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ORIGINAL ARTICLE

Obsidian in the Caribbean islands? Mysterious Ceramic Age glass artefacts in the Lesser Antilles

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Abstract

The Caribbean islands witnessed a population expansion of ceramic-using horticulturalists during the Early Ceramic Age (ca. 500 BC to 750 AD) from the Orinoco Valley to Puerto Rico. We examined 18 lithic artefacts from Guadeloupe and Dominica initially thought to be obsidian, a material believed to be absent from those islands. We investigated the volcanic or meteoritic origin of this unique and yet unknown material through observation (binocular, SEM, microtomography) and geochemical analyses (PIXE, SEM-EDS, ED-XRF, EPMA, LA-ICP-MS). Elemental analyses rule out the hypothesis of an origin from a meteoritic impact (i.e. identification as tektites). Most of the artefacts have an andesitic composition (<63% SiO₂), which appears to be unique among 'massive' glasses. The only artefact with a rhyolitic composition has been traced back to the Guadeloupe's Volcan du Tuf, where glassy fragments have been collected and analysed. The geological source of the other vitreous artefacts that exhibit an andesitic composition could be from a sublocal subduction-arc volcanism (maybe from Martinique), although no volcanic vitreous material of this kind has ever been reported worldwide. These results once again highlight the regional mobility of Early Ceramic populations and the production of standard lithic products using a highly original, albeit low-quality, local lithic resource, and provide valuable references for future identification of similar materials.

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KEYWORDS

andesite, Ceramic Age, impactites, Lesser Antilles, obsidian, sourcing, tektites

INTRODUCTION

The archipelago of Caribbean islands was settled late in the history of the peopling of the Americas, first by hunter-gatherers during what is called the Archaic Age, and from ca. 500 BC onward, by ceramic-using horticulturalists originating from the Lower Orinoco during what is called the Ceramic Age (Keegan et al., 2013; Reid, 2018). This migration of Amerindians during the Early Ceramic period, producing specific ceramics grouped in the Saladoid series (Rouse, 1992), was very successful in colonising the archipelago up North to Puerto Rico while remaining strongly homogeneous in their way of life as seen in their settlement patterns, food production and consumption, and production of goods such as ceramics and lapidary ornaments (Bérard, 2013; Queffelec, 2023). Their culture underwent a transformation from ca. 750 AD, evolving into more distinct regional groups marked by varying settlement patterns, shifts in material culture, and alterations in societal organisation (Hofman, 2013). Then, during the Recent Ceramic period, the remaining part of the Caribbean archipelago was settled until the arrival of Europeans in the late 15th century.

During the Ceramic Age, the nonperishable material culture is mainly formed of ceramics for diverse types of pots, shells for tools and adornments, and lithics for cutting, scraping, and grinding tools, as well as personal adornments. The production of flaked stone tools, of particular interest for this work, is mainly driven by production of small unretouched flakes by bipolar percussion (Bérard, 2011; Walker, 1979) to be used for different activities including the production of teeth for manioc graters (Walker, 1979). The principal raw materials for flaked industry are flint and jaspers, respectively coming mainly from Antigua and from local sources near the archaeological sites (Knippenberg, 2007; Queffelec et al., 2023). The provenance of the lithic raw materials used to produce tools, ornaments such as beads and pendants, or other symbolic artefacts like trigonoliths have been one of the main proxies employed by archaeologists to study the regional mobility and networks in the Caribbean islands (e.g. Cody, 1993; Garcia-Casco et al., 2013; Knaf et al., 2021; Knippenberg, 2007; Queffelec, 2022; Queffelec, 2023).

When investigating the origins of lithic raw materials to gain a deeper understanding of the ‘litho-space’ of ancient societies (Delvigne et al., 2021), one invaluable resource on a global scale is the natural glass known as obsidian. This vitreous silica-rich volcanic rock originates from the cooling of a very viscous magma, preventing its crystallization (Tuffen et al., 2020). It has a geochemical composition specific to the volcano—and even to the eruption—which produced the magma (Shackley, 1998, p. 5). Obsidian has served a dual purpose, having been widely utilised by past civilizations for its knapping capabilities and by archaeologists for its high potential in geochemical fingerprinting (e.g. Cann & Renfrew, 1964; Poupeau et al., 2010). Despite the extensive use of obsidian in various regions across the Americas, proving highly effective in unveiling insights into the past networks and mobility of people (Glascok, 2002; Golitko et al., 2012; Mills et al., 2013), it was a prevailing consensus among archaeologists and geologists that the Caribbean islands are devoid of such raw material despite the volcanic origin of most islands in the Lesser Antilles. The volcanic arc of the Lesser Antilles originates from the subduction of the Atlantic plate beneath the Caribbean plate. This geological process predominantly produces andesitic magma, which differs from the silica-rich rhyolitic magma essential to the formation of obsidian *sensu stricto*. To the best of our knowledge, no andesitic volcanic

glass suitable for flaking has been documented in archaeological or geological literature. Such andesitic glass is exceptionally rare and typically forms exclusively due to cosmic impacts, either as impactites or tektites. Impactites are terrestrial rocks formed by the high energy and pressure generated by a high velocity, massive asteroid or comet impact (Osinski & Pierazzo, 2013). They primarily record the geochemistry of the impacted material and can thus exhibit considerable compositional variation. With large impacts, some material may be ejected into the atmosphere, where it rapidly cools and vitrifies before falling back to Earth hundreds or even thousands of kilometres from the impact site—these are known as tektites (Osinski & Pierazzo, 2013). Tektites, as microfragments in deep-sea deposits, have been identified in the southern Caribbean (Barbados), marking the southernmost extent of the North-East American tektite field (Glass et al., 1984). Certain rocks discovered in Haiti could also be linked to a meteorite impact, one that occurred around the Cretaceous-Tertiary transition and was responsible for the Chicxulub crater in the Yucatan. However, this interpretation remains contentious (Jéhanno et al., 1992; Kring & Boynton, 1992), and in any case, these rocks are far too small to be used for knapping. The recent discovery of a new field of tektites of andesitic composition in Belize, referred to as ‘belizites’, calls this into question: the largest of these tektites can form nodules a few centimetres in diameter (Rochette et al., 2021 fig. 2).

In this article, we present the first identification of archaeological stone tools made of natural glass coming from several archaeological sites in Guadeloupe and Dominica. A thorough description of these artefacts is presented, as well as geochemical and mineralogical characterization combining several analytical techniques. Most of these artefacts being vitreous andesite, major questions have been raised about their geological and geographical origin. As one of the artefacts was made of obsidian, we collected a sample of rhyolitic glass identified in the eastern part of Guadeloupe. We were also able to obtain a sample of rhyolitic glass from Puerto Rico to determine whether it was the source material used. Based on the detailed characterization achieved, we are investigating the origin of this hitherto unknown raw material and its distribution in several archaeological sites from the Ceramic Age.

MATERIAL AND METHODS

For the sake of reproducibility and transparency, note that we use throughout the text the MeRIT system as per Nakagawa et al. (2023): Initials of authors, but also of acknowledged people, are used to precisely identify individual contributions.

Archaeological artefacts

Nineteen archaeological artefacts from eight Ceramic Age sites, made of natural glass, were identified in the literature (Table 1 and Figure 1). We were able to access and scrutinize 16 of these (Figure 2). Most of them come from development-led archaeology and were initially identified thanks to the vigilant efforts of CS in monitoring grey literature sources.

