on current literature, these figures have never been dated (Fig. 5). Their potential chronology is based upon the stratigraphy of two nearby human burials where red and orange haematite were found, along with diagnostic lithics and the remains of floral evidence (Santos 2013). The human occupation in Toca

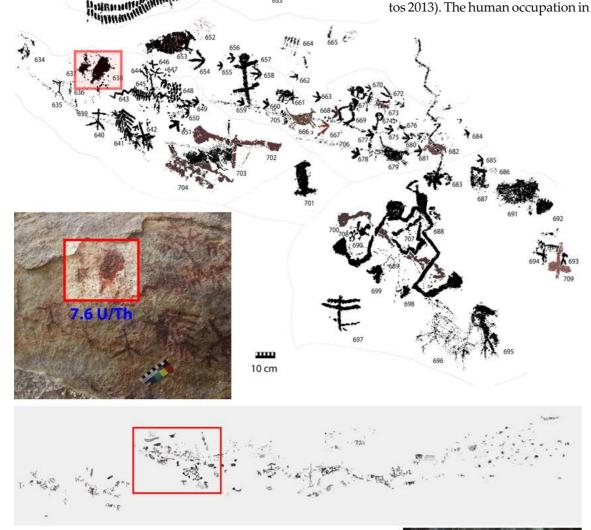
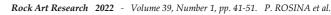


Figure 5. Panels sections with paintings from Toca do Paraguaio shelter. Position of the dated figure within the shelter. © TDS, 2021.



Excavation Unit	Layer	Radiocar- bon ages	Mate- rial	Labora- tory	Sample	Calibrated ages/Cal- ib software	Calibrated ages/Ox- cal software
Excavation 1	B1	8600±100 bp	Charcoal	MC-2510	PR1-B1	9310–9892 Cal BP	9311–9894 Cal BP
Excavation 1	B2	8670±120 bp	Charcoal	MC-2480	PR2-B2	9427–10142 Cal BP	9430–10145 Cal BP
Excavation 1	B2	8780±120 bp	Charcoal	MC-2511	PR3-B2	9537–10158 Cal BP	9537–10160 Cal BP
Excavation 1	С	7000±100 bp	Charcoal	MC-2509	PR4-C	7612–970 Cal BP	7612–7972 Cal BP
Excavation 2	Level 3	7040±50 bp	Charcoal	BETA- 232672	PR5-C	7699–7937 Cal BP	7698–7938 Cal BP

 Table 1. Toca do Paraguaio, calibrated ages.

do Paraguaio is dated to two periods; an earlier one includes dates of 9537-10158 Cal BP, 9427-10142 Cal BP and 9311–9894 Cal BP, and a second one with dates of 7612–7970 and 7699–7937 Cal BP (the conventional <sup>14</sup>C ages from Toca do Paraguaio have been calibrated at 2 sigma with 95.4% probability on the Calib 8.2 software developed by Stuiver and Reimer). The calibration curve used in the process was the SHCal20 (Hogg et al. 2020). More details can be seen in Table 1. However, we stress caution in connecting the rock art, the pigments used and the burials.

Regarding the geochemical analysis on applied pigments, several studies have been undertaken in several sites whose most famous are the Boqueirão da Pedra Furada (BPF), Toca do Baixão do Perna I, Toca do Vento and Toca do Arapoá do Gongo (Lage 1990, 1997, 1998). In this study, the authors sought to understand more precisely the pigments used in the rock art of southeast Piauí, their chemical and mineralogical composition and their relationship with the rock support. Problems related to the origin of the raw material used in the manufacture of these pigments were also addressed, together with the reconstruction of the preparation techniques and their application on the rock support (Lage 1997).

Recent fieldwork and research at the Boqueirão da Pedra Furada and Toca do Paraguaio shelters have included the analysis of extracted pigment samples, and the results were previously published by the authors (Gomes et al. 2019). The samples were taken using sterile tungsten scalpels, latex gloves and masks to avoid any possible contamination and then stored in Eppendorf tubes. All sample sizes were kept to a minimum, according to the standards of recent advances in analytic techniques (Gomes et al. 2013, 2015). All samples were taken in the form of powder and followed the code of ethics and guidelines for the practice of the American Institute for Conservation, A.I.C. 2015.

Those samples were analysed employing a multiproxy archaeometric approach using x-ray micro-fluorescence, Raman spectroscopy, scanning electron microscopy, stereomicroscopy observation and high temperature-gas chromatography (HT-GC) (for a full description of samples, methods and results, please refer to Gomes et al. 2019). At Boqueirão da Pedra Furada shelter, three samples were extracted from figures

that were made of grey, red and white pigments. Not surprisingly, each sample had distinctive characteristics. Sample BPF1 was taken from a grey pigment that depicted a 'deer'. Sample BPF2 was extracted from a white motif, defined as a circular abstract mark. Elements of this composition were superimposed over a red figure. Sample BPF3 was taken from a red figure that was interpreted as another 'deer'.

