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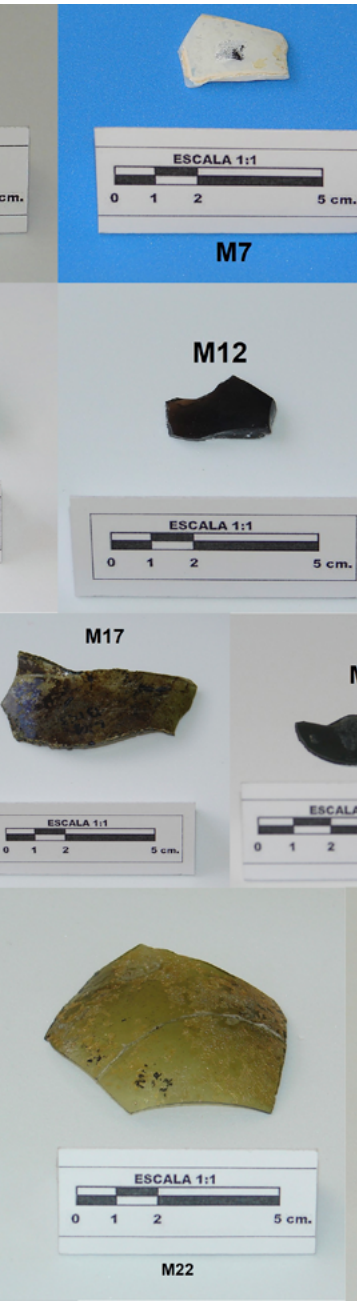

Analysing the Physicochemical Characteristics of an Archaeological Glass Collection from Mexico City, Mexico

Ir a la versión en español

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Translation by Richard Addison

ABSTRACT

This paper is a preliminary study detailing the results of a chemical-physical analysis executed on 20 fragments of archaeological glass from Mexico City. The analysis was carried out using a scanning electron microscopy (SEM) and X-ray microanalysis, two techniques not previously applied in Mexico for this purpose. Both the content of oxides and the sample's physical characteristics allowed us to determine that approximately seventeen coincided with the types of components identified in European glass prior to the 18th century. The other three were identified as having come from a later period. The physical characteristics on the microscopic level were diverse and showed adhesions resulting from the effects of environmental degradation. This facilitates an analysis of oxide content and mechanisms of glass degradation that will allow us to further our understanding of this material's production process in our country moving forward.

KEYWORDS

archeometry, barrilla, tequesquite, glaziers, vitrifiers, fluxes, leaching

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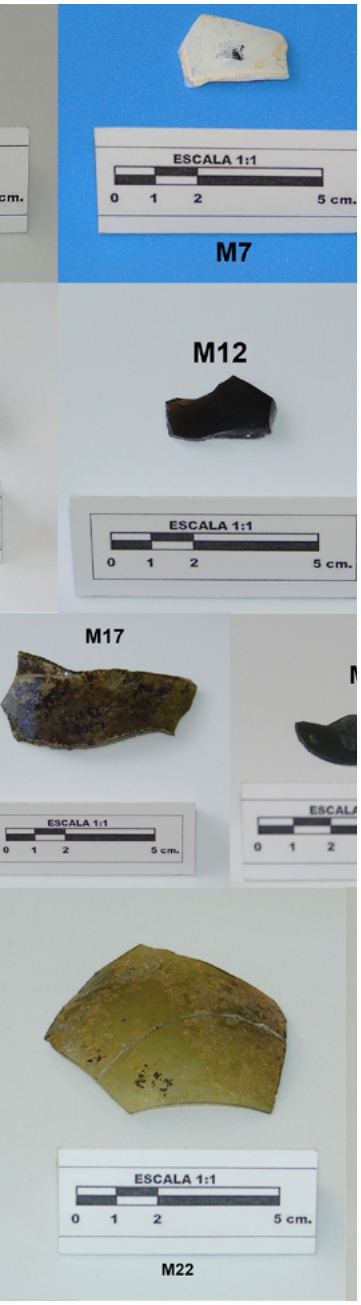

INTRODUCTION

Mexico City, the New Spanish capital, quickly established itself as an important political and economic center in which much of the country's population came to reside, increasing the demand for a variety of products, including glass, initially of sumptuary type. Between 1530 and 1534, the first glaziers arrived in New Spain; so by the end of the 16th century at least eight of them were working in the country, leading to the creation of two ordinances enacted to regulate the use of raw materials. In the 17th and 18th centuries, the number of artisans increased (Peralta, 2011, pp. 34-72; 2014, pp. 73-78). The existence of these and the identification, through the recognition of written and pictorial testimonies, of furnaces in production sites are evidence of the local manufacture of glass. They made flat glass stills, sublimators, mortars, flasks, vessels with particular characteristics, antlers, candlesticks with mirrors, chandeliers, glass beads, eyeglasses and other objects of sumptuary use, at the request of apothecaries, metal separators and, in general, society as a whole (Peralta, 2011, pp. 34-72; 2013, [23], pp. 2-25; 2014, pp. 73-78).

Production persisted despite the instability of the country during the independence movement, resulting in the establishment of approximately seven manufacturing centers within Mexico City's perimeter by the 19th century. Their products were highly sought after by the food and beverage industries, professionals, and the general populace.

Between the 16th and 19th centuries, goods made from this material came from Spain, the Czech Republic, France, England, and what today is the United States (Peralta, 2011, pp. 34-72; 2021, pp. 5-14). From the outset, glass production was driven by the needs of human beings, as well as its aesthetic qualities, and the raw material's geographical availability (the latter two determined its manufacture and if the process of elaboration was to be kept secret, due to the sumptuary value given to the objects). The Industrial Revolution and the resulting advancements in scientific knowledge made it possible to interpret the interaction of the chemical compounds that integrated the glass material, which led to the production process being significantly modified. Even today, we see that industries seek to protect certain aspects of their specific production methods (Alvizar, 2007, pp. 38-46; Peralta, 2018, pp. 3-29).

In Mexico City, the legacy of glass production was left by the Iberian artisans, with everything suggesting that Mexico employed similar methods of manufacturing, since currently there is no written documentation, formula, or exact recipe for the proportions of

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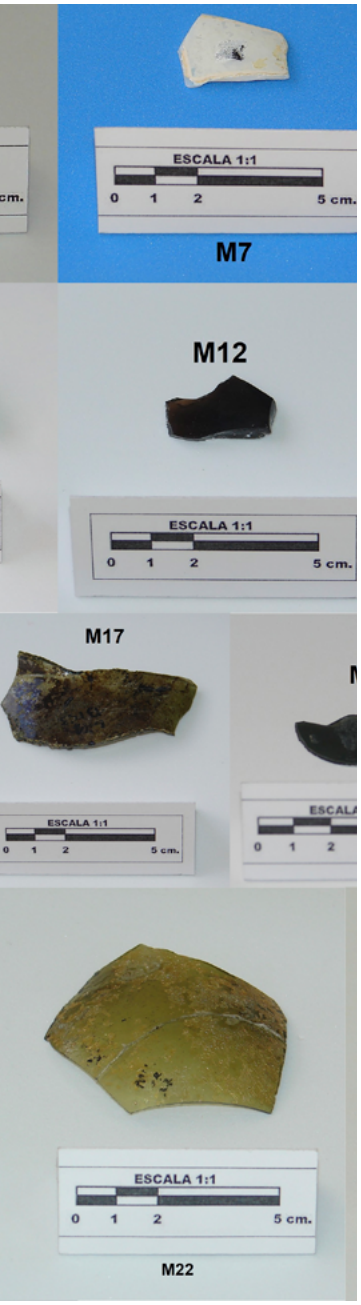

the raw material's components used in the pre- and post-colonial era. On the other hand, the investigation of urban glass objects thus far has been conducted from the standpoint of the aesthetic characteristics of complete pieces, which are compared to those of European origin in order to locate them in a specific period of time. However, the physicochemical analysis of fragments and unconnected pieces of this material recovered in archaeological preservation efforts may prove advantageous in determining their historical context.

It is currently possible to chemically analyze all types of archaeological material through various methodologies to identify the molecular components that constitute them, with which the raw material used can be recognized which, in turn, contributes to the understanding of the manufacturing processes and the evolution of its production (Cadena, 2018, pp. 28-32; Cárdenas, 2020, pp. 70-72).

Since little has been developed here in Mexico—and, even more scarcely, from the perspective of a proper chemical analysis—the manufacture of glass, this work aims to perform a preliminary analysis of the composition of oxides and the microscopic state of archaeological glass fragments. This, in conjunction with the review and observation of macroscopic physical characteristics, facilitates the determination of the period of production, whether colonial or post-colonial.

The proposed objectives are:

1) First, to recognize in archaeological glass fragments located in two areas of the Historic Center of Mexico City the composition of vitrifying, silicon (SiO_2), and phosphoric (P_2O_5) oxides; fluxes such as sodium (Na_2O) and potassium (K_2O) oxide; stabilizers such as calcium oxide (CaO), magnesium oxide (MgO) and aluminium oxide (Al_2O_3); coloring agents such as iron oxide (Fe_2O_3); and decolorizing agents such as manganese oxide (MnO) by scanning electron microscopy, in its X-ray energy emission variant.

