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Glass from the 11th–13th century medieval castle of San Giuliano (Lazio Province, Central Italy)

Colleen Zori[a,*], James Fulton[b], Peter Tropper[c], Davide Zori[d]

[a] Baylor Interdisciplinary Core and Department of Anthropology, Baylor University, 1 Bear Place #97350, Morrison Hall 231, Waco, TX 76798-7350, United States
[b] Department of Geosciences, Baylor University, 1 Bear Place #97354, Waco, TX 76798-7354, United States
[c] Institute of Mineralogy and Petrography, Universität Innsbruck. Innrain 52f, 6020 Innsbruck, Austria
[d] Baylor Interdisciplinary Core and Department of History, Baylor University, 1 Bear Place #97350, Morrison Hall 231, Waco, TX 76798-7350, United States

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ABSTRACT

Compositional analysis of glass from the medieval castle of San Giuliano (Lazio, Italy), occupied from approximately CE 1050–1250, sheds light on the financial wherewithal and integration of the castle's elite inhabitants into wider economic networks. Portable X-ray fluorescence (pXRF) of 261 shards was used to select 32 for further analysis using wavelength dispersive electron microprobe analysis (EMPA-WDS). This compositional analysis documents the pervasive recycling of earlier glass cullet, some of which pre-dates the 4th century CE. Around 20% of the sample comprised primarily plant ash glass, evidencing the penetration of plant-ash glasses into inland sites on the western central Italian peninsula. Almost 60% of the shards were an intermediate glass combining more recent plant ash glasses with recycled natron-based glass cullet derived from the Roman and Early Medieval periods. Compared to glass assemblages from contemporaneous sites, the levels of both recycled and intermediate glasses are quite high, with a concurrent incidence of trace elements that further precluded the manufacture of perfectly translucent glass vessels. This suggests that while the residents of the castle desired glass as a symbol of prestige, they may not have had the economic resources to obtain glass of the highest quality.

1. Introduction

Glass in Italy underwent significant transformation from the Early Middle Ages (400–1000) to High Middle Ages (1000–1300), both in raw material usage and the spatial organization of production. In Roman times, raw glass fluxed with natron and decolorized using antimony was primarily produced in the eastern Mediterranean and imported by secondary production centers on the Italian peninsula (Gorin-Rosen 2000; Nenna 2014; Tal et al. 2004). There, craftspeople shaped the glass into vessels and other items, sometimes adding colorants and otherwise modifying the chemical composition of the glasses. Around the mid-4th century CE and for reasons that remain unclear, antimony was replaced by manganese, among a few other options, as a decolorant (Jackson 2005; Paynter and Jackson 2016). The use of natron as a flux then began to decline in the 7th-8th centuries AD, due to a combination of environmental changes and political instability in Abbasid-controlled Egypt, the only documented source of the evaporitic alkali natron (Shortland et al. 2006; Whitehouse 2002). By the end of the 9th century, natron had been largely replaced by sodium-rich ashes of halophytic plants found in the eastern Mediterranean (Henderson 2013). Documentary sources and archaeological data indicate that after several centuries of importing raw plant-ash glass, production centers in Italy—first in Veneto and later in Tuscany, Liguria, and elsewhere—began to manufacture their own glass, initially importing plant ash and high-silica beach sand from the east (mid-13th century) and later developing local and/or western Mediterranean sources for both silica and plant ash (mid- to late-13th century and after; Cagno et al. 2008, 2010, 2012; Fenzi et al. 2013; Occari et al. 2021; Verità 2013; Verità et al. 2002; Verità and Zecchin 2007).

The transition between natron and plant ash fluxed glass was gradual on the Italian peninsula. Samples from Venice indicate the presence of only natron glass in the 5th-7th centuries, while the assemblages from the 8th-11th centuries and the 10th-12th centuries show plant ash glasses comprising around 15 % and then 45 % of the assemblages, respectively (Verità 2013: 520). By the 13th-14th centuries, the glass assemblage was over 90 % plant ash glass. At the same time,

* Corresponding author.
E-mail addresses: Colleen_Zori@baylor.edu (C. Zori), James_Fulton@baylor.edu (J. Fulton), peter.tropper@uibk.ac.at (P. Tropper), Davide_Zori@baylor.edu (D. Zori).