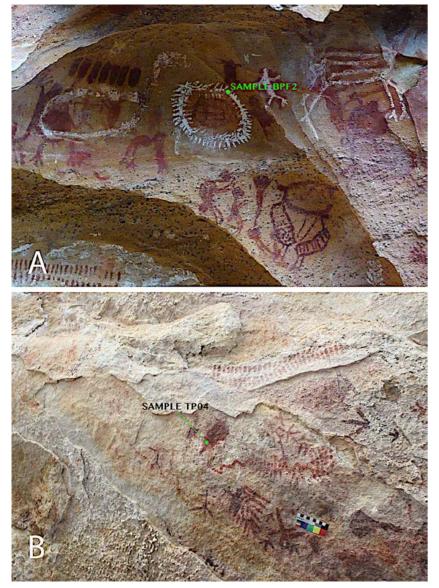
Four samples were also extracted from Toca do Paraguaio shelter. They were from figures within the lower and upper sectors of the site. Sample TP01 was extracted from an anthropomorphous figure, whilst sample TP02 was from a zoomorphic figure. Within the upper section of the site, sample TP03 was secured from a pigment smear that represented a geometric form, and sample TP04 was from a clear infilled geometric dark-red motif. Based on the Raman, XRF and SEM/EDS sample results published in the previous studies (Gomes et al. 2019), the dominant pigment material used for paintings was red ochre (haematite).

For samples TP03 and TP04 from Toca do Paraguaio, the main characteristic was the significant presence of calcium (Ca), particularly within sample TP04, in which calcium is the most abundant chemical element detected (Gomes et al. 2019).

The presence of calcium and haematite has also been detected in samples from nearby Toca da Pinga da Escada (Cavalcante et al. 2008) and earlier studies in the Serra de Capivara pigments in Toca do Boqueirão do Sítio da Pedra Furada (BPF) (Lage 1998; Lage et al. 2007). When considering the preservation of each panel, we postulate that the presence of calcium (Ca) within our samples could be associated with a natural concretion formation; this is despite the negative results for oxalate in the micro-Raman spectra analysis. An alternative (but weaker hypothesis) is that calcite has been deliberately used in the pigment preparation process (for whatever reason) (Gomes et al. 2019).

### Methods and materials

Two of these samples from the cited previous publication (Gomes et al. 2019) were found to contain enough preserved material to be applied to uranium-series disequilibrium (U/Th) dating methods. The dating process was decided only after the geochemical results. The samples that revealed carbonate were used for the dating process: sample 4 from Toca do Para-



*Figure 6.* Sampled figures that were dated through uranium-series disequilibrium (U/Th) dating methods: A) sample 2 from Toca do Boqueirão da Pedra Furada (white circular figure); B) sample 4 from Toca do Paraguaio (dark-red geometric figure). © TDS, 2013.

guaio (TP04) (a dark-red geometric figure), and sample 2 from Toca do Boqueirão da Pedra Furada (BPF2) (a white circular figure) (Fig. 6). The concretions positioned in both previously identified samples were thus dated despite the known analytical problems of dating gypsum speleothems with the U-series technique (Sanna et al. 2010).

The carbonate samples were first cleaned with a dental drill to remove the contaminations on the surfaces. Then, samples were washed ultrasonically in 18.2 M $\Omega$  ultrapure water and dried overnight in an oven at 60°C. The sample was weighed (15-35 mg for each) and dissolved in 7N HNO, in a Teflon beaker containing a known quantity of a <sup>229</sup>Th-<sup>233</sup>U-<sup>236</sup>U triple spike. One drop of HClO was added to the sample solution to decompose the organic material. The sample-spike mixture was heated overnight on a hot plate at 120°C to equilibrate. U and Th were then separated from each other and other cations, bypassing the sample solution through a U-TEVA resin column following the procedure of Douville et al. (2010). Sample matrix elements were first excluded by rinsing with 3N HNO<sub>2</sub>. Subsequently, Th was eluted using 3N HCl, and finally, U was eluted using 0.5N HCl. One drop of HClO, was added to the U and Th fractions, respectively, to remove any organic material derived from the U-TEVA resins. The U and Th solutions were evaporated to dryness and then dissolved in a mixture of 0.5 N

	.ab. Code	Shelter	Refe- rence sample	Sam- pling	Figure	<sup>238</sup> U [ng/g]	<sup>232</sup> Th [ng/g]	δ <sup>234</sup> U [‰]	( <sup>230</sup> Th/ <sup>238</sup> U)	( <sup>230</sup> Th/ <sup>232</sup> Th)	Age [ka] ª	$ \begin{array}{c} \delta^{234}U_{i} \\ [\%]^{b} \end{array} $	Corr. Age [ka BP] °
ę	SP-1	Toca Pa- raguaio	Sample 4	Dark red pig- ment	Geo- metric figure	162.9±0.1	28.47±0.05	3549.6±4.8	0.3570±0.0013	6.24±0.03	8.75±0.04	3627±8	7.6±0.6
9	SP-4	Bo- queirão Pedra Furada	Sample 2	White pigment	Cir- cular figure	115.3±0.2	61.31±0.11	4057.9±11.0	0.6100±0.0033	3.50±0.02	13.72±0.08	4182±21	10.7±1.5

<sup>a</sup> U-series ages were calculated with half-lives of 75 584 yr for <sup>230</sup>Th (Cheng et al. 2013) and 245 620 yr for <sup>234</sup>U (Cheng et al. 2013),  $1.4 \times 10^{10}$  yr for <sup>232</sup>Th (Holden 1990), and  $4.47 \times 10^9$  yr for <sup>238</sup>U (Goldstein et al. 1989).