Out of the 19 artefacts listed in literature, 16 originate from archaeological sites situated in Guadeloupe. Bisdary is 1 km from the sea, on the foothills of the Caribbean Mountains (Romon, 2004). This site underwent surveying in 2004 and subsequent excavation in 2006 revealing the remains of a Cedrosan Saladoid site dating back to approximately 500–800 cal. AD. In addition to its unique geographical location, quite far from the shore for this period, this site yielded five archaeological objects recorded as being made of ‘obsidian’ (of which only four were found in the curated collection so far) (Fouéré, 2006, p. 34). The site of Tourlourous, located near the southwestern shore of Marie-Galante, is also attributed to the Cedrosan Saladoid (Colas, 2002). Seven archaeological artefacts made of natural glass were identified during

TABLE 1 List of archaeological and geological sites with volcanic glass artefacts or glassy geological samples and their location (+; yet to be confirmed, *; lost). MGCT02–3 and PSF-1 were analysed in PIXE in 2007 but are now lost. SJB-1 could only be analysed by pXRF during a preliminary study and could not be brought to the laboratory for logistical reasons. The nucleus from Antigua is only listed in literature, we were not able to study it.

Artefacts from archaeological sites							
Sample code	Site	City	Island	Longitude	Latitude	Period	Bibliography
Bis-01	Bisdary	Gourbeyre	Basse-Terre	-61.709	15.988	Early Ceramic	Romon et al., 2006
Bis-02	Bisdary	Gourbeyre	Basse-Terre	-61.709	15.988	Early Ceramic	Romon et al., 2006
Bis-03	Bisdary	Gourbeyre	Basse-Terre	-61.709	15.988	Early Ceramic	Romon et al., 2006
Bis-04	Bisdary	Gourbeyre	Basse-Terre	-61.709	15.988	Early Ceramic	Romon et al., 2006
RRC-01	Ruelle Roches Caraïbes	Baillif	Basse-Terre	-61.736	16.038	Early Ceramic	Bonnisset & Chancelerel, 2001
RRC-02	Ruelle Roches Caraïbes	Baillif	Basse-Terre	-61.736	16.038	Early Ceramic	Bonnisset & Chancelerel, 2001
MGCT02-1	Tourlourous	Capesterre-de-Marie-Galante	Marie-Galante	-61.224	15.889	Early Ceramic	Colas, 2002
MGCT02-2	Tourlourous	Capesterre-de-Marie-Galante	Marie-Galante	-61.224	15.889	Early Ceramic	Colas, 2002
MGCT02-3 *	Tourlourous	Capesterre-de-Marie-Galante	Marie-Galante	-61.224	15.889	Early Ceramic	Colas, 2002
MGCT02-4	Tourlourous	Capesterre-de-Marie-Galante	Marie-Galante	-61.224	15.889	Early Ceramic	Colas, 2002
MGCT02-5	Tourlourous	Capesterre-de-Marie-Galante	Marie-Galante	-61.224	15.889	Early Ceramic	Colas, 2002
MGCT02-6	Tourlourous	Capesterre-de-Marie-Galante	Marie-Galante	-61.224	15.889	Early Ceramic	Colas, 2002
MGCT02-7	Tourlourous	Capesterre-de-Marie-Galante	Marie-Galante	-61.224	15.889	Early Ceramic	Colas, 2002
CB-050a	Caillie à Bélassé	Terre de Bas	Ilets de Petite Terre	-61.11	16.17	Late Ceramic	Gagnepain, 2007
CB-051a	Caillie à Bélassé	Terre de Bas	Ilets de Petite Terre	-61.11	16.17	Late Ceramic	Gagnepain, 2007

TABLE 1 (Continued)

Artefacts from archaeological sites										
Sample code	Site	City	Island	Longitude	Latitude	Period	Bibliography			
DEL-3-1	Delices 3	Saint Patrick Parish	Dominique	-61.256	15.275	Early Ceramic	Shearn, 2014			
SJB-1	Stade José Bade	Capesterre-de-Marié-Galante	Marié-Galante	-61.224	15.889	Early Ceramic	Serrand, 2016			
PSF-1 *	Place Saint François	Basse-Terre	Basse-Terre	-61.73	15.995	Early Ceramic	Sellier-Ségaard, 2001			
Not included yet	Mill Reef+	Saint Philip	Antigua	-61.679	17.057	Middle/Late Ceramic	Murphy, 1999			
TABLE 1 (Continued)										
Artefacts from archaeological sites										
Sample code	Mass(g)	Type	SEM-EDS	PIXE	EPMA	LA-ICP-MS	ED-XRF	Raman	Microtomography	
Bis-01	1.312	Flake		X			X		X	
Bis-02	2.21	Flake	X	X			X		X	
Bis-03	4.318	Nucleus		X	X		X	X	X	
Bis-04	2.367	Flake	X	X			X	X	X	
RRC-01	2.733	Flake		X			X	X	X	
RRC-02	5.504	Undetermined	X	X			X	X	X	
MGCT02-1	1.394	Flake		X			X	X	X	
MGCT02-2	4.063	Flake	X	X			X		X	
MGCT02-3 *	N.A.	Undetermined		X						
MGCT02-4	2.792	Nucleus		X			X		X	
MGCT02-5	0.156	Flake	X	X			X		X	
MGCT02-6	16.896	Flake		X	X		X		X	
MGCT02-7	2.682	Flake	X	X			X		X	
CB-050a	0.901	Flake	X	X			X	X	X	

TABLE 1 (Continued)

Artefacts from archaeological sites											
Sample code	Mass(g)	Type	SEM-EDS	PIXE	EPMA	LA-ICP-MS	ED-XRF	Raman	Microtomography		
CB-051a	0.299	Flake	X	X			X		X		
DEL-3-1	0.373	Flake	X				X		X		
SJB-1		Flake					pXRF				
PSF-1 *		Flake		X							
Not included yet		Nucleus									
Samples from geological formations											
Sample code	Site	City	Island	Longitude	Latitude	Type of rock	SEM-EDS	PIXE	EPMA	LA-ICP-MS	ED-XRF
Volc.Tuf	Volcan du Tuf	Vieux-Habitants	Basse-Terre	-61.734	16.051	Rhyolite	X	X	X	X	X
Riv.Capesterre	Rivière de Capesterre	Capesterre-Belle-Eau	Basse-Terre	-61.609	16.072	Andesite					X
Est.Carbet	Embouchure du Grand-Carbet	Capesterre-Belle-Eau	Basse-Terre	-61.574	16.021	Andesite					X
BoisJoli	Bois Joli	Terre-de-Haut	Les Saintes	-61.609	16.072	Andesite					x
ObsPR	Yunque National Forest		Puerto Rico	-65.777	18.299	Rhyolite	X	X	X	X	X

