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Colonial Glass Beads (16th-Cent.) from Indigenous Localities of the Tamtoc Peninsula (San Luis Potosí), Mexico

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Abstract. We have investigated two Nueva Cádiz tubular glass beads (16th-Century) from the indigenous localities of the Tamtoc Peninsula, Huasteca Potosina (Mexico). It was part of an exceptional discovery of 96 European glass beads found in archaeological contexts of the Early Colonial period (dated ¹⁴C 1512 AD ± 30 years, before 1560 AD). They correspond typologically to the Kidd's typology's colour variants IIIc1 and IIIc2. These multilayered beads are made from a gob formed by three successive dips, then blown up, drawn, and hot-formed before being sectioned. PIGE/PIXE (Particle-induced Induced Gamma and X-Ray Emissions) and Raman spectroscopy analyses reveal that the glass type is an ashes soda-lime-silica glass. Trace elements associated with the metal oxides used to tint and opacify these glasses are also significant. The outer layer of both types is blue-coloured by copper oxide while a mixture of tin and lead oxides opacifies the middle layer in white through cassiterite (SnO₂) formation. As, Ni, Bi, and U contents reveal this cobalt, mixed with manganese, which colours the inner layer of type IIIc1 beads in purple, comes from the Freiberg mine in Southeastern Germany (Saxony). Compared with other Pan-American finds from the same period, these Mexican Nueva Cádiz beads have similar chemical compositions to specimens discovered in the colonial city 'ruins of Nueva Cádiz (Cubagua Island, Venezuela). Finally, we question 16th-Century texts on how Mesoamerican peoples might have culturally perceived these blue beads made with a material unknown to them. By studying Mexico's colonial history, we propose several Spanish expeditions that may have introduced these beads to the Huasteca.

Keywords: Nueva Cádiz Multilayered Glass Bead, Mexico Huasteca Tamtoc, Chemical Analyses, Raman Spectroscopy, Spanish Colonial Expeditions.

1. Introduction

This typological, archaeometrical, and technical study of glass ornaments, a material alien to Mesoamerican cultures, is part of the Mexican archaeological program *Origen y Desarrollo del Paisaje Urbano de Tamtoc, San Luis Potosí* (hereafter ODPUT-SLP) [1]. Directed since 2008 by the Maestra E. Martínez Mora and supervised by Mexico's *Instituto Nacional de Antropología e Historia* (INAH), this program focuses on the study of the pre-Hispanic city of

Tamtoc, its peripheral sites, and its wider region, the Potosinan Huasteca (see Figures 1. [a] and [b]). European tubular glass beads were discovered during excavations in Tamtoc's Architectural Group F and Mound 1-Norte of a peripheral settlement on the *Rancho Aserradero*. Mass-produced in Renaissance Europe, glass beads like these small, brightly blue ornaments became authentic chronological markers in the Americas and, as we shall see, more specifically in the Huasteca region. All Spanish maritime and land expeditions imported them during the first decades of the Conquest (1500-1560). Discovered from Florida to the Central Andes (Tiahuanaco), the natives immediately adopted them, and as soon as they came into their possession, they were redistributed from town to village. In the southeastern United States, these beads enriched the diversity of goods traded along ancestral exchange roads [2, pp. 45-52]. Later, from the second half of the 16th and into the 17th-Century, rosaries, and prayer beads made in the Indian Christian missions of Mexico with these European beads were even sent back to Europe [3].

This research aims to identify the glasses' composition and the nature of their colouring agents, with their set of trace elements. The results obtained can contribute to tracing the European origin of these glass beads, reconstructing the *chaîne opératoire* involved in their manufacture, and bringing new data to the troubled period of the Huasteca Conquest.

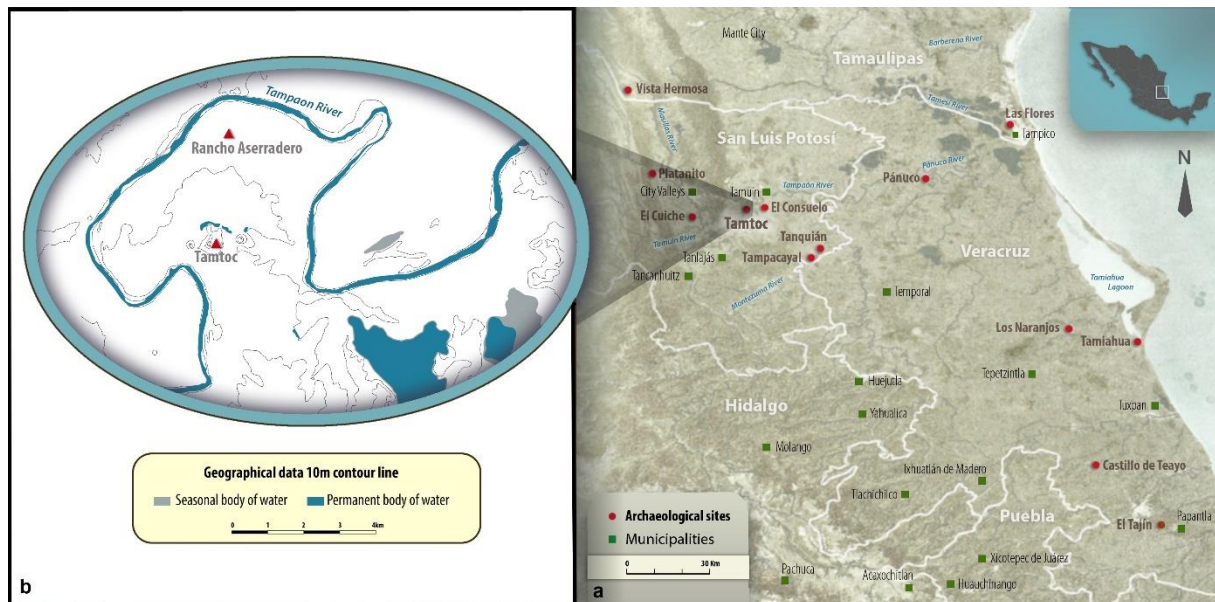


Figure 1. (a), and (b). The central Huasteca region with toponyms and hydronyms cited in the text and Tamtoc peninsula (Municipio of Tamuín) cut by the Tampoán river in the Huastecan peneplain, with localities of glass beads discoveries; Map Jimena Reyes Pimentel.

2. Archaeological contexts

The city of Tamtoc is located (N 21° 55' 28.38" - W 98° 48' 46.88"; alt. 40 m. a. s. l.) in the *Municipio* of Tamuín (SLP), on a Peninsula (6.300 x 4.040 km) cut by the Tampoán River in the sediments forming the Huastecan peneplain (see Figure 1. [b]). It was inhabited from 400 BC (Pre-urban I Period) until the invasion of the Spanish conquistadors and their allied Indians (1525 AD, Urban Apogee Period; [4]). This ceremonial center is the best studied of the Potosinan Huasteca and has been described as " *the pre-Hispanic capital of the Huasteca region* " [5, pp. 67]. The multi-layered glass bead INV.145-CAT.138 was discovered on the periphery of Mound F-1, one of a group of six mounds excavated in 2004 (*Tamtoc* archaeological Program, [6]) and again in 2011 (ODPUT-SLP archaeological Program).

Sample BRA-01 belongs to a collection of 95 glass beads (INV.140-CAT.133) found in the female burial RA12A [7, pp. 9-11] of the pre-Hispanic *Poblado* known as " *Conjunto Norte Rancho Aserradero* " (hereafter CNRA). This is a group of around 50 tumuli built on 80 ha to the north of the Tamtoc peninsula and 2.5 km northeast of it (N 21° 56' 41.64" - W 98° 48' 45.20"; alt. 33 m. a. s. l.) [6, pp. 17] [8, pp. 29]. Burial RA12A was one of 19 graves in Tumulus 1-Norte excavated in 2004 by G. Ahuja Omicochea and D. Lozano Briones (Figure 2). Due to the poor state of preservation, Burial RA12A was recovered in 2012 by the physical anthropologist P. Hernández Espinoza (INAH) as a sedimentary block and further studied in the laboratory [7, pp. 5]. It is the skeleton of a woman aged 45-49 years in a lying supine position of high social status, which is reflected both in the intentional deformations of her skull [9, pp. 155-164] and in the presence of a rich set of funerary offerings: bicolor and polychrome ceramics of various types, jadeite-jade ornaments, turquoise beads, red *Spondylus crassisquama* shells, and copper beads covered with gold leaf (see Figures 3 and 4).