2) We also seek to compare the percentage of oxides mentioned with those reported in previous European pieces.

3) Additionally, the aim is to examine the state of microscopic physical deterioration and correlate it with previously documented findings and finally,

4) to correlate the chemical composition and macroscopic physical characteristics of the studied fragments.

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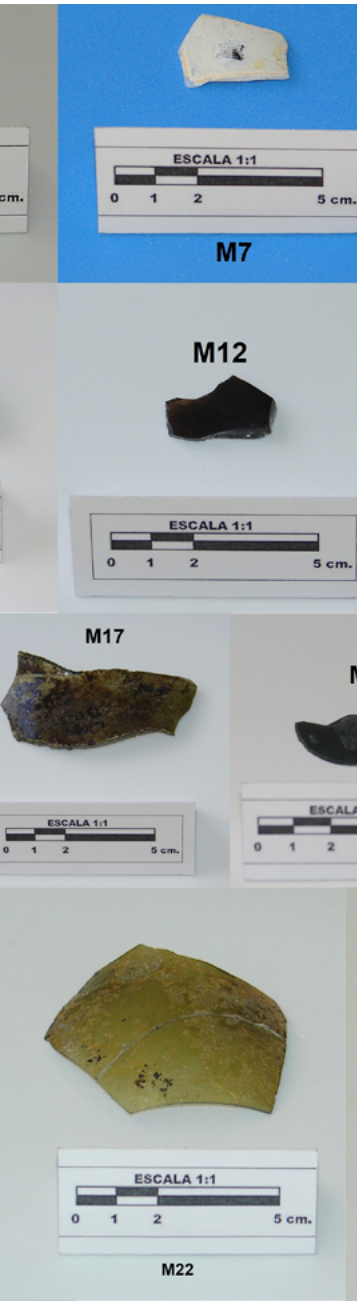

The chemical components used to manufacture glass are oxides; the basic ones constitute the group that allows the reticular molecular structuring, so they are called *vitrifiers*. The *fluxes* are those that facilitate the liquefaction of the raw material, and the *stabilizers* consolidate the chemical structure; those of the secondary group are those that give coloration or suppress it.

Such chemical compounds came from geological and vegetal material; both types required washing, crushing, sifting, and even calcination to achieve their molecular integration at temperatures higher than 1000 °C. The lack of care in the process of selection, purification, and liquefaction of the raw material had an impact on the characteristics of transparency, brightness, and resistance of the glass (Peralta, 2018, pp. 3-29).[1]

MATERIAL AND PROCEDURE USED

A total of 55 fragments of glass pieces, belonging to the Museo del Templo Mayor (MTM) in Mexico City, were obtained from the archaeological rescue work done in the streets of Corregidora and Lic. Primo de Verdad, in the Historic Center. Of these, 20 were selected based on the following criteria:

- they were part of a container or vase,
- colored often associated with the 17th and 18th centuries
- be no larger than 10 cm.

Each one was photographed and numbered for control, with the location in relation to the rescue site, their physical characteristics, and their color, to conform the registry. Small samples were obtained from them for chemical analysis (Figure 1).

The oxides analysed semi-quantitatively were: SiO_2, P_2O_5, Na_2O, K_2O, CaO, MgO, Al_2O_3, Fe_2O_3, and MnO and were reported as a weight percentage (wt.%). An EDAX X-ray spectrometer was used, coupled to the high-resolution scanning electron microscope (SEM) FEI, model SFEG-Sirion XL30. The accelerating voltage used was 5 kV to obtain the images with secondary electrons, however for the microanalyses 20 kV was used, to excite the K_α lines of all the elements present, including that of Fe; the spectra were quantified using the Genesis-spectrum software and all the images were obtained with secondary electrons. The analysis was performed at

[1] This research was supported by the Secretaría de Investigación y Posgrado (SIP) and the Comisión de Fomento de Actividades Académicas of the Instituto Politécnico Nacional (IPN), registration number: 20100708.

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FUNCTION*	SAMPLE / COLOR / OXIDE	4 Strong green	5 Dark grey	6 Hyaith red	7 Opaline	8 Honey yellow	9 Ligth green	10 Opaque olive green	11 Aquamarine green	12 Smoke gray	13 Olive green	14 Dark	15 ultramarine blue	16 Amber	17 Olive green	18 Green	19 Aquamarine blue	20 Opaque light blue	21 Olive green	22 Green olive-yellow	23 Opaque olive green
1	SiO_2	41.21	93.2	65.25	74.71	76.79	71.53	64.57	66.07	57.59	62.55	50.35	62.46	58.16	61.82	71.83	63.80	66.95	64.23	64.44	71.01
	P_2O_5	---	---	0.14	2.98	1.29	1.25	0.16	0.10	0.65	1.11	0.72	---	0.50	1.66	0.06	0.04	0.63	1.43	1.54	0.13
2	NaO_2	21.66	---	17.49	5.61	7.41	10.31	21.86	23.79	27.03	6.66	7.23	22.41	26.22	12.03	20.18	23.52	21.06	10.62	0.18	9.38
	K_2O	0.72	---	3.61	2.24	0.71	2.26	8.19	0.55	1.28	0.96	1.82	0.41	1.69	0.86	0.37	1.09	2.06	0.86	7.21	1.56
3	CaO	1.04	---	0.20	4.21	1.48	2.01	0.80	3.55	3.60	6.92	4.45	0.72	2.56	7.13	4.68	2.29	1.09	26.04	26.04	2.49
	MgO	5.07	2.59	1.10	0.8	5.89	4.79	2.76	1.33	2.73	9.68	6.36	0.20	5.85	6.56	0.26	2.01	4.89	6.97	6.94	3.21
	Al_2O_3	6.45	3.63	10.30	7.86	5.75	0.88	5.74	3.71	5.60	10.02	23.61	1.29	3.97	8.75	1.54	0.25	2.86	6.63	10.28	8.24
4	Fe_2O_3	23.52	---	---	0.32	0.20	0.44	2.08	0.48	2.19	0.44	0.66	0.12	0.48	0.08	0.16	0.64	0.22	0.25	0.39	1.25

*

1= VITRIFYING

2= FLUX

3= STABILIZER

4= COLORING

FIGURE 1. Total number of glass fragments analyzed (Sample record number, sample =M#) (Photograph: M.Sc. José Roberto Peralta Rodríguez, 2019; courtesy: Escuela Superior de Medicina, IPN, Mexico).

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the Materials Science Area of the Higher Education Physics and Mathematics School of the Instituto Politécnico Nacioanl (IPN).

RESULTS AND DISCUSSION

Figure 2 shows the content of oxides quantified by fragment and color. To facilitate the analysis and comparison of figures with other works, tables were prepared by function of each one of them (not included, in order to moderate the length of the paper).

FIGURE 2. Average concentration of oxides in wt.% and color of the samples analyzed (Table: M.Sc. José Roberto Peralta Rodríguez, 2020; courtesy: Escuela Superior de Medicina, IPN, Mexico, 2024).

A) Chemical composition

One of the vitrifiers in the samples was SiO_2, 13 of these samples fell within the range of 55 to 65 wt.%, figures similar to those quantified by other authors in 12th to the 18th centuries glass (Carmona, García-Heras, Gil & Villegas, 2005, pp. 251-258; Müller, Torge &

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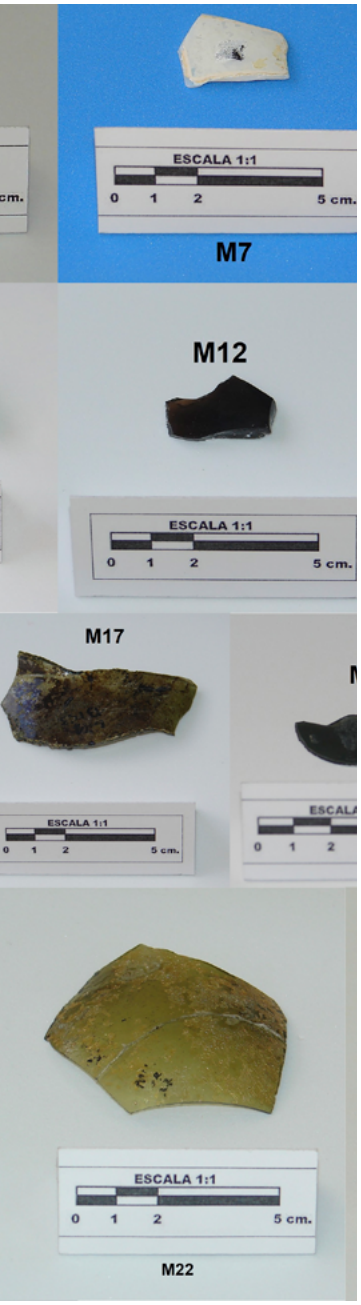

Adam, 1994, pp. 45-48; Schalm, 2004, pp. 1647-1656; Schalm Janssen, Wouters & Caluwé, 2007, pp. 663-668), one of these is below the minimum value; five, slightly above the maximum; and one, significantly exceeds it (the latter is considered to be of more recent production). This indicates that the samples studied were made with original recipes between the 9th and 18th centuries that did not change over time. In relation to the presence of P_2O_5, considered vitrifying since the 20th century (Kreidl & Weyl, 1941, pp. 372-378), was found in 13 of the samples in concentrations between 0.04 and 0.72 wt.%, figures that do not agree with those of the 9th and 18th centuries glass, as they exceed the figures of 2.1 wt.%. Sample 7 contained 2.8 and 18, 6.0 wt.%. This discrepancy in the content of P_2O_5 could reflect the type of raw material used or the type of their production site; therefore, it is ruled out that they were made using modern production processes.

