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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 horti-ceramicists 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). Elementary 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 sub-local 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, 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.

Keywords

Obsidian, Andesite, Lesser Antilles, Ceramic Age, Impactites, Tektites, Sourcing

Introduction

The archipelago of Caribbean islands was settled late in the history of the peopling of America, firstly by hunter-gatherers during what is called the Archaic Age, and from ca. 500 B.C onwards, by horti-ceramicists 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 serie (Rouse 1992), has been 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 non-perishable 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 (Walker 1979; Bérard 2011) 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 proxy employed by archaeologists to study the regional mobility and networks in the Caribbean islands (e.g. Cody 1993; Knippenberg 2007; Garcia-Casco et al. 2013; Knaf et al. 2021; 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 crystallisation (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 and 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 (Glascock 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 and 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 they rapidly cool and vitrify before falling back to Earth hundreds or even thousands of kilometres from the impact site - these are known as tektites (Osinski and Pierazzo 2013). Tektites, as micro-fragments 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 and Boynton 1992), and in any case, these rocks are way 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”, shakes things up: the largest of these tektites can form nodules a few centimetres in diameter (Rochette et al., 2021 figure 2).

In this article, we present the first-ever 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 proposed, 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 that had been identified in the eastern part of Guadeloupe 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 scrutinise sixteen 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.

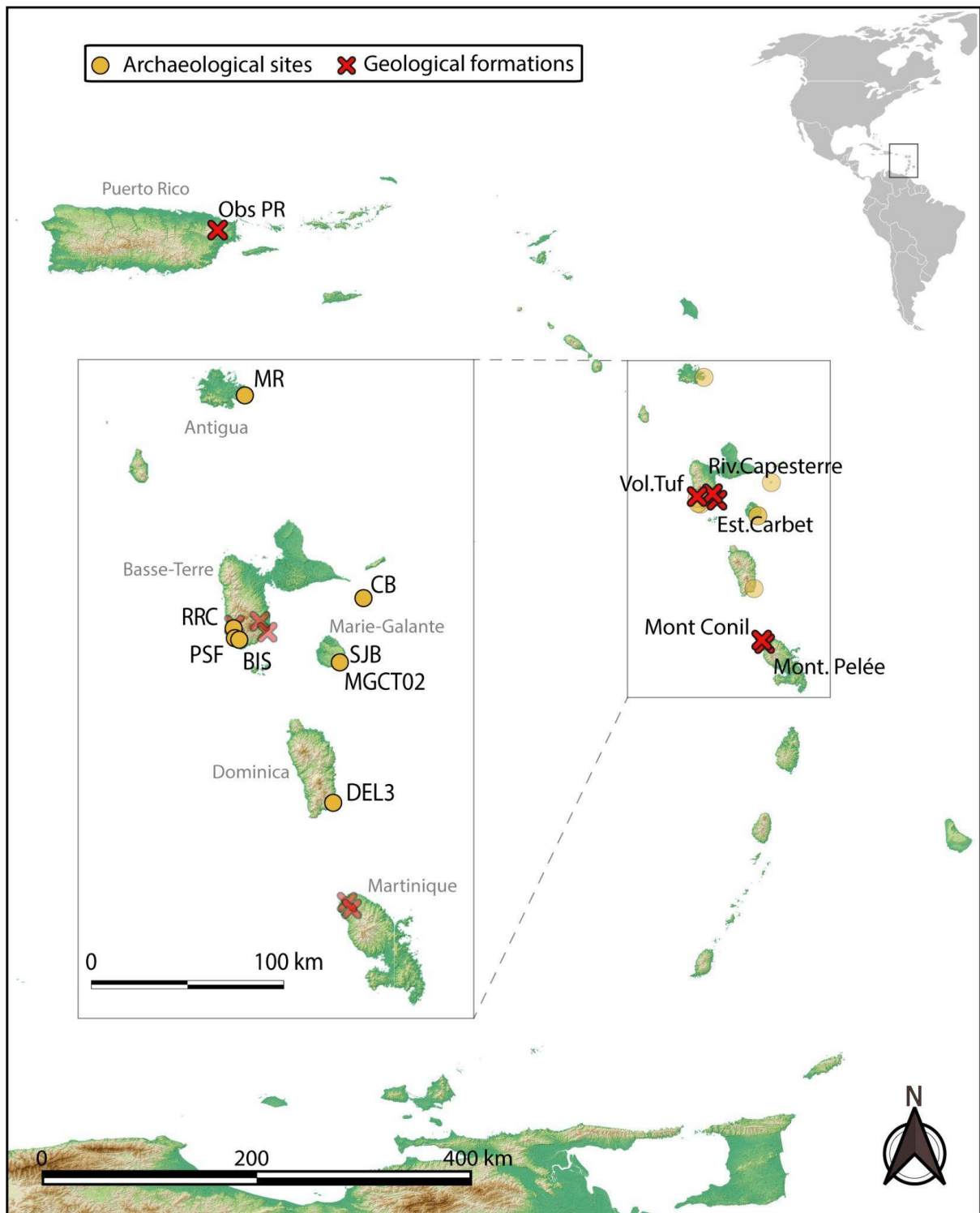


Figure 1 Map indicating the locations of the archaeological sites from which the artefacts originate, and the locations of the geological deposits that have been sampled (Guadeloupe and Puerto Rico) or are scheduled for sampling but already used as comparative data (Martinique). SJB and MGCT02 are located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

<i>Artefacts from archaeological sites</i>																
Sample Code	Site	City	Island	Longitude	Latitude	Period	Bibliography	Mass (g)	Type	SEM-EDS	PIXE	EPMA	LA-ICP-MS	ED-XRF	Raman	Microtomo graphy
Bis-01	Bisdary	Gourbeyre	Basse-Terre	-61.709	15.988	Early Ceramic	Romon et al., 2006	1.312	Flake		X			X		X
Bis-02	Bisdary	Gourbeyre	Basse-Terre	-61.709	15.988	Early Ceramic	Romon et al., 2006	2.21	Flake	X	X			X		X
Bis-03	Bisdary	Gourbeyre	Basse-Terre	-61.709	15.988	Early Ceramic	Romon et al., 2006	4.318	Nucleus		X	X	X	X	X	X
Bis-04	Bisdary	Gourbeyre	Basse-Terre	-61.709	15.988	Early Ceramic	Romon et al., 2006	2.367	Flake	X	X			X	X	X
RRC-01	Ruelle Roches Caraïbes	Baillif	Basse-Terre	-61.736	16.038	Early Ceramic	Bonnissent & Chancerel, 2001	2.733	Flake		X			X	X	X
RRC-02	Ruelle Roches Caraïbes	Baillif	Basse-Terre	-61.736	16.038	Early Ceramic	Bonnissent & Chancerel, 2001	5.504	Undetermined	X	X			X	X	X

MGCT02-1	Tourlourous	Capesterre-de-Marie-Galante	Marie-Galante	-61.224	15.889	Early Ceramic	Colas, 2002	1.394	Flake		X			X	X	X
MGCT02-2	Tourlourous	Capesterre-de-Marie-Galante	Marie-Galante	-61.224	15.889	Early Ceramic	Colas, 2002	4.063	Flake	X	X			X		X
MGCT02-3	Tourlourous	Capesterre-de-Marie-Galante	Marie-Galante	-61.224	15.889	Early Ceramic	Colas, 2002	N.A.	Lost							
MGCT02-4	Tourlourous	Capesterre-de-Marie-Galante	Marie-Galante	-61.224	15.889	Early Ceramic	Colas, 2002	2.792	Nucleus		X			X		X
MGCT02-5	Tourlourous	Capesterre-de-Marie-Galante	Marie-Galante	-61.224	15.889	Early Ceramic	Colas, 2002	0.156	Flake	X	X			X		X
MGCT02-6	Tourlourous	Capesterre-de-Marie-Galante	Marie-Galante	-61.224	15.889	Early Ceramic	Colas, 2002	16.896	Flake		X	X	X	X		X
MGCT02-7	Tourlourous	Capesterre-de-Marie-Galante	Marie-Galante	-61.224	15.889	Early Ceramic	Colas, 2002	2.682	Flake	X	X			X		X
CB-050a	Caille à Bélasse	Terre de Bas	Ilets de Petite Terre	-61.11	16.17	Late Ceramic	Gagnepain, 2007	0.901	Flake	X	X			X	X	X
CB-051a	Caille à Bélasse	Terre de Bas	Ilets de Petite Terre	-61.11	16.17	Late Ceramic	Gagnepain, 2007	0.299	Flake	X	X			X		X

DEL-3-1	Delices 3	Saint Patrick Parish	Dominique	-61.256	15.275	Early Ceramic	Shearn, 2014	0.373	Flake	X				X		X
SJB-1	Stade José Bade	Capesterre-de-Marie-Galante	Marie-Galante	-61.224	15.889	Early Ceramic	Serrand, 2016		Flake					pXRF	X	
PSF-1	Place Saint François	Basse-Terre	Basse-Terre	-61.73	15.995	Early Ceramic	Sellier-Ségard, 2001		Flake (Lost)		X					
Not Included Yet	Mill Reef+	Saint Philip	Antigua	-61.679	17.057	Middle/Late Ceramic	Murphy, 1999		Nucleus							

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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

110 Table 1 List of archaeological and geological sites with volcanic glass artefacts or glassy geological samples (+: yet to be confirmed) and their location. Bis-05 and MGCT02-3
111 were inventoried but are now lost. PSF-1 was analysed in PIXE but is now lost. SJB-1 could only be analysed by pXRF during a preliminary study and could not be brought to
112 the laboratory for logistical reasons.

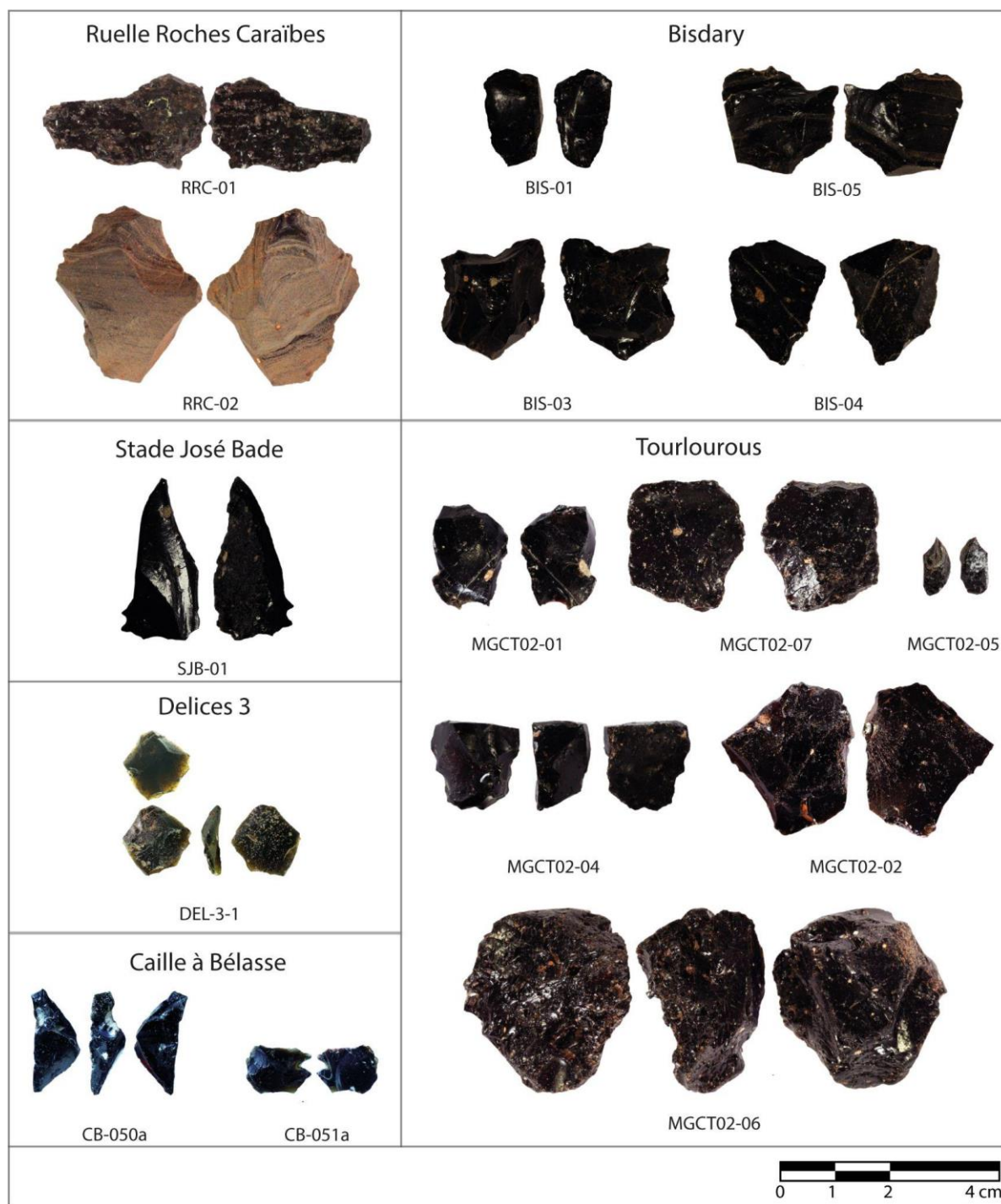


Figure 2 Macrophotographs of the sixteen glassy artefacts recovered from six archaeological sites.