The bones of this beaded Lady were triple radiocarbon dated in Mexico at the *Laboratorio de Espectrometría de Masas con Aceleradores* of the UNAM's *Instituto de Física* (LEMA-298,1,1, bone, cal. 2 Σ : 1505 \pm 30 years AD and LEMA-298,1,2, bone, cal. 2 Σ : 1512 \pm 30 years AD) and in Florida (USA), at the Beta Analytic Inc. laboratory (Beta 516,910 - 015, tooth, cal. 2 Σ : 1538 - 1635 AD). The Mexican dates obtained place her death in the decade corresponding to the first Spanish settlement attempts (1518-1520) on the Huasteca coast, followed by the brutal conquest of this cultural region (1522-1530). In contrast, the Floridian date gives a broader time frame. This last dating is inconsistent with two historical events: the conquest of the Huasteca which began in 1522, and the manufacturing chronology (1500-1560 AD) of this kind of multilayered beads in Europe. That's why we have therefore decided to reject this last date.

The remarkable antiquity of the archaeological context in which the set beads 140-133 were discovered, suggests a link with the first Spanish expedition to the Tamaulipecan Huasteca between 1518 and 1520 A.D. As for the single specimen 145-138 illustrates a late attendance at the ancient pre-Hispanic ceremonial center of Tamtoc. To scientifically valorize these historically important discoveries, we submitted a sampling of them to a chemical and non-invasive analysis program.



Figure 2. From left to right, southern and eastern facades of Edifice 1-Norte of CNRA; credit photo E. Martínez Mora/INAH.

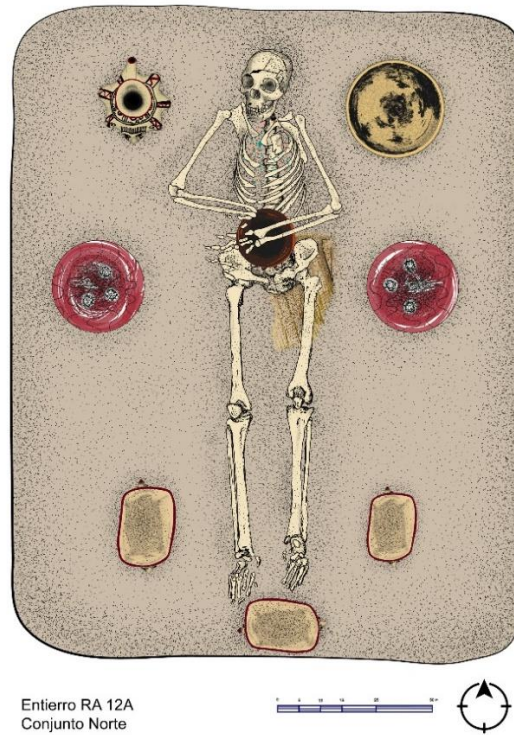


Figure 3. Edifice 1-Norte of the CNRA, the burial of the beaded Lady (Burial RA12A). She is lying in a supine position, surrounded by Huastecan-style ceramics: Huasteca polychrome and Huasteca black-on-white types, red slip with fine paste; drawing E. Martínez Mora/INAH, digitization F. Gomezcana Martínez.



Figure 4. Museographic display of Nueva Cádiz, *Spondylus crassisquama* red shell and gold beads found in the burial of the beaded Lady (Burial RA12A), Edifice 1-Norte, CNRA (1518-1522 AD); photo E. Martínez Mora/INAH.

3. Material and experimental methods

3.1 Typologies applied to glass beads from the Tamtoc Peninsula

The glass bead 145-138 is discovered on the periphery of Mound F-1 at Tamtoc. Like all those (140-133) deposited as offerings in Burial RA12A at CNRA, it is tubular in form and quadrangular in cross-section. They are made from the concentric superposition of three layers of stretched glass following the terminologies of Loewen and Dussubieux [10, pp. 64-65] and of Walder et al. [11, p. 86]. The only morphological variables are their dimensions and the layer's color. We used the revised typological system published in 2012 by K.E. et M.A. Kidd [12] to describe European colonial beads introduced to the Americas to characterize them. We have supplemented this first system with that of K. Karklins [13], published in the same review volume, which offers improvements to characterize shapes, dimensions, and the color system.

The large 145-138 bead (see Table 1, and Figures 5 [a, b, c, and d]) with a circular cavity of 3.25 mm consists of a transparent colorless inner glass core, a middle layer of opaque white glass, and an outer layer of translucent shadow blue glass. Bubbles close to the surface are stretched parallel to the length of the bead. According to Kidd's typological characterization system for American colonial beads [12, pp. 54, Table 5, Plate VII], 145-138 is a Type IIIc2 bead. In other words, a Class III tubular bead is made up of alternating layers, and the c2 variety corresponds to the colorless, white, and shadow blue alternation from the inner to the outer layer. For Karklins, this is a Type IIIc of hand-drawn bead [13].

All the multilayered glass beads (140-133) found in Burial RA12A are made of the same colour alternation as the medium sample BRA-01 (see Table 1, and Figures 6. [a, b, and c]). The core is amethyst and opaque. In the case of the specimen loaned for analysis, the central circular threading void has a diameter of 0.9 mm. The middle layer is white and opaque, and the outer layer of glass is bright blue and translucent. On the surface, as on other specimens in this set, this glass shows a slight orange alteration cape. In the typology of American colonial beads, all these specimens belong to class III and variety c1 [12, pp. 54, Table 5, Plate VII] and class III variety c for Karklins [13, pp.65, Figure 3]. These are tubular beads with a quadrangular cross-section made up of three alternating glasses: amethyst, white, and bright blue.

Table 1. Dimensions and typology of glass beads from the Tamtoc Peninsula localities.

NO. Bead	Long. (mm)	Width (mm)	Weight (gr)	Kidd and Karklins Types	Munsell Colors			Observations
					Outside/ outer layer	Middle layer	Core	
145-138	7.35	7.64-7.24	1.2	IIIc2-IIIc	shadow blue (2.5PB 5/4) translucent	white (N 9/0) opaque	colorless (N 7/0) transparent	-
*BRA-01 (set 140-133)	5.71	3.65-3.95	0.3	IIIc1-IIIc	bright blue (5.0B 5/7) translucent	white (N 9/0) opaque	amethyst (7.5P 4/8) opaque	orange surface alteration

*BRA: Bead Rancho Aserradero.