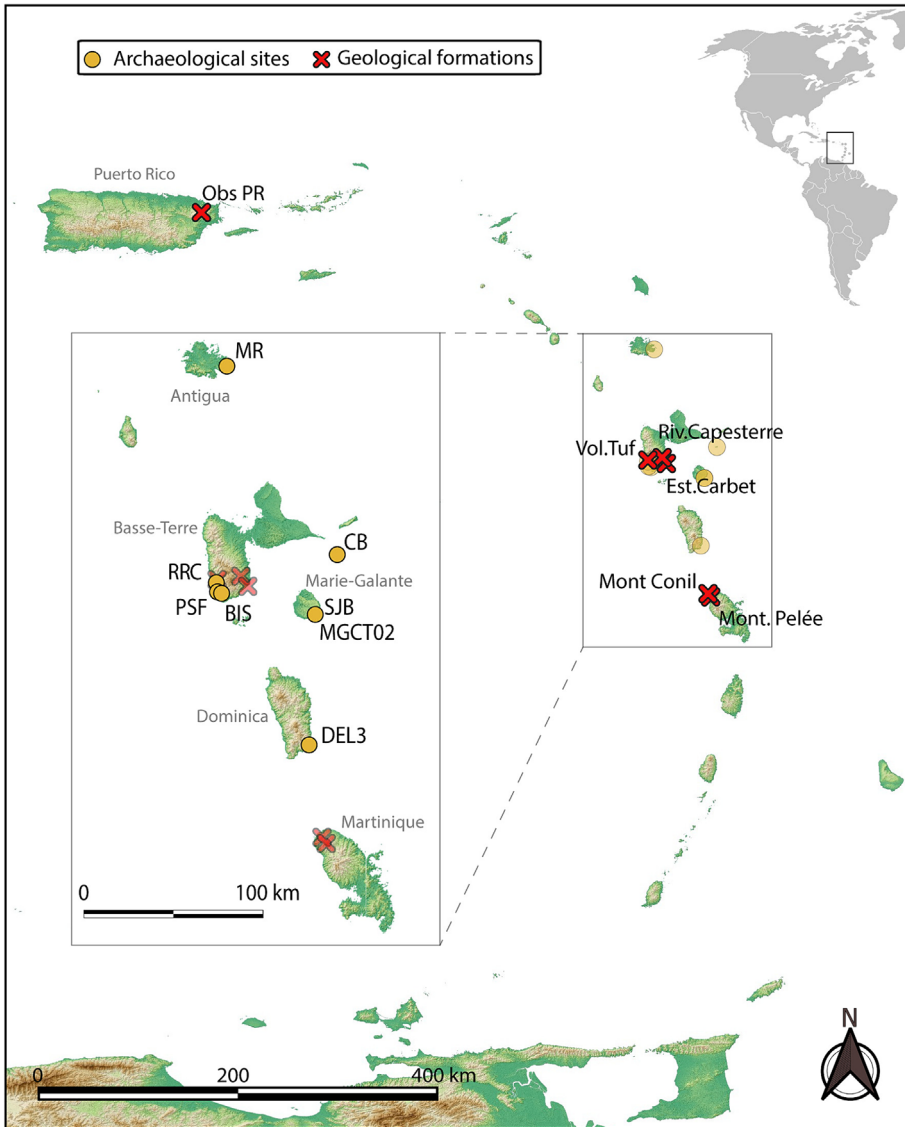


FIGURE 1 Map indicating the locations of the archaeological sites from which the artefacts originate, and the locations of the geological deposits that have been sampled (Guadeloupe and Puerto Rico) or are scheduled for sampling but already used as comparative data (Martinique). SJB and MGCT02 are located on the same spot as the sites are only a few tens of metres apart.

the excavation and were analysed in 2007 by PIXE in a preliminary project, but we only recovered six of them during our recent study. Excavated in the adjacent parcel of Tourlourous in 2014, the Stade José Bade is considered to be part of the same site (Sellier-Segard & Casagrande, 2016). One flake made of a similar glassy material has been found on the site, regrettably, it comes from levels containing a mixture of Cedrosan Saladoid and Troumassan Troumassoid artefacts. We had access to this artefact during a preliminary study conducted in Guadeloupe, with only pXRF available at this time, but the object was no longer available when we conducted more detailed laboratory analyses. Place Saint François was excavated in

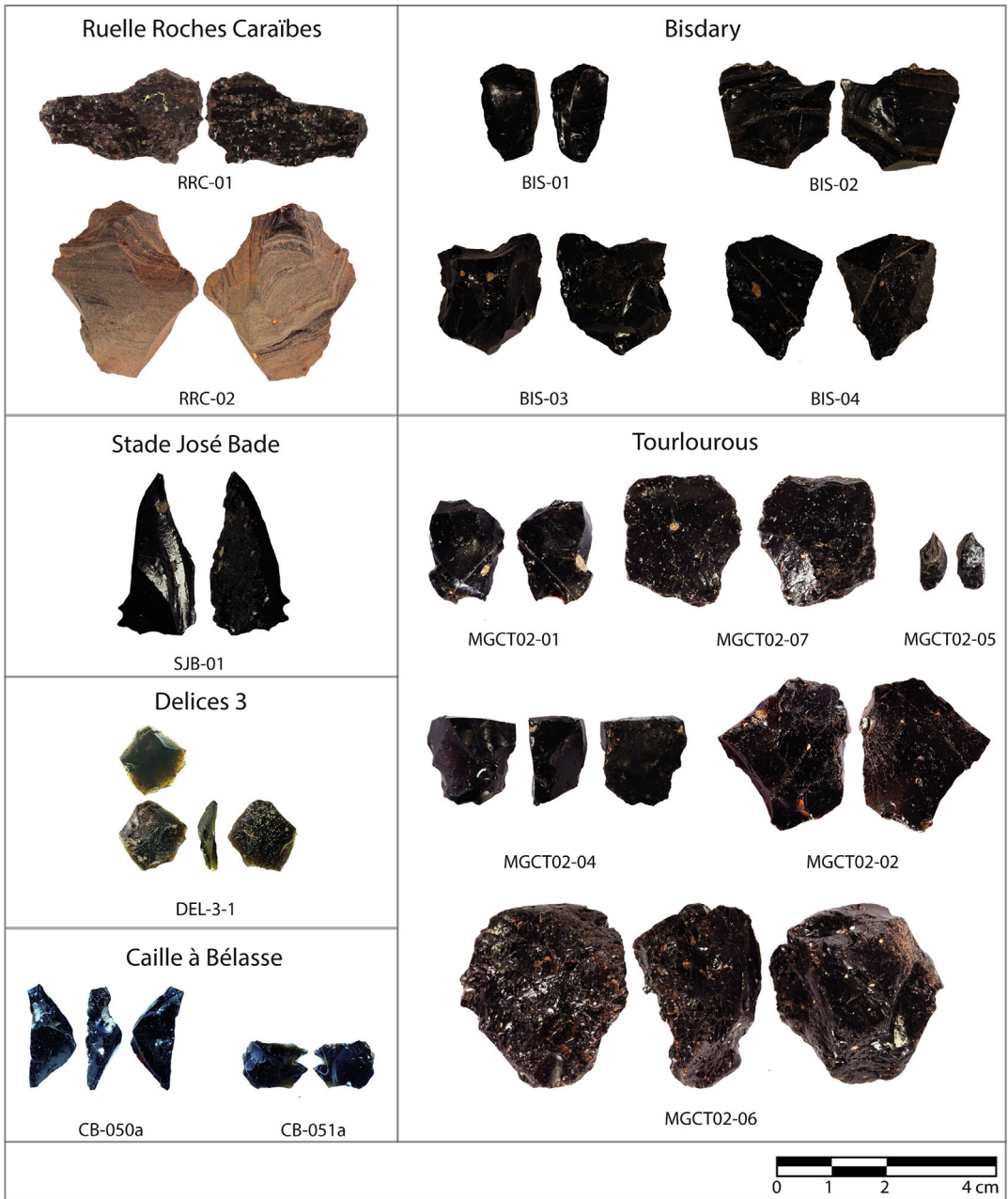


FIGURE 2 Macrophotographs of the sixteen glassy artefacts recovered from six archaeological sites that we were able to access.

2001, just in front of the cathedral of Basse-Terre, and is interpreted as a Cedrosan Saladoid occupation (Sellier-Ségard, 2001). One artefact made of glassy rock comes from this site and was analysed in 2007 by PIXE and has since been lost. Similarly, the Ruelle des Roches Caraïbes site, excavated in 2001, a few kilometres south of Volcan du Tuf, yielded two glassy artefacts from the Cedrosan Saladoid levels (Bonnisent & Chancerel, 2001). The Caille à Bélasse site is the only Late Ceramic site having yielded glassy artefacts. It is interpreted as a

Troumassan Troumassoid settlement and located on the Ilets de Petite Terre in the southwest part of the Guadeloupean archipelago (Gagnepain, 2007).

Two artefacts were discovered outside Guadeloupe. One was unearthed 20–30 cm below the surface by IS, in the site called *Delices 3*, on the southwest coast of Dominica (Shearn, 2014, p. 318). It is attributed to a Cedrosan Saladoid occupation. Additionally, there is a ‘small reduced core’ designated as ‘obsidian’ at the Mill Reef site (Antigua) (Murphy, 1999, p. 208), which could not be included in the present study. It is worth noting that there are mentions of other volcanic glass in the literature, but it seems that these are all misidentifications. A blade from the Maruca site in Puerto Rico was described as obsidian (Febles, 2004; Rodríguez Ramos, 2013) despite being identified initially as black chalcedony (Rodríguez, 2001) and subsequently confirmed as such (Rodríguez Ramos, comm. pers. to AQ 2020). In Martinique, a report on the Early Saladoid site of Fond-Brûlé listed nine ‘volcanic glass’ artefacts (Mattioni, 1982). However, upon a thorough examination of the complete archaeological collection, this identification can be discarded, because every artefact photographed in the report has been found in the curated collection, and there is not a single volcanic glass artefact (black jaspers and flints are present).