Out of the nineteen artefacts, seventeen originate from archaeological sites situated in Guadeloupe. Bisdary is "located at an elevation of 100 metres on the foothills of the Caribbean Mountains, 1 kilometre from the sea" (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 the excavation, and were analysed in 2007 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 and 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. Place Saint François was excavated in 2001, just in front of the cathedral of Basse-Terre, and is interpreted as a Cedrosan Saladoid occupation (Sellier-Ségard 2001). 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 (Bonnissent and 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 southwestern 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's 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, since 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 sixteen studied artefacts 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. Technical drawings and surface views from the 3D models acquired by microtomography are presented in Supplementary Information (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) never saw 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 (Smith et al. 1980; Le Guen de Kerneizon et al. 1982). The only described obsidian, 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). Our 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. While there is documented evidence of a silicic suite in the northeastern Antilles, primarily dating back to the Albian age (113.0 ± 1.0 Ma 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 non-destructive 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).

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, while 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 (50kV/1mA) and a Vortex SDD detector, and a specialised protocol was developed specifically for the study of silica-rich archaeological artefacts (Leck et al. 2018; Leck 2022). The PCA was carried out using CoDaPack software (Comas and 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 big 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 sub-samples 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 Ouest* 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], etc.), 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 2 to 6 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 twelve 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, 5mJ 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 seconds and the acquisition of 9 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 devitrifications 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 micro-sampling for EPMA and LA-ICP-MS, 3D reconstruction of the surface and the inner structure of the samples was realised 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 µm³ per voxel. AQ extracted isosurfaces from these 3D models using the Avizo software.

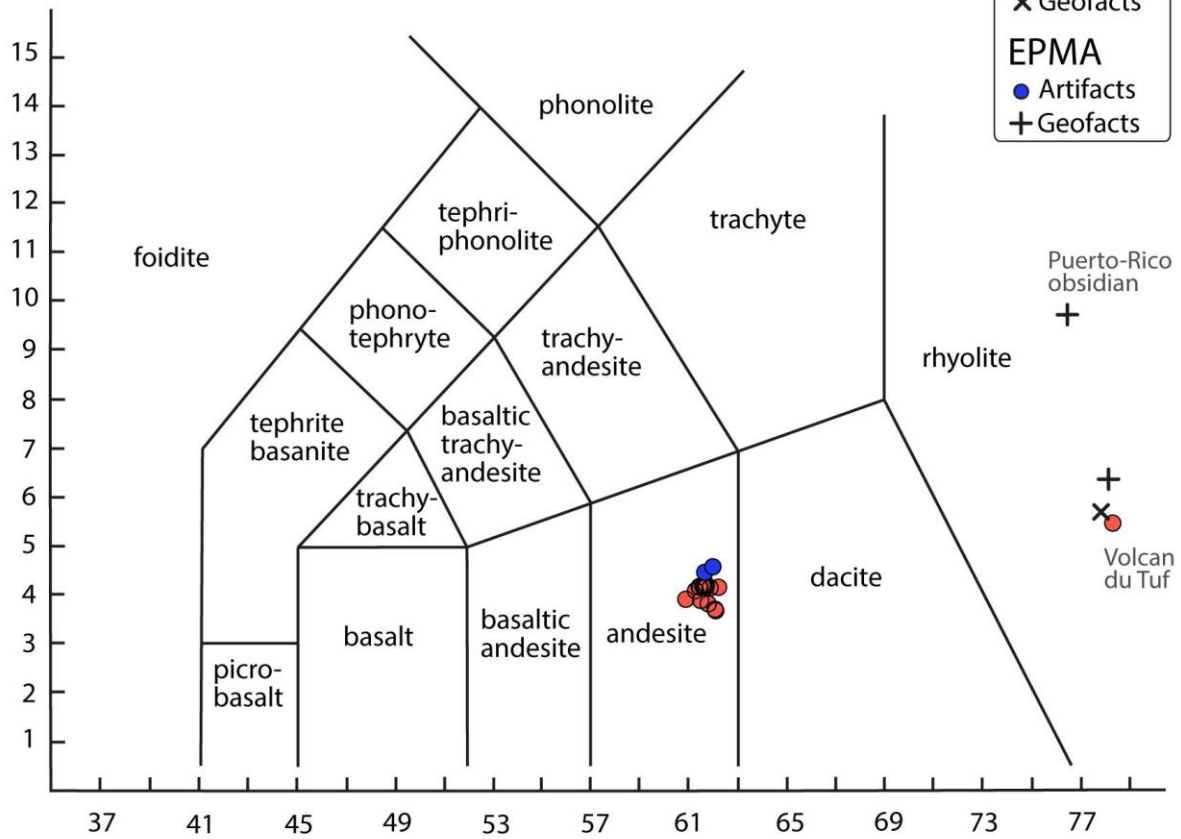
Results

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 bi-pyramidal, showing fluidity and probably resulting from crystallisation in the magma chamber or chimney, others are definitely devitrification spherules (Figure 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. Lastly, 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 five centimetres. It is also conceivable that these blocks were likely collected in a primary or sub-primary 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 sub-millimetric 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.

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

Na₂O + K₂O (wt%)



SiO₂ (wt%)

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, while the geological samples are composed of obsidian, a more typical rhyolitic glass.

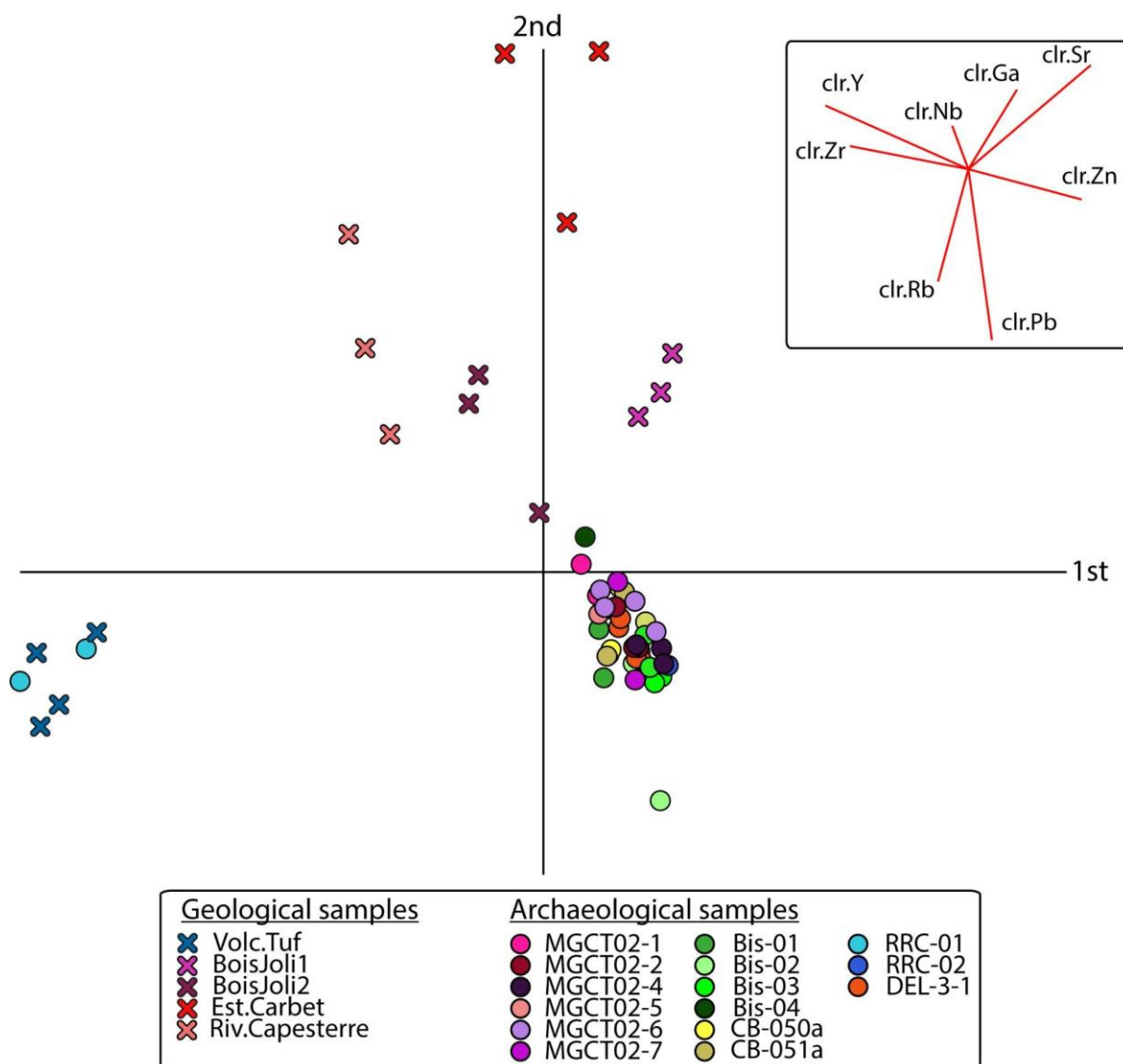


Figure 4 PCA on ED-XRF data (trace elements only) from all artefacts as well as geological samples collected in the Guadeloupean archipelago (rhyolitic and andesitic). Each point on the graph relates to one measurement on the sample.

Based on these non-invasive 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 non-archaeological 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).

EPMA (%)	ObsPR	VolcanTuf	MGCT02-6	BIS-03
SiO₂	76,36	78,02	61,74	62,00
TiO₂	0,22	0,15	0,57	0,57
Al₂O₃	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
Na₂O	4,92	4,22	3,35	3,48
K₂O	4,83	2,22	1,29	1,29
P₂O₅	0,02	0,04	0,23	0,23
SO₂	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
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

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

For the andesitic archaeological artefacts, no enrichment in Cr, Ni, or Co are visible in the archaeological artefacts, indicating that no extra-terrestrial 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 (Labanieh 2009; Germa et al. 2011; Howe et al. 2015; Fedele et al. 2021) and if the 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).