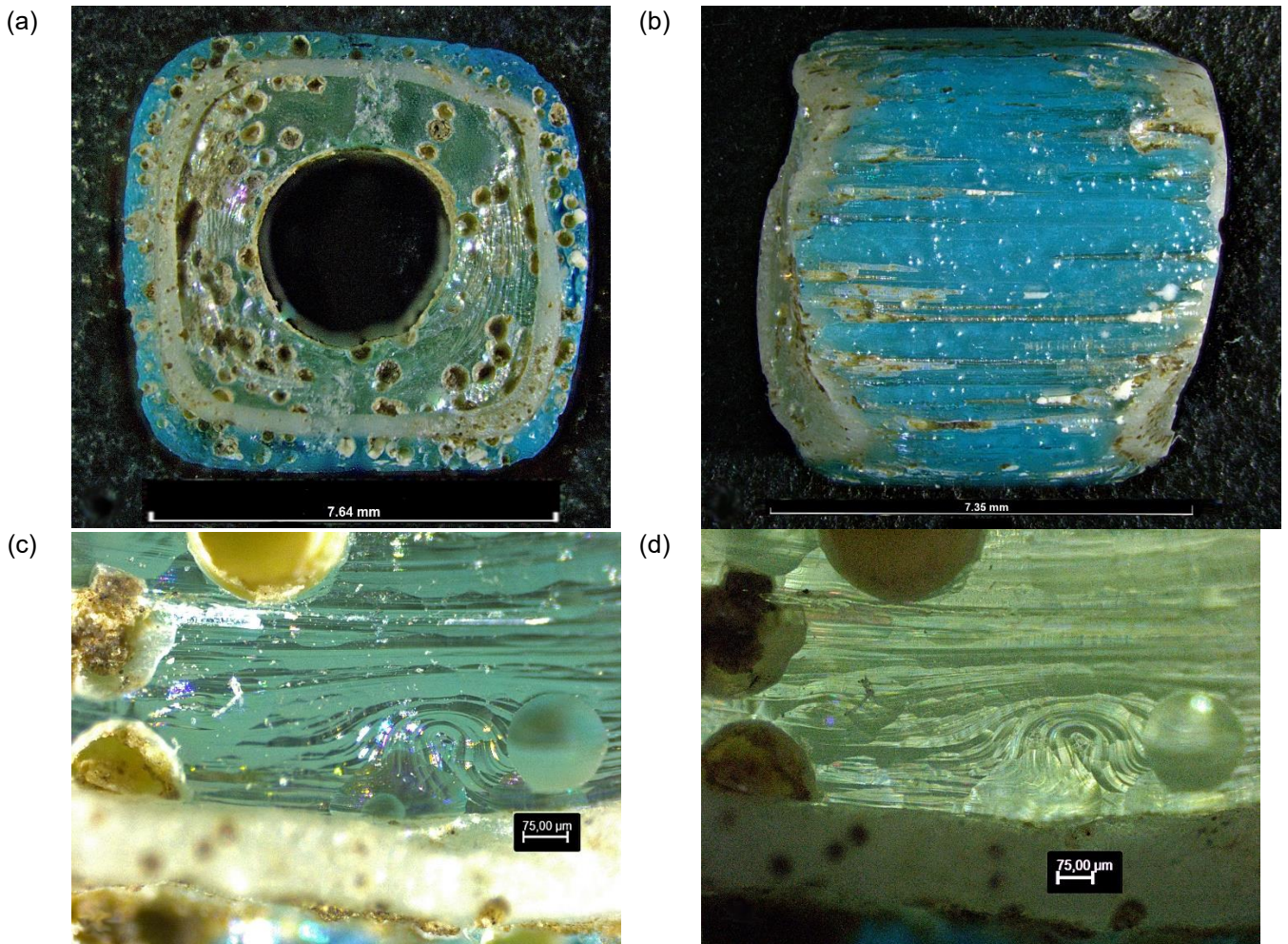


Figure 5. (a) Nueva Cádiz bead 145-138 (Tamtoc), type IIIc2 of Kidd's typology [12] and IIIc of Karklins [13]; (b) view of its outer shadow blue glass surface. Glass is stretched parallel to the length of the bead; (c and d) enlarged views (x8) of the bead edge 145-138 (type IIIc2-IIIc), syrupy zone forming a knot in the light grey transparent glass of the core; credit photos F. Gendron/MNHN.

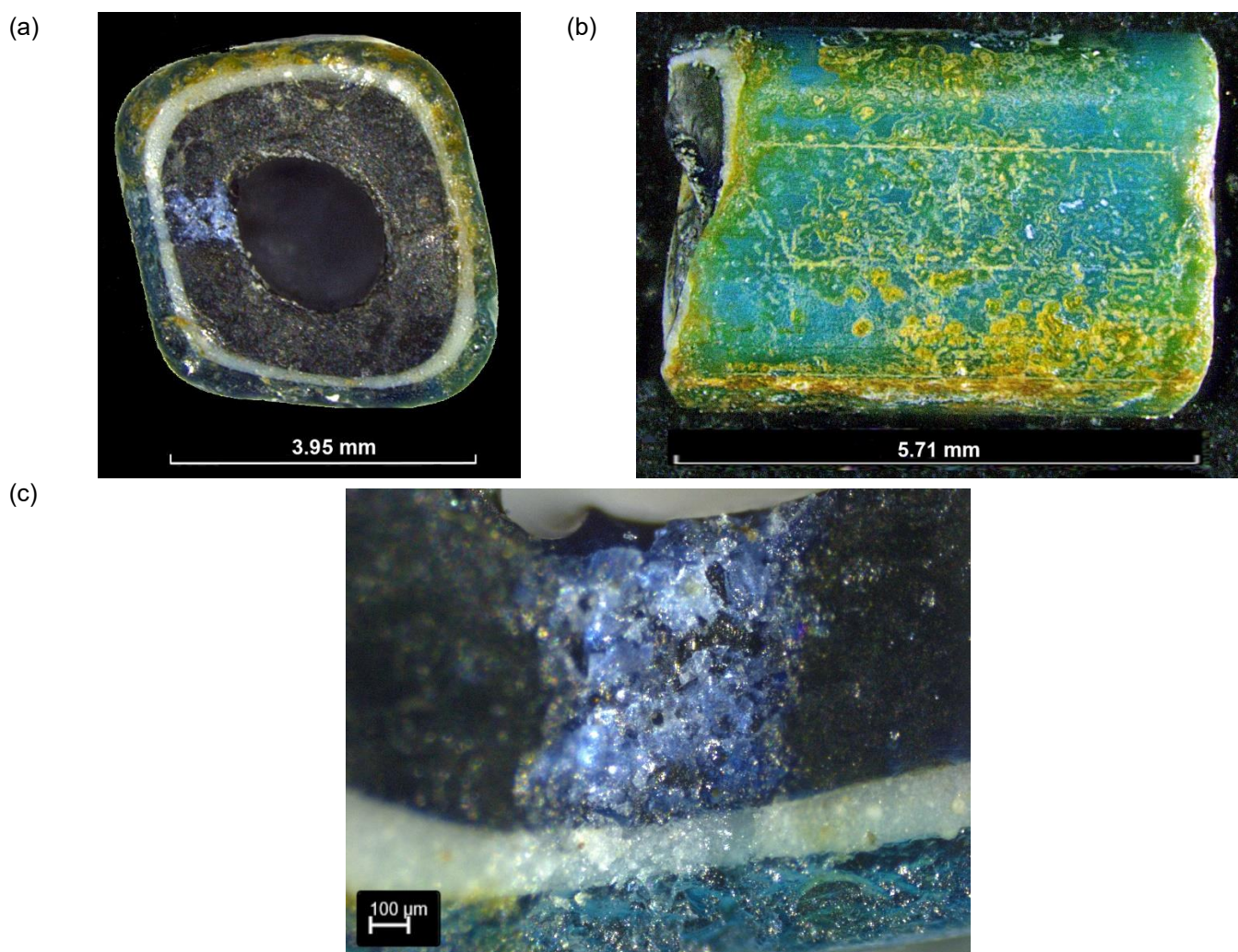


Figure 6. (a) Nueva Cádiz bead BRA-01 (CNRA), type IIIc1 of Kidd's typology [12], and IIIc of Karklins [13]; (b) enlarged view of its outer bright blue surface. The glass is stretched parallel to the length of the bead; (c) Enlarged view (x8) of the amethyst inner core of BRA-01 (type IIIc1-IIIc); credit photos X. Bai/C2RMF and F. Gendron/MNHN.

3.2 Experimental methods

3.2.1 PIXE/PIGE – Chemical characterization of glasses

Particle Induced X-ray Emission (PIXE) technique coupled with Particle Induced Gamma Emission (PIGE) analysis was carried out with the external beam of the NewAGLAE accelerator [14] at the *Centre de Recherche et de Restauration des Musées de France* (C2RMF). The analytical setup included four detectors. The artefacts were subjected to a 3 MeV proton beam of 1 nA intensity for a few minutes allowing the chemical composition of the samples to be derived from two PIXE spectra. The first spectrum was devoted to the major elements (from sodium to iron, $10 \leq Z \leq 26$) measurement; it was recorded in a helium atmosphere allowing the measurement of sodium, magnesium, aluminum, and silicon. The second spectrum was devoted to the measurement of trace elements. It was recorded with three detectors screened with a 50-μm aluminum absorber to attenuate the X-ray of major constituents. The analysis was recorded by scanning the 50-μm beam on a $1 \times 1.5 \text{ mm}^2$ area, to account for the composition of each glass layer of the multi-layered beads. The major element concentrations were determined from the first spectrum. The matrix composition determined in this step was used to process the second spectrum by adjusting trace element concentrations to consider the level of iron determined in the first spectrum [15]. PIGE spectra (in particular, the 440 keV

gamma ray prompt-induced by the ^{23}Na ($p, p_1\gamma$) ^{23}Na nuclear reaction) were used to derive the sodium content of glasses to have an accurate composition minimizing the impact of possible glass alteration; Brill A, B, C and D glasses from Corning Museum of Glass were used as references. The quantitative processing was achieved using the TRAUPIXE program [16] developed at the C2RMF based on GUPIXWIN software [17]. Before the analysis of archaeological artifacts, the full processing chain was carefully checked on pellets of reference targets (DR-N from *Centre de Recherche Pétrographique et Géochimique* [CRPG], Corning Museum of Glass Brill A, B, C and D). The main composition of these two European archaeological multi-layered beads found in Mexico is shown in Table 2.