<sup>b</sup> Back-calculated initial  $\delta^{234}$ U.

<sup>c</sup> U-series ages were corrected with the assumption of the initial <sup>230</sup>Th/<sup>232</sup>Th atomic ratio of  $4.4\pm2.2\times10^6$ , a value for a material at secular equilibrium, with the bulk earth <sup>232</sup>Th/<sup>238</sup>U value of 3.8 and with an assumed error of 50%. The ages are given at the 'BP' scale, before 1950 CE.

 Table 2. MC-ICPMS U-series dating results obtained on the carbonate samples from Toca do Paraguaio and Boqueirão da Pedra Furada.

  $HNO_3$  and 0.01 N HF for U and Th isotopic analysis using a Neptune MC-ICPMS with the combination of Faraday cups and an ion counter system, similar to the protocol described by Shao et al. (2019).

#### **Results and discussion**

The Th/U series dates are presented in Table 2. All the <sup>230</sup>Th/<sup>232</sup>Th activity ratios are much lower than 20, indicating severe detrital contamination. Given the low <sup>230</sup>Th/<sup>232</sup>Th activity ratios, the obtained ages probably represent the maximum ages for the samples.

U-series dating of the carbonate samples associated with the abstract figures from BPF and TP provided an age spanning from  $10.7\pm1.5$  (from BPF) to  $7.6\pm0.6$ years (from TP) that are according with previous chronological attributions by style.

Serra da Capybara National Park research includes the fundamental question of who the first settlers on the American continent were and when this initial colonisation phase occurred (Nash 2009; Lahaye et al. 2013; Lourdeau 2019). The rock art paintings found in large numbers in the region are considered the richest testimonies of symbolic behaviour within the Serra da Capivara. The very rich rock art palimpsest in this remote region of northeast Brazil and the potential of the indirect dating from several rock art shelters suggests that colonisation and subsequent settlement were indeed complex (Lahaye et al. 2013, 2015, 2019). The dating of several major rock art sites, including Toca do Boqueirão da Pedra Furada, by one of the

Year	Site	Material	Type of rock art	Method	Results	Reference	
1991	Toca da Bastiana	Calcite	Red painted anthro- pomorphous figure	EPR	27 ka	Baffa (1991); Wata- nabe et al. (2003)	
2003	Toca da Bastiana	Calcite	Red painting	XRF, TL, EPR	36 ka	Watanabe et al. (2003)	
		Calcite extracted from the calcite layer dated by	Red painting	Plasma-chem- ical extraction	First measurement: $2540\pm60$ years BP (AA-42663 on 07-07-01), corrected for a $\delta^{13}$ C of -11.7 %; second measurement: $2470\pm40$ years bp (AA-42664 on 08- 05-01), corrected for a $\delta^{13}$ C of -11.6 %: combined age of 2490±30 years bp.	Steelman et al. (2002); Rowe and Steelman (2003)	
	Toca da Bastiana	Wata- nabe et al. (2003)		Plasma-chem- ical extraction + AMS radio- carbon	3730±90 BP		
			Sample 1		1880±60 BP		
		Calcite	Sample 2		2280±110 BP		
		Calcite	Sample 3		2970±300 BP	Rowe and Steel- man (2003)	
			Sample 4		3320±50 BP		
	Toca do Sítio do Meio	Calcite	Pigment	AMS radio- carbon	2700±110 BP		
	Pedra	Calcite	Pigment		2120±110 BP		
	Furada	Calcile	Pigment		3570±50 BP		
Toca da Extrema		Calcite	Pigment		1230±50 BP		

authors (NG) has managed, through deep open area excavation, to date fallen painted rock fragments to at least 29 000 years BP (Guidon and Delibrias 1986; Lage 1997).