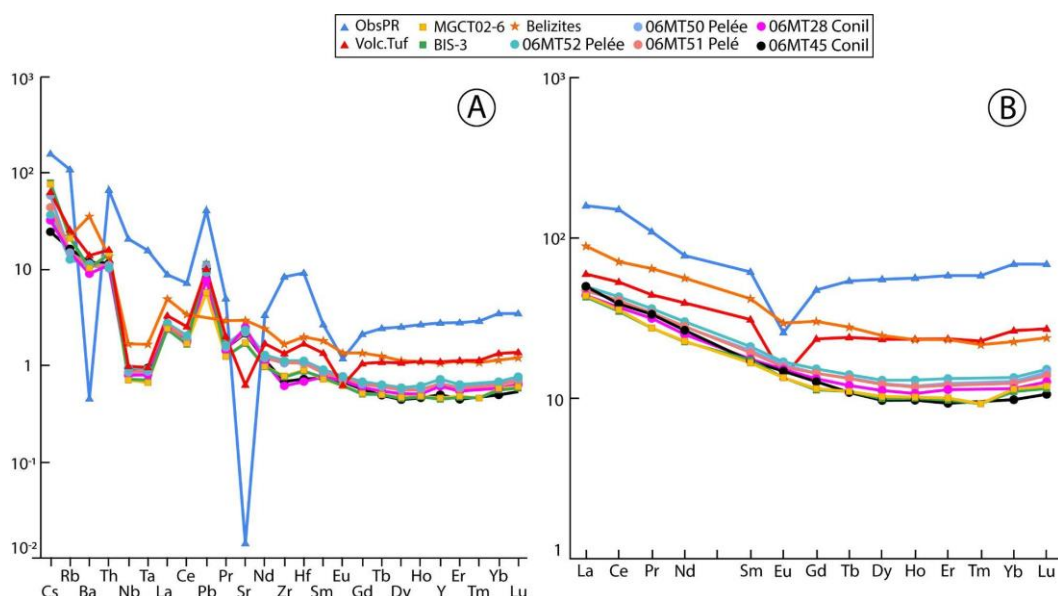


Figure 5 Comparison of both archaeological artefacts, the two obsidian samples analysed by LA-ICP-MS, with Belizites (from Rochette et al. 2021, Pb is below detection limit in this analysis) and some andesites from northwest Martinique (from Labanieh, 2009, Tm are lacking in these analysis). A. N-MORB-normalised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

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 eighteen elements 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). While 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 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 (Figure 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 kilometres 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. Although 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 (van Gijn 1993; Knippenberg 2007; de Waal and Knippenberg 2020; Queffelec et al. 2023). 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 they used to produce, 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 and Casagrande 2017).

While 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) were 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 archaeology dig 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 (Walker 1979; Rodriguez Ramos 2001; Bérard 2011). 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 hypothesis: 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; Rehren et al. 2013; Comelli et al. 2016; Hofmann et al. 2023), 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 (Hildebrand et al. 1994; Cornec et al. 2013; Marwick et al. 2021).

The only mentions of naturally occurring andesitic composition glass we could locate all pertained to 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. Firstly, as mentioned earlier, the raw nodules were likely generally spherical, akin to many tektites. However, their dimensions would have approached approximately five centimetres in diameter, which is an exceptionally large size for tektites especially if they were far from the impact (Montanari and 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. While 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. While 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 Antiguan 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 litho-spaces of different sites (Delvigne et al. 2021). It is only by conducting and combining the analysis of the techno-economical pattern of different litho-spaces 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.

453

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471

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472

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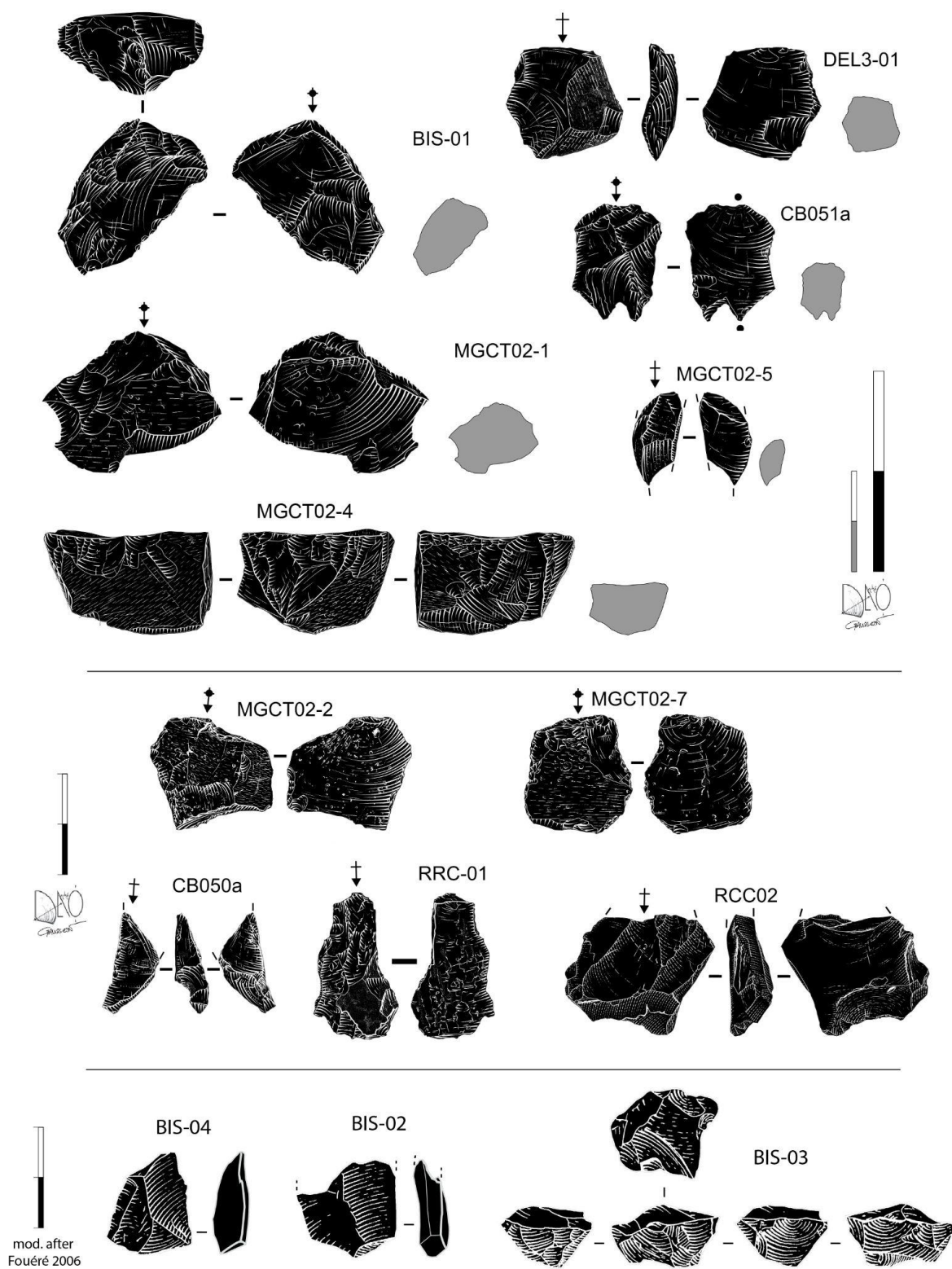
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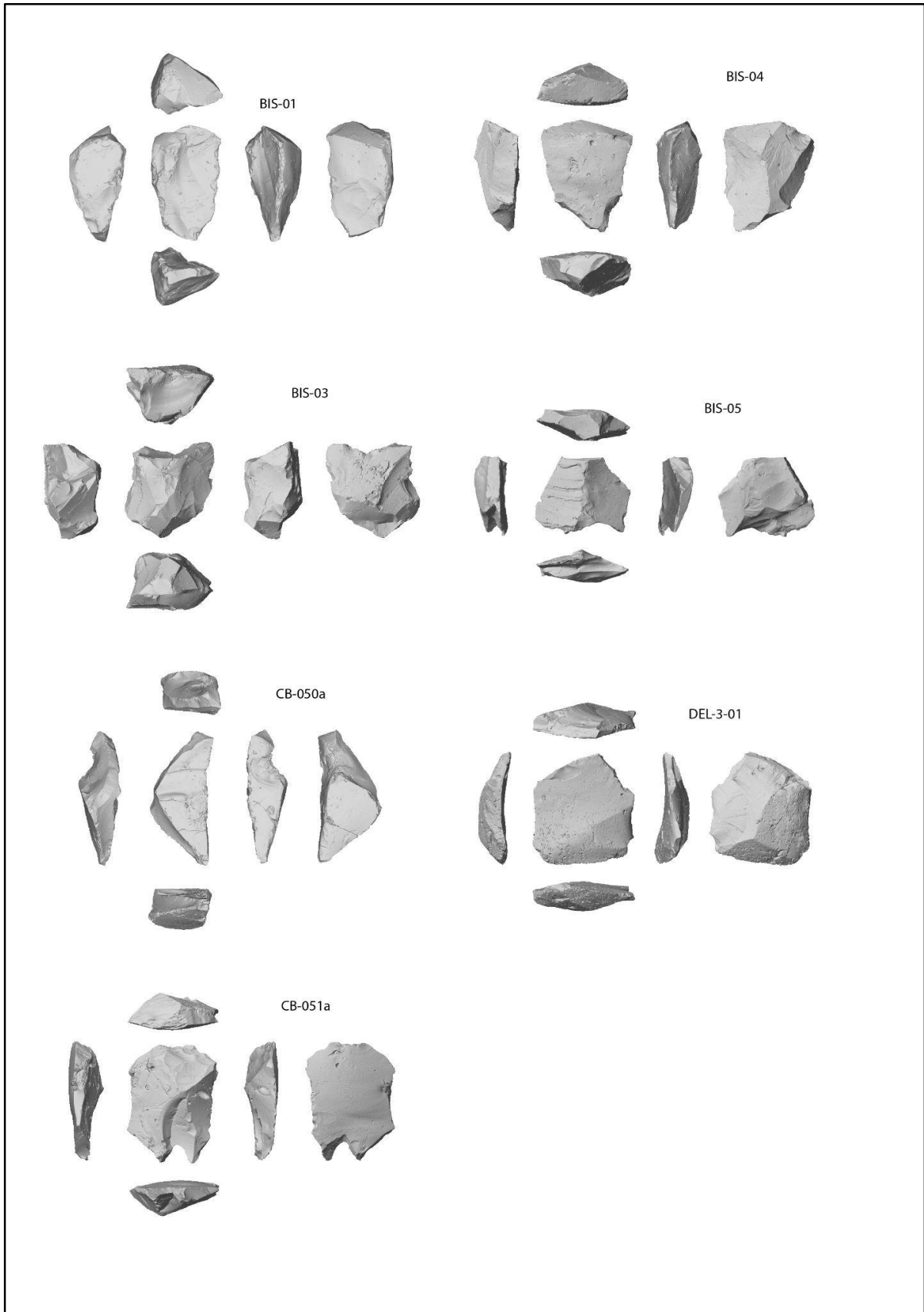
Supplementary information



mod. after
Fouéré 2006

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707 Figure S1 Technical drawing of a selection of the most interesting artefacts on the technological point of view.
708 Drawings by P. Gaussein except BIS-02, BIS-03, and BIS-04 by P. Fouéré. Infographic by AQ.