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glassworkers also recycled natron glass, mixing it sometimes with natron glass cullet or combining it with new plant ash glass. In the Venetian samples, recycled natron glass increased slightly over time, from 21 % of the 5th-7th century assemblage, to 24 % and 27 % of the 8th-11th and 10-12th century assemblages, respectively (Verità 2013: 520). In the 10th-12th centuries, the Venetian assemblage also included about 4 % "intermediate" glasses, created when natron and plant ash glasses were melted together (Verità 2013). While recycling glass likely reflects a downturn in *trans*-Mediterranean trade in Late Antiquity and the Early Middle Ages (Mirti et al., 2001; Schibille and Freestone 2013), it is nevertheless notable that plant ash glass was already arriving at sites like Venice, Nogara, Grado, Comacchio, and Bari by the 8th-9th centuries (Bertini et al. 2020; Neri et al. 2019; Silvestre and Marcante, 2011; Silvestri et al. 2005). This testifies to continued trade links between the Italian peninsula and the eastern Mediterranean, as well as a growing demand for prestigious glassware as economic conditions improved at the end of the first millennium. Analyses of glass from sites spanning the end of the early Middle Ages and first part of the High Middle Ages thus stand to make important contributions to understanding the nature of these technological transformations and how widely the products penetrated the economic landscape of medieval Italy.

The present study examines the assemblage of glass fragments from the site of San Giuliano, located in Lazio province about 75 km northwest of Rome. Excavations conducted 2016–2019 focused on a medieval hall that was part of a fortified castle complex comprising a curtain wall, dry moat, collapsed tower, and a mortuary structure associated with a probable chapel (Fig. 1; Zori et al. 2017, Zori et al., 2018). The glass was recovered from secure habitation surfaces or trash deposits of the hall, contexts yielding calibrated radiocarbon dates spanning approximately CE 1050–1250 (Fig. 2).

Chemical analyses of a subsample of 32 shards reveal that while much of the glass was fluxed with plant ash, recycled natron glass cullet had been introduced into many of the glass batches, producing glass of intermediate chemical composition. While this has been observed at contemporaneous Italian sites, the continued incorporation of recycled natron glass and the high percentage of intermediate glass in this assemblage suggest that residents of San Giuliano did not have access to glassware made from pure and unrecycled glass. Even though glass clarity and quality may have been compromised by the prevalence of recycling and mixing of different glass types, glassware was nonetheless integral to demonstrating prestige for San Giuliano elites aspiring to greater social stature.

2. Materials: The glass sample from San Giuliano

Excavation of approximately 186 m^3, comprising the interior of the medieval hall and its immediate environs, yielded 386 glass shards. The majority are transparent glass ranging from clear to weakly colored