3.2.2 Raman spectroscopy – Mineralogical characterization of glasses

Raman spectroscopy was used to characterize glasses composition and structure and Q^n speciation of the glass [18]. Raman spectra were obtained using a LabRAM HR Evolution Raman spectrometer (Horiba) equipped with a Peltier-cooled CCD and 1800 lines per mm grating at the *Institut de Physique du Globe de Paris* (IPGP). For excitation, a Coherent laser LM405-180 with a wavelength of 405 nm and a nominal laser power of 180 mW was used. The spectra were acquired with a 50x objective and a 50-pinhole in a frequency range between 20 and up to 1200 cm^{-1} . The spectral resolution of the setup is $\sim 1.7 \text{ cm}^{-1}$ and the spatial resolution is $\sim 1 \mu\text{m}$. The laser power at the exit was adjusted to 10 mW on the sample. The spectra were acquired in three windows from 20 to 1500 cm^{-1} . We decided to use the 405 nm because the glass composition is relatively rich in Mn^{2+} which can produce a large fluorescence band with higher excitation frequency as shown by Schibille et al. [19]. Acquisition time was 60 seconds with 3 repetitions for each window.

Table 2. Chemical composition of multilayered beads 145-138 and BRA-01 (wt.% and ppm).

Bead NO.	Layer	Na ₂ O*	MgO	Al ₂ O ₃	SiO ₂	P ₂ O ₅	Cl	K ₂ O	CaO	Fe ₂ O ₃	Ti (ppm)	Zn (ppm)	Sr (ppm)	Ba (ppm)
145-138	Outer	9.04	1.37	3.84	69.70	0.37	0.96	2.24	8.76	0.74	1179	84	426	381
	Central	9.77	1.37	1.87	56.01	0.26	0.84	1.84	7.09	0.47	1041	74	451	760
	Core	11.91	1.94	1.25	70.97	0.18	1.00	2.31	9.24	0.48	676	43	601	479
BRA-01 (140-133)	Outer	6.86	0.75	3.80	73.52	1.92	0.70	1.73	5.36	1.14	883	110	369	41
	Central	8.10	1.30	2.98	61.71	1.62	0.48	1.88	5.45	0.76	985	91	390	376
	Core	7.63	0.62	2.57	72.71	2.57	0.21	1.35	5.32	1.32	860	239	580	205

* This data was obtained by PIGE.

Table 3. Trace-elements composition (wt.%) of multi-layered beads BRA-01 and 145-138.

Bead NO.	Layer	SO ₃	V ₂ O ₃	Cr ₂ O ₃	MnO	CoO	NiO	CuO	As ₂ O ₅	SnO ₂	PbO	Bi ₂ O ₃	UO ₃
BRA-01 (140-133)	Outer	0.46	0.000	<i>0.002</i>	0.083	0.035	0.015	<i>1.100</i>	0.063	<i>1.194</i>	0.892	<u>0.198</u>	0
	Central	0.00	0.013	0.001	0.705	0.184	0.036	0.250	0.120	7.484	6.158	<u>0.536</u>	0
	Core	0.00	0.004	<i>0.002</i>	<i>1.092</i>	<i>0.566</i>	<i>0.198</i>	0.126	<i>0.760</i>	0.208	0.255	<u>2.267</u>	<u>0.002</u>
145-138	Outer	0.39	0.001	<i>0.002</i>	0.048	0.003	0.003	<i>1.427</i>	0.004	<i>0.502</i>	0.389	0.001	0
	Central	0.00	0.016	0.000	0.278	0.002	0.007	0.177	0.019	8.954	10.715	0.021	<u>0.002</u>
	Core	0.28	0.001	<i>0.002</i>	0.130	0.002	0.002	0.006	0.001	0.048	0.047	0.000	0

Italic numbers*: the colorant elements; **Bold numbers: the opaque elements; Underlined numbers: the unusual elements.

4. Results

4.1 Glass beads' chemical compositions

From the microscopic images, surfaces of the bead 145-138 appear very altered, so we need to locally remove this degraded layer using fine sandpaper. This operation allows us to obtain a better analysis of the material of the bead. The glasses lose their sodium along the alteration, therefore, the 145-138 that has been cleaned seems to have more sodium than the BRA-01. This last did not seem altered, but it appears that the outside glasses (central and outer) are slightly altered.

The compositional data obtained by PIXE/PIGE show that the base glass characteristics of the three layers in bead 145-138 and BRA-01 are similar in Na₂O, MgO, Al₂O₃, SiO₂, P₂O₅, Cl, K₂O, and Ca₂O as well as in trace-elements Ti, Zn, Sr, and Ba, but with significantly different concentrations. Judging from the elevated MgO and K₂O contents (> 1.5 wt. %), 145-138 have been produced with sodium flux issued from ashes halophytic plants such as *Salicornia (Salicornia europaea)* whose cultivation for this purpose is documented from Catalonia to Italy, as early as the 13th-Century [20] [21] [22] [23]. It can be assumed that the same is true for bead BRA-01, even though soda and magnesium oxide are somewhat low due to a high degree of surface alteration (see Table 2). Phosphor concentrations are significantly higher in BRA-01 than in 145-138, which may again be an effect of corrosion or residues of contaminations caused by secondary working practices.

Different colouring elements/agents have been identified that underlie the different layers (see Table 3). The shadow and bright blue colours of the outermost layers in both beads are probably due to copper in its oxidized state, with cobalt possibly contributing to the colouring of bead BRA-01. Cobalt underlies the inner layer of BRA-01. The cobalt colourant is associated with elevated levels of Mn, Ni, As, Bi, and U [24]. The central layer in both seems to be coloured and opacified by a combination of lead and tin-oxides, which was confirmed by Raman spectroscopy.

4.2 Opacifying agents and microstructure

Figures 7., and 8. show Raman spectra for 145-138 and BRA-01 beads respectively. Several spectra were obtained in each of the three glass layers, outer, central, and inner corresponding to the core zone of the beads. Alteration products are visible in Figure 6. (b). We have not analyzed them using Raman spectrometry, and have focused on the analysis of pristine glass. However, one of the spectra for the middle layer in Figure 7. shows the start of a peak at 1100 cm⁻¹ characteristic of a carbonate, which may be due to the onset of alteration of the middle layer. In some cases, traces of carbonates may be evidence of bad melting of the raw materials but this generally favours the alteration process.

In Figures 7., and 8., which correspond to the two beads, we can see that each layer has a different glass Raman spectrum. Several Raman spectra were produced for each layer, clearly showing that each bead is composed of three different homogeneous glasses, confirming the PIXE and PIGE analyses. The compositions of the six glasses making up the six different layers correspond to soda-lime silicate glasses, with slight differences from one glass to the next as is relatively well known in this sort of bead [25]. For example, the inner and outer layers of the two beads show fairly similar spectra, while the Raman spectra of the middle layer are more different. One Raman spectrum of the outer layer of the bead 145-138 shows a small peak which can be attributed to the beginnings of cassiterite (SnO₂) crystallization or it may be the result of contamination between the outer and middle layers when the beads were made. The addition of lead and tin in the middle layer was used to opacify the glasses, as clearly shown by the two intermediate layers of the two beads. This opacification is achieved by the crystallization of cassiterite, characterized by the peaks at 635 and 775 cm⁻¹ and the light

changes in the area ratios of the bending and stretching bands near 1100cm^{-1} as shown by Koleini et al. [25].