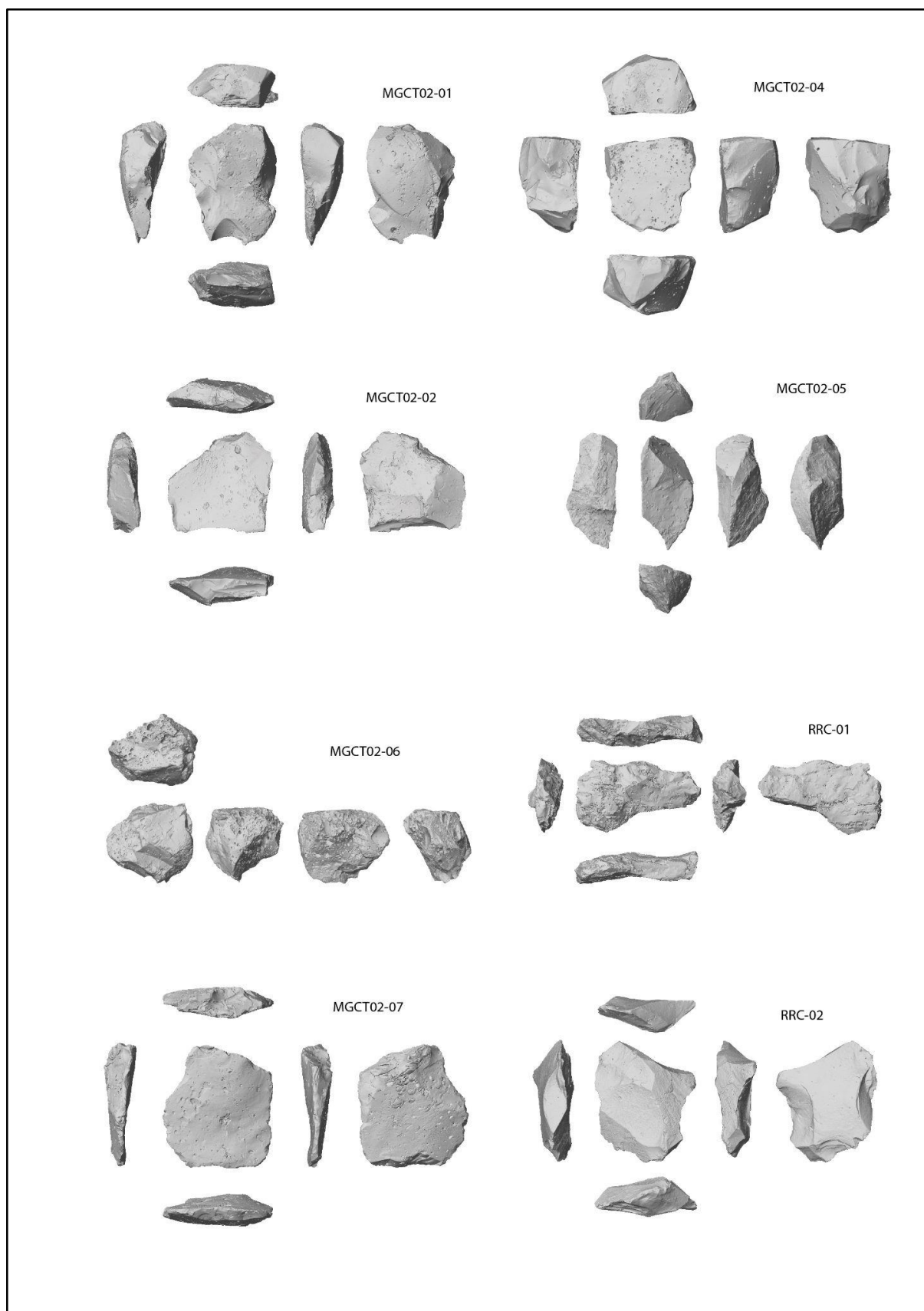
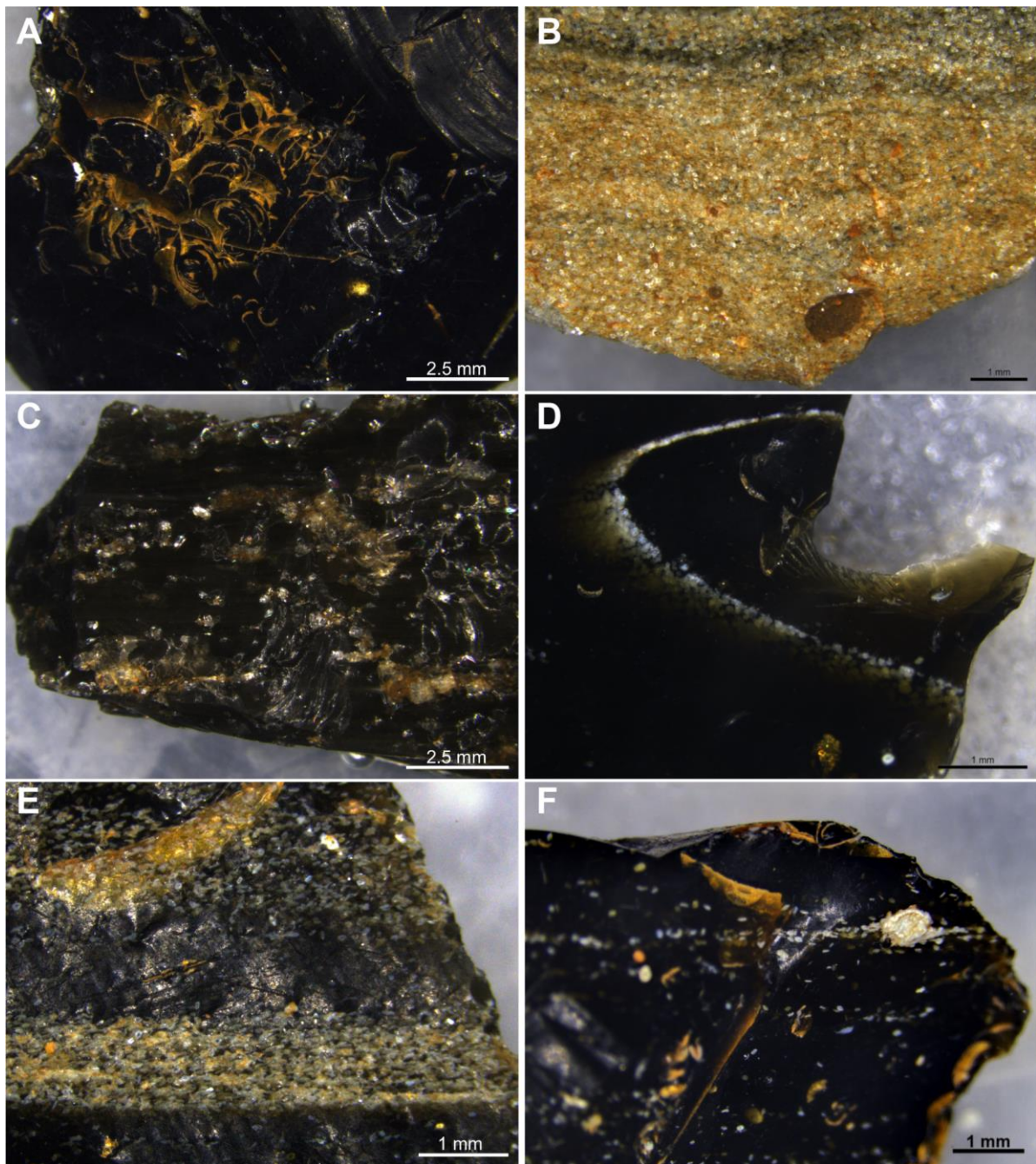


Figure S2 Developed view of the surface of artefacts extracted from the 3D models from microtomography acquisitions prior to the microsampling of two samples. Acquisition by NV, Surface extraction and infographic by AQ



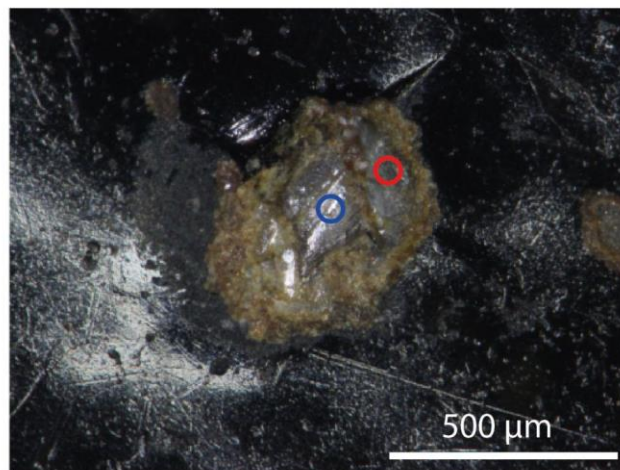
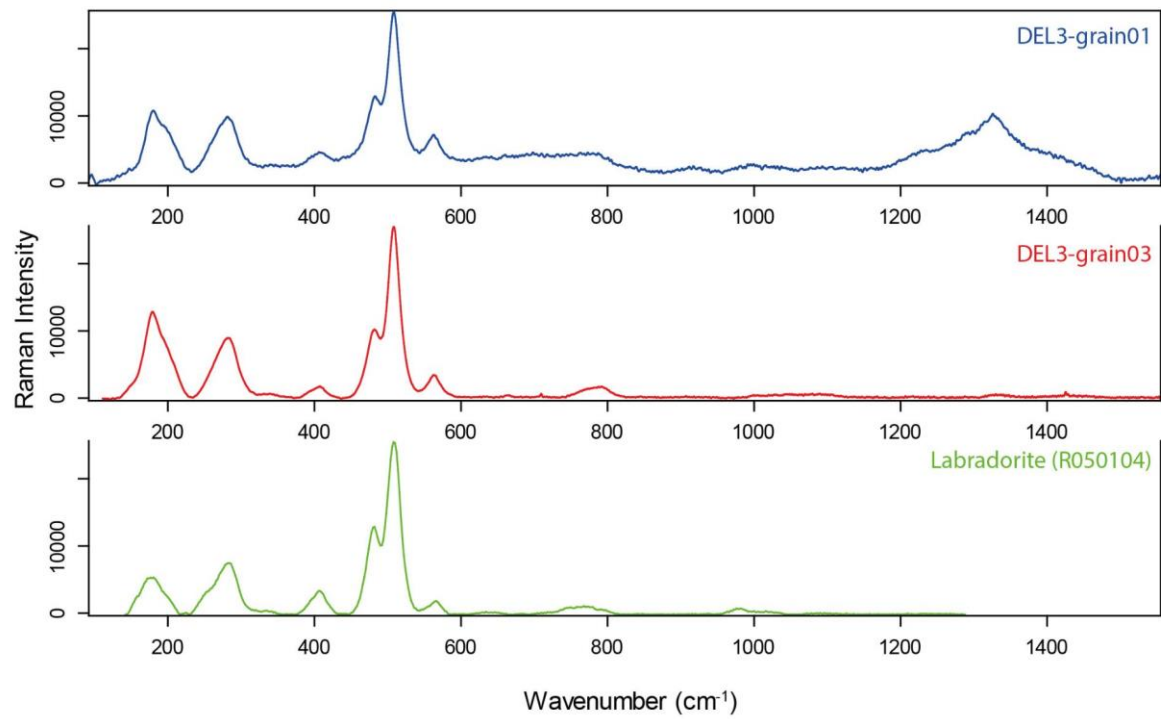
Figure S3 (A) Glassy pumice in an ash tuff in the process of weathering, Volcan du Tuf. (B) Block of tuff from the Volcan du Tuf containing numerous glassy inclusions, some blocks must have contained inclusions large enough to be cut. (C) Porphyritic andesites from Bois Joli (Les Saintes) containing large quantities of glass but not enough to make tools.



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721 Figure S4 Microphotographs of archaeological samples, showing perlitic texture in BIS-03 (A), the high quantify
 722 of crystals in RRC-02 (B), the quasi-absence of crystals in RRC-01 (C), and the planar distribution of crystals in
 723 CB051 (D), BIS-02 (E) and BIS-01 (F). Pictures and infographic by AQ

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Figure S5-1 Raman spectra acquired on two different grains (one in photo) in the DEL-3-1 flake.