Regarding the flux material, 19 samples correspond to the sodium glass variant and one—sample 22—, to the potassium variant, while the high concentration of K_2O of the latter suggests that it corresponds to an object manufactured in the 18th century. Most likely, dry oak, poplar, pine, birch, and ashes were used as raw materials (Peralta, 2018, pp. 3-29), a primitive recipe of European origin. It is considered that most of the sodium-type pieces used ashes from the *Salsola tragus*, known as *barrilla* in Spanish (a plant of the Chenopodiaceae family that grows in salty soils) or *tequesquite* (from Nahuatl *tequixquitl*) (Peralta, 2018, pp. 3-29), material obtained from the vicinity of Lake Texcoco (Tylor, 1861, pp. 129-161) (there was also a glass factory near the lake since mid-18th century), or sodium carbonate, obtained by the Solvay process, used since 1861.

The use of wood ash and other components to produce glass was made in some European regions, while the use of *barrilla* plants, common in Eastern cultures since Mesopotamian times, was spreading (Tait, 2004, pp. 78-79). With the latter, the melting temperature was reduced; however, the generated vitreous mass was more viscous, and the pieces produced were more fragile, but with potassium oxide, the glass acquired greater brightness (Fernández, 2002, pp. 315-330). The use of the *barrilla* was gradual since this knowledge was reserved, and it was not until the early 19th century that its use was replaced by the Leblanc system, and this, in turn, by the Solvay. Meanwhile, some glassmakers used the K_2O present in the ashes of shrubs.

The considered stabilizing components contributed to the reinforcement of the molecular structure generated by the alkaline

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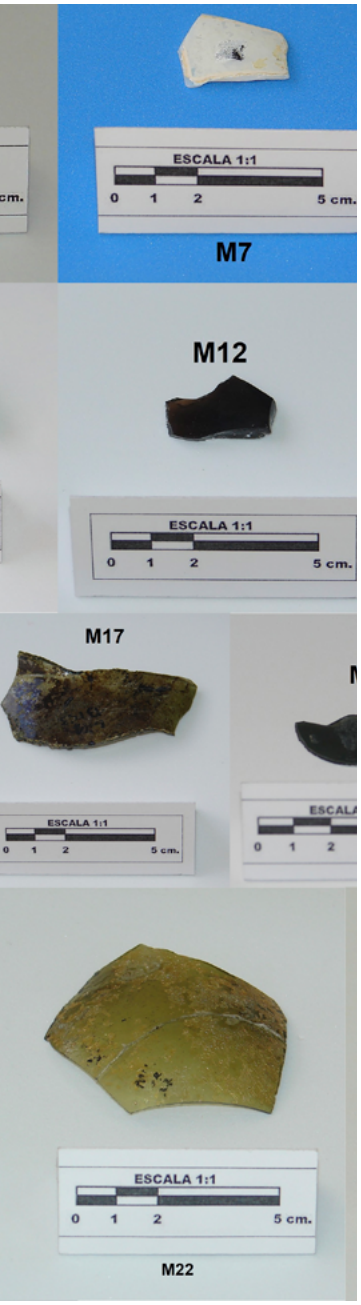

ions (Na and K). In relation to CaO, it appears to have a higher concentration in samples 21 and 22, which is consistent with what is recognized in glass from the 9th and 18th centuries originating in Germany, France, and Spain (Carmona, García-Heras, Gil & Villegas, 2005, pp. 251-258; Müller, Torge & Adam, 1994, pp. 45-48; Schalm, Janssens, Wouters & Caluwé, 2007, pp. 663-668). On the other hand, in samples 6, 10, and 15 the concentration was below 1 wt.%, 14 of these presented it within the range of 1.48 to 7.13 wt.%, reduced values, but not identical to those identified in samples of the mentioned centuries (Cagno, Janssens & Mendera, 2008, pp. 1389-1395; Lima, Medici, Pires de Matos & Verita, 2012, pp. 1238-1248; Marrocchino et al., 2020, pp. 819-827; Verita, Reiner & Zecchin, 2002, pp. 261-271; Wolf et. al, 2015, pp. 660-667); however, studies of Italian samples from the 13th to 16th and 16th to 17th centuries reported ranges lower than those from the 9th to 18th centuries, also of the same provenance (Cagno, Mendera, Jeffries & Janssens, 2010, pp. 3030-3036; Raedt et al., 2002, pp. 1912-1917); only sample 5 lacks that oxide, so, it can be deduced that a more recent recipe was used for its elaboration. From the above, it can be inferred that the source of calcium extraction was different according to the artisan's experience, incorporating a greater or lesser amount.

In relation to magnesium oxide, the values recognized in the fragments were greater than the unity, regardless of whether they were sodium or potassium glass; the same is true in Italian and European glass from the 9th to the 18th centuries (Cagno, Janssens & Mendera, 2008, pp. 1389-1395; Cagno, Mendera, Jeffries & Janssens, 2010, pp. 3030-3036; Carmona, García-Heras, Gil & Villegas, 2005, pp. 251-258; Lima, Medici, Pires de Matos & Verita, 2012, pp. 1238-1248; Marrocchino et al., 2020, pp. 819-827; Müller, Torge & Adam, 1994, pp. 45-48; Schalm, Wouters & Caluwé, 2004, pp. 1647-1656, Schalm Janssens, Wouters & Caluwé, 2007, pp. 663-668). Exclusively, two previous studies of Italian glass from the 6th to the 12th centuries and from the late 13th century report that they do not exceed the unity. On the other hand, the values of Al_2O_3 are remarkably high in soda glass, except in samples 9 and 19, which do not exceed unity in wt.%; 6, 13, and 22 presented it above 10 wt.%; and the remaining in a range of 1.5 to 6 wt.%. Most studies about this oxide from the 8th to 18th centuries show that it has a weight of between 2.0 and 7.0 grams. This is similar to what other studies have found, which usually have values of between 1.50 and 1.50 wt.% (Barrera & Velde, 1989, pp. 48-54; Cagno, Janssens & Mendera, 2008, pp. 1389-1395; Carmona,

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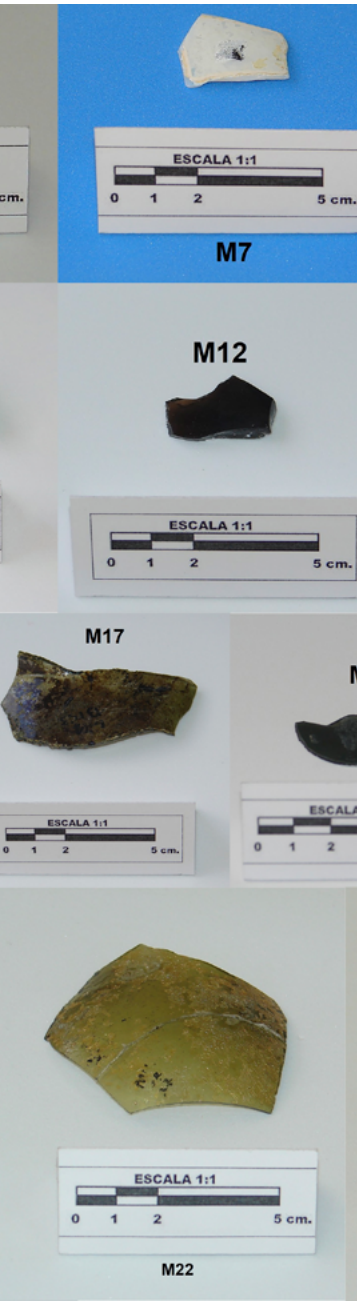

García-Heras, Gil & Villegas, 2005, pp. 251-258; Marrocchino et al., 2020, pp. 819-827; Müller, Torge & Adam, 1994, pp. 45-48; Raedt et al., 2002, pp. 1912-1917; Schalm, Janssens, Wouters & Caluwé, 2007, pp. 663-668). The above could be because the raw material was of different provenance or, as with the Italian glass, was intentionally changed to produce glass of different aesthetic characteristics.

With respect to Fe_2O_3, it was absent in samples 5 and 6, 14 samples were between the range of 0.08 and 1.00 wt.%, 3 between 1.25 and 2.19 wt.%, and one sample with an elevated value of 23.52 wt.%. This last value is partly similar to what has been detected in Italian glass (Cagno, Janssens & Mendera, 2008, pp. 1389-1395; Lima, Medici, Pires de Matos & Verita, 2012, pp. 1238-1248; Marroscchino, 2020, pp. 819-837; Raedt et al., 2002, pp. 1012-1017; Verita, Reiner & Zecchin, 2002, pp. 261-271), dated between the 7th and 17th centuries, and, to a lesser extent, in Belgian, Swiss, and Spanish glass from the 8th to the 18th centuries (Carmona, García-Heras, Gil & Villegas, 2005, pp. 251-258; Schalm, Janssens, Wouters & Caluwé, 2007, pp. 663-668; Wolf et al., 2015, pp. 660-667).