The central layer of both beads shows relative variability in the Raman spectra, which correspond to a combination between the glass spectrum of the middle layer and the cassiterite Raman spectrum characterized by the intense peak at 635cm^{-1} . The Raman spectra of the six glasses (three per bead) show a peak centered around 1100cm^{-1} essentially characteristic of SiO_2 in Q^3 species, a peak around 950cm^{-1} which can be attributed to Q^2 species, and a small peak around 1000cm^{-1} often observed in mixed alkaline/alkaline-earth aluminosilicates [18]. Taken together, these Raman peaks show that the compositions of the glasses used to manufacture the three bead layers correspond to sodic ash silicate glasses [26].

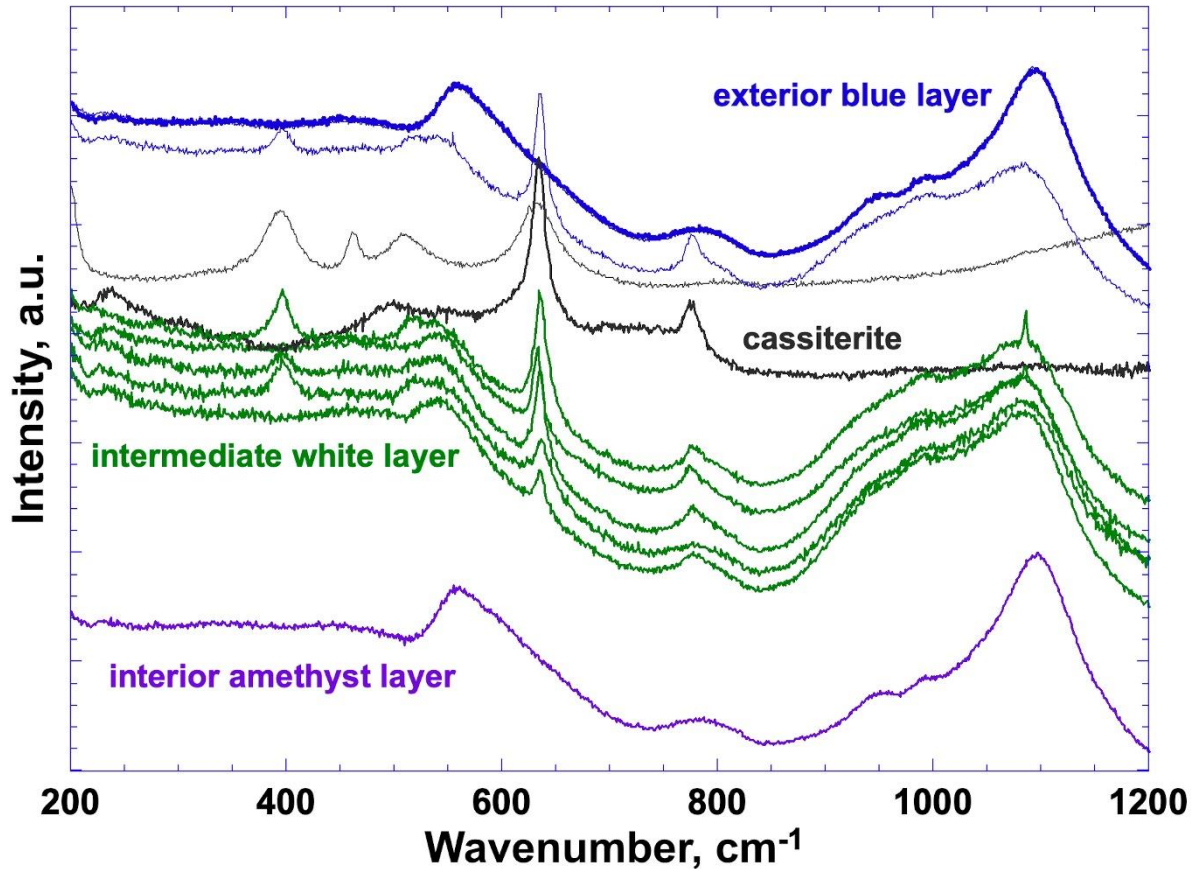


Figure 7. Raman spectra of 145-138 bead (Tamtoc).

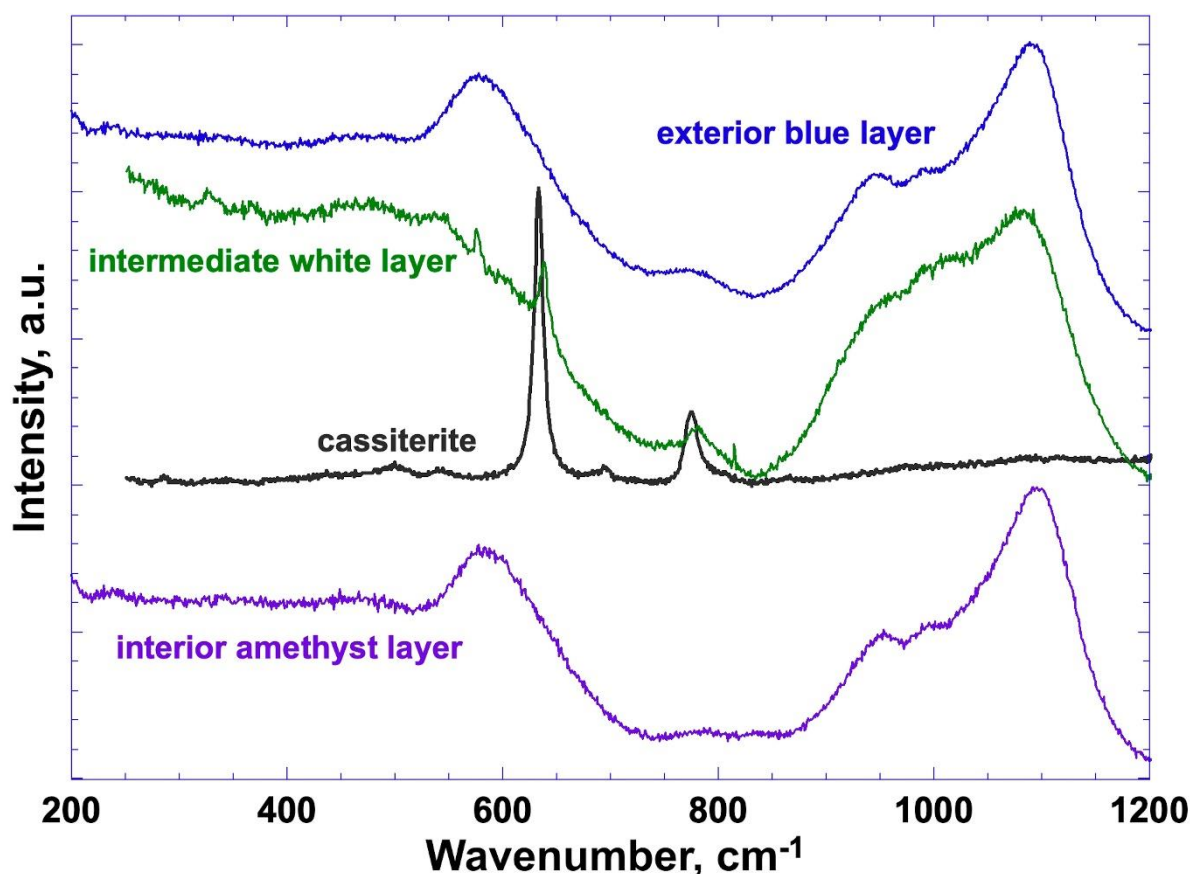


Figure 8. Raman spectra of BRA-01 bead (CNRA).