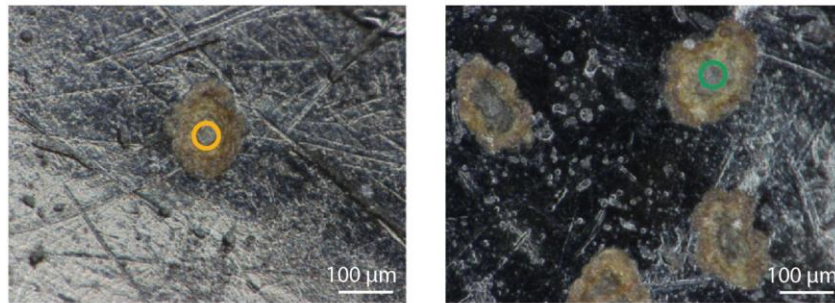
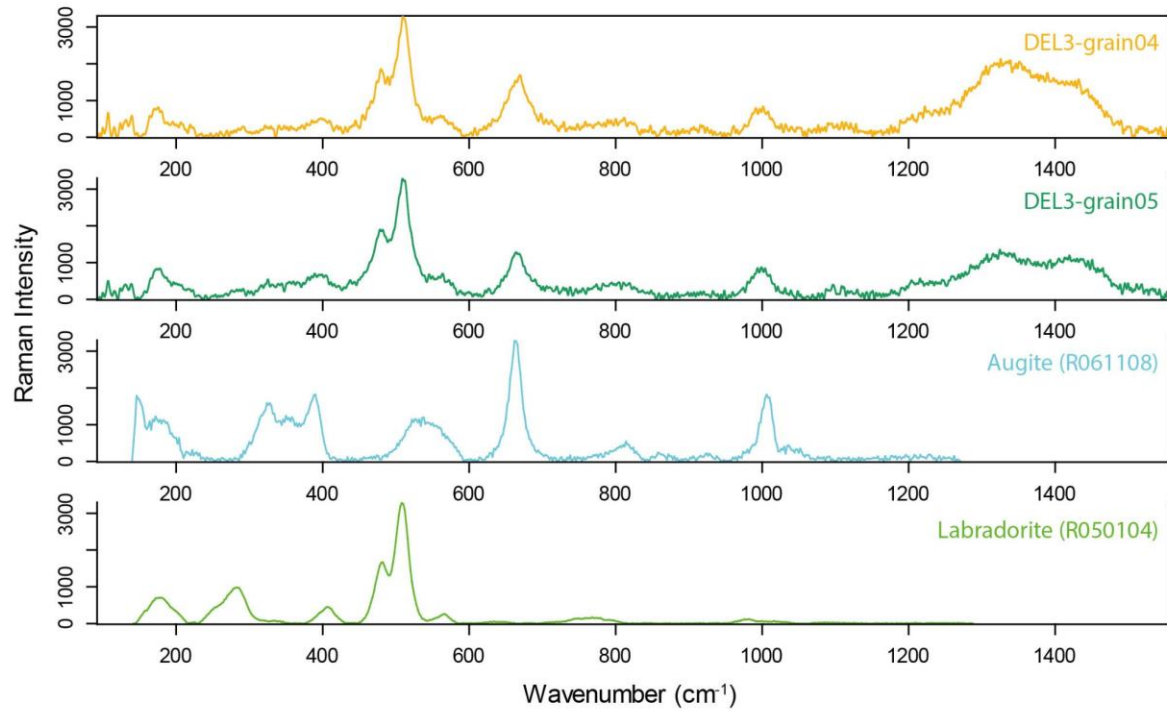


Figure S5-2 Raman spectra acquired on two different grains (colours of circle in picture is equivalent to colours of spectra) in the DEL-3-1 flake. Despite the measured spot size of a few microns, both spectra exhibit a mix of labradorite and augite.

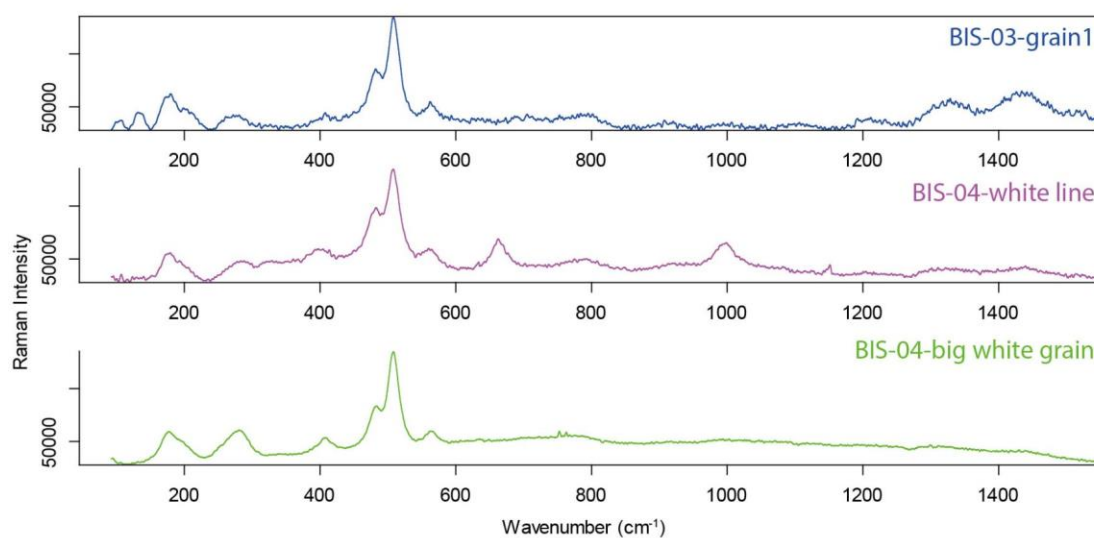
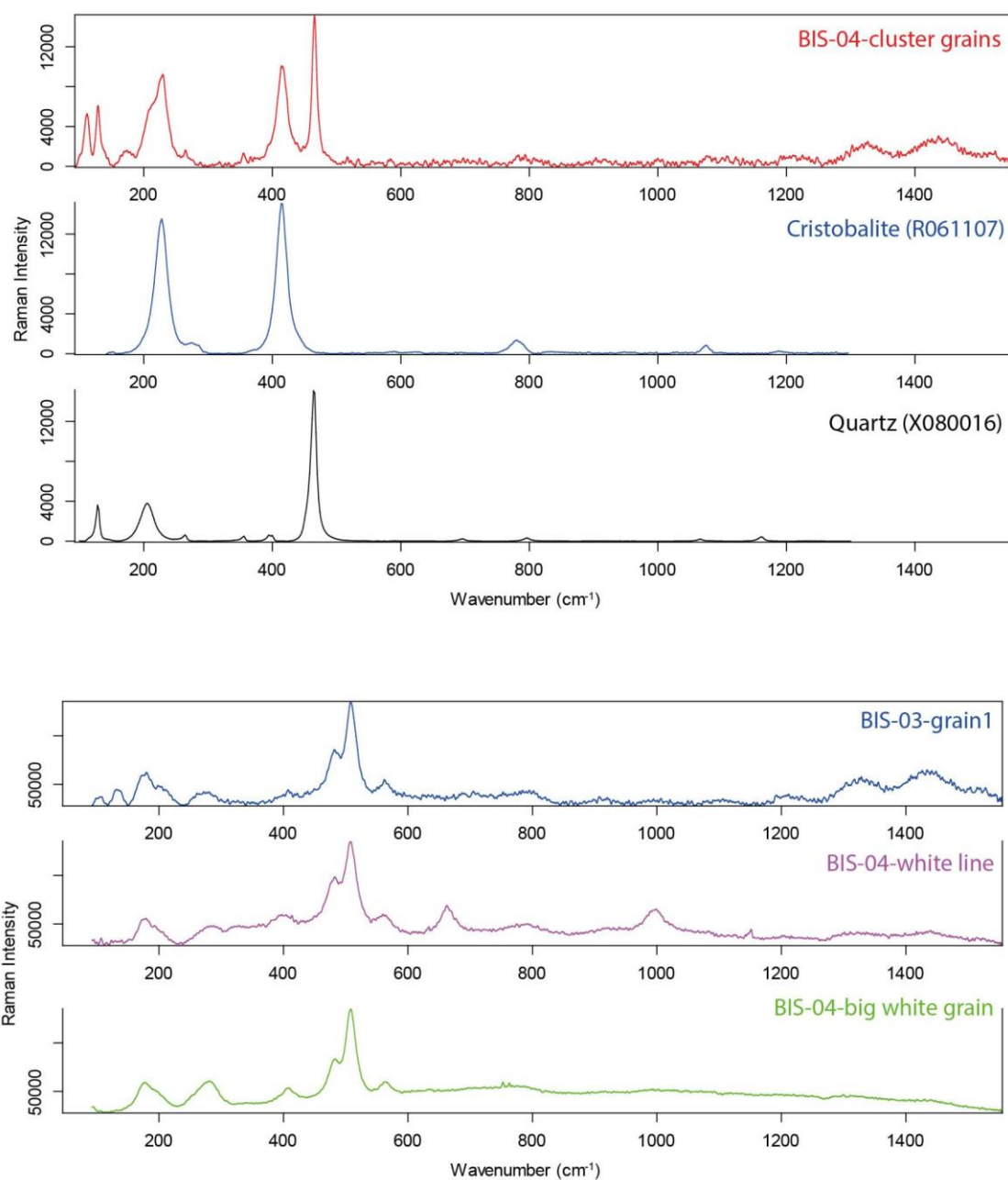


Figure S5-3 Raman spectra acquired in clusters of grains on the BIS-04 and BIS-04 artefacts, showing the presence of quartz and cristobalite, as well as labradorite.

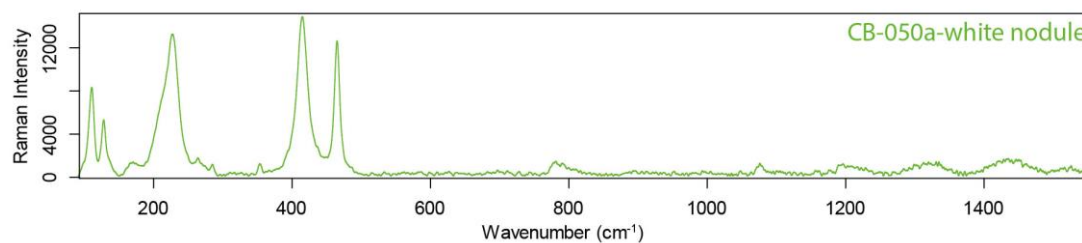


Figure S5-4 Raman spectrum acquired on a white nodule on the CB-050a artefact, showing the presence of both quartz and cristobalite in the same analysed spot.

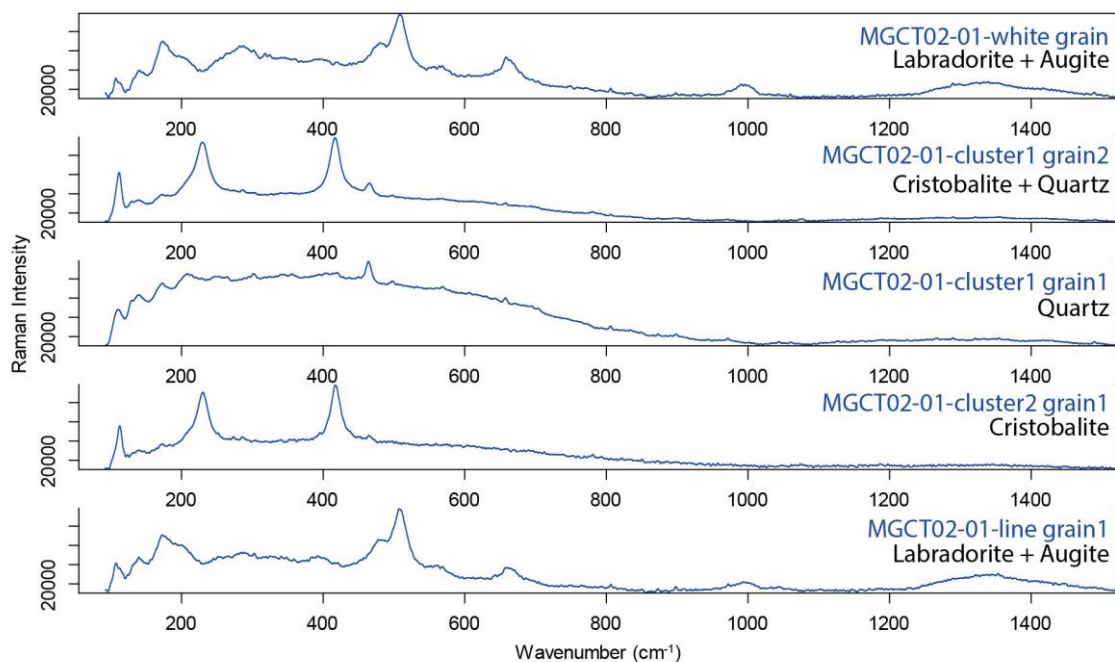


Figure S5-5 Raman spectra acquired in several clusters or lines of grains on the MGCT02-01 artefact showing the presence of quartz, cristobalite, augite, and labradorite in these crystallisation spots.