Some dates have been obtained from various sites within the Serra da Capivara rock art using various methods (Table 3). One of the first attempts (Watanabe et al. 2003) reports TL and EPR dating to obtain a minimum age of 36 ka BP. Around the same time, Steelman et al. (2002) secured a <sup>14</sup>C age of 3730±90 BP on organic material in the same painting that Watanabe et al. (2003) used in their TL and EPR dating. In addition, Steelman et al. (2003) obtained a minimum age of 2490±30 years BP for an oxalate accretion overlying the same painting. Radiocarbon dates on paint residues from four other images in the same shelter were 1880±60, 2280±110, 2970±300 and 3320±50 BP. Dates on four other paintings from nearby shelters yielded ages of only a few thousand years: at Toca do Sitio do Meio, 2700±110 BP; at Pedra Furada, 2120±110 and 3570±50 BP; and at Toca do Extreme, 1230±50 BP (Rowe and Steelman 2003). Later, <sup>14</sup>C analysis was used to date thin calcite precipitates covering paintings that were cross-dated using uranium-series disequilibrium in three rockshelters (Toca da Bastiana, Toca do Serrote de Moendas and Toca da Gameleirinha). Fontugne et al. (2013) obtained ages ranging between 1770 and 4390±30 <sup>14</sup>C yr BP (1554-5031 cal BP) and between 4520±30 and 11805±35 <sup>14</sup>C yr BP for Toca da Bastiana and Toca da Gameleirinha, respectively.

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τu								
2006	Toca do		Black Pigment	?	?			
2008	Serrote das Moendas	Calcite	?	?	?			
			Calcite overlying external paintings 1	AMS ra- diocarbon dcf=5%	?			
			Calcite overlying external paintings 3	AMS ra- diocarbon dcf=5%	1178–1296 (1252) BP			
			Calcite overlying external paintings 4	AMS ra- diocarbon dcf=5%	4418–4783 (4499) BP			
	Toca da Gamelei- rinha	melei- Calcite	Sample 1 subsample A - calcite at the bedrock contact	AMS ra- diocarbon dcf=5%	13 471–13 789 (9863) BP			
			Sample 1 subsample B - compact calcite fraction overlying contact with the	AMS ra- diocarbon dcf=5%	9696–10 149 (9863) BP	Fontugne et al. (2013)		
				U/Th series	12.28±0.186 – 10.56±0.289 ka bp			
			bedrock		12.28±0.206 - 10.56±0.309 BP			
2013			Sample 1 subsample C - calcite with a cau- liflower-like surface	AMS ra- diocarbon dcf=5%	7507–7656 (7579) BP	(2013)		
				U/Th series	12.24±0.761-4.81±1.163 BP			
			Sample 2 subsample D - compact calcite fraction overlying contact with the bedrock		9312–9524 (9453) BP			
			Sample 2 subsample E - compact calcite fraction overlying contact with the bedrock	AMS ra- diocarbon dcf=5%	4430–4801 (4552) BP			
	Toca do Serrote das Moendas	ote das Calcite	Calcite overlying external paintings 4	AMS ra- diocarbon dcf=5%	964–1168 (1028) BP			
			Compact calcite fraction overlying contact with bedrock	AMS ra- diocarbon dcf=5%	?			

*Table 3.* Dates from various sites within the Serra da Capivara rock art using a variety of methods.

These results were considered in good agreement with those obtained by Rowe and Steelman in 2003. At Toca do Serrote de Moendas 2, contrasting dates were obtained: 31860±210 and 1590±30<sup>14</sup>C yr BP (Fontugne et al. 2003).

#### Proposed date rages based on stylistic analysis

The chrono-stylistic classification of the rock art in the Serra da Capivara is based upon excavated rockshelters in which remains of paintings and panels have been dated in Toca do Boqueirão do Sítio da Pedra Furada (BPF), Toca do Baixão do Perna I (TBP-I), Toca do Fundo do Baixão da Pedra Furada (TFBPF), Toca do Sítio do Meio (TSM), Toca do João Leite (TJL) and many others (Buco 2012; Pessis 1992; Dos Santos 2020). The cited authors assume that figures displaying certain distinct styles emerged during the Pleistocene-Holocene transition, dated between 12000–10000 BP or maybe during the early Holocene, between 10 000-8000 BP (Santos 2020).

The figures considered the oldest graphic manifestations in the area comprise the Nordeste tradition, dating between 12000 and 6000 BP (Pessis 1999). These figures and motifs are based on several distinct styles that include the Serra da Capivara style, which falls within the Nordeste tradition (12000 and 6000 BP); the Serra Talhada style, between 10 000 and 7000 BP; the Serra Branca style, between 9000 and 6000 BP; and the Angelim style, between 6000 and 4000 BP) (Pessis 1987, 1992, 1999; Guidon 1989; Morales 2002, 2005; Buco 2012).

Based upon these date ranges, one can assume that some form of artistic endeavour was occurring within this region of South America between c. 12000 and 4000 BP; however, the authors express caution when considering such a wide date range. There is a requirement to obtain data based on direct dating of organic residues from paint samples, especially from

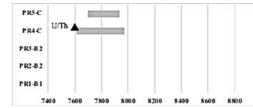


Figure 7. Updated chronology of Toca do Paraguaio with calibrated radiocarbon ages represented by the grey rectangles and the U/Th date for a red abstract figure seen in the black triangle.

sites that have undergone systematic research, such as the Toca do Paraguaio rockshelter.