5. Discussions

5.1 Nueva Cádiz beads' Historiography

Our bibliographical research about the diversity of European glass beads introduced into the Americas during the first centuries of the Conquest (1492-1600) reveals morphologically similar types to the specimens discovered on the Tamtoc Peninsula. These types are referred to as "*Nueva Cádiz plain*" by the North American archaeologist J. Mann Goggin (1916-1963). This name is borrowed from the colonial city of Nueva Cádiz, in whose ruins these multi-layered beads were first discovered and published [10, pp. 66] [27], [28, pp. 258, no. 1133a]. The history of this Spanish city began around 1500 A.D. onwards on the Cubagua island, which lies in the Caribbean Sea off the Venezuelan coast. The Spaniards' decision to settle on this arid and isolated land was motivated by the lure of profit, the presence of fantastic pearl oyster beds in the coastal waters and those of the neighboring islands (Margarita, Coche, La Tortuga, La Blanquilla). From 1508 to 1513, the Spanish crown incorporated these islands and the continental coastal strip between Cape Vela (Colombia) and Cape Maracapaná (Venezuela) into the Governorate of Urabá. Then, from 1528 to 1546, the Governorate became the German colony of *Klein-Venedig* (Little Venice or Poor Little Venice) or *Welserland* (Land of the Welser [bankers]) [29, pp. 309]. But, on December 25, 1541, *temporum titulum*, Nueva Cádiz was destroyed by a tidal wave and never rebuilt. Based on the excavations carried out between 1954 and 1958 by Venezuelan archaeologist J.M. Cruxent (1911-2005), Goggin adds a census of Pan-American finds of plain and twisted Nueva Cádiz beads. At the time, he reported only one Mexican specimen but did not specify where or by whom it was found [27, p. 7]. Since then, new, better-documented discoveries have been made, such as in the cemetery of the San Gabriel de Tacuba church (México D.F.) [30] and in the Chinantla Baja and Chinantla

Media Oaxacan regions [31, pp. 88 quoted by 28, pp. 22-23]. Finally, other specimens come from the colonial villages (16th-17th-Cent. A.D.) of Lamanai and Tipu in Belize [32].

Before the discovery of the CNRA, the number of Nueva Cadiz beads found in Mesoamerica was limited. Adding up the numbers quoted in previous cited publications, we can confirm that there are barely a dozen specimens.

According to Loewen and Dussubieux [10, pp. 64-65] and Walder *et al.* [11, pp. 86], tubular beads with alternating amethyst, white, and bright blue, like the set 140-133 (Type IIIc1), were imported into the Americas from 1500 until around 1560 A.D. Radiocarbon dates from the RA12A burial from CNRA confirm their statement and make the 95 Nueva Cádiz beads that were attached to it, the oldest currently discovered and ¹⁴C dated in the Americas. They come from an archaeological context older than those discovered in the Nueva Cádiz ruins in Venezuela, or at least contemporary. As for the 145-138 bead, according to the same authors [10, 11], this type IIIc2 was only produced until around 1560. Our typo-chronological study had therefore helped to place the context of the 145-138 bead between 1518 and 1560 AD. This period can be extended by some years due to the time of commercial exchanges, but the old ceremonial center of Tamtoc was quickly abandoned during the brutal Huasteca conquest, at an unknown date after 1522.

5.2 Chemical Analysis and Raman Spectroscopy

PIXE, PIGE, and Raman analysis confirm that the glasses of the Nueva Cádiz beads discovered in the Tamtoc peninsula result from the fusion of silica sands mixed with sodium-rich ashes of halophytic plants, typical of glass produced during the Late Bronze Age and revive in the 9th-Century AD [20]. Raman spectra confirm that these two beads are made up of the superposition of three glass layers of soda-lime silicate with minor compositional differences. Cassiterite (SnO₂) is the main opacifier and colourant in the central white glass layer. The presence of lead oxide in the two intermediate layers of glass was likely added intentionally to reduce the viscosity [33] of the crystal/liquid mixture at high temperatures, facilitating the formation of cassiterite crystals [25]. In turn, this would increase the viscosity [33] [34], and the final glass will be white as opacified glass ceramics.

Differences in Al₂O₃, Fe₂O₃, Ti, and Zr support the hypothesis of different silica sources. Considering the levels of alumina as an impurity of the silica source in the 145-138 layers (3.84 wt. %, 1.87 wt. %, and 1.25 wt. %) we can distinguish two compositions. According to Friedrich and Degryse [35, p. 145], the intermediate and internal layers fall into Group A.1 (Al₂O₃: 0.63 - 2.76 wt. %) which corresponds to glasses for which the source of the silica is quartzite pebbles, with low amounts of feldspars, reduced to powder. This is the technique that was practiced by Venetian glassmakers. The 145-138 outer layer corresponds to their second group (A.2) [35] characterized by higher contents of alumina (3.53 – 5.07 wt. %) with iron oxide (1.25 - 1.45 wt. %), more characteristic of medieval glassworking in Tuscany and the exploitation of local silica sources [36, pp. 73], [37, pp. 349], [38].

As for the three BRA-01 glasses, with Al₂O₃ contents of 3.80 wt.%, 2.98 wt.% and 2.57 wt.% and Fe₂O₃ contents of 1.14 wt.%, 0.76 wt.% and 1.32 wt.%, they correspond more closely to the group A.2 [35, pp. 145].

The use of cassiterite as a glass opacifier is a chronological indicator. At the beginning of the 16th-Century, tin oxide was used in Italian and Spanish glassmaking (Murano, Venice, Veneto and Altare Liguria; Seville and Granada, Andalusia), and was Portuguese practices [2, pp. 46-47] [28, pp. 65]. In the 14th-15th-Centuries, traders from Majorca (Balearic Islands, Spain) introduced it to Italy from where it passed to France (Lyon, Nevers, Rouen, Saint-Porchère) during the 16th-Century [39]. This tin-oxide glass technique will then spread throughout Europe (Holland, Germany, England, and Eastern Europe).

Finally, the notable presence of bismuth in the inner layer of the BRA-01 bead is an authentic geographical marker. It is associated with the cobalt source, more precisely with the cobalt ores in southern Germany (Saxony) in Schneeberg and Freiberg mines which have been mined since the 15th-Century [24]. The mineral exploited is asbolane ($[\text{Co,Ni}]_x\text{Mn}[\text{O,OH}]_4 n\text{H}_2\text{O}$), a cobalt-bearing manganese oxide, which appears in the form of black kidneys [40]. These masses contain high amounts of cobalt, nickel, manganese, and other elements such as arsenic, bismuth, and some uranium for the Freiberg mine.

As a conclusion to these historiographical and analytical researches, we hypothesize that the colour variations observed within the BRA-01 and 145-138 beads can correspond to two distinct glass factories.

5.3 Comparison of analytical results

The composition of ashes soda-lime-silica glass from Nueva Cádiz beads from the Tamtoc peninsula is therefore comparable to that of other specimens found in Venezuela (Nueva Cádiz ruins' [10, pp.70, Table 4]) and Bolivia (Tiahuanaco region? The Jones-Avent collection [11, pp.89, Table 1]) [28] [41] (Table 4). We only regret the absence of analysis of Mexican beads from the San Gabriel de Tacuba cemetery (México D.F.) and the Chinantla Baja and Media regions [30] [31]. This sodium-rich composition of halophytic plant ashes is also found in specimens discovered in 17th-Century contexts, such as the Santiago del Baradero cemetery (Argentina) and Huron sites in Simcoe County (Ontario, Canada) [11] [42].

We also note that only Mexican and Argentine specimens are melted with silica sands rich in aluminum oxide impurities. Compared with European glass compositions from the 15th and 16th-Centuries, the best coincidences are with glass from the Iberian Peninsula (Andalusia and Portugal) and, above all, with that from the Ligurian village of Altare (Italy) [43, pp. 2193, Table 2] and Cucagna Castle (Friuli, Italy) [35, pp. 144, Table 2]. Historically, the furnaces of the Altare glassworks were lit in medieval times (1179 AD) and were not extinguished until 1978 [44]. However, as the archives of this locality have been destroyed, it is impossible to know whether these glassworks produced anything other than flat glass (windows, mirrors) and hollow glass (glassware, containers). The glassworks at Cucagna Castle, lit in the 12th-Century, were finally extinguished in 1522 when the Republic of Venice conquered Friuli.

Table 4. Comparative compositions for Nueva Cádiz beads and glasses of different origins (wt. % and ppm).