The above indicates that, most likely, the difference lies in the concentration of other oxides that were not identified in the present study. The manganese detected in 13 samples was in the range of 0.01-0.20 wt.%, 4 between 0.21-1.00 wt.%, and it was absent in sample 5. The low range mentioned is similar to that detected in the Italian glass, and higher in the rest of the European countries discussed. Although it is considered that the presence of iron alone gives the glass its green coloration, this is relative since it will be found in different concentrations when reacting with oxygen. Generating other oxides, such as ferrous and ferric, which, depending on the proportion in which they are found, will give the glass a blue-green to green or yellow-green to yellow coloration. Manganese can provide violet coloration depending on other oxides present and not only act as a decolorant (García, Gil, Carmona & Villegas, 2003a, pp. 21-34; Mirti, Davit & Gulmini, 2020, pp. 221-239). Other metals present in the sand can generate diverse tonalities. Therefore, it is necessary to expand the detection of more oxides to correlate them with the color of the glass or, otherwise, with the action of other metals with decolorizing functions.

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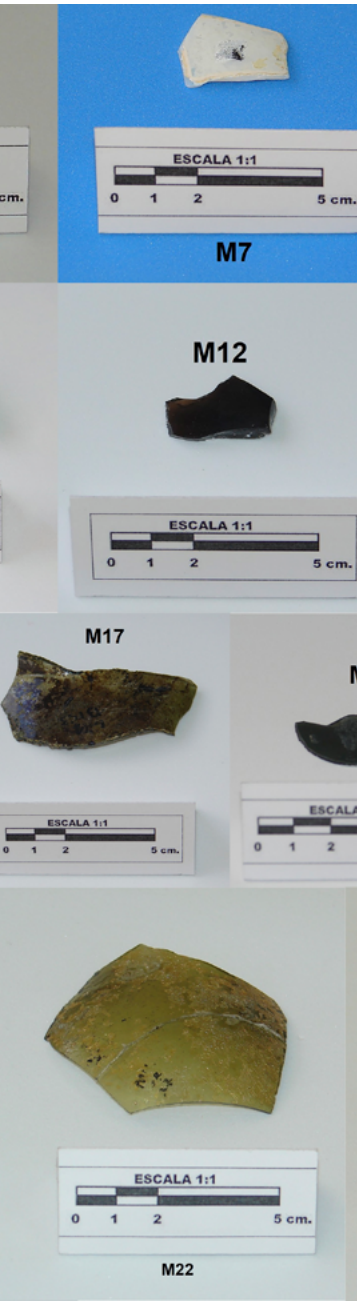

B) Microscopic physical deterioration

Glass is apparently considered to be resistant to chemical agents; however, when subjected to environmental changes, such as humidity, temperature, pH, and microorganisms, its integrity is affected. Prolonged exposure to humidity causes the formation of a silica gel layer or film on its surface. The environment and the temperature generate hydronium ions (H_3O^+), and along with them, an acid medium that interacts with the alkaline ions (Na and K) of the glass matrix. The removal of the latter (de-alkalinization) determines the concentration of OH^- ions, which increases the pH and breaks the bond between silica and oxygen that forms the molecular lattice, or the cause of glass degradation. However, continued deterioration through an influx of heavy metals from the subsurface has an effect on the physical appearance (García Rincón, Jimeno & Villegas, 2003b, pp. 173-81; García, Gil, Carmona & Villegas, 2003a, pp. 21-34; Silvestri, Molin & Salviulo, 2005, pp. 1338-1349). Previous observations have revealed notable morphological modifications attributed to microorganisms that exhibit affinity and adhesion to the glassy surface. This results in the formation of a biofilm comprising single cells, hyphae, and filaments, a polymeric extracellular material that, in conjunction with the slime, retains moisture and favors the release of metals. Consequently, the chemical degradation induced by acid and chelate production alters the pH, thereby favoring the persistence of microorganisms and the establishment of others (Rölleke et al., 1999, pp. 107-114; Stockmann et al., 2012, 1-18).

With the naked eye, there were detected yellowish and bluish tonalities on the surface of the samples analyzed in this study, as well as a detachment of thin fractions in the form of flakes, light-brown adherence, and scratches. When small fractions were observed under the scanning electron microscope, manifestations in the relief of the surface were detected. Samples 5 and 6 presented irregular surfaces with an angulation pattern (Figure 3) and curvilinear shapes in the manner of puzzle pieces, an aspect that was not observed in the rest of the fragments. It is probable that the prominent variations in the chemical constituents detected in these samples have an impact on the molecular organization and, consequently, on the microscopic physical structure, as evidenced by the high percentage of SiO_2 in sample 5 and the *hyalith* glass fragment in sample 6.

A portion of sample 7 classified as opal glass presented, due to its macroscopic characteristics, a rough surface very similar to a sample previously studied (García, Rincón, Jimeno & Villegas,

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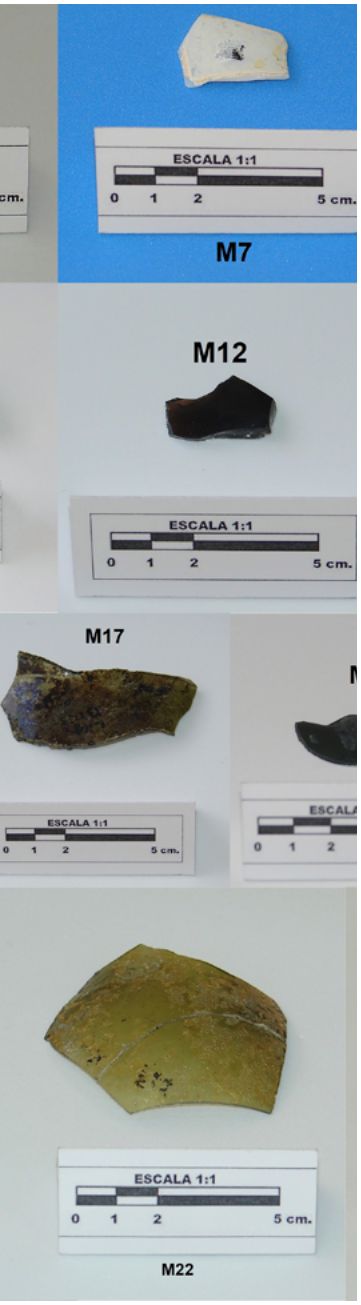

2003b, pp. 173-181). Its authors attribute this characteristic to the high content of SiO_2 and CaO, which coincides to some extent with the values of SiO_2 in the samples analyzed in the present investigation, but not with those of CaO, which are much lower.

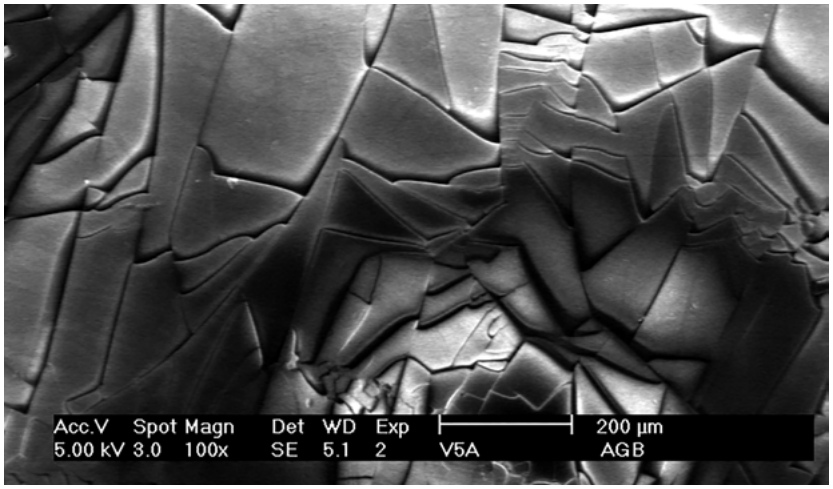

FIGURE 3. Photomicrograph of sample 5 at 100X, corresponding to the base portion of a dark gray container with a decanter. Its high SiO_2 content gives it the molecular arrangement shown in this image, which does not resemble the rest of the samples analyzed (Micrographs: Sc. D. Arturo García Bórquez, 2020; courtesy: Escuela Superior de Física, IPN, Mexico).

The rest of the samples, regardless of whether they are soda or calcium glass, show modifications in the relief of the surfaces caused by small punctiform excavations, some with regular edges as in sample 13 (Figure 4A) and others with irregular edges interposed between tortuous grooves, as in samples 10 and 19 (Figure 4B). Previous works have reported that soda glass is more resistant to the environment than potassium glass (Bettembourg, 1976, pp. 36-42); however, this was not the case in the present study, which could be due to the characteristics of the medium and the time of exposure to it.