Despite earlier results being considered problematic as they were perceived to be younger than the most recent occupations dated in the SCNP (Fontugne et al. 2013), the results presented here are considered consistent with the period statistically stipulated for the representations of geometrics (Table 1 and Fig. 7).

Although there are pre-Clovis dates for some archaeological layers in Pedra Furada and generally in the Serra da Capivara (Parenti 2001; Lahaye et al. 2019), the rock paintings that were dated seem to belong to a more recent phase.

#### Conclusion

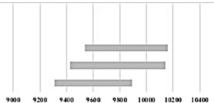
The U-series dating of the carbonate samples associated with the geometric figures from BPF and TP provided an age spanning from 10.7±1.5 ka (from BPF) to 7.6±0.6 ka (from TP), placing the rock art within the early Holocene. These results are roughly synchronous with dates obtained from the archaeological excavations, and the dating indicates an early Holocene presence, in good agreement with some previous studies (Rowe and Steelman 2003; Watanabe et al. 2003; Pessis and Guidon 2009; Fontugne et al. 2013).

However, the authors express caution as the dates obtained for the rock art only provide maximum dates. It could be the case that the rock art is considerably younger.

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#### REFERENCES

- ARNAUD, M. B., L. EMPERAIRE, N. GUIDON and J. PELLERIN 1984. L'Aire archéologique du sud-est du Piauí (Brésil). Edited by Recherche sur les civilisations. Paris. Synthèse 16(1): 1-118.
- BAFFA, O. Jr 1991. Unpubl. pers. comm. apud Watanabe, S., W. E. F. Avta and H. Hamaguchi 2003.
- BEDNARIK, R. G. 2012. U-Th analysis and rock art: a response to Pike et al. Rock Art Research 29(2): 244-246.
- BOËDA, E., C. LAHAYE, G. D. FELICE, N. GUIDON, S. HOELTZ, A. LOURDEAU et al. 2014. The peopling of South America: expanding the evidence. Antiquity 88(341): 954–955. https://doi.org/10.1017/S0003598X00050900.
- BOËDA, E., A. LOURDEAU, C. LAHAYE, G. D. FELICE, S. VIANA, I. CLEMENTE-CONTE et al. 2013. The Late-Pleistocene industries of Piauí, Brazil: new data, Paleoamerican Odyssey. In Paleoamerican Odyssey, pp. 445-465.
- BRUMM, A., A. A. OKTAVIANA, B. BURHAN, B. HAKIM, R. LEBE, JIAN X. Z. et al. 2021. Oldest cave art found in Sulawesi. Science Advances 7(3): 1-13. https://doi.org/10.1126/ sciadv.abd4648.
- BUCO, C. DE ANDRADE 2012. Arqueologia do movimento. Relações entre arte rupestre, arqueologia e meio ambiente, da pré-História aos dias atuais, no vale da Serra Branca. Parque Nacional Serra Da Capivara, Piauí, Brasil. Unpubl. Ph.D. thesis, Universidade de Trás-os-Montes e Alto Douro.
- CAPUTO, M. V. and E. CONDE LIMA 1984. Estratigrafia, idade e correlação do grupo Serra Grande-Bacia do Parnaíba. In Anais do XXXIII Congresso Brasileiro de Geologia, Rio de Janeiro, pp. 58-67.
- CAVALCANTE, L. C. D., M. C. S. M. LAGE and J. D. FABRIS 2008. Chemical analysis of red pigment in human bone. Quimica Nova 31(5): 1117-1120. https://doi.org/10.1590/ s0100-40422008000500034.