All the 16 artefacts available for study are small flakes and nuclei originating from bipolar percussion technique (Figure S1), as already noted by Fouéré (2006) for those found in Bisdary site. They do not differ significantly from artefacts made of other raw materials in the respective sites, which are mainly made of flint from Antigua, and are also mainly produced by bipolar technique (Bérard, 2011; Davis, 2002; Knippenberg, 2007; Queffelec et al., 2023). Based on ethnographic comparisons and traceological study (Walker, 1979), the main hypothesis for the function of small-sized flakes produced by bipolar percussion in the Ceramic Age of the Lesser Antilles is their use as teeth for cassava grater boards. Some authors recommend a more site-specific interpretation and not a general application of this hypothesis (Bérard, 2004; Crock & Bartone, 1998), and because no traceological study has been conducted on these artefacts, we prefer not to make any hypothesis on this subject. Technical drawings and surface views from the 3D models acquired by microtomography are presented in Figure S1 and S2 to support this interpretation.

Finally, it must be noted that AQ conducted a survey of the Caribbean archaeological community by email and during the IACA 2022 congress. None of the respondents (see Acknowledgements) reported ever seeing a single black vitreous artefact during their career in the Caribbean islands, and no participant of the conference came forward either to say so.

Geological volcanic glass

Volcanic glass worldwide is mostly of rhyolitic composition and is referred to as obsidian. Rhyolitic rocks are very scarce in the Lesser Antilles, with only three to four occurrences with no description of obsidian (Guen et al., 1982; Smith et al., 1980). The only obsidian described in geological literature, dated to 325 ka, is located in the Volcan du Tuf area (Basse-Terre, Guadeloupe) and consists of a brecciated dyke containing perlitic obsidian originating from maar eruptive activity (Gadalia et al., 1988). Fieldwork enabled us to identify the location of this obsidian source in the commune of Vieux-Habitants (Basse-Terre, Guadeloupe), on the edge of the Volcan du Tuf maar (Table 1). Several samples were taken, all with very poor knapping qualities (Figure S3).

A lone obsidian block from Puerto Rico is known to exist, having been discovered by a local worker while clearing a drainage ditch beside the PR 191 road in the El Yunque national forest (Walker pers. com. to AQ 2022). Despite additional fieldwork conducted in the area, no other blocks matching this description were located (Knippenberg pers. com. to AQ 2022). Regrettably, the precise geological origin of this high-quality obsidian block, provided by JW

and SK, therefore remains unknown. Although there is documented evidence of a silicic suite in the northeastern Antilles, primarily dating back to the Albian age (113.0 ± 1.0 to 100.5 ± 0.9 Ma) and found in Puerto Rico and the Virgin Islands (Jolly et al., 2008), the composition of these rocks, predominantly rhyolites, does not align with that of our sample (ObsPR). Even though these Albian rocks have likely undergone significant devitrification due to their age, this observation underscores the capacity of the tectonic conditions in this particular region of the Antilles to generate high-silica rocks similar to the ObsPR sample analysed in this study.

In addition to our search for obsidian from the Volcan du Tuf, we carried out a prospecting campaign in the Guadeloupe archipelago to locate vitreous andesitic material. Based on data from the geological map and indications from local prospectors, we identified and collected three deposits of glassy andesitic rocks trapped in ash deposits in the Capesterre river and at the estuary of the Grand-Carbet river (Capesterre-Belle-Eau, Basse-Terre, Guadeloupe) and at Bois Joli (Terre-de-Haut, Saintes Archipelago, Guadeloupe) (Table 1). Unfortunately, none of these deposits contained glassy rocks massive enough to be knapped and potentially matching the material of the studied artefacts.

Methods

All samples underwent macroscopic and binocular observations. The various methods of analysis applied to each sample are reported in Table 1. The artefact from Stade José Bade (SJB) could only be analysed by portable X-Ray Fluorescence (pXRF) because it remained in Guadeloupe. This pXRF analysis confirmed its andesitic composition.

AQ conducted binocular observations using a Leica Z16APO to observe the texture and the structure of samples, as well as the distribution of phenocrysts. Photographs were taken with a Leica Flexacam C1 camera and the LAS X 5.0 software.

LBG and CL performed nondestructive elemental analysis by Particle-Induced X-ray Emission (PIXE) on the AGLAE facility (Calligaro et al., 2002) using the procedures presented in Bellot-Gurlet et al. (1999). The AGLAE facility of the Laboratoire de Recherche des Musées de France is based on a two MV tandem accelerator Pelletron 6SDH-2 built by National Electrostatics Co. For obsidian analyses, LBG and CL used an external proton beam with an energy of 3 MeV before its exit outside the accelerator vacuum. The entire X-ray spectrum is recorded by means of two Si (Li) detectors, for matrix and trace elements respectively. The instrument is calibrated with obsidian reference materials.

AL and AQ observed and analysed the artefacts by scanning electron microscope (SEM) coupled with energy dispersive spectroscopy (EDS) in a JEOL IT500HR electron microscope (FEG source). The SEM images and EDS spectra were acquired without carbon coating, in a low-vacuum mode (here 40 Pa). The images were acquired with a backscattered electron detector, whereas the EDS analysis were performed with two Oxford Ultimex 100 detectors at 20 kV for 30 s, producing spectra with 4.0–4.5 million counts.

AL conducted an analysis of the artefacts and geological samples using energy dispersive x-ray fluorescence (ED-XRF) on a Hitachi SEA 6000vx device. This device is equipped with a rhodium source (50 kV/1 mA) and a Vortex SDD detector, and a specialised protocol was developed specifically for the study of silica-rich archaeological artefacts (Leck, 2022; Leck et al., 2018). The PCA was carried out using CoDaPack software (Comas & Thió-Henestrosa, 2011). The data were prior transformed by a centred log ratio transformation (Aitchison, 2003).

Four micro-samples were prepared for electron microprobe analyses (EPMA) and laser ablation—inductively-coupled plasma—mass spectrometry (LA-ICP-MS) analyses by AQ. EPMA and LA-ICP-MS analyses were only conducted on a very limited selection of samples: three of them were cut with a wire saw (Well3500) and epoxy resin (Araldite 2020): two

from two rather large archaeological artefacts for which the shape would not be altered by the sampling (BIS-03 and MGCT02–06), one from the natural glass from Puerto Rico. Several glass fragments from Volcan du Tuf were also impregnated in epoxy resin. All samples were abraded with silicon carbide discs and polished with diamond paste. We obtained the official authorization of cutting small subsamples from both archaeological artefacts via the French Ministry of Culture.

EPMA and LA-ICP-MS were conducted to obtain accurate geochemical composition of both major, minor and trace elements. JL determined the major-element compositions of the glasses by EPMA analysis using a Cameca SX100 from the *Microsonde Ovest* platform using wavelength dispersive spectrometers at 15 KV accelerating voltage, 10–12 nA beam current, and a spot size ranging from 10 to 20 μm . Minerals (wollastonite [Si, Ca], orthoclase [K], albite [Na], apatite [P]), oxide (MnTiO₃ [Mn, Ti]), Al₂O₃ [Al], Fe₂O₃ [Fe], Cr₂O₃ [Cr], FeS₂ [S] and so forth, and metal standards (Ni) were used for calibration. In order to avoid the heterogeneity effects induced by small crystals or inclusions, the compositions of the glasses given here are averages of two to six analyses depending on the sample. BG performed the LA-ICP-MS measurements using a Thermo Fisher Scientific ELEMENT XR mass spectrometer with a linear dynamic range of 12 orders of magnitude thanks to the combination of a Faraday detector and a dual-mode SEM detector. The laser was a Resonetics M50E excimer laser operating at a wavelength of 193 nm, 5 mJ and a repetition rate of 10 Hz. The sampling cell is flushed with an argon/helium glass flow of 1 L/min Ar + 0.65 L/min He. The analytical protocols followed are those typically employed for the analysis of glass (Gratuze, 2016), which include a pre-ablation time of 20 s and the acquisition of nine mass scans for 58 elements. Trace elements data were normalised to N-MORB (Gale et al., 2013) and normalised to chondrite (Barrat et al., 2012).