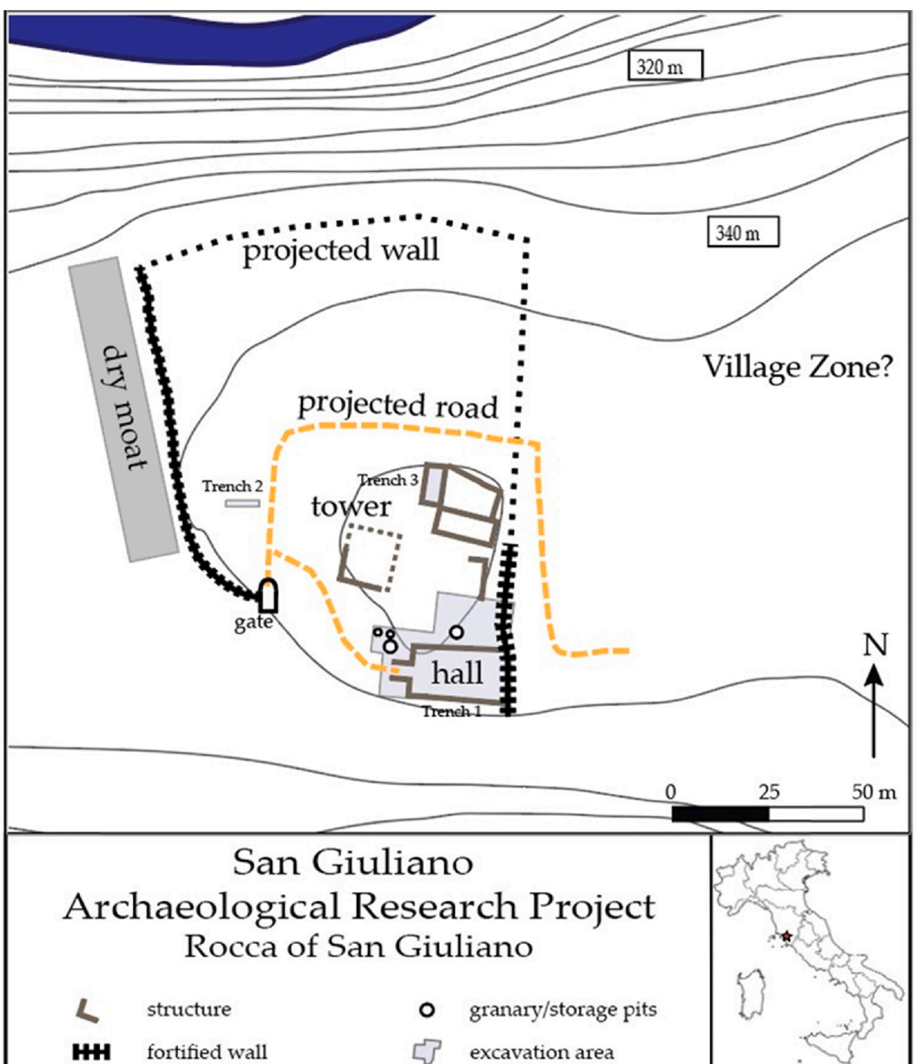
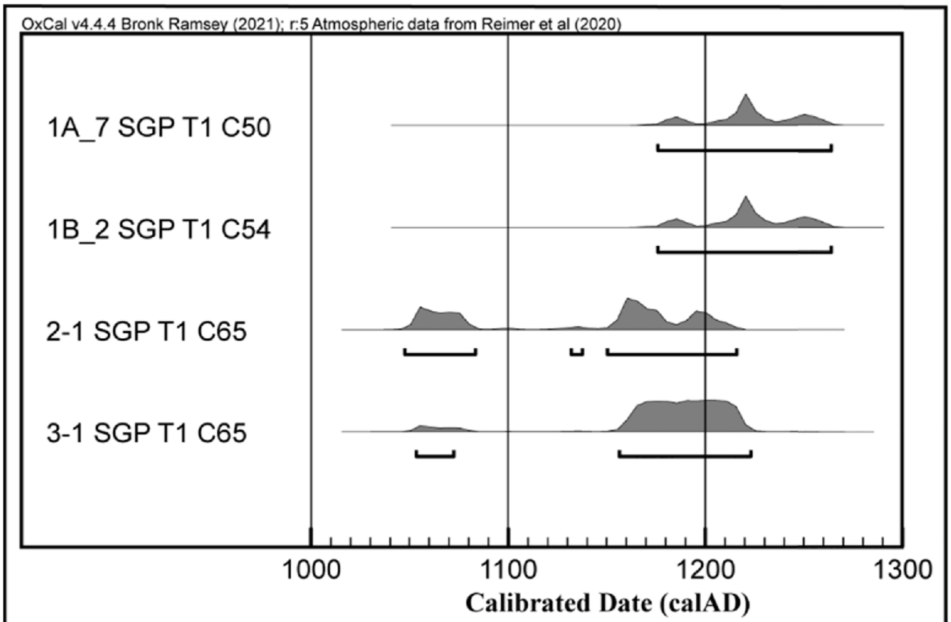


Fig. 1. The fortifications and structures of La Rocca, showing the area excavated.



Sample ID	14C Age	Calibrated date
1A_7 SGP-T1-C50	835+/-15	1176-1264 (95.4%)
1B_2 SGP-T1-C54	835+/-15	1176-1264 (95.4%)
2-1 SGP-T1-C65	900+/-15	1047-1083 (33.0%) 1132-1137 (1%) 1150-1216 (61.5%)
3-1 SGP-T1-C65	875+/-20	1053-1072 (3.6%) 1165-1223 (91.8%)

Fig. 2. Radiocarbon dates from San Giuliano, deriving from a former granary used as a trash pit (C50 and C54) and the occupation surface of the hall (C65). Calibration of the radiocarbon dates and figure by B. Damiata.

(light green, yellow, or pink). Fragments of dark olive green, amber, and other colors are also present in smaller quantities, as well as opaque glass in purple, red, and brown. Colored decorative elements include blue rims or diffuse blue swirls, yellow trails, and fragments decorated with preformed cannulas made with the *reticella* technique, documented at sites dating to as early as the late 6th century but becoming more common in the 9th-12th centuries (see e.g. Schibille and Freestone 2013; Silvestre and Marcante, 2011; Uboldi and Verità 2003).