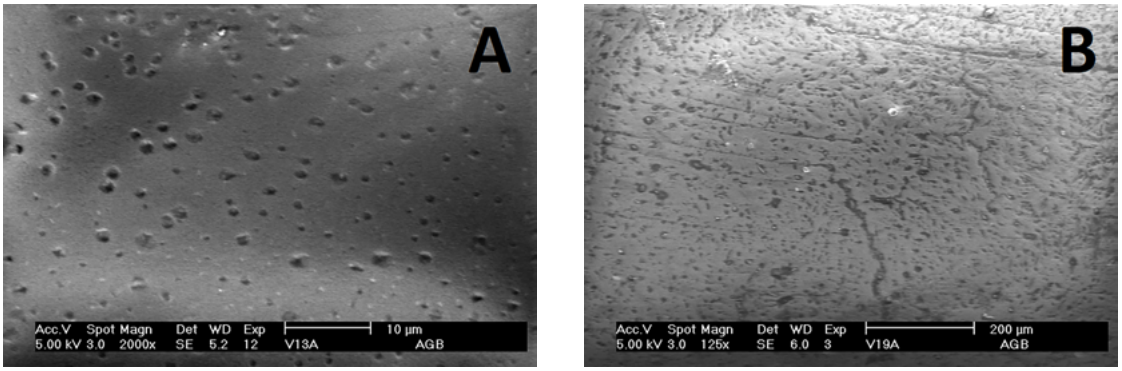

FIGURE 4. Photomicrograph A, of sample 13 at 2000X, of a small portion of the neck of a bottle with low NaO_2 content exhibits surface deterioration that differs from that of image B, which is of sample 19, at 125X, of the neck and shoulder fraction of a container with much higher content of the referenced oxide. The difference in deterioration is probably dissimilar due to the time of exposure to moisture and subsoil components (Micrographs: Sc. D. Arturo García Bórquez, 2020; courtesy: Escuela Superior de Física, IPN, Mexico).

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Sample 11 showed areas with wide excavations of uniformly curved edges, which, presumably, originated from air bubbles that were the result of inadequately mixing the raw material, or of insufficient temperature for the melting of the glassy material.

In samples 4 and 20 (Figures 5A and 5B respectively), excavations occupied by polyhedral structures can be seen, which may be inorganic crystals of calcium, oxalates, oxides, sulphates or other components formed by the interaction of the environment. Likewise, on the smooth surfaces of samples 6 and 10, adhesion in the form of a bar is observed, and on sample 14, a spherical structure. In samples 10, 16, 17, and 18 (Figure 6) the adhesions are of irregular shape, so, it is assumed, they are likely of biological origin. Previous research on this matter does not report adhesions of this type; however, the photographic records do not present similarities to what was observed in the present investigation, so it is possible that the variations, result from the presence of different bacteria or other microorganisms that should be cultivated in similar conditions for their identification. The fragment of sample 9 presents lumpy and filamentous adherence on its surface, which could be the manifestation of a fungal colony (Figure 7), similar to that reported in previous research (Andrejevna & Aldona, 1999, pp. 181-191).

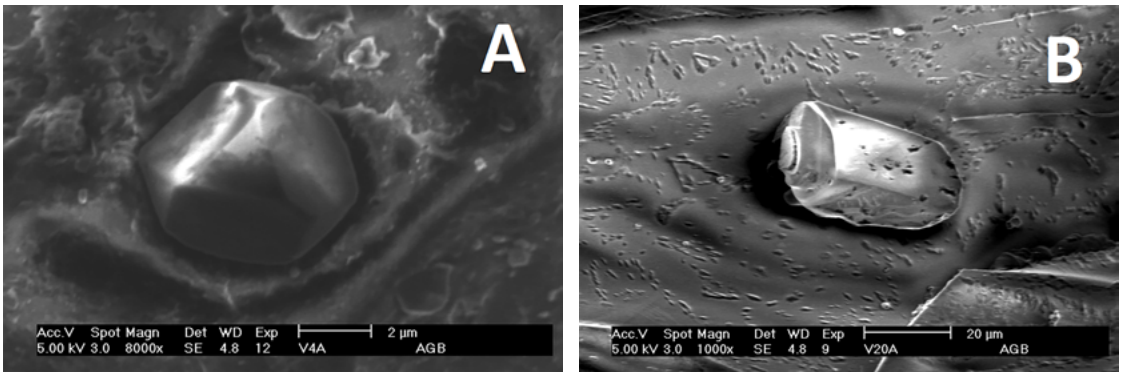
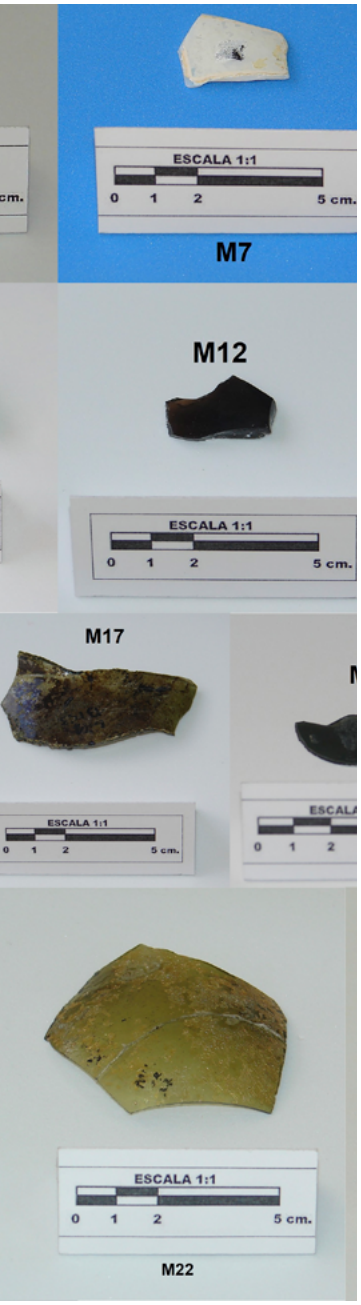


FIGURE 5. Photomicrograph A is of sample 4 at 8000X, a portion of a liquorice vat of hemispherical shape with ample deterioration of its surface; it has a reduced content of SiO_2 and a higher content of Fe_2O_3 than sample 20, whose image B is at 500X and belongs to the liquor fraction. The incrustations on its surfaces are polyhedral (Micrographs: Sc. D. Arturo García Bórquez, 2020; courtesy: Escuela Superior de Física, IPN, Mexico, 2024).



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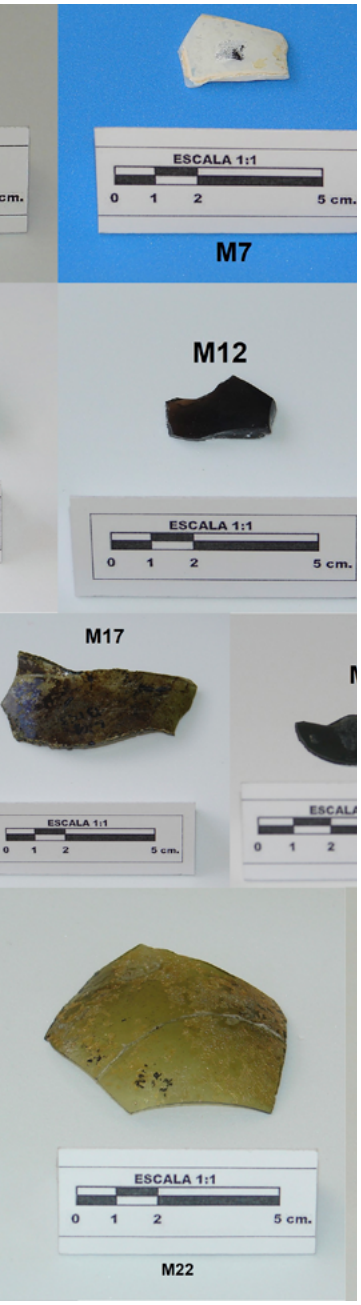