AQ performed micro-Raman spectroscopy analysis on phenocrysts located inside the devitrification centres in several samples with a Bruker SENTERRA Raman microspectrometer equipped with a 532 nm laser and a 1200-line diffraction grating resulting in a resolution of 3 to 5 cm^{-1} , after cleaning the samples with hydrogen peroxide and ultrasonic bath. Spectra were interpreted using the RRUFF database (Lafuente et al., 2016).

Before microsampling for EPMA and LA-ICP-MS, 3D reconstruction of the surface and the inner structure of the samples was realized by NV via X-ray microtomography using a GE VTOMEX-S 240/180. Depending on the size of the artefacts and on the fact that they would suffer or not the microsampling for geochemical analysis, the microscan performed have variable resolutions between 6 and 21 μm^3 per voxel. AQ extracted isosurfaces from these 3D models using the Avizo software.

RESULTS

The andesitic artefacts consist of a matrix that is entirely translucent, with a slight brownish-orange hue (see Figure 2). A perlitic structure is evident across all pieces, suggesting quenching, as this is the most likely way this material with this chemical composition formed naturally. A few phenocrysts of various morphologies can be observed on the surface: Some are bipyramidal, showing fluidity and probably resulting from crystallization in the magma chamber or chimney, others are definitely devitrification spherules (Figures 2, S4, and S5). Some pieces exhibit significant vesiculation, with vesicles accounting for approximately half the volume of certain pieces (Figure S4). Microtomography scanning reveals that these vesicles are of various types, sizes, shapes, and orientations (Figure S6), signifying different degassing and solidification events. Last, the size of the artefacts and the occasional presence of cortical surfaces lead us to estimate that the original blocks were likely rounded and had a diameter of approximately 5 cm. It is also conceivable that these blocks were likely collected in a primary or subprimary

position, possibly within an ash layer, as the cortical surfaces show no signs of erosion caused by transport.

The rhyolitic artefact, in contrast, features a dark grey vitreous matrix with a banded texture highlighted by submillimetric phenocrysts of feldspar (Figure S4). The perlitic structure is remarkably prominent, to the extent that it probably hindered the development of a useful conchoidal fracture for knapping.

Noninvasive techniques—to ensure the conservation of these rare artefacts—were used on most samples to assess the homogeneity of the collection. SEM-EDS and PIXE data showed a very homogeneous glass composition and revealed the andesitic composition of most samples (Figure 3, Table S1 & S2). ED-XRF analysis confirmed the overall high homogeneity among trace elements for the archaeological andesitic artefacts and the close proximity of the rhyolitic artefact with the Volcan du Tuf source, as PIXE already did in the initial study (Figure 4 and Table S1, S3 & S4). Andesites containing vitreous phases sampled in Guadeloupe do not match the archaeological artefacts' composition. SEM-EDS analysis detected plagioclase feldspar and rare Fe-Ti oxides. It revealed the fibrous structure of plagioclase feldspars within voids (Figure S7), likely formed due to matrix devitrification. These spherulites displayed bow-tie and spherical shapes. Their alignment is probably due to the original alignment of crystallisation nuclei in fluidal planes (Figure S4). Raman spectroscopy (Figures S5) confirmed the identification of most crystals as labradorite, indicating their formation in fluidal planes likely occurred within the magma chamber prior to the eruption, rather than through devitrification, due to the

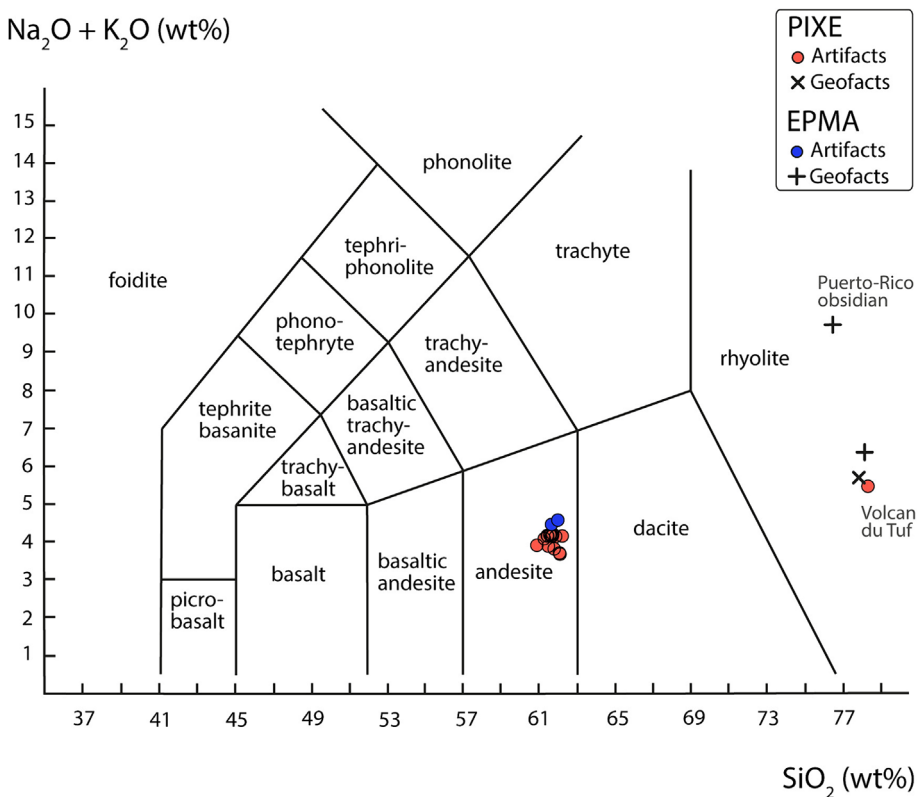


FIGURE 3 Total alkali silica (TAS) diagram (Le Maitre et al., 2005) illustrates the elemental composition measured using PIXE and EPMA on glassy archaeological and geological samples. All the artefacts, with the exception of one, consist of an unusual andesitic glass, whereas the geological samples are composed of obsidian, a more typical rhyolitic glass.