- CHAVES, S. 2000. Estudo palinológico de coprólitos pré-Históricos Holocenos coletados na Toca do Boqueirão do Sítio da Pedra Furada: contribuições paleoetnolôgicas, paleoclimáticas e paleoambientais para a região sudeste do Piauí - Brasil. Revista do Museu de Arqueologia e Etnologia 10: 103–120.
- CLOTTES, J. 2012. U-series dating, evolution and Neandertal. International Newsletter on Rock Art 64: 1–6.
- DOUVILLE, E., E. SALLÉ, N. FRANK, M. EISELE, E. PONS-BRAN-CHU and S. AYRAULT 2010. Rapid and accurate U-Th dating of ancient carbonates using inductively coupled plasma-quadrupole mass spectrometry. Chemical Geology 272(1-4): 1-11. https://doi.org/10.1016/j.chemgeo.2010.01.007.
- FELICE, G. D. 2002. A controvérsia sobre o sítio arqueológico Toca do Boqueirão da Pedra Furada, Piauí-Brasil. Fumdhamentos 1(2): 142–178.
- FONTUGNE, M., Q. SHAO, N. FRANK, F. THIL, N. GUIDON and E. BOEDA 2013. Cross-dating (Th/U-14C) of calcite covering prehistoric paintings at Serra da Capivara National Park, Piaui, Brazil. Radiocarbon 55(3): 1191-1198. https://doi. org/10.1017/s0033822200048104.
- GILLESPIE, R. 1997. On human blood, rock art and calcium oxalate: further studies on organic carbon content and radiocarbon age of materials relating to Australian rock art. Antiquity 71(272): 430-437. https://doi.org/10.1017/ S0003598X00085033.
- Gomes, H., P. Rosina, N. Guidon, C. Buco, T. dos Santos, L. VOLPE, C. VACCARO, G. H. NASH and S. GARCÊS 2019. Identification of organic binders in pre-Historic pigments through multiproxy archaeometric analyses from the Toca do Paraguaio and Boqueirão da Pedra Furada shelters (Serra da Capivara National Park, Piauí, Brazil). Rock Art Research 36(2): 214–221.
- GUÉRIN, C., M. A. CURVELLO, M. FAURE, M. HUGUENEY AND C. MOURER-CHAUVIRÉ 1996. A fauna Pleistocênica do Piauí (nordeste do Brasil): relações paleoecológicas e biocronológicas. Fundhamentos 1(1): 55-103.
- GUIDON, N. 1989. Tradições rupestres na área de São Raimundo Nonato. Clio Arqueológica 5: 5-10.
- GUIDON, N. and G. DELIBRIAS 1986. Carbon-14 dates point to man in the Americas 32,000 years ago. Nature 321(6072): 769-771. https://doi.org/10.1038/321769a0.
- GUIDON, N., F. PARENTI, M. F. DA LUZ, C. GUÉRIN and M. FAURE 1994. Le plus ancien peuplement de l'Amérique: le Paléolithique du nordeste Brésilien. Bulletin de La Société Préhistorique Française 91(4): 246–250; https://doi. org/10.3406/bspf.1994.9732.
- GUIDON, N. 1975. Peintures rupestres de Varzea Grande Piauí, Brésil. Edited by École des hautes études en sciences Sociales. Paris.
- HOGG, G. Alan, J. H. Timothy, H. Quan, G. P. Jonathan, C. S. M. TURNEY, J. SOUTHON, A. BAYLISS et al. 2020. SHCal20 Southern Hemisphere calibration, 0-55,000 Years Cal BP. Radiocarbon 62 (4): 759-78; https://doi.org/10.1017/ RDC.2020.59.
- JONES, T., A. L. VLADIMIR, P. L. KING, U. TROITZSCH, D. WES-LEY, A. A. WILLIAMS and A. NAYINGULL 2017. Radiocarbon age constraints for a Pleistocene-Holocene transition rock art style: the northern running figures of the East Alligator River Region, western Arnhem Land, Australia. Journal of Archaeological Science: Reports 11: 80-89. https:// doi.org/10.1016/j.jasrep.2016.11.016.
- LAGE, M. C. S. M. 1990. Etude archéométrique de l'art rupestre du sud-est du Piauí - Brésil. Unpubl. PhD thesis, Université Paris 1, Paris.

- LAGE, M. C. S. M. 1997. Análise química de pigmentos de arte rupestre do sudeste do Piauí. Revista Do Museu de Arqueologia e Etnologia Suplemento 2: 89–101.
- LAGE, M. C. S. M. 1998. Datações de pinturas rupestres da área do parna Serra da Capivara. Clio Arqueológica 13: 203-213; https://periodicos.ufpe.br/revistas/clioarqueologica/article/view/247061/35949.
- LAGE, M. C. S. M., L. C. D. CAVALCANTE and J. S. SANTOS 2007. Estudo químico de sedimentos arqueológicos do Parque Nacional Serra Da Capivara, Piauí – Brasil. Fundhamentos 6:106-114.
- LAHAYE, C., G. GUÉRIN, E. BOËDA, M. FONTUGNE, C. HATTÉ, M. FROUIN, I. CLEMENTE-CONTE et al. 2015. New insights into a Late-Pleistocene human occupation in America: the Vale da Pedra Furada complete chronological study. Quaternary Geochronology 30: 445-451; https://doi. org/10.1016/j.quageo.2015.03.009.
- LAHAYE, C., G. GUÉRIN, M. GLUCHY, C. HATTÉ, M. FONTUGNE, I. CLEMENTE-CONTE, J. C. SANTOS et al. 2019. Another site, same old song: the Pleistocene-Holocene archaeological sequence of Toca da Janela da Barra do Antonião-North, Piauí, Brazil. Quaternary Geochronology 49: 223-229; https://doi.org/10.1016/j.quageo.2018.03.006.
- LAHAYE, C., M. HERNANDEZ, E. BOËDA, G. D. FELICE, N. GUIDON, S. HOELTZ, A. LOURDEAU et al. 2013. Human occupation in South America by 20,000 BC: the Toca da Tira Peia Site, Piauí, Brazil. Journal of Archaeological Science 40(6): 2840-4287; https://doi.org/10.1016/j.jas.2013.02.019.
- LOURDEAU, A. 2019. The Serra da Capivara area and the first settlements in South America: a bibliographical review. Boletim do Museu Paraense Emilio Goeldi: Ciencias Humanas 14(2): 367-398; https://doi.org/10.1590/1981.81 222019000200007.
- MAIDA, G. DI, M. A. MANNINO, J. ZILHÃO, D. L. HOFFMANN, M. García-Diez, A. Pastoors, C. D. Standish et al. 2020. Radiocarbon and U-series age constraints for the lateglacial rock art of Sicily. Quaternary Science Reviews 245: 106524; https://doi.org/10.1016/j.quascirev.2020.106524.
- MAZEL, A. and A. WATCHMAN 1997. Accelerator radiocarbon dating of Natal Drakensberg paintings: results and implications. Antiquity 71(272): 445-449; https://doi. org/10.1017/S0003598X00085069.
- MAZEL, A. and A. WATCHMAN 2003. Dating rock paintings in the UKhahlamba-Drakensberg and the Biggarsberg, KwaZulu-Natal, South Africa. Southern African Humanities 15: 59-73.
- MORALES JR., R. 2002. The nordeste tradition: innovation and continuity in Brazilian rock art (viewed 9/2/2003). http:// www.ditomorales.com/dissintro.htm.
- MORALES, R. 2005. The Angelim style and northeast Brazilian rock art. In J. K. K. Huang and E. V. Culley (eds), Making marks: graduate studies in rock art research in the new millennium, pp. 27–39. Occasional Paper, American Rock Art Research Association.
- MOTA, L. and R. SCHEEL-YBERT 2019. Landscape and firewood use in Toca Do Boqueirão da Pedra Furada (Piauí, Brazil) during early and mid-Holocene. Journal of Archaeological Science: Reports 23: 281-290; https://doi.org/10.1016/j. jasrep.2018.10.034.
- NASCIMENTO, A. C. C. 2009. Engraved world: a contextual analysis of figures and markings on the rocks of south-eastern Piaui, Brazil. Unpubl. thesis, University of Newcastle.
- NASH, G. H. 2009. Serra da Capivara. America's oldest art? Current World Archaeology 37: 41-46.
- PARENTI, F. 2001. Le gisement Quaternaire de Pedra Furada