Origin	Layer	Na ₂ O*	MgO	Al ₂ O ₃	SiO ₂	P ₂ O ₅	SO ₃	Cl ₂ O	K ₂ O	CaO	Fe ₂ O ₃	Ti ppm	MnO
Nueva Cádiz, Venez. XVI th -Cent. [10, Table 3]	External Turquoise	13.7	3.1	1.1	71.2	-	-	-	2.9	7.6	0.4	168- 282	
	Intermediate White	13.0	3.1	1	71.8	-	-	-	3.3	7.2	0.6		
	Internal	12.5	2.5	0.9	73.2	-	-	-	3.6	6.4	0.9		
TiahuanacoBoliv. XVI th -Cent. [11, Table 1]	External Turquoise	12.7	2.8	0.9	67.8	0.3	-	-	3.8	7.3	0.5	217- 331	
	Intermediate White	10.1	2.2	0.7	51.8	-	-	-	2.7	6.0	0.4		
	Internal	13.6	2.8	1.0	70.6	-	-	-	3.2	7.6	2.4		
San. Bara. Argentina. XVII th - Cent. [42, Table 6]	External	6.9	1.23	7.03	78.47	-	-	0.64	1.97	3.0	0.6	-	
	Turquoise												
Simcoe Co. Ont., Cand. XVII th - Cent. [11, Table 1]	External Turquoise	9.0	3.2	1.0	68.3	0.3	-	-	6.3	7.2	0.6	0.09	
	Intermediate White	8.4	2.6	1.7	48.3	0.4	-	-	2.8	6.8	0.7		0.11
	Altare, Liguria, Italy	-	-	2.1-7.8	-	-	-	-	1.1- 7.5	-	-		-
14-16 th -Cent. glasses with known origins [43, Table 2] [35, Table 2] [10, Table 5]	Cucagna Cast., Friuli, Italy	12.71	2.95	1.70	68.20	0.16	0.30	0.93	2.38	9.06	0.51	0.07	1.05
	Grenada, Spain	-	-	2.1-4.2	-	-	-	-	5.7- 6.9	-	-	-	600- 1350
	Portugal	-	-	1.8-6.1	-	-	-	-	2-6.9	-	-	-	370- 750

5.4 Nueva Cádiz beads manufacturing processes

The basic materials used to manufacture Nueva Cádiz glass beads are silica sand (SiO_2), soda (Na_2O) derived from halophyte plant ashes, potash (K_2O) combined with magnesium (MgO), and lime (CaO). Thanks to this mixture, the viscosity point, in particular that of silica sand, drops from 1730 °C to 1400 °C, resulting in significant fuel savings over time. During this dynamic melting process, the glasses are coloured and opacified by the addition of copper, cobalt, manganese, tin, and lead oxides. In the case of multilayered, polychrome beads such as Nueva Cádiz, their manufacture requires the firing of a sufficiently large furnace or battery of furnaces capable of holding three glass crucibles simultaneously in a viscous state. Once these mixtures have been brought to and maintained above their viscosity point, the manufacture of the glass canetilles begins. This technical operation can be carried out according to two processes described in Neri's glass treatise of 1612 [45], but, according to our observations, the beads from the Tamtoc peninsula were made by hot stretching a canetille.

5.4.1 Observations and technical traces on beads from the Tamtoc Peninsula

The deformation of the diameter, the very regular tubular appearance of the beads in our study, the unidirectional orientation of the air bubbles trapped in the outer layer, and the literature, prove that they were made using the drawn glass process. Observation suggests that the cane was made using the coiling technique if the bubbles were oriented in a radial direction. On the other hand, if the bubbles follow the main stretching orientation, as in the case of the specimens studied here [Figure 5. (b), and 6. (b)], the original tube will have been formed by the three-color glass gob stretching technique.

5.4.2 A canetille of drawn glass

Indian and Near East glassmakers' traditions were sources of inspiration for Murano glassmakers who learned an older, semi-industrial process in the 14th-Century [28]. This process is more cost-effective because it allows the production of beads in large batches by stretching the glass. The chain of operations for manufacturing begins with the fusion, in three crucibles, of glasses of different colors. In the first crucible, the glassmaker gathers a sufficient quantity of glass, or glass parison, using a hollow metal cane. Then quench this glass parison which will correspond to the internal layer or core of the beads - the colorless layer in the case of the specimen from Tamtoc (145-138) and the amethyst for those from the CNRA (140-133) – in the second crucible that contains the opaque white glass. This operation is repeated in the crucible containing the blue glass which will form the outer layer. Without letting up, the glassmaker blows into the cane to introduce an air bubble into this multi-layered and polychrome glass parison which inflates into a hollow sphere. An assistant or “cane puller boy” [45] intervenes, attaching a solid cane or rod called a pontil to the other end of this viscous glass ball. The two operators then move in the opposite direction to stretch the glass sphere into a long tube [46]. As long as the glass retains its viscosity, it remains possible to stretch it but, depending on the written sources, the size of this hollow rod, or tube, varies considerably, even excessively. After solidification, the large tube is cut into the desired lengths. For this, written sources mention wet blades and therefore thermal shock. It is also possible that it is scratched with a mineral harder than glass, then split as is done for flat glass, or even that it is cut on a block with a hammer as with mosaicists.

All subsequent and special shaping and finishing, such as quadrangular profiling and twisting, is carried out on segments from the main original tube [28] [46]. The difficulty hidden beneath the apparent simplicity of this shaping is to obtain a square profile containing a perforation, with this external shaping having to impact as little as possible on the circular shape of the inner hole. If the square profile is produced by blowing the glass into a mold, the air pressing the glass against the walls will produce not only a square outer profile but also an inner

profile of the same shape, as confirmed by contemporary glassblowers [2]. The bottles blown in square molds reflect the effects of this process.

However, some sources claim that it is possible to mold the glass parison before stretching the rod or to give the softened rod this shape, without the inner diameter undergoing deformation [12]. Alternatively, the segment is softened by annealing and then pressed into a mold - made of plaster, ceramic, wood, or metal [46] - with a square profile. However, this operation also has the effect of pressing the diameter, which collapses and takes on the shape of an ovoid. Observation of the edges of the two specimens from the Tamtoc peninsula confirms that this process may have been used to shape their original segment. However, this process leaves the mold's seams visible where its two parts meet, but we do not observe them here. Finally, there remains the option of flattening the softened segment with a flat or palette tool, against an angled shape [45] or not [13]. The latter process leaves no seam but, like that produced by in-mold pressing, it causes the central hole to collapse.

Then, the twisting operation seen on the Nueva Cádiz beads from the churchyard in Tacuba (Mexico) [30], can take place. This is a simple concept, but only possible if the glass is viscous. Finally, whatever shaping and finishing operations are carried out on these segments of the original rod, will be performed before the final annealing. In other words, any hot operation on glass requires subsequent annealing to release the stresses accumulated in the material. Depending on the type of glass, this annealing is carried out at between 450 and 550°C, followed by a long cooling period. Finally, these sections of rod or tube are reduced to beads a few centimeters to a few millimeters long. When cold, this cutting produces sharp but random breaks in the canetille, as can be seen on Mexican type IIIc1 beads (BRA-01). On the other hand, if this cutting is carried out under heat with a blower chisel, the cuts will be clean and relatively flat, with more prominent edges like on 145-138 bead. Here, briefly summarized, are the production processes for the little blue beads that enabled the conquistadores to get rich more cheaply when bartering with the natives of the Americas or Africa. Faced with the infatuation of the peoples of the Mesoamerican area for these blue glass beads, we also wondered about the anthropological foundations of this colour attraction.