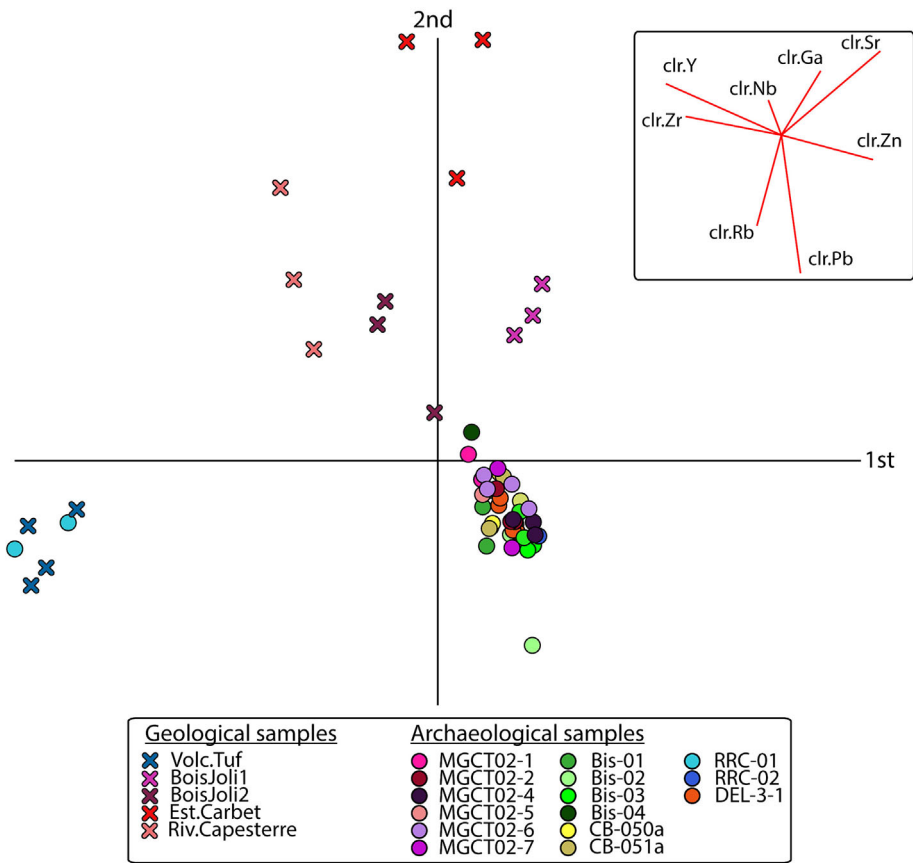


FIGURE 4 PCA on ED-XRF data (trace elements only) from all artefacts as well as geological samples collected in the Guadeloupean archipelago (rhyolitic and andesitic). Each point on the graph relates to one measurement on the sample.

low calcium concentration of the obsidian. It also identified the presence of augite, cristobalite, and quartz in the clusters or lines of minerals.

Based on these noninvasive results and in order to obtain high accuracy measurements from these surprising artefacts and potential sources, we conducted EPMA and LA-ICP-MS analysis (Table 2). The homogeneity and the andesitic nature of the archaeological was confirmed, as well as the rhyolitic composition of both nonarchaeological glass (Figure 3). The homogeneity of the two archaeological artefacts is incredibly high, for all the major and trace elements that we were able to measure (51 in total), clearly indicating that these two artefacts, unearthed from two different archaeological sites, originate from the same volcanic event, probably from the andesitic volcanism of the central Lesser Antilles subduction context (Figure 5).

For the andesitic archaeological artefacts, no enrichment in Cr, Ni, or Co are visible, indicating that no extraterrestrial contamination can be suspected from this data (Koeberl, 1998; Koeberl, 2014; Rochette et al., 2021).

On the other hand, the trace elements and REE spectra suggest an origin from an andesitic volcanism that could be related directly to central Lesser Antilles context like the andesites from the northwestern part of Martinique, where the Mont Conil and the Montagne Pelée have been sampled (Figure 5). Other andesitic rocks from the region have been sampled and analysed previously (Fedele et al., 2021; Germa et al., 2011; Howe et al., 2015; Labanieh, 2009), and if the

TABLE 2 Major and minor elements composition measured by EPMA and trace elements abundances measured by LA-ICP-MS.

EPMA (%)	ObsPR	VolcanTuf	MGCT02-6	BIS-03
SiO ₂	76.36	78.02	61.74	62.00
TiO ₂	0.22	0.15	0.57	0.57
Al ₂ O ₃	11.54	12.58	16.94	16.99
FeO	2.16	1.80	5.49	5.40
MnO	0.12	0.06	0.13	0.12
MgO	0.07	0.14	2.66	2.65
CaO	0.11	1.51	6.87	6.72
Na ₂ O	4.92	4.22	3.35	3.48
K ₂ O	4.83	2.22	1.29	1.29
P ₂ O ₅	0.02	0.04	0.23	0.23
SO ₂	0.03	0.02	0.01	0.00
F	0.14	0.01	0.01	0.00
Cl	0.20	0.24	0.06	0.06
Total	100.72	101.01	99.35	99.51
LA-ICP-MS (µg/g)	ObsPR	VolcanTuf	MGCT02-6	BIS-03
V	3.98	0.24	124	124
Cr	0.31	0.48	8.54	8.57
Co	0.02	0.88	12.9	13.8
Ni	<LOD	0.33	4.51	4.59
Cu	0.04	13.9	142	147
Zn	236	35.7	70.4	69.7
Ga	30.1	11.6	17.2	17.2
As	3.45	0.97	2.00	1.66
Rb	200	47.2	38.8	39.6
Sr	1.82	80.5	221	218
Y	92.2	36.4	15.3	14.9
Zr	852	136	79.5	77.3
Nb	75.1	3.57	2.57	2.62
Mo	5.66	1.18	2.34	2.40
Sn	5.23	1.48	1.22	1.35
Sb	0.28	0.07	0.39	0.30
Cs	3.81	1.52	1.84	1.91
Ba	8.87	273	203	202
La	36.9	13.9	10.2	9.97
Ce	89.3	31.5	21.2	20.7
Pr	9.85	3.98	2.47	2.47
Nd	35.7	18.1	10.4	10.4
Sm	9.29	4.69	2.52	2.62
Eu	1.49	0.79	0.78	0.78
Gd	9.66	4.78	2.34	2.30
Tb	2.00	0.89	0.41	0.41
Dy	13.9	5.87	2.58	2.50

TABLE 2 (Continued)

LA-ICP-MS ($\mu\text{g/g}$)	ObsPR	VolcanTuf	MGCT02-6	BIS-03
Ho	3.15	1.31	0.57	0.56
Er	9.57	3.86	1.65	1.60
Tm	1.51	0.59	0.24	0.24
Yb	11.4	4.39	1.90	1.83
Lu	1.67	0.66	0.29	0.28
Hf	22.6	4.15	2.20	2.16
Ta	3.77	0.23	0.16	0.17
W	0.58	0.28	0.37	0.39
Pb	20.9	5.12	2.92	5.78
Th	16.8	4.01	3.69	3.60
U	6.32	1.30	1.14	1.13

general shape of the diagram is similar, the absolute values do not match the results from the archaeological artefacts (Figure S8). There is apparently a large enough variability in trace elements and REE in the Lesser Antilles context, between islands, and between periods of volcanic activity to propose a potential provenance of the archaeological material from Martinique. The samples of rhyolites (ObsPR and VolcanTuf) exhibit typical compositions of differentiated volcanic rocks, such as a negative anomaly in Eu. However, it can be observed that these two rocks are not derived from the same type of magma. The Puerto Rico obsidian is low in alkaline earth elements and does not display anomalies in Nb and Ta, which may indicate a significant contribution from the oceanic crust in the formation of the magma during subduction (Briqueu et al., 1984; Jolly et al., 2008). The composition of the obsidian from the Volcan du Tuf corresponds, in turn, to the ultimate term of a tholeiitic arc series, as previously suggested by Gadalia et al. (1988).

DISCUSSION

The results presented here introduce, for the first time, fragments of glassy material found in several archaeological sites from the Ceramic Age in the Caribbean islands. These 19 pieces were discovered within an area of only a 50 km radius. Our extensive review of the literature and survey of the Caribbean archaeologist community has confirmed that these are the only artefacts of this kind currently known. The thorough analysis of these objects, from both an archaeological and geological perspective, aims to document them and bring them to the attention of the community. It also sought to determine their origin, as this type of material is traditionally used in archaeology to study the mobility of materials, people, and exchange networks. Two different materials have been identified in this archaeological corpus: obsidian *sensu stricto* and vitreous andesite, a material hitherto unknown.