(Piaui, Brésil). Stratigraphie, chronologie, évolution culturelle. Éditions Recherche Sur Les Civilistions, Paris.

- PEARCE, D. G. and A. BONNEAU 2018. Trouble on the dating scene. Nature Ecology and Evolution 2(6): 925–926; https:// doi.org/10.1038/s41559-018-0540-4.
- Pellerin, J. 2014. Unidades de relevo e formação superficiais na região do Parque Nacional Serra Da Capivara. In A.-M. Pessis, G. Martin and N. Guidon (eds), Os biomas e as sociedades humanas na pré-História da região do Parque Nacional Serra da Capivara, Volume II-A, pp. 58-67. FUM-DHAM, São Paulo.
- PESSIS, A.-M. 1987. Art rupestre préhistorique: premiers registres de la mise en scène. Micro Edition Université de Paris X, Institut d'Ethnologie, Paris.
- PESSIS, A.-M. 1992. Identidade e classificação dos registros gráficos pré-Históricos do nordeste do Brasil. CLIO - Série Arqueológica 1(8): 35-68.
- PESSIS, A.-M. 1999. Pré-História da região do Parque Nacional Serra da Capivara. In M. C. Tenório (ed.), Pré-História da Terra Brasilis, pp. 61–74. Universidade Federal Rio de Janeiro, Rio de Janeiro.
- PESSIS, A.-M. and N. GUIDON 2009. Dating rock art paintings in Serra de Capivara National Park combined archaeometric techniques. Adoranten September: 49-59.
- PESSIS, A.-M., G. MARTIN and N. GUIDON 2010. Datations des peintures rupestres du Parc National Serra da Capivara: une construction issue de la confrontation des techniques archéométriques. In Pre-Actas IFRAO Congress, Symposium Pleistocene Art of the Americas, pp. 2-10.
- Plagnes, V., C. Causse, M. Fontugne, H. Valladas, J. M. CHAZINE and L. H. FAGE 2003. Cross dating (Th/U-14C) of calcite covering prehistoric paintings in Borneo. Quaternary Research 60(2): 172-179. https://doi.org/10.1016/ S0033-5894(03)00064-4.
- Pons-Branchu, E., R. Bourrillon, M. W. Conkey, M. Fon-TUGNE, C. FRITZ, D. GÁRATE, A. QUILES et al. 2014. Uranium-series dating of carbonate formations overlying Paleolithic art: interest and limitations. Bulletin de La Societe Prehistorique Francaise 111(2): 211-24; https://doi. org/10.3406/bspf.2014.14395.
- ROSENFELD, A. and C. SMITH 1997. Recent developments in radiocarbon and stylistic methods of dating rockart. Antiquity 71(272): 405-411; https://doi.org/10.1017/ S0003598X00085008.
- Rowe, M. W. and K. L. STEELMAN 2003. Comment on 'Some evidence of a date of first humans to arrive in Brazil. Journal of Archaeological Science 30(10): 1349-1351; https:// doi.org/10.1016/S0305-4403(03)00021-9.
- RUIZ, J. F., A. HERNANZ, R. A. ARMITAGE, M. W. ROWE, R. VIÑAS, J. M. GAVIRA-VALLEJO and A. RUBIO 2012. Calcium oxalate AMS 14C dating and chronology of post-Palaeolithic rock paintings in the Iberian Peninsula. Two dates from Abrigo de Los Oculados (Henarejos, Cuenca, Spain). Journal of Archaeological Science 39(8): 2655-2667; https://doi.org/10.1016/j.jas.2012.02.038.
- SANNA, L., F. SAEZ, D. SIMONSEN, S. CONSTANTIN, J. M. CALA-FORRA, P. FORTI and S. E. LAURITZEN 2010. Uranium-series dating of gypsum speleothems: methodology and examples. International Journal of Speleology 39(1): 35-46; https://doi.org/10.5038/1827-806X.39.1.5.