Visual inspection and comparison of shards recovered from the 2016–2019 seasons have provided an estimate of approximately 75 distinct vessels represented in the assemblage. Apart from one mold-blown ring base, it appears the glass found at San Giuliano was free-blown. Many of the shards derive from drinkware such as beakers and goblets, but also bottles and at least one lamp. At least 35 of the vessels were prunted beakers typical of contemporaneous medieval sites (Fig. 3): the prunts are arranged in staggered horizontal rows, usually below a horizontal trail that separates the body from the flaring rim, itself typically reworked, fire-rounded, and averaging 6–7 cm in diameter (see Newby 1999). As is characteristic of the type, the prunted and plain beakers of San Giuliano have ring bases and often a high, concave kick.

2.1. Evidence for glass-working

No unequivocal evidence of glassworking, such as furnace settings, wasters, crucibles, or glassworking tools have been encountered at San Giuliano. Excavations, however, have yielded a small quantity of indirect evidence suggesting the possibility of local glassworking (compare with e.g. Villa Magna [Lepri 2016:328]). Evidence for attempts at glassworking include three opaque heterogeneous vitreous masses, irregularly shaped and with a granular, glassy appearance (Fig. 4a; see Fenzi et al. 2013: 481-2 and their figure 6.1.4.b). Other debris, including a rounded drop of blue glass and a tear-drop shaped dripping with an irregular, bubbly surface, may be the result of glassworking efforts (Fig. 4b and c; see Fenzi et al 2013, their Fig. 6.1.4c and 6.1.9a). Chemical analysis of these materials is forthcoming.

3. Methods

Of the 386 glass shards recovered from excavations in and around the medieval hall of San Giuliano, 261 were analyzed in the field using a Bruker Tracer 5i energy-dispersive portable X-ray fluorescence spectrometer (pXRF). pXRF is suitable for field analysis because it can be used to quickly and non-destructively analyze a large number of samples and tests a comparatively broad analytical area (8 mm spot size). In our analysis, we used a dual beam method with a duration of 60 s (15 kV, 55μA, no filter) for major element analysis and 30 s (40 kV, 14μA, no filter) for trace elements. The Bruker Mudrock Dual Calibration was applied to the data, and reference samples of tin bronze, cupronickel, 14 K gold, and silver using the Precious Metal 2 Calibration were analyzed at the beginning and end of each day to ensure the stability of the

Fig. 3. Base of a prunted beaker from San Giuliano. Photo credit: Marco Cesare (ArchaeoMatica).

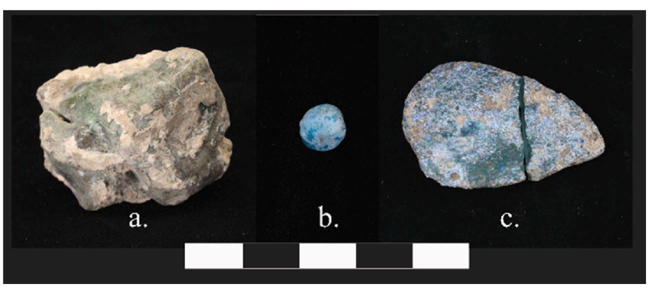


Fig. 4. Indirect evidence of glass-working from San Giuliano, left to right: a.) opaque heterogeneous vitreous mass; b.) drop of blue glass; and c.) dripping of aqua glass with bubbly surface and earthen encrustations. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

instrument calibration during fieldwork (Supplemental Figure 1). All data are reported as oxide weight percent of the bulk material. Note that Mg is the lowest mass element that can be detected using the pXRF instrument with an air atmosphere, and concentrations for elements with lower atomic masses (Mg, Al, Si, P, and S) have greater uncertainly. The pXRF analysis is most useful for analyzing minor and trace element concentrations, and the focus for this part of the analysis was on identifying a representative sample, as well as outliers, for EPMA analysis. We report data here for TiO_2, MnO, Fe_2O_3, CoO, NiO, CuO, ZnO, As_2O_3, Rb_2O, SrO, ZrO_2, and PbO (see Supplemental Figures 2–4 and Supplemental Data 1).