A single obsidian artefact

Obsidian *sensu stricto* is a vitreous volcanic rock, oversaturated with silica and typically of rhyolitic composition (Tuffen et al., 2020). Although all the archaeological samples from this study were initially recognized as such on the field, their geochemical analysis using PIXE yielded unexpected results by unveiling the presence of a single rhyolitic sample within the

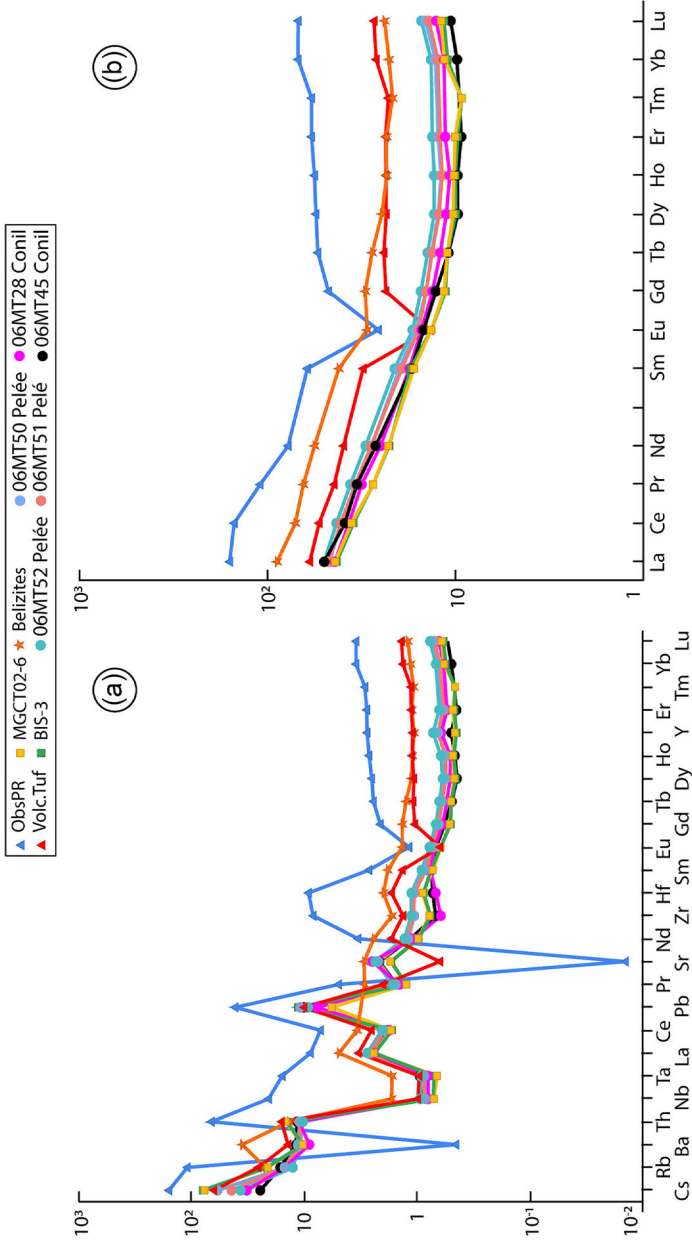


FIGURE 5 Comparison of both archaeological artefacts, the two obsidian samples analysed by LA-ICP-MS, with Belizites (from Rochette et al., 2021, Pb is below detection limit in this analysis) and some andesites from Northwest Martinique (from Labanieh, 2009, tm are lacking in these analysis). (a) N-MORB-normalised trace elements patterns. (b) Chondrite-normalised REE patterns.

corpus. These findings were subsequently confirmed through the application of other analytical techniques. The RRC-01 sample sets itself apart from other glassy artefacts in our study due to its distinct geochemical composition and its dark grey coloration (Figures 3 and 4). The combination of its chemical profile and macroscopic features strongly suggests it originates from the Volcan du Tuf, the sole known rhyolitic volcano in Guadeloupe, situated at a mere distance of less than 2 km from the archaeological site where it was unearthed.

Despite its uniqueness in the archaeological record, this artefact provides valuable insights. It attests the choice of experimenting with local materials when the knappable lithic raw materials in Guadeloupe are scarce. The low knapping quality of this obsidian underscores the Early Ceramic Age inhabitants' ability to work with relatively poor quality raw materials. Most of the lithic tools produced in the Lesser Antilles are based on good quality flint from Antigua, jasper from several islands, and radiolarite from La Désirade (de Waal & Knippenberg, 2020; Knippenberg, 2007; Queffelec et al., 2023; van Gijn, 1993). Interestingly, they decided to produce some flaking products with a poor quality material, while they could have used this shiny and unique material to produce other kind of artefacts such as the diverse beads or pendants made using other hard and difficult to work stones such as rock crystal, amethyst or nephrite (Queffelec, 2022; Raymond et al., 2022), including some rare black ones (Brock et al., 2020; Queffelec et al., 2020). We think that this novelty could be related to the exploration of yet uncharted areas of the islands, as Early Ceramic occupations are predominantly coastal, at least as evidenced by the archaeological record (Stouvenot & Casagrande, 2017).

Although it is possible that this particular raw material may never be encountered again in any other archaeological site, our work has established a geochemical reference for both a plausible source of volcanic glass in the Caribbean islands and for the artefact. Furthermore, the search for the source of this artefact has allowed us to discover the existence of a likely obsidian deposit in Puerto Rico, analyse a sample from it, and thereby provide a reference for the composition of this obsidian. This may prove highly valuable for future provenance studies if similar materials are uncovered in the Caribbean again.

Andesitic glass artefacts

The confirmed andesitic glass artefacts were recovered in a 50 km radius area, where the southern site is located on the southeast coast of Dominica and the northernmost site is located in the southern half of the island of Basse-Terre (Guadeloupe). If the artefact from Mill Reef (Antigua) was to be confirmed, it would significantly modify the geographical extent even if keeping it constrained to the central Lesser Antilles. Apart from two exceptions, they were all recovered in Early Ceramic context. Two samples originate from an archaeological project at the Caille à Bélasse site, on the Ilet de Petite Terre, which regrettably remains incomplete due to the passing of the excavator. This site, despite remaining unpublished, is considered to be Late Ceramic in age (Gagnepain, 2007).

The technological analysis conducted on the artefacts connects them all to a *chaîne opératoire* using bipolar percussion techniques. They do not differ in this way from the artefacts made from other raw materials in the sites where they were unearthed and in many sites from this period (Bérard, 2011; Rodriguez Ramos, 2001; Walker, 1979). It is therefore interesting to note that despite being a very unusual material, probably seen for the first time by the crafter, the method employed was exactly the same as with the jasper or the flint they usually knapped.

Given the poor quality of the material and the simplicity of the products, it seems unlikely that these rocks were part of an exchange network. They would therefore suggest that the same

group was highly mobile within different islands of the Guadeloupe archipelago, Dominica, and perhaps Antigua.

Despite intensive literature research and discussion with volcanologists, including glass specialists, and archaeologists specialised in obsidian, no such andesitic glass has been ever identified, neither in the Caribbean nor anywhere else in the world. This vitreous andesite, and its origin in particular, is thus of tremendous interest, both for archaeologists and geologists. There are two concurrent hypotheses: It was produced by volcanoes but never identified or studied by geologists, or it originates from an impact and would therefore add to the list of meteoritic material used by people in the past, beside meteoritic iron in Arctic, Egypt, or Europe (Buchwald, 1992; Comelli et al., 2016; Hofmann et al., 2023; Rehren et al., 2013), and impactites, especially Libyan glass (e.g. Lull García, 2014). Surprisingly, despite tektites being found in many archaeological sites, including some where obsidian was used as raw material for knapping, no mention of modified tektites has been found (Cornec et al., 2013; Hildebrand et al., 1994; Marwick et al., 2021).