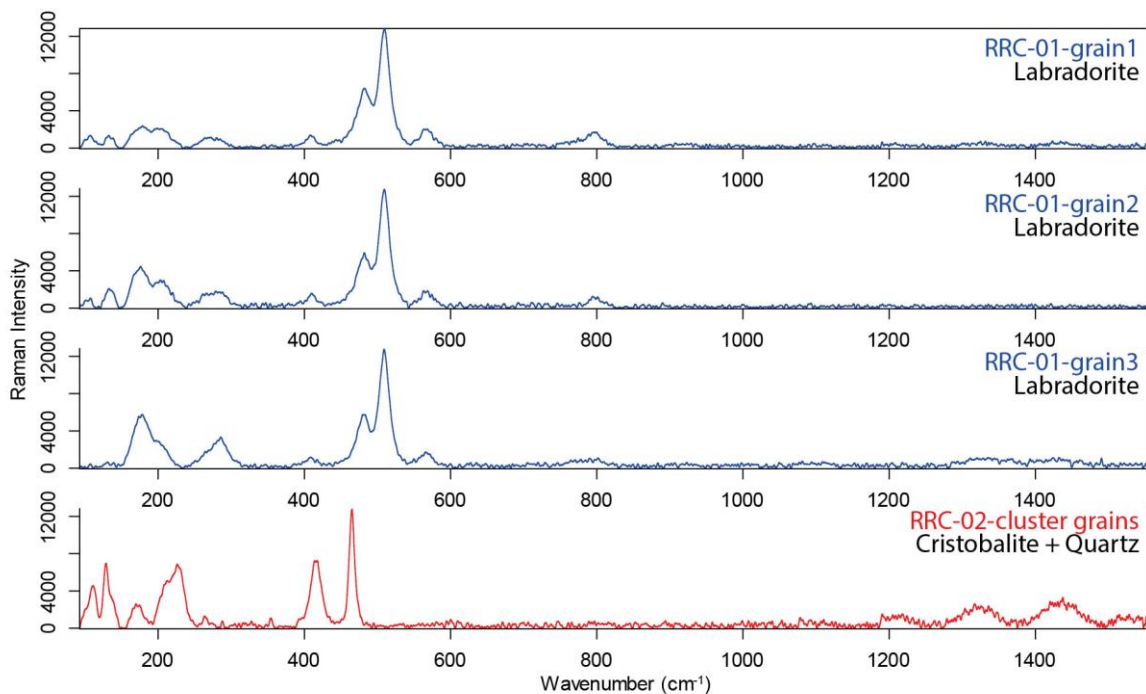
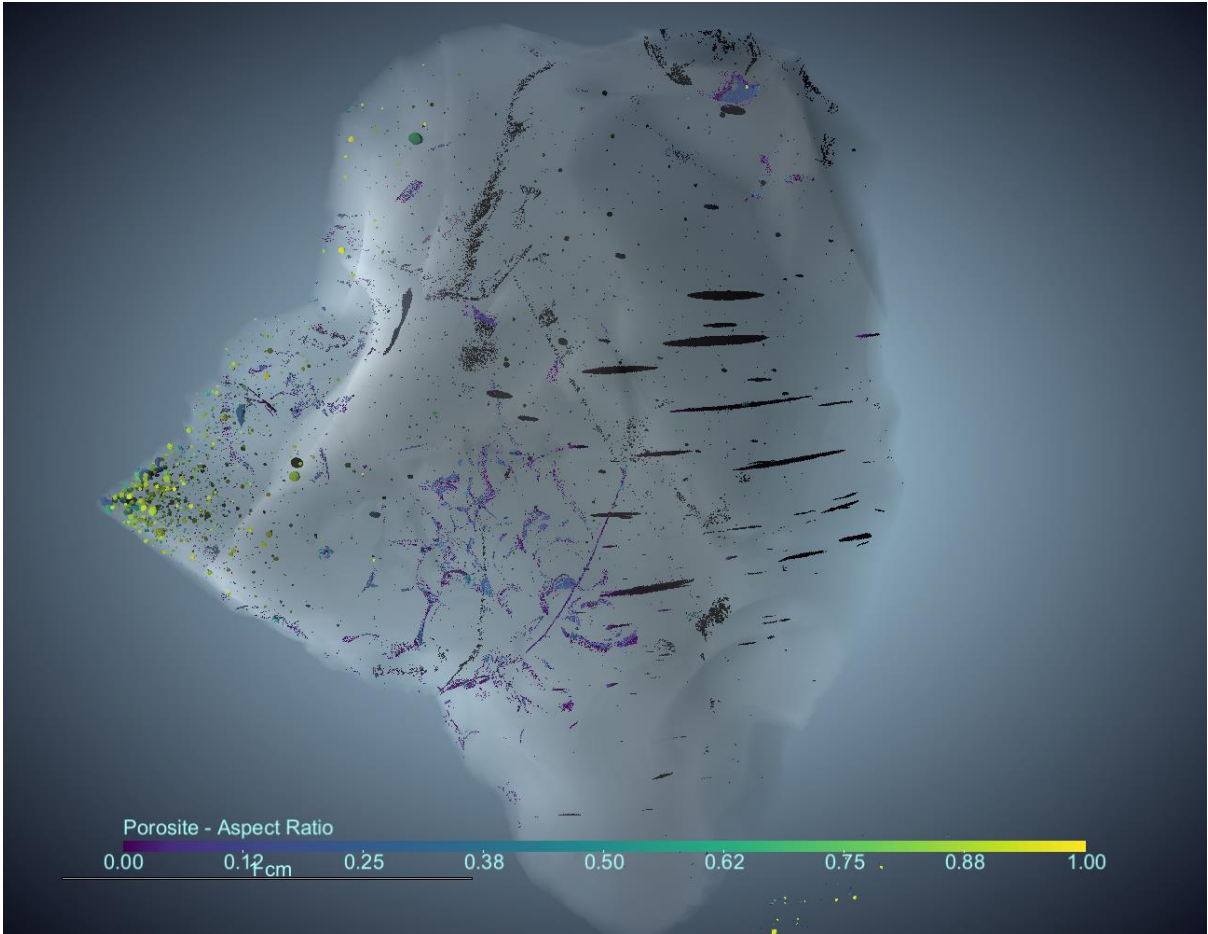


Figure S5-6 Raman spectra acquired on several grains on the RRC-01 and RRC-02 artefacts, labradorite on the former, and of quartz and cristobalite in the later

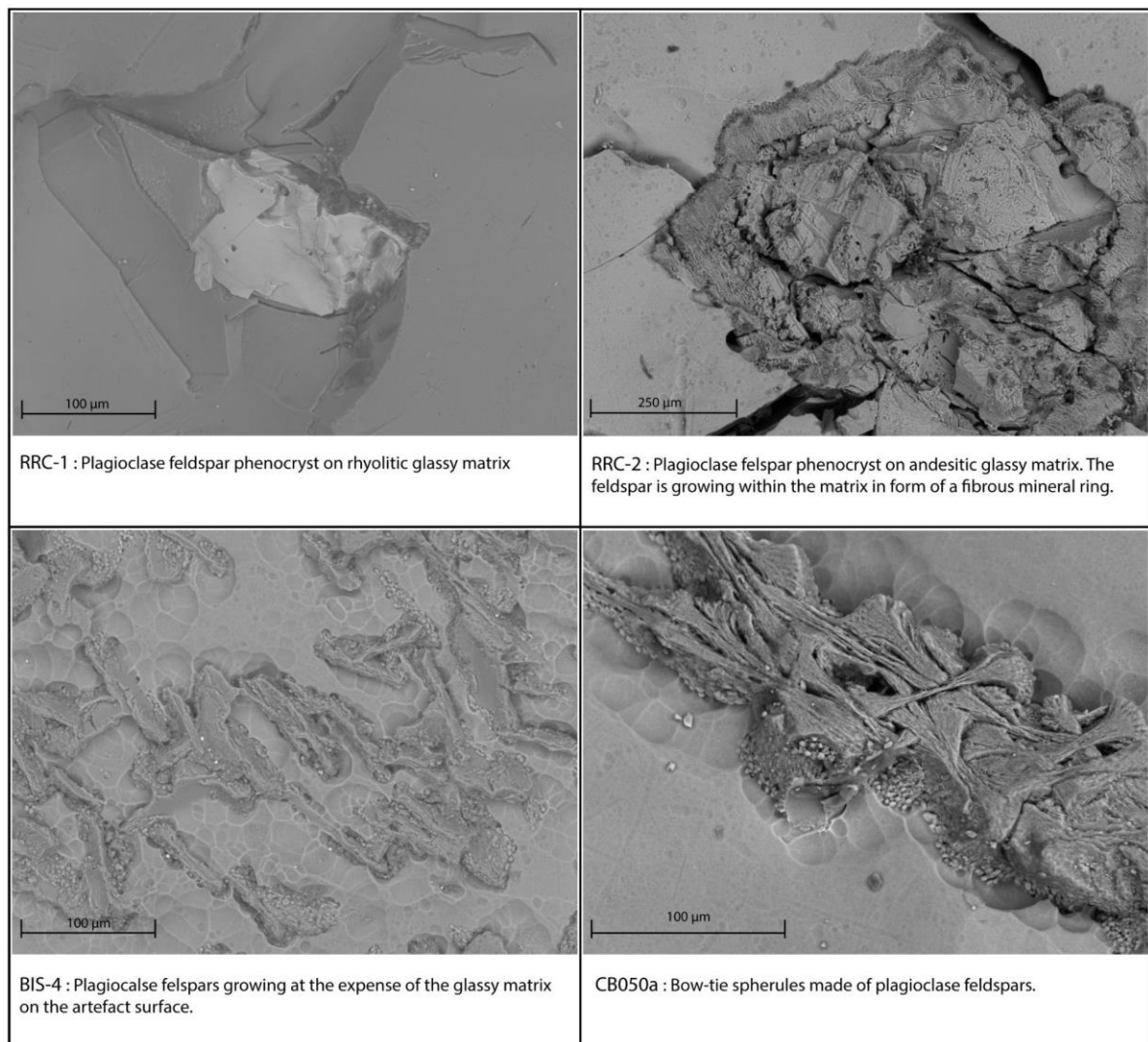
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745 Figure S6 Microtomography view of the internal distribution of bubbles in the artefact BIS-03. Pores are
746 colourized by their sphericity. Figure by NV.