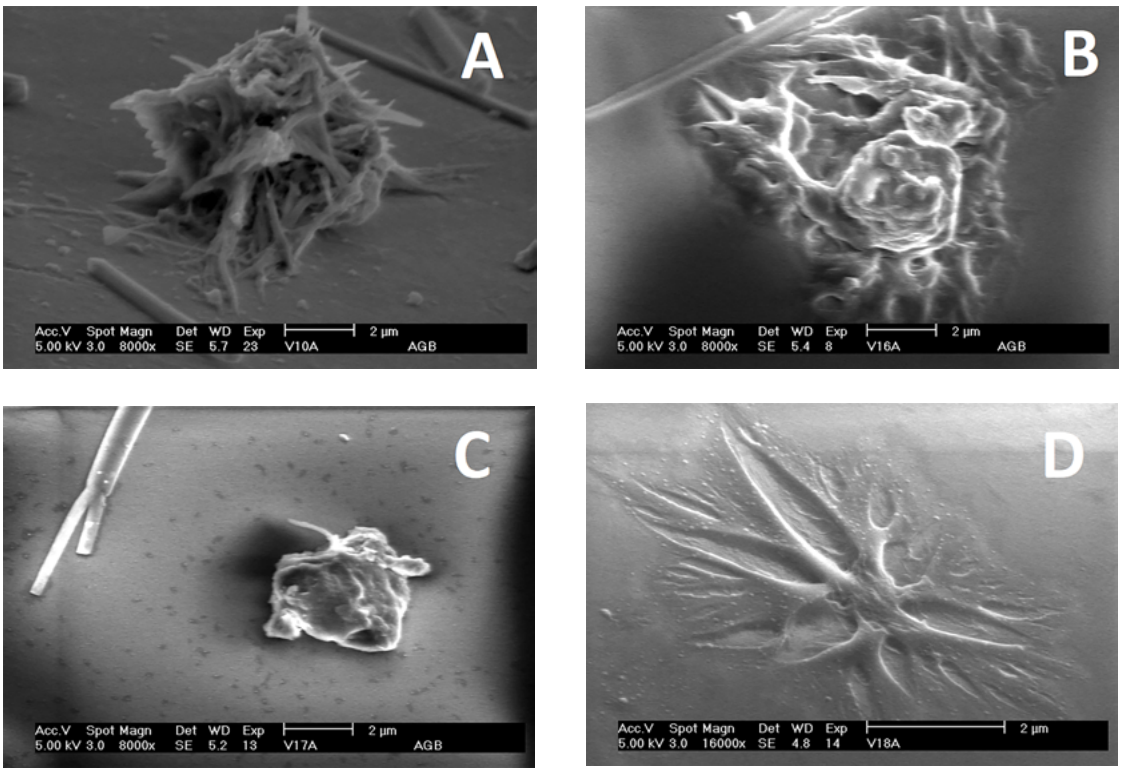
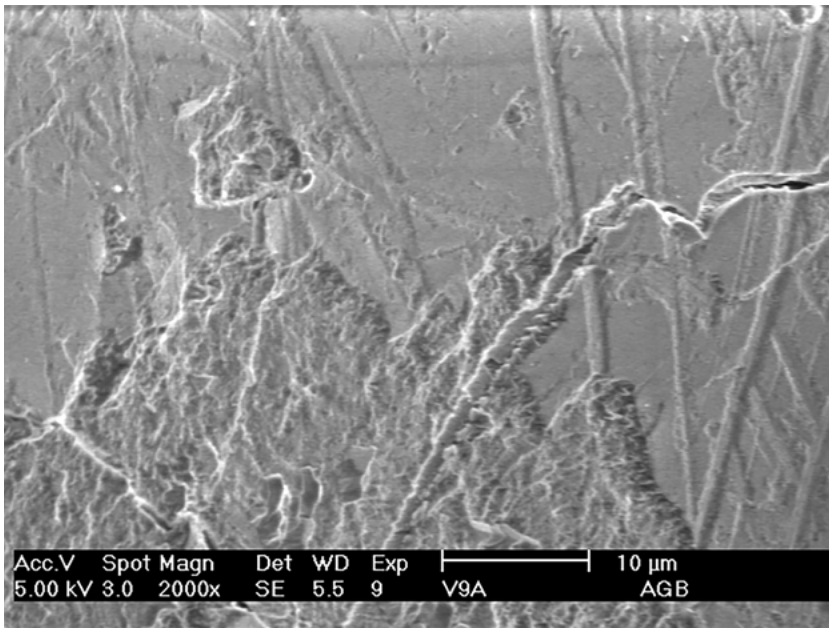


FIGURE 6. Micrographs A, B, C, and D, respectively, of samples 10 (bottom of liquor box), 16 (portion of knitting needle), 17 (fragment of container), and 18 (fraction of lens). They exhibit surface adhesions of particular morphology that could be of biological type; the first three images are 8000X and the last one is 16000X (Micrographs: Sc. D. Arturo García Bórquez, 2020; courtesy: Escuela Superior de Física, IPN, Mexico, 2024).

FIGURE 7. Photomicrograph of sample 9 at 2000X, apparently part of the base of a cup and with high and K_2O content, showing hyphal growth (Micrographs: Sc. D. Arturo García Bórquez, 2020; courtesy: Escuela Superior de Física del IPN, Mexico, 2024).



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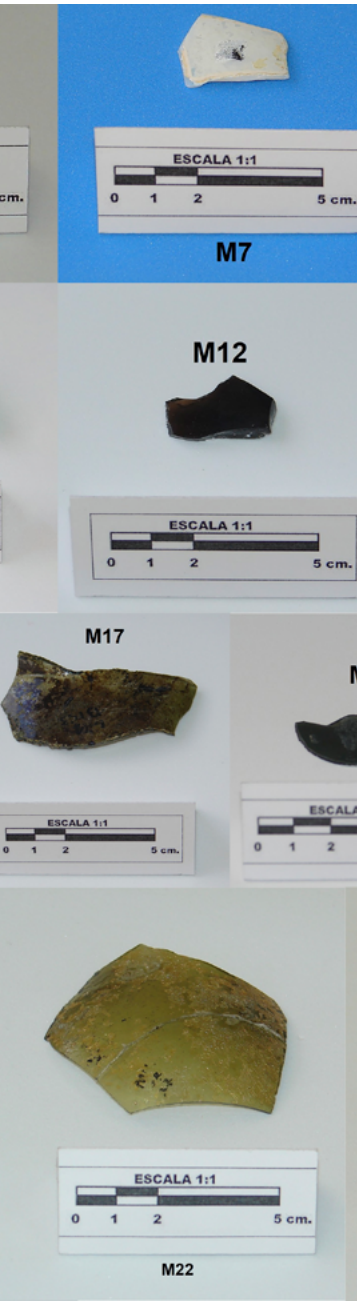

A particular characteristic was observed in samples 9, 19, 22, and 23: this was the straight laminar organization, while in samples 8, 18, and 20 they were concentric with a wide radius (Figures 8A and 8B). In the work of Cox and Ford (1993, pp. 5637-5647) this characteristic was reported in potassic glass, but was concentric with a short radius, so it is proposed that it is a consequence of moisture erosion in punctual zones by sodium, potassium, and magnesium leaching. However, the variants observed in this research could correspond to the area of the fragment of the piece of glass; that is, if the laminar organization is straight, it could be a piece of the wall of a bottle, and if the distribution is concentric, it could be a piece belonging to the area of the shoulder or lip of a bottle.

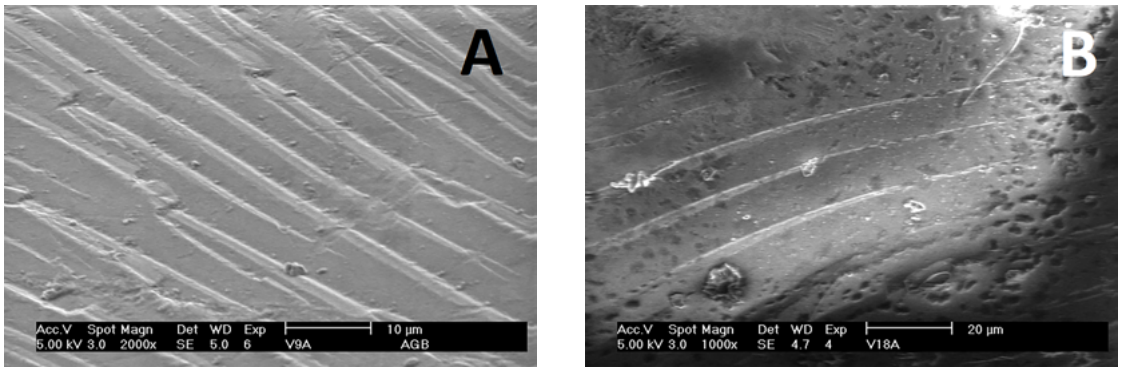

FIGURE 8. Micrographs of samples 8:A, which corresponds to the bottom of a circular decanter, and 18:B, which is the base of a cup, show that the first is sodic and the second is potassic with high SiO_2 content. In both images at 1000X, the molecular arrangement is evident. In image A, the straight arrangement is observed in the center and without cleavage, and in image B, the convex linear arrangement is the probable result of the shape of the zones of each one of the pieces (Micrographs: Sc. D. Arturo García Bórquez, 2020; courtesy: Escuela Superior de Física del IPN, Mexico, 2024).

C) Description of fragments and color

The determination of the tonality of the samples was considered as a macroscopic particularity of reference, and not with the purpose of determining the oxide or oxides that generate it.

Single color samples

Fragment 7, for its milky and opaque hue, was considered as *opaline*; its slight convexity suggests that it corresponds to a vessel. This type of glass was produced in France between 1810 and 1890, although since the 19th century it has been manufactured in Venice

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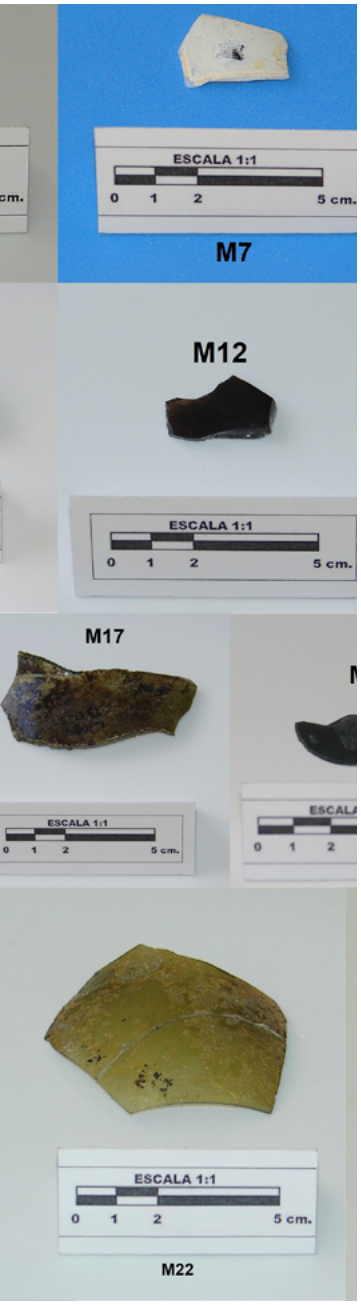

(Corning Museum of Glass, 2023). Sample 6 was catalogued as *red hyalith*; a variant of opaque glass produced in Bohemia between 1830 and 1840 (Stanislas, n. d.); a portion that has smooth faces and some curvature, so it could correspond to a fragment of glass or container. The fragments mentioned were probably from pieces produced abroad and not nationally.