5.5 Blue color symbolism in Mesoamerica

Now that the typology, analyses, technical reconstructions, and hypotheses of origin have been established, the question remains as to the specific success of these blue beads with the indigenous populations of the Americas. For the conquistadors, they were nothing more than modest blue glass beads that allowed them to make a shameful 1000% profit when exchanged for native gold. Although at the same period, they were being exported to Asia and Africa, for the peoples of the Mesoamerican geocultural area, these beads take another resonance. These multicolored beads have the appearance of their precious stones. The blue of the outer layer of the Nueva Cádiz beads makes them look like turquoise or amazonite, minerals from deposits in Southwestern USA (New Mexico, California, Colorado) for the former, and from Chihuahua (*Municipio Coronado*) for the latter [47] [48]. During the 16th-Century, with the help of Nahuatl scholars, the Franciscan Bernardino de Sahagún (1500-1590) drew up a list of gems known and named by the pre-Hispanic populations of the Mexican Central Plateau. This inventory includes some blue gems such as the *xihuitl*, *teoxihuitl*, *xoxouqhui itztli*, which "is sometimes blue", *tolteca itztli*, *matlalitzli* and *xiuhmatlalitzli* [49, Book XI, Ch. 8] [50]. While it would be risky to attribute contemporary mineralogical equivalents to these ancient Nahuatl appellations, these stones do reveal that the color blue was highly symbolic in Mesoamerica. In a vast semantic field, it was linked to the South, to the heat, to the color of the sky, to the zenithal sun named *Xiuhpiltontli* (Child of Turquoise), to the solar year, and the annual cycle of plants. Blue gems like turquoise symbolized dry grass, as the Aztec century lasted 52 solar years and ended with the binding of a bundle of 52 reeds, the *xiuhmolpilli*. Since a plant's cycle of growth and decline lasts one solar year, turquoise was used to metaphorically express this heliacal duration. Rare, valuable, and from faraway regions, blue gems adorned the attributes of the heliac gods. The term *xihuitl* was used in the syllabic construction of terms designating the

precious or the divine. Coincidentally, Nueva Cádiz beads carry this precious blue color a coincidence that would explain the greed with which members of the Floridian Indian nations (Cayuse) bordering the Gulf of Florida plundered the cargoes of Spanish ships grounded on their shores [3]. The recovered glass beads were then integrated into their ancestral trading system and redistributed to the cities of the peninsula's interior.

5.6 Spaniards in the Huasteca, 1518-1530

The first contact between Spanish conquistadors and the main indigenous populations (Teenek and Nahua) of the Huasteca took place in 1518. These coincide with the stopover of J. de Grijalva's (1490-1527) expedition at the mouth of a wide *río* that the Spaniards named "*de los Canoas*". Perhaps this is the *río* Tanhuijo (Veracruz)? From the coasts of the Yucatan peninsula to those of the northern Veracruz, the members of the expedition tried to establish trade relations with the Indians. According to B. Diaz del Castillo [51], in San Juan de Ulua (Veracruz) 2,000 green glass beads, part of what was bought in Cuba were exchanged for fine cotton clothes and a few gold artefacts. However, during their brief stopover at the mouth of this *río*, the situation degenerated, forcing the Spaniards to withdraw, abandoning one of their ships with its trading cargo.

In late 1518, Captain and cartographer A. Álvarez de Pineda (1494-1520) was sent by F. de Garay (1475-1523), Spanish Governor of Jamaica, to explore the coastline of Florida. After several unsuccessful attempts, he set course for the west, following the coastline of the Gulf of Mexico southwards and entering in the mouth of the *río* Pánuco (Tamaulipas). The conquistadors discovered a large town (perhaps the site of Las Flores?) and spent some forty days at rest without encountering any animosity. Pineda even had his ships careened and sailed six leagues up the river, discovering some forty villages. Before returning to Jamaica, he chose to establish a colony. But, when Captain D. de Camargo (?-1520) supplied the colony in 1520, he discovered the place besieged by native warriors. Pineda and Camargo attempted a rescue operation in which the cartographer and almost all the colonists were killed, while their meager possessions fell into the Indians' hands [52].

Finally, in 1522, a year after the fall of Mexico-Tenochtitlan (August 13, 1521), the land conquest of the Huasteca began. H. Cortès (1485-1547) entered the region from the west to consolidate his territorial hold on the Huasteca against the ardor of F. de Garay. He led a troop of conquistadores, 300 *peones*, 120 horses, a few artillery pieces, and 40,000 allied Indians commanded by the *tlatoani* H. Ixtlilxóchitl (1500-1550). The column followed the ancestral salt route that had long linked the Gulf Coast to the cities of the Mexican Central Plateau. This territorial intrusion quickly took on the appearance of a raid and lasted several years. Cultural damages caused by this expedition were followed from May 1527 by Governor N. Beltrán de Guzmán (1490-1558) who controlled cutting of the autonomous Pánuco territory. This dark and troubled period in the history of the Huasteca was hardly conducive to trade between the natives and the new masters of Mexico, and complete the dating of the Tamtoc peninsula archaeological contexts. These Huastecan glass beads undoubtedly arrived from the Gulf coast and, if so, are linked to the brief stay, in 1518 and 1520, of two historic Spanish expeditions.

6. Conclusion

The 96 European glass beads of Nueva Cádiz type found on indigenous sites in the Potosinan Huasteca (Mexico) fall into typological variants IIIc1 (95 specimens) and IIIc2 (1 specimen) of the classification established by Kidd and Kidd [12] and Karklins [13]. ¹⁴C dating of the remains of the deceased (RA12R), to which the 140-133 set of beads was attached, reveals a very early colonial context (1512 AD ± 30 years), while the undated context of the specimen 145-138 cannot be later than 1560. Otherwise known as "Nueva Cádiz" in the literature, the beads from the CNRA would therefore be the oldest ever discovered in the Americas.

The chemical (PIXE and PIGE) and mineralogical (Raman microprobe) analyses carried out on these beads confirm that their glass is indeed European, as all are ash soda-lime in nature and of Mediterranean manufacture type. The soda used is derived from the ash of halophytic plants, and the silica sand (SiO₂) from the crushing of quartzite pebbles with amounts of feldspars. The oxides used to color these glasses are copper for the outer layer of glass, mixing tin and lead for the inner glass, and magnesium blended with cobalt for the BRA-01 inner glass. Cobalt, a rare metal on a global scale, is an essential geographical marker here. In the 15th-16th-Century, it was mined in southern Germany at the Schneeberg and Freiberg mines in the Erzgebirge (Saxony). It is one of the structural elements of asbolane, a cobalt-rich manganese hydroxide containing traces of other metalloids and metals such as arsenic, bismuth, and uranium, which we find traces in the inner layer of BRA-01, which links it to the Freiberg mine. A comparison of the glass composition of these Colonial Mexican beads with that of glass produced in various European glassworks in the 16th-Century reveals similarities with Italian glassware from Liguria and Friuli. To narrow down the possibilities, it would be necessary to analyze more beads from the 140-133 set, and, to compare results, analyze other Nueva Cádiz beads found in Mexico (Tacuba cemetery and Chinantla regions).

Mass-produced from canetillas and acquired in great quantities by the conquistadors, Nueva Cádiz beads were imported in big quantities to the Americas in the first half of the 16th-Century. As in the case of Florida, these glass beads were very popular with the indigenous population. The specimens discovered on the Tamtoc peninsula, whose presence is probably linked to the Grijalva and Alvarez Pineda maritime expeditions, appear to be a southern extension of the lucrative activity developed by the Cayuse Indians of Florida. The spread of these beads into the Huastec hinterland may have been supported by their blue color, whose heliac symbolism is evident throughout Mesoamerica.

Data availability statement

The Nueva Cádiz glass beads found on the pre-Hispanic sites of the Tamtoc peninsula (SLP) are available for consultation in Mexico City, Mexico, and on request from **Ms. Estela Martínez Mora** estela_martinez@inah.gob.mx

Underlying and related material

The other Nueva Cádiz beads discovered at *Conjunto Norte Rancho Aserradero*, as well as the Tamtoc specimen, are currently housed at INAH's Cuicuilco site in Mexico City, Mexico.

Author contributions

E. Martínez Mora: Archaeological Investigations – Writing – Original Draft, Funding acquisition, Resources. **X. Bai** and **Q. Lemasson:** Methodology, Formal analysis, Writing - Original Draft. **C. Bachelot:** Glassmaking techniques, Writing - Original Draft. **D.R. Neville:** Methodology, Formal analysis, Writing - Review & Editing - Original Draft. **F. Gendron:** Conceptualization, Archaeological Investigation, Writing – Review & Editing, Funding acquisition, Resources.

Competing interests

The authors declare no competing interests.

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