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Figure S7 Plagioclase phenocrysts and fibrous feldspar spherules in the RRC-1, RRC-2, BIS-04 and CB050a samples (after H₂O₂ + ultrasound cleaning)

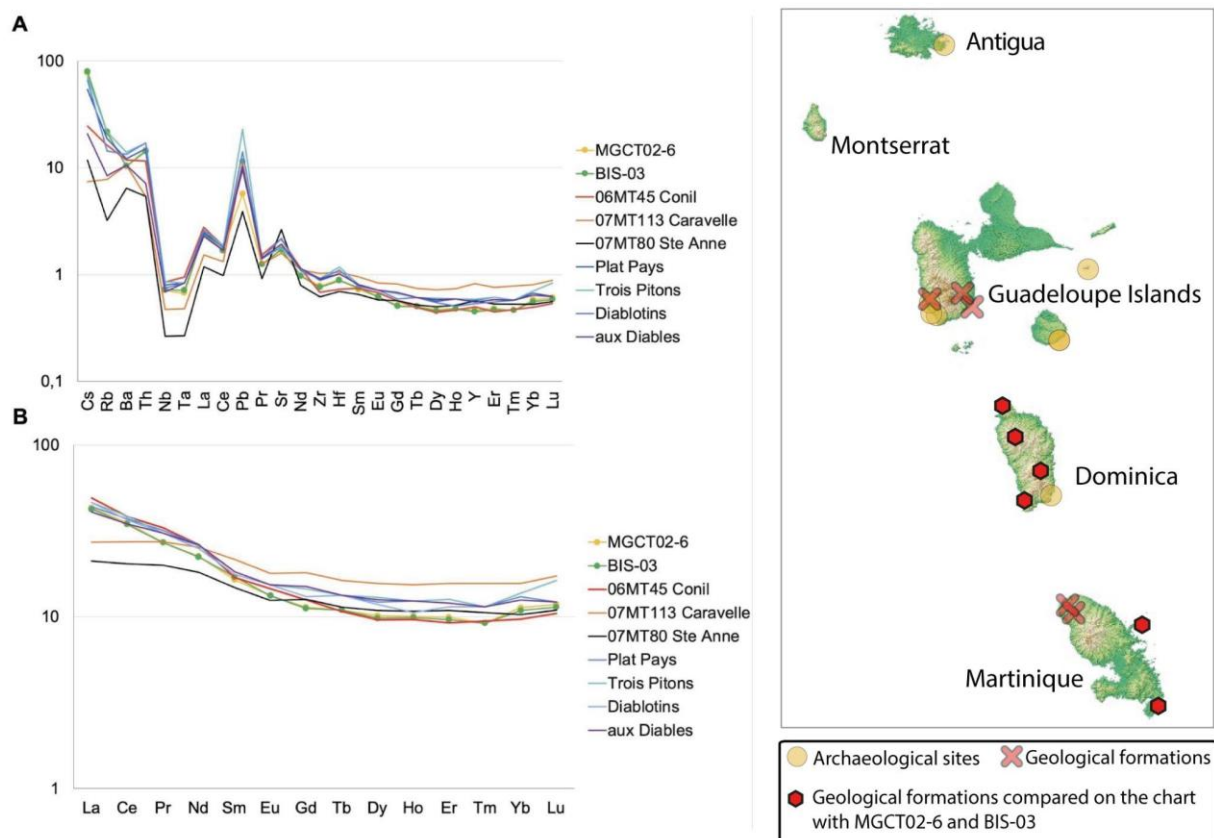


Figure S8 Comparison of archeological samples and eruption products from the east and south of Martinique (from Labanieh, 2009) and the island of Dominica (from Howe et al., 2015). A. N-MORB-normalised trace elements patterns. B. Chondrite-normalised REE patterns.

Sample	Analysis per sample	SiO ₂ (%)	Al ₂ O ₃ (%)	Fe ₂ O ₃ (%)	MnO (%)	CaO (%)	Na ₂ O (%)	K ₂ O (%)	TiO ₂ (%)	Zn (µg/g)	Ga (µg/g)	Rb (µg/g)	Sr (µg/g)	Zr (µg/g)
Volc.Tuf	2	77.84	12.43	2.04	0.07	1.33	3.85	1.8	0.12	39	14	62	106	174
RRC-01	2	78.31	12.58	1.71	0.06	1.3	3.56	1.89	0.12	34	12	56	100	173
RRC-02	2	60.92	17.91	5.61	0.12	5.5	2.82	1.06	0.49	62	15	78	185	93
PSF-01	2	61.83	17.11	5.66	0.12	5.7	2.73	1.06	0.49	65	18	96	237	96
MGCT02-01	2	61.68	17.05	5.22	0.11	5.61	3.07	1.06	0.48	60	16	79	231	99
MGCT02-02	1	61.31	16.92	5.57	0.12	5.64	3.01	1.05	0.49	64	18	73	237	92
MGCT02-03	1	62.1	17.11	5.53	0.11	5.71	2.61	1.07	0.5	68	19	80	275	109

MGCT02-04	1	61.52	17	5.4	0.11	5.65	2.57	1.29	0.49	60	21	85	263	114
MGCT02-05	1	61.9	17.04	5.11	0.11	5.59	3.05	1.07	0.49	59	16	90	240	102
MGCT02-06	1	61.62	16.99	5.26	0.11	5.64	3.1	1.05	0.49	60	17	68	243	112
MGCT02-07	1	61.6	17.05	5.24	0.11	5.62	3.1	1.06	0.49	59	17	89	242	110
BIS-01	1	62.25	17.17	4.82	0.1	5.55	3.07	1.06	0.47	54	16	85	227	102
BIS-02	1	61.49	17.02	5.38	0.11	5.59	3.06	1.07	0.49	62	15	75	232	106
BIS-03	1	62.13	17.33	5.37	0.11	5.73	2.58	1.07	0.48	62	15	64	229	92
BIS-04	2	61.73	17.08	5.23	0.11	5.58	3.07	1.06	0.47	59	16	74	219	94
CB-051a	1	61.75	17.06	5.19	0.11	5.57	3.11	1.07	0.47	56	16	76	200	89

CB-050a	1	61.46	17.01	5.38	0.12	5.64	3.07	1.07	0.49	57	17	81	202	91
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Table S1 Compositions measured in 2007 by PIXE on archaeological artefacts and a geological sample from the Volcan du Tuf.

Sample	Na₂O (%)	MgO (%)	Al₂O₃ (%)	SiO₂ (%)	K₂O (%)	CaO (%)	TiO₂ (%)	MnO (%)	Fe₂O₃ (%)
BIS-01	3.5	2.7	17.4	62.6	1.1	6.3	0.5	0.1	5.5
BIS-01	3.5	2.7	17.6	62.7	1.2	6.3	0.5	0.1	5.3
BIS-01	3.5	2.7	17.6	62.7	1.1	6.2	0.5	0.1	5.3
BIS-01	3.5	2.7	17.5	62.8	1.1	6.2	0.5	0.1	5.3
BIS-02	3.6	2.7	17.6	62.8	1.1	6.2	0.5	0.1	5.0
BIS-02	3.2	2.7	17.9	62.7	1.2	6.1	0.5	0.1	5.5
BIS-02	2.9	2.6	17.9	62.9	1.1	6.0	0.5	0.1	5.6
BIS-02 polished	3.5	2.7	17.3	62.7	1.1	6.4	0.5	0.1	5.5
BIS-02 polished	3.5	2.7	17.3	62.4	1.2	6.5	0.5	0.1	5.7
BIS-02 polished	3.3	2.6	17.3	62.5	1.2	6.6	0.6	0.2	5.7
BIS-02 polished	3.5	2.7	17.4	62.8	1.1	6.3	0.5	0.1	5.5
BIS-02 polished	3.2	2.7	17.4	62.7	1.2	6.4	0.6	0.1	5.6

BIS-02 polished	3.0	2.6	17.2	62.5	1.2	6.7	0.6	0.1	6.0
BIS-02 polished	3.4	2.8	17.5	62.8	1.2	6.3	0.6	0.1	5.3
BIS-03	3.5	2.7	17.5	62.9	1.1	6.2	0.5	0.1	5.3
BIS-03	3.6	2.7	17.4	62.7	1.1	6.2	0.5	0.1	5.3
BIS-03	3.8	2.8	17.4	62.5	1.1	6.3	0.5	0.1	5.3
BIS-03	3.6	2.7	17.4	62.7	1.1	6.2	0.5	0.2	5.3
BIS-03	3.5	2.7	17.3	62.7	1.2	6.3	0.6	0.1	5.5
BIS-03	3.7	2.8	17.6	63.1	1.1	6.0	0.5	0.1	4.9
BIS-04	3.5	2.7	17.5	63.1	1.1	6.2	0.5	0.1	5.1
BIS-04	3.5	2.7	17.4	63.0	1.1	6.2	0.5	0.1	5.1
BIS-04	3.4	2.7	17.4	62.6	1.2	6.4	0.5	0.2	5.5
BIS-04	3.5	2.7	17.4	62.7	1.2	6.3	0.5	0.1	5.5
BIS-04	3.5	2.7	17.4	62.7	1.2	6.3	0.5	0.1	5.4
BIS-04	3.4	2.7	17.6	62.8	1.2	6.2	0.5	0.1	5.3
BIS-04	3.8	2.8	17.8	63.5	1.1	5.8	0.5	0.1	4.5

BIS-04	3.5	2.7	17.4	62.7	1.2	6.3	0.6	0.1	5.4
BIS-04	3.6	2.7	17.5	63.1	1.1	6.1	0.5	0.1	5.0
BIS-04	3.5	2.7	17.3	63.2	1.1	6.2	0.5	0.1	5.0
BIS-04	3.4	2.2	15.2	64.4	1.2	5.6	0.9	0.2	6.6
BIS-04	3.4	2.7	17.5	62.6	1.2	6.4	0.5	0.1	5.4
BIS-04	3.4	2.7	17.5	62.7	1.2	6.3	0.5	0.1	5.4
BIS-04	3.6	2.7	17.6	62.9	1.1	6.1	0.5	0.1	5.1
BIS-04	3.6	2.7	17.7	63.0	1.1	6.1	0.5	0.1	5.0
BIS-04	3.6	2.7	17.4	62.6	1.1	6.3	0.5	0.1	5.4
CB-051a	3.6	2.7	17.4	62.8	1.1	6.2	0.5	0.1	5.3
CB-051a	3.6	2.8	17.5	63.1	1.1	6.1	0.5	0.1	5.1
CB-051a	3.6	2.8	17.4	62.8	1.2	6.1	0.5	0.2	5.3
CB-051a	3.5	2.7	17.4	62.6	1.1	6.3	0.5	0.1	5.4
CB-050a	3.5	2.7	17.6	62.8	1.1	6.2	0.5	0.1	5.2
CB-050a	4.0	2.8	17.3	61.9	1.2	6.3	0.5	0.1	5.1
CB-050a	3.6	2.7	17.6	62.8	1.1	6.2	0.5	0.1	5.2

CB-050a	3.7	2.8	17.9	63.2	1.1	5.8	0.5	0.1	4.7
DEL-03-1	3.4	2.7	17.5	62.6	1.2	6.2	0.5	0.1	5.4
DEL-03-1	3.4	2.7	17.6	62.5	1.2	6.3	0.5	0.2	5.5
DEL-03-1	3.4	2.7	17.5	62.4	1.2	6.3	0.5	0.1	5.6
DEL-03-1	3.4	2.7	17.7	61.8	1.2	6.3	0.5	0.1	5.7
DEL-03-1	3.4	2.7	17.6	62.4	1.2	6.2	0.5	0.1	5.6
MGCT02-01	3.5	2.7	17.6	62.9	1.1	6.1	0.5	0.1	5.2
MGCT02-01	3.6	2.7	17.5	62.9	1.1	6.1	0.5	0.1	5.2
MGCT02-01	3.5	2.7	17.6	62.5	1.2	6.2	0.6	0.1	5.5
MGCT02-01	2.8	2.3	16.4	55.8	1.1	5.6	0.5	0.2	5.6
MGCT02-01	3.6	2.7	17.6	63.1	1.1	6.1	0.5	0.1	5.1
MGCT02-02	3.5	2.7	17.4	62.6	1.1	6.3	0.5	0.1	5.5
MGCT02-02	3.5	2.7	17.4	62.7	1.1	6.3	0.5	0.1	5.4

MGCT02-02	3.4	2.7	17.3	62.6	1.2	6.3	0.5	0.1	5.6
MGCT02-02	3.5	2.7	17.4	62.4	1.2	6.4	0.6	0.1	5.6
MGCT02-02	3.8	2.8	17.7	63.4	1.1	5.8	0.5	0.1	4.7
MGCT02-02	3.7	2.8	17.6	63.2	1.1	5.9	0.5	0.1	4.9
MGCT02-02	3.5	2.7	17.3	62.7	1.2	6.2	0.5	0.2	5.4
MGCT02-02	3.5	2.7	17.3	62.5	1.2	6.3	0.5	0.1	5.6
MGCT02-02	3.8	2.8	17.8	63.5	1.1	5.7	0.5	0.1	4.5
MGCT02-02	3.5	2.7	17.4	62.7	1.2	6.3	0.5	0.1	5.5
MGCT02-05	3.6	2.7	17.6	62.3	1.1	6.4	0.5	0.1	5.3
MGCT02-05	3.7	2.8	17.8	63.0	1.1	6.0	0.5	0.1	4.7
MGCT02-06	3.5	2.7	17.5	62.9	1.1	6.2	0.6	0.1	5.3
MGCT02-06	3.1	2.7	17.5	63.0	1.1	6.3	0.6	0.2	5.4