Sample 8, with a *honey-yellow* tone, corresponds to the bottom of a circular liquor jar, with deterioration and several light brown adherences.

Sample 16, amber in color, is a fragment of a knitting needle. Its thickness and straight shape are similar to the complete blue specimen previously found in a coffin of a nun from the convent of Santa Teresa la Antigua from the late 18th or early 19th century (Fernández, 1990, p. 297); coincidentally, the fragment analyzed here was found on the street where this convent was located. This type of pieces are small in number: so far, we have identified other similar blue specimens of flattened width and varied lengths, which, along with other pieces of glass, make up a necklace ornament on display at the Museo del Vidrio (Museum of glass) in Monterrey, Nuevo Leon, Mexico.

Multiple staining samples

The samples of grey tonality, such as number 5, of dark color and corresponding to the incomplete bottom of a small circular bottle with a conical decanter ("*kick up*" or "*push up*") without the mark of the tip, which implies the use of a mold for its manufacture; this type of particularity was introduced at the beginning of the 19th century (Dungworth, 2012, p. 38). Sample 12, of "smoke" tonality, is the fragment of a bottle body, which presents a thin portion, corresponding to the body wall, and the thicker one, to the edge of its base.

The black-colored samples are: 10, 13, and 14. The first one is the fraction of the bottom of a liquor tank; 13 corresponds to a small portion of a necklace and, in its upper part, to a thin lip; and 14, classified as glass slag, is a vitreous mass of rocky appearance and rough surface with small punctual zones of red color, which in the microphotography are perceived as adhesions of spherical form.

Three catalogued fragments of blue color and of different tonalities are: 15, which has a flat bottom, perhaps from an ultramarine blue decanter; 19, corresponding to the upper portion of an aquamarine blue bottle, with a short neck, non-uniform lip, and a portion

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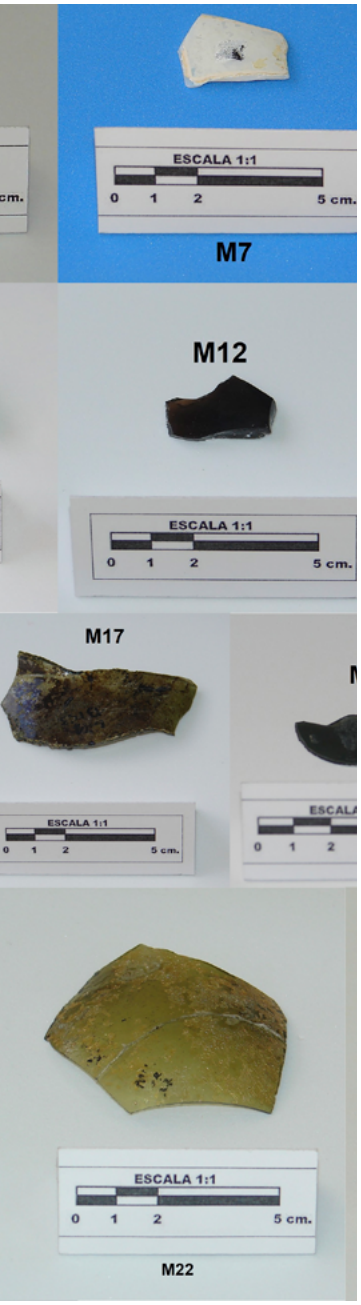

of the flat shoulder in decline. In this one, elongated bubbles are observed with the naked eye, caused by the incomplete fusion of the vitreous material. The remaining fragment, 20, is light blue and opaque, rectilinear in shape, and with one of its edges of some concavity that suggests that it corresponds to the quadrangular bottom of a container. The opposite side to this edge would be part of the body of a liquor container and shows flaking and slight iridescence as well as small bubbles; under the electron microscope, adherences in the form of small lumps are observed.

The most numerous group concerns the green color in different tonalities; the samples were: 4, 9, 11, 17, 18, 21, 22, and 23. Number 4 is a strong green and corresponds to a hemispherical portion of the shoulder of a liquor jar with severe deterioration on its surface and detachment of small slabs. Sample 9, is light green, irregularly shaped, and decorated with three white lines of intermediate thickness, which seems to be due to its moderate concavity, the base of a cup. Sample 11, aquamarine green, is a fragment of the bottom of a decanter. Sample 17, olive green in color and irregular in shape, suggests that it is a fraction of a liquor jar shoulder, whose surface is rough with adherences and iridescence in shades of blue. Sample 18, of dark green color, is of oval shape, barely visible convexity and thin thickness, with deteriorated surfaces: due to these characteristics, it would correspond to a piece of lens. Sample 21, deep green, is a wide fragment of a bottle with a non-uniform lip and short neck, so it was part of a bottle containing some laboratory material, whose inner side is rough and with slight iridescence. Sample 22, yellowish olive green, is supposed to be, due to its convexity,a bottle fragment: its external surface shows iridescence and shapeless adherences. Sample 23, opaque green in color, which, because of its convexity, is presumed to be a bottle fragment, has small perforations and numerous bubbles.

In relation to the color classification of the fragments, it is necessary to rely on a standardized procedure, such as spectrophotometry, or catalogues such as Gaffer® Casting Color or SPEC-TRUE. With respect to glass fragments, the characteristics of some of them do not permit us to specify the type of vessel to which they belong; therefore, as a single piece, it is necessary to reconsider their storage, unless they were found with other objects that are part of a whole context.

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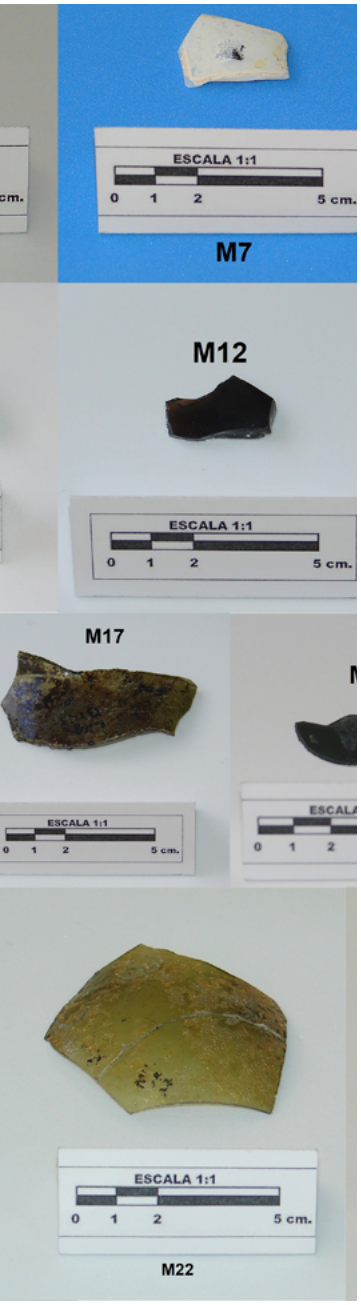

CONCLUSIONS

The determination of the temporality of glass can be partially imprecise because, in general terms, the recipes or formulas for making the glass have a period of conservation of several years, due to the interest and efforts to preserve the production substrate. In addition to this, the raw material sources differ enormously, depending on the concentration values of oxides identified and compared in the present work, which are only partially coincident. On the other hand, Glaziers also have the flexibility of adjusting the proportion of basic components or introducing additional elements to achieve the desired qualities in their pieces. The diversification of raw materials and their utilization notably began in the latter part of the 16th century, with significant challenges emerging in incorporating new chemical elements thereafter. For instance, while seventeen analyzed fragments date back to the 18th century or earlier, three are from later periods.

From this initial study, we highlight the importance of expanding the analysis of oxides of pieces with known temporality and national origins to establish a reliable reference for accurately distinguishing production periods. It's crucial to include more oxides responsible for specific colors to objectively classify the tonality of glass pieces.

The analysis of the microscopic characteristics of glass samples is essential for understanding the physical composition of the molecular arrangement formed by fluxes, stabilizers, and vitrifying agents. It also provides insight into structural and surface modifications observable at a macroscopic level. Additionally, it aids in understanding the mechanism behind the adherence of biological material to glass through experimental models, facilitating the study of glass material degradation due to environmental interactions.

Considering the macroscopic characteristics of disjointed glass pieces alongside microscopic and chemical analyses is crucial, particularly for fragments representing significant portions of glass pieces. However, smaller fragments also contribute valuable information, primarily through a particular color, engravings, or surface decorations, warranting their safe extraction for their analysis.