- SANTOS, G. M., M. I. BIRD, F. PARENTI, L. K. FIFIELD, N. GUIDON and P. A. HAUSLADEN 2003. A revised chronology of the lowest occupation layer of Pedra Furada rock shelter, Piauí, Brazil: the Pleistocene peopling of the Americas. Quaternary Science Reviews 22(21-22): 2303-2310; https:// doi.org/10.1016/S0277-3791(03)00205-1.
- SANTOS, T. 2013. Rock-art of Toca Do Paraguaio (Piauí, Brazil). A morpho-technique approach. Unpubl. MA thesis, 2 vols, Instituto Politécnico de Tomar and Universidade de Trás-os-Montes e Alto Douro.
- SANTOS, T. 2020. Contexto arqueológico da Toca do Paraguaio e as ocupações do Holoceno antigo no sudeste do Piauí, Brasil. CLIO Arqueológica 34(3): 17; https://doi. org/10.20891/clio.v34n3p17-44.
- SHAO, Q., C. H. LI, M. J. HUANG, Z. B. LIAO, J. ARPS, C. Y. HUANG, Y. C. CHOU and X. G. KONG 2019. Interactive programs of MC-ICPMS data processing for <sup>230</sup>Th/U geochronology. Quaternary Geochronology 51: 43-52; https://doi.org/10.1016/j.quageo.2019.01.004.
- STEELMAN, K. L., R. RICKMAN, M. W. ROWE, T. W. BOUTTON, J. Russ and N. GUIDON 2002. AMS radiocarbon ages of an oxalate accretion and rock paintings at Toca do Serrote da Bastiana, Brazil. In K. Jakes (ed.), Archaeological chemistry, pp. 22–35. American Chemical Society, Washington, DC.
- TANG H. and R. G. BEDNARIK 2021. Rock art dating by <sup>230</sup>Th/<sup>234</sup>U analysis: an appraisal of Chinese case studies. Archaeological and Anthropological Sciences 13(1): 1-10; doi: 10.1007/s12520-020-01266-0.
- WATANABE, S., W. E. F. AYTA and H. HAMAGUCHI 2003. Some evidence of a date of first humans to arrive in Brazil. Journal of Archaeological Science 30: 351-54; https://doi. org/10.1016/S0305-4403(03)00021-9.
- WATCHMAN, A. 1991. Age and composition of oxalate-rich crusts in the Northern Territory, Australia. Studies in Conservation 36(1): 24-32; https://doi.org/10.1179/ sic.1991.36.1.24.
- WATCHMAN, A. 2004. Minimum age for a petroglyph on a boulder of significance in southern Kakadu National Park, Northern Territory, Australia. Rock Art Research 21(2): 187-195.
- WATCHMAN, A. and J. B. CAMPBELL 1996. Micro-stratigraphic analyses of laminated oxalate crusts in northern Australia. In 22nd International Symposium, The Oxalate Films in the Conservation of Works of Art, pp. 409–422. Milan, Italy.
- WATCHMAN, A., WALSH M. J., M. J. MORWOOD and C. TUNIZ 1997. AMS radiocarbon age estimates for early rock paintings in the Kimberley, N.W. Australia: preliminary results. Rock Art Research 14(1): 18-26.
- WATCHMAN, A., S. O'CONNOR and R. JONES 2005. Dating oxalate minerals 20–45 ka. Journal of Archaeological Science 32(3): 369-374; https://doi.org/10.1016/j.jas.2004.10.007.
- WATCHMAN, A., P. TAÇON, R. FULLAGAR and L. HEAD 2000. Minimum ages from north Western Australia for pecked rock markings Jinmium. Archaeology in Oceania 35: 1-10.
- WHITE, R., R. BOURRILLON, J. CLOTTES, M. W. CONKEY, S. C. RODRIGUEZ, M. CORTÉS-SÁNCHEZ et al. 2020. Still no archaeological evidence that Neanderthals created Iberian cave art. Journal of Human Evolution 144: 102640; https:// doi.org/10.1016/j.jhevol.2019.102640.