Data from the initial pXRF screening was used to select a subsample of 32 glass fragments that were then subjected to further analysis using an electron probe microanalyzer (EPMA; see Table 1 for sample information; Table 2 for averaged wt.% results; Table 3 for averages, SD, maximum and minimum measures of the glass groupings; Supplemental Figure 5 for image of the shards and Supplemental Data 2 for unaveraged EPMA data). EPMA is a good complement for pXRF analysis because it provides accurate quantifications of both light and heavy elements, although detection limits for trace elements can be problematic. The San Giuliano samples were selected for both archaeological context and glass color. Shards included in the subsample were recovered from the occupation surface inside or outside the hall or from

Table 1
Color and vessel type, if known, of the samples considered in the EPMA analysis.

Sample #	Context	Color	Vessel Type
1	SGP-T1-C9	Light olive green	Beaker (prunt)
2	SGP-T1-C13	Weakly colored green	Beaker
3	SGP-T1-C13	Weakly colored green	Beaker (prunt)
4	SGP-T1-C13	Weakly colored green	Beaker (?)
5	SGP-T1-C50	Weakly colored green	Bottle
6	SGP-T1-C50	Weakly colored green	Beaker (?)
7	SGP-T1-C54	Weakly colored green	Beaker
8	SGP-T1-C54	Colorless	Beaker (prunt)
9	SGP-T1-C61	Colorless	Beaker
10	SGP-T1-C65	Weakly colored green	Unknown
11	SGP-T1-C65	Weakly colored pink	Beaker (prunt)
12	SGP-T1-C65	Weakly colored yellow	Beaker
13	SGP-T1-C252	Weakly colored green	Beaker (prunts)
14	SGP-T1-C258	Weakly colored green	Beaker (prunts)
15	SGP-T1-C258	Colorless	Beaker (prunt)
16	SGP-T1-C297	Colorless	Unknown
17	SGP-T1-C297	Weakly colored green	Unknown
18	SGP-T1-C283	Weakly colored green	Unknown
19	SGP-T1-C283	Weakly colored green	Beaker (prunt)
20	SGP-T1-C317	Weakly colored green	Beaker (mold-blown ring base)
21	SGP-T1-C49	Dark blueish green	Unknown
22	SGP-T1-C49	Opaque red glass	Unknown
23	SGP-T1-C49	Opaque red glass	Unknown
24	SGP-T1-C49	Green	Unknown
25	SGP-T1-C49	Blueish green glass	Unknown
26	SGP-T1-C53	Colorless with white reticello and yellow trail	Goblet
27	SGP-T1-C69	Weakly colored green with blue rim	Beaker/goblet
28	SGP-T1-C81	Weakly colored green	Beaker(?)
29	SGP-T1-C014	Green	Biconical beaker
30	SGP-T1-C252	Weakly colored green with blue swirls	Beaker
31	SGP-T1-C279	Weakly colored green with blue rim	Beaker/goblet
32	SGP-T1-C317	Amber	Unknown

subsurface features, such as a granary subsequently used for trash deposition. Radiocarbon dates obtained for these layers provide secure chronological reference points (see Fig. 2). As for color, 27 of these shards are colorless or naturally colorized light green/pink/yellow shards, while five are more strongly colored. Distinct and strongly colored elements, such as blue rims, white *reticella* decoration, and a yellow trail, were also sampled and are discussed in a separate section below (Table 4).

Table 2
Averaged wt.% chemical composition of the San Giuliano glass samples, as per the EPMA-WDX analysis.