The only mentions of naturally occurring andesitic composition glass we could locate all pertained impactites and tektites. Several rocks of this type exhibit significant similarities with our vitreous andesites: automorphic feldspars, originating from the pre-existing target rock of the meteorite impact, embedded within the perlite-structured impact glass (French, 1998, p. 90), as well as closely related elemental compositions (Izett, 1991): The chemical composition of the rock mainly copy the chemical composition of the impacted rock, it can therefore be glass of a very diverse range of compositions. Recently, a new field of tektites has been identified in Belize, of which unmodified elements have been found in some Mayan archaeological sites (Rochette et al., 2021). These rocks display elemental compositions quite similar to the andesites we are studying and are notably located in the Caribbean region. Several lithic materials found in the Caribbean islands already pointed to Central America, such as part of the jadeite jade celts (Garcia-Casco et al., 2013; Knaf et al., 2021) but also, as a working hypothesis, the green lapidary material sudoite, which could come hypothetically from Costa Rica (Queffelec et al., 2021; Queffelec et al., 2023).

We focused our efforts on investigating various indicators that could help us identify an extraterrestrial origin for these artefacts or, conversely, confirm a volcanic origin whose exact source might potentially be traced. First, as mentioned earlier, the raw nodules were likely generally spherical, akin to many tektites. However, their dimensions would have approached approximately 5 cm in diameter, which is an exceptionally large size for tektites especially if they were far from the impact (Montanari & Koeberl, 2000, p. 58). Furthermore, the chemical composition of the artefacts, even though they could be derived from several blocks, is highly uniform. Moreover, no variation in the chemical composition of the glass is observed at the microscopic scale with the SEM-EDS (Table S2). This characteristic is more commonly shared by volcanic rocks than by impact glasses, whether tektites or impactites, which can exhibit significant heterogeneity (French, 1998). Although some tektites can be relatively homogeneous (Rochette et al., 2021), their level of homogeneity is not comparable with what we observe here (Table S5).

No positive anomalies in chromium, cobalt, or nickel were observed, which are typically indicators of contamination by meteoritic material, frequently found in impactites (Koeberl, 2014). The profiles of trace elements and rare earth elements are, in turn, consistent with arc volcanism, specifically aligning with recent volcanic activity in the central part of the Lesser Antilles (Figure 5) where the artefacts were recovered.

A volcanic origin therefore appears to be the most likely scenario. However, as of now, we have not been able to trace the exact source of this material. The distribution of artefacts suggests a probable origin in the southern part of the Guadeloupean archipelago. Yet, this may be influenced by the robust state of archaeology in Guadeloupe due to standard and development-led archaeology, compared to neighbouring islands. In 2021, we conducted a survey in this

region, guided by a thorough review of geological literature, geological maps, and their descriptions. However, we did not find any vitreous andesite blocks; only porphyritic andesites were discovered, with a matrix that is largely vitrified but unsuitable for toolmaking. These rocks were analysed, and their compositions do not fit with the same volcanic event (Table 1, Figure 4). Comparisons with rocks from Guadeloupe or Dominica revealed pattern similarities in the general, qualitative shape of trace and REE spectra, pointing to the possibility of finding such patterns in andesites originating from subduction in the region. It is only with rocks from northern Martinique that qualitative and quantitative very good fits were observed (Figures 1 and 5). This region is particularly challenging to access, mostly uninhabited, and no surveys have been possible thus far. However, we plan to conduct explorations in the coming years to search for a source of vitreous andesite.

Another hypothesis that can be considered is that the original deposit has now vanished or is inaccessible. Indeed, erosion is quite pronounced in this tropical climate, where raining events can be important and even catastrophic in case of hurricanes. The deposit of these highly distinctive vitreous andesites is, or was, almost certainly very limited and may have been eroded. Additionally, the region experiences very active volcanic activity, and large portions of the land available to Early Ceramic inhabitants have been covered by recent eruption products. The fact that almost all artefacts were unearthed in sites dated to the Early Ceramic period may indicate that the source disappeared around 500 CE. Recent volcanic eruptions on the islands of Guadeloupe, Dominica, and Martinique are well known and may have covered the potential source, as, for example, the well-known P2 Plinian eruption, dated to 280 AD, which entirely covered archaeological sites in Martinique, like the site of Vivé, under a few cm of dilute pyroclastic flow deposits overlain by 10 to 50 cm of pumice fallout deposits (Carazzo et al., 2019).

CONCLUSION

We present here the first identification of natural glass artefacts in the Ceramic Age context in the Caribbean Islands. One among these samples is identified as obsidian *sensu stricto*, for which the origin is the Volcan du Tuf, located near the archaeological site, on the Western part of Basse-Terre island in Guadeloupe. This raw material, despite its bad quality for knapping, seems to have been attractive for inhabitants of Guadeloupe during the Early Saladoid, with which they produced at least a small flake by bipolar percussion, as they usually do with other raw materials. Looking for the obsidian sources in the Caribbean islands led us to identify an obsidian deposit in Puerto Rico, which does not appear to have been utilised by any population because of its current absence in the archaeological series. The deposit is only known through secondary sources but provides a massive and impurity-free material of very good quality for crafting. We provide here a geochemical reference framework that will make it possible to properly identify this material if it is ever found in an archaeological site. We present a geochemical reference framework here that will enable the proper identification of this material if it is ever found at an archaeological site.

The other artefacts have been identified as vitreous andesites, which are highly atypical rocks whose origin is a real challenge to identify. Although a formation related to a meteorite impact has been extensively considered, our observations and analysis lead us to favour the hypothesis of a volcanic origin. Geochemical similarities with volcanic deposits from Martinique demonstrate that the volcanism from the central part of the Lesser Antilles could produce andesitic lava of such a composition. However, despite geologists' work conducted in Guadeloupe and Martinique for decades, and despite some field surveys in Guadeloupe, we have not been able to locate the original deposit or even find a few secondary vitreous andesites. Therefore, we seriously consider the possibility that the source, if it was located in Guadeloupe or Martinique, could now be lost, perhaps covered by more recent volcanic deposits. Additional

field surveys on the Northwestern coast of Martinique might be interesting to organise in the future. The thorough mineralogical and geochemical description of the artefacts will allow to easily connect any new discovery of artefacts or raw material to these samples.

Surveying the literature and the community of archaeologists in the Antilles, we confirmed that the use of volcanic glass is extremely rare and that, outside the Guadeloupean artefacts, currently only one artefact is confirmed in Dominica and one still needs to be confirmed in Antigua. This constrained geographical distribution allows us to make the hypothesis that the source material would be located in Guadeloupe or a neighbouring island. Although we have not yet identified the original source of these vitreous andesites, this study allows us to include this material in the limited list of materials discovered in Antillean archaeological sites for which provenance can be traced. The list is currently rather short and is composed of the Antigua flint, the St. Martin calci-rudite and cherto-tuffite, and the numerous lapidary raw materials. By adding two new geochemically fingerprinted raw materials to this list, this study may facilitate future endeavours to determine the provenance of similar artefacts and help reconstruct the lithospaces of different sites (Delvigne et al., 2021). It is only by conducting and combining the analysis of the techno-economical pattern of different lithospaces of sub-contemporaneous sites that one can approach a dynamic vision of the structure and relations that existed during the Early Ceramic inhabitants of the Caribbean islands.

AUTHOR CONTRIBUTIONS

Conceptualization: Alain Queffelec, Arthur Leck, and Christian Stouvenot. *Funding acquisition:* Alain Queffelec. *Investigation:* Arthur Leck, Alain Queffelec, Bernard Gratuze, Céline Leandri, Isaac Shearn, Ludovic Bellot-Gurlet, Guillaume Carazzo, and Jessica Langlade. *Methodology:* Arthur Leck, Alain Queffelec, and François-Xavier Le Bourdonnec. *Formal analysis:* Arthur Leck. *Project administration:* Alain Queffelec. *Resources:* Alain Queffelec, Bernard Gratuze, Christian Stouvenot, François-Xavier Le Bourdonnec, Isaac Shearn, and Ludovic Bellot-Gurlet. *Supervision:* Alain Queffelec. *Visualisation:* Arthur Leck and Alain Queffelec. *Writing—original draft:* Arthur Leck and Alain Queffelec. *Writing—review and editing:* All authors.

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DATA AVAILABILITY STATEMENT

The data that supports the findings of this study are available in the supplementary material of this article

PEER REVIEW

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