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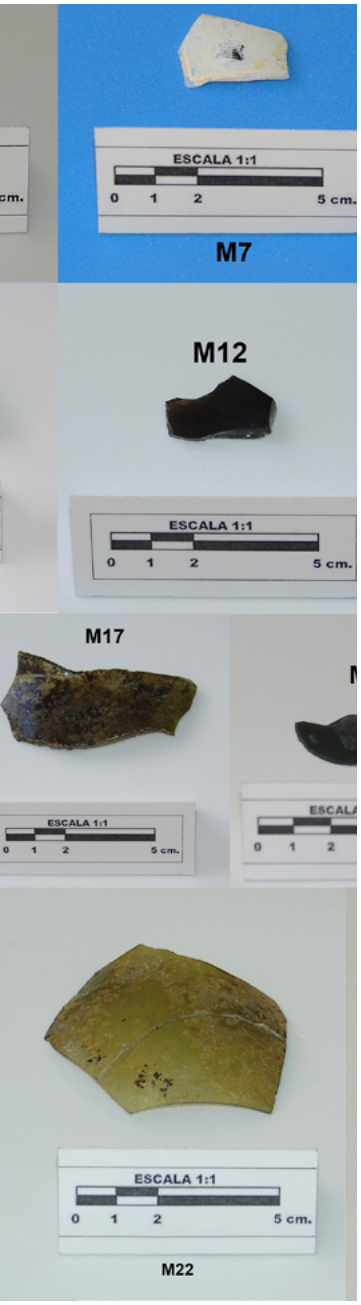

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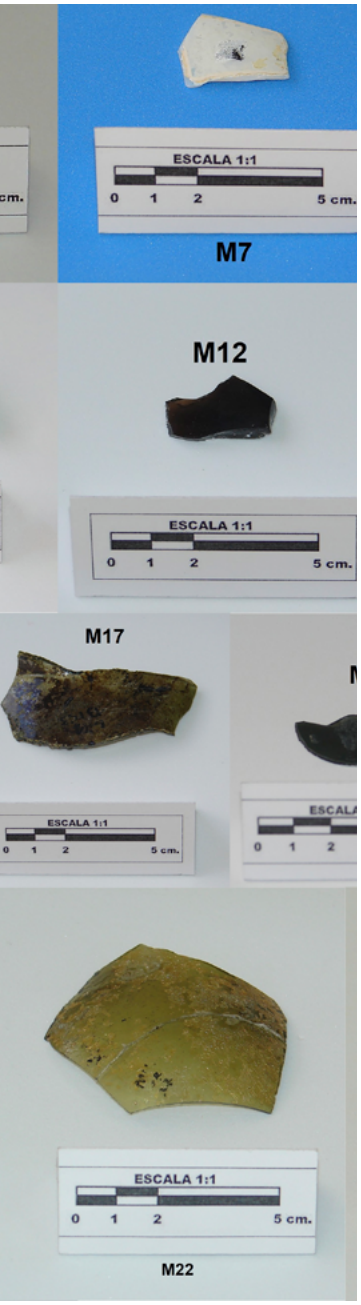

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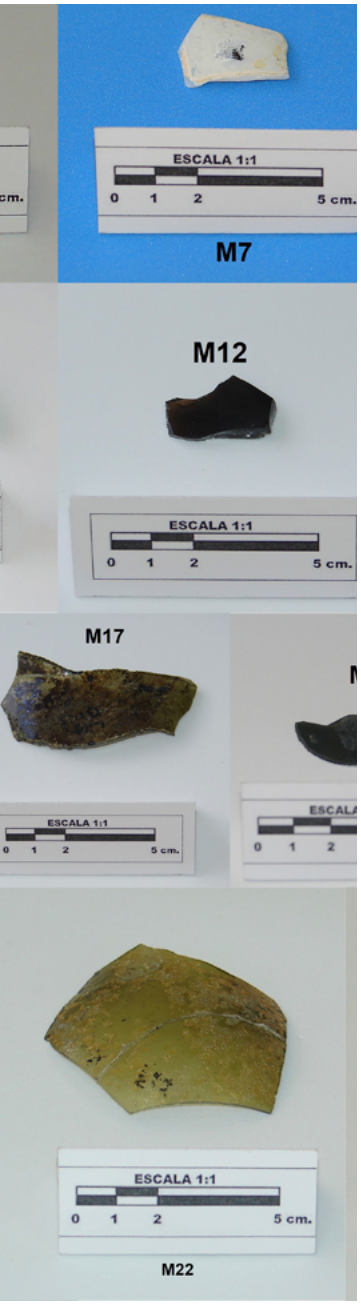

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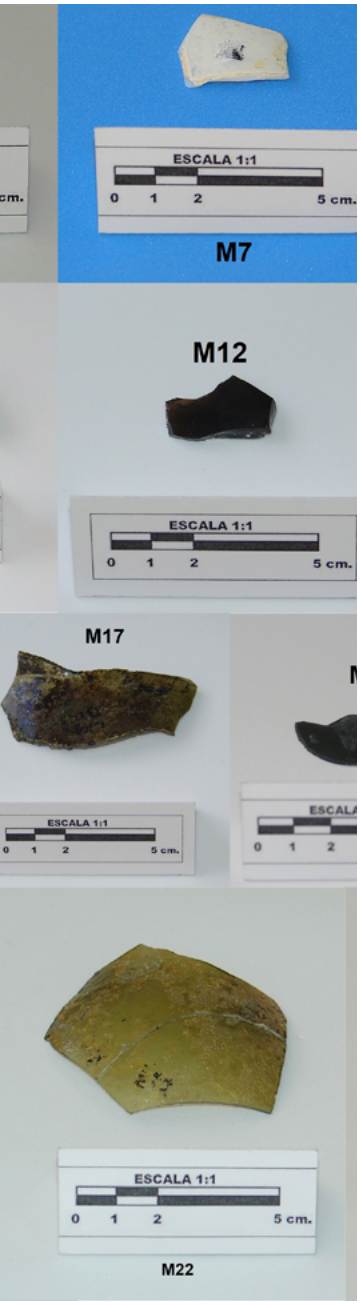

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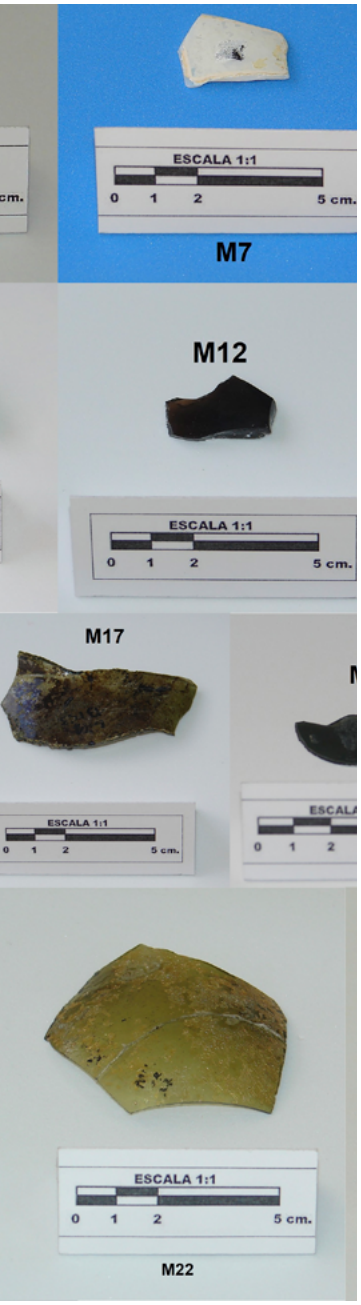

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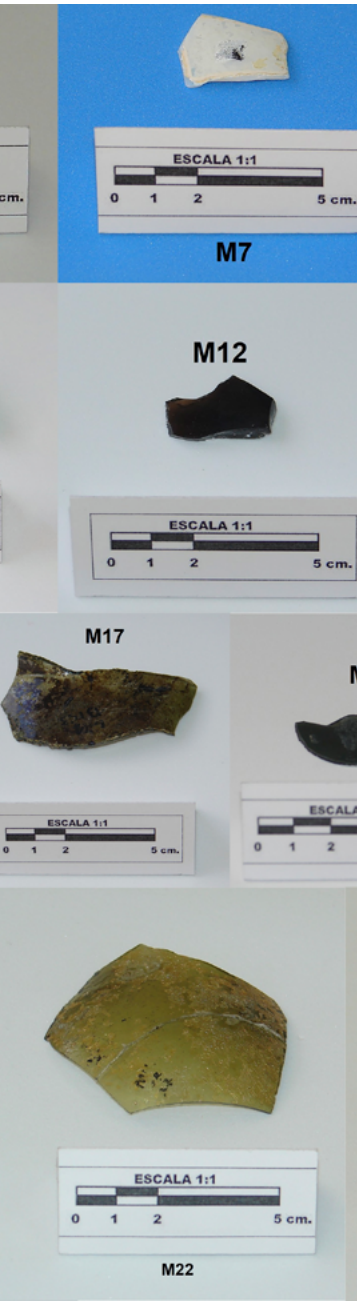

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