MGCT02-06	3.5	2.7	17.5	62.8	1.1	6.2	0.5	0.1	5.3
MGCT02-06	3.5	2.7	17.5	62.8	1.1	6.2	0.5	0.1	5.3
MGCT02-06	3.6	2.7	17.5	62.9	1.1	6.1	0.5	0.1	5.2
MGCT02-06	3.6	2.7	17.4	62.5	1.1	6.3	0.5	0.1	5.4
MGCT02-06 polished	3.5	2.7	17.5	62.9	1.1	6.2	0.5	0.1	5.3
MGCT02-06 polished	3.4	2.6	17.2	62.5	1.2	6.5	0.6	0.2	5.8
MGCT02-06 polished	2.9	2.6	17.2	62.8	1.2	6.6	0.6	0.1	5.8
MGCT02-06 polished	3.1	2.4	16.7	63.6	1.3	6.3	0.5	0.1	5.8
MGCT02-06 polished	3.0	2.6	17.1	62.7	1.2	6.6	0.6	0.1	6.0
MGCT02-06 polished	3.1	2.7	17.6	63.2	1.1	6.2	0.5	0.1	5.3
MGCT02-06 polished	2.9	2.6	17.2	62.9	1.2	6.6	0.6	0.2	5.9
MGCT02-06 polished	3.2	2.7	17.5	63.1	1.1	6.3	0.6	0.1	5.4

MGCT02-06 polished	3.2	2.6	17.2	62.4	1.2	6.6	0.6	0.1	5.9
MGCT02-06 polished	3.1	2.7	17.4	62.7	1.2	6.5	0.5	0.1	5.7
MGCT02-06 polished	2.7	2.5	16.9	62.4	1.2	7.0	0.6	0.1	6.5
MGCT02-07	3.5	2.7	17.4	62.6	1.1	6.3	0.5	0.1	5.5
MGCT02-07	3.4	2.7	17.3	62.6	1.2	6.4	0.6	0.1	5.6
MGCT02-07	3.5	2.7	17.4	62.6	1.2	6.3	0.5	0.1	5.4
MGCT02-07	3.4	2.7	17.4	62.6	1.2	6.3	0.5	0.1	5.4
RRC-01	4.6	0.1	12.7	77.4	2.0	1.4	0.1	0.1	1.5
RRC-01	4.5	0.1	12.6	77.0	2.0	1.4	0.2	0.1	1.9
RRC-01	4.3	0.1	12.6	77.2	2.1	1.4	0.1	0.1	1.7
RRC-01	4.5	0.1	13.2	76.4	2.0	1.7	0.1	0.1	1.8
RRC-01	4.7	0.2	14.2	73.7	2.0	2.7	0.1	0.1	1.9
RRC-01	4.5	0.1	12.8	77.4	2.0	1.4	0.1	0.1	1.4

RRC-01	4.5	0.1	12.8	77.6	1.9	1.3	0.1	0.1	1.4
RRC-01	4.3	0.1	12.9	76.8	1.9	1.6	0.1	0.1	1.8
RRC-01	4.8	0.1	12.8	76.3	2.0	1.5	0.1	0.1	1.9
RRC-01	4.6	0.1	12.7	76.9	2.0	1.4	0.1	0.1	1.7
RRC-01	3.4	0.2	13.0	77.3	1.9	1.5	0.3	0.1	2.0
RRC-01	4.2	0.1	12.8	77.5	2.0	1.4	0.1	0.1	1.6
RRC-01	3.2	0.2	13.2	77.3	1.8	1.7	0.2	0.1	2.1
RRC-02	3.3	2.7	17.5	62.4	1.2	6.3	0.6	0.1	5.6
RRC-02	3.3	2.6	17.7	62.5	1.2	6.3	0.6	0.1	5.5
RRC-02	3.2	2.6	17.6	62.0	1.3	6.5	0.6	0.2	6.0
RRC-02	3.4	2.7	17.5	62.7	1.2	6.3	0.5	0.1	5.5
RRC-02	3.4	2.6	17.3	61.8	1.3	6.6	0.6	0.2	6.2
RRC-02	3.5	2.0	18.4	60.6	1.0	7.1	0.6	0.1	6.4
RRC-02	2.8	2.6	17.5	62.5	1.3	6.4	0.6	0.1	5.9
RRC-02	3.4	2.1	18.7	60.9	1.0	6.7	0.6	0.1	6.3
RRC-02	2.8	2.6	17.5	62.4	1.3	6.5	0.6	0.1	6.0

RRC-02	3.1	2.6	17.5	62.7	1.3	6.4	0.5	0.1	5.6
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758 Table S2 Compositions measured by SEM-EDS on the glassy matrix of some archaeological artefacts.

Sample	MnO (µg/g)	Fe₂O₃ (µg/g)	Zn (µg/g)	Ga (µg/g)	Rb (µg/g)	Sr (µg/g)	Y (µg/g)	Zr (µg/g)	Nb (µg/g)	Pb (µg/g)
BoisJoli1	553	16498	45	21	19	305	18	100	19	4
BoisJoli1	892	23696	64	20	23	253	20	110	20	4
BoisJoli1	1543	34378	72	19	27	252	25	127	20	5
BoisJoli1	525	18725	61	17	19	250	17	111	18	3
BoisJoli2	575	19565	34	19	31	325	31	100	18	4
BoisJoli2	2982	66435	76	15	30	220	30	98	18	4
BoisJoli2	993	28943	38	17	33	310	30	106	19	4
Est.Carbet1	1159	47658	31	13	17	252	22	74	15	1
Est.Carbet1	982	42236	28	15	14	266	18	62	15	1
Est.Carbet1	1322	56028	32	14	15	251	20	72	15	2
Riv.Capesterre 1	2125	67503	66	15	20	188	46	107	18	4
Riv.Capesterre 1	1745	66798	65	13	20	180	45	107	17	3
Riv.Capesterre 1	1736	66898	65	14	20	182	46	107	18	2

Volc.Tuf	882	21571	35	11	45	114	42	163	20	5
Volc.Tuf	1003	23981	38	12	50	131	48	194	23	5
Volc.Tuf	798	19817	32	12	42	130	40	149	20	4
Volc.Tuf	1173	28389	37	11	43	106	43	179	20	5

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Table S3 Trace elements measured by ED-XRF on geological samples.

Sample	MnO (µg/g)	Fe₂O₃ (µg/g)	Zn (µg/g)	Ga (µg/g)	Rb (µg/g)	Sr (µg/g)	Y (µg/g)	Zr (µg/g)	Nb (µg/g)	Pb (µg/g)
BIS-01	1514	57634	52	12	30	203	19	76	17	4
BIS-01	1516	58066	51	12	31	204	19	76	16	5
BIS-02	1658	62582	65	13	34	220	20	82	17	5
BIS-02	1732	65767	69	16	36	237	21	87	18	5
BIS-02	1644	63785	63	14	36	239	22	88	19	5
BIS-02	1278	58544	60	14	34	207	19	82	8	6
BIS-03	1520	59139	63	13	31	206	19	77	16	4
BIS-03	1538	62115	73	14	33	217	20	82	17	5
BIS-03	1508	60754	73	13	33	220	20	82	17	5
BIS-04	1580	60276	63	13	32	213	19	78	16	5
BIS-04	1560	59883	56	13	33	217	20	80	17	4
CB-050a	1556	58582	58	13	31	204	20	75	16	4
CB-050a	1544	58444	51	13	31	206	19	75	16	5
CB-051a	1489	56283	50	11	30	200	18	74	16	4

CB-051a	1802	68212	68	16	37	246	22	91	19	4
DEL-03	1697	64528	70	16	35	227	22	84	19	6
DEL-03	1604	60521	63	13	33	212	20	79	18	4
DEL-03	1639	62521	65	15	33	220	21	82	18	5
DEL-03	1575	60149	57	13	32	211	19	78	17	4
RRC-01	886	22406	37	13	48	142	45	179	21	5
RRC-01	915	23305	37	13	47	122	46	220	21	5
RRC-02	1580	63696	71	14	34	229	21	85	18	5
RRC-02	1625	65392	73	15	35	236	21	88	19	6
RRC-02	1556	62200	70	15	33	223	20	83	18	5
MGCT02-01	1502	57323	51	12	31	204	19	76	17	3
MGCT02-01	1531	58557	51	13	32	210	19	77	17	4
MGCT02-02	1663	62659	64	15	35	228	21	83	17	5
MGCT02-02	1536	58059	59	13	31	205	18	76	17	4
MGCT02-02	1514	57639	52	13	31	203	18	75	16	4
MGCT02-04	1618	61878	75	15	34	222	21	82	18	5

MGCT02-04	1629	61497	70	15	34	223	20	81	17	5
MGCT02-04	1613	61317	64	13	33	219	19	81	17	5
MGCT02-05	1521	58075	52	13	31	204	19	77	17	4
MGCT02-06	1572	59431	60	14	32	210	19	77	16	4
MGCT02-06	1615	61752	64	16	34	224	20	82	17	5
MGCT02-06	1522	57965	53	13	31	205	19	75	16	4
MGCT02-06	1572	59902	56	13	32	213	20	79	17	4
MGCT02-07	1691	64238	67	14	35	227	21	84	18	6
MGCT02-07	1509	56944	58	13	31	200	19	76	16	3

Elts	Na ₂ O	MgO	SiO ₂	Al ₂ O ₃	K ₂ O	CaO	FeO
EPMA analysis, from this work (ObsPR, Volc.Tuf, MGCT02-6 and BIS-03) and Rochette et al. 2021 (Belizite)							
Mean ObsPR	4,9	0,1	76,4	11,5	4,8	0,1	2,2
Min ObsPR	4,8	0,1	75,6	11,5	4,8	0,1	2,1
Max ObsPR	5,0	0,1	76,9	11,6	4,9	0,1	2,2
Mean Volc.Tuf	4,2	0,1	78,0	12,6	2,2	1,5	1,8
Min Volc.Tuf	4,1	0,1	77,3	12,4	2,2	1,5	1,7
Max Volc.Tuf	4,3	0,2	78,6	12,7	2,3	1,6	1,9
Mean MGCT02-6	3,4	2,7	61,7	16,9	1,3	6,9	5,5
Min MGCT02-6	3,2	2,6	61,5	16,9	1,2	6,7	5,4
Max MGCT02-6	3,5	2,7	62,1	17,0	1,3	7,1	5,8
Mean Bis-03	3,5	2,7	62,0	17,0	1,3	6,7	5,4
Min Bis-03	3,4	2,6	61,7	16,8	1,3	6,6	5,3
Max Bis-03	3,6	2,7	62,3	17,2	1,3	6,9	5,5
Mean Belizite	3,6	1,8	62	17,2	2	4,7	6,4
Min Belizite	2,7	1,5	60,3	16,1	1,7	4	5,5
Max Belizite	4,2	2,1	63,3	17,9	2,4	5,4	7,4
% variability (1-min/max)x100							
MGCT02-6	7,46	4,28	0,99	0,91	6,73	5,24	6,69
Bis-03	4,51	1,73	0,93	2,05	1,77	3,51	3,15
MGCT02-6+Bis-03	9,85	4,28	1,31	2,05	6,73	6,42	7,92
Belizites _a	37,3	25,4	4,6	10,1	28,8	26,6	25,3
Belizites _b	15,6	13	1,2	3,4	6,8	5,8	9,6
Belizites _c	23,3	18,1	2,3	6,1	9,1	9,7	20,6
Ivoirites	36,9	32,8	3,3	7,6	17,9	55,9	11,3
Australasites	60,3	83,5	18,7	49,7	52,3	81,1	58,6
Moldavites	–	58,8	12	47	41,5	76	69,1
Bediasites-georgiites	45,7	–	14	46	36,3	–	68,2

Table S5 Major element concentrations and compositional variability of vitreous rocks analysed in this study and tektites from different strewn fields, particularly belizites. According to Rochette et al. (2021, Table 1).