ID #	SiO_2	Na_2O	CaO	K_2O	MgO	PbO	CuO	Al_2O_3	Fe_2O_3	Sb_2O5	SO_3	ZnO	La_2O_3	P_2O_5	Cl	ZrO_2	TiO_2	ThO_2	SrO	MnO	Cr_2O_3	CoO	Total
Primarily natron glass																							
5	67.42	17.05	7.15	1.10	1.20	0.19	0.16	2.62	0.97	0.37	0.28	0.03	0.08	0.25	0.87	0.01	0.18	0.04	0.12	0.88	0.07	n.d	101.04
18	66.74	17.91	5.71	1.24	1.10	0.32	0.22	2.85	1.24	0.43	0.19	0.03	0.04	0.20	0.87	0.00	0.23	0.03	0.07	0.89	0.02	n.d	100.33
24	66.35	17.58	6.60	0.95	0.93	0.68	0.28	2.58	1.11	0.45	0.29	0.05	0.07	0.17	0.86	0.05	0.15	0.03	0.05	1.05	0.03	n.d	100.29
25	66.11	17.65	6.49	0.63	0.81	0.83	0.58	2.41	0.92	0.46	0.32	0.03	0.09	0.10	0.80	0.07	0.16	0.01	0.07	0.95	0.08	n.d	99.58
26	69.92	18.72	5.92	0.58	0.52	0.00	0.02	1.98	0.38	0.44	0.35	0.11	0.07	0.10	0.84	0.00	0.10	0.04	0.13	0.40	0.08	n.d	100.69
29	66.98	17.45	6.58	1.10	1.16	0.18	0.19	2.87	1.10	0.26	0.29	0.08	0.00	0.26	0.82	0.04	0.21	0.01	0.10	0.98	0.04	n.d	100.68
AVE.	67.25	17.73	6.41	0.93	0.95	0.37	0.24	2.55	0.96	0.40	0.29	0.05	0.06	0.18	0.84	0.03	0.17	0.03	0.09	0.86	0.05	0.00	
Primarily plant-ash glass																							
6	65.12	14.19	8.94	2.31	3.36	0.04	0.02	2.56	1.19	0.18	0.14	0.04	0.06	0.51	0.71	0.00	0.13	0.01	0.05	1.31	0.08	n.d	100.93
7	65.35	13.65	8.73	2.29	3.66	0.03	0.01	2.75	1.22	0.10	0.14	0.08	0.06	0.57	0.77	0.12	0.13	0.00	0.05	1.43	0.00	n.d	101.11
11	67.53	13.88	8.86	2.09	2.82	0.02	0.01	1.44	0.65	0.08	0.18	0.11	0.04	0.53	0.92	0.06	0.09	0.00	0.03	0.43	0.03	n.d	99.79
16	67.41	15.49	7.08	2.18	2.05	0.08	0.03	2.64	1.22	0.14	0.16	0.03	0.03	0.35	0.80	0.05	0.12	0.04	0.10	1.20	0.03	n.d	101.22
27	67.69	15.44	6.78	2.13	2.32	0.08	0.04	2.58	1.10	0.17	0.13	0.06	0.02	0.39	0.64	0.10	0.13	0.00	0.18	1.56	0.07	n.d	101.60
30	66.79	14.52	8.56	2.95	2.33	0.13	0.05	1.66	0.71	0.19	0.13	0.03	0.05	0.67	0.88	0.02	0.08	0.02	0.14	0.70	0.04	n.d	100.64
32	60.37	20.10	4.59	1.99	2.59	0.07	0.00	4.70	3.07	0.10	0.08	0.11	0.02	0.38	1.00	0.22	0.40	0.03	0.10	1.62	0.03	n.d	101.58
AVE.	65.75	15.32	7.65	2.28	2.73	0.06	0.02	2.62	1.31	0.14	0.14	0.07	0.04	0.49	0.82	0.08	0.15	0.02	0.09	1.18	0.04	0.00	
Intermediate glass (combining natron and plant ash glasses)																							
1	66.56	16.39	7.58	1.64	1.90	0.11	0.02	2.65	1.19	0.59	0.20	0.07	0.13	0.23	0.78	0.10	0.16	0.02	0.06	1.14	0.02	n.d	101.08
2	66.84	16.15	7.37	1.73	1.87	0.16	0.03	2.68	1.25	0.18	0.22	0.06	0.08	0.28	0.76	0.10	0.18	0.04	0.13	1.09	0.02	n.d	101.21
3	71.00	14.50	6.21	1.85	2.77	0.04	0.01	1.58	0.74	0.13	0.06	0.04	0.04	0.36	0.96	0.04	0.12	0.04	0.15	0.71	0.03	n.d	101.36
4	68.13	15.68	7.05	1.74	2.18	0.11	0.02	2.30	1.06	0.30	0.16	0.06	0.08	0.29	0.83	0.08	0.15	0.03	0.11	0.98	0.02	n.d	101.22
8	70.11	14.81	6.15	1.94	2.66	0.08	0.05	1.59	0.70	0.07	0.10	0.10	0.05	0.39	0.91	0.04	0.07	0.00	0.07	0.71	0.04	n.d	100.65
9	68.04	15.07	7.59	1.88	3.10	0.07	0.04	2.06	0.91	0.12	0.16	0.12	0.11	0.59	0.82	0.02	0.16	0.01	0.04	0.76	0.05	n.d	101.70
10	64.62	14.28	9.14	1.56	4.27	0.01	0.00	2.75	1.04	0.09	0.12	0.07	0.09	0.39	0.86	0.10	0.15	0.00	0.08	0.95	0.06	n.d	100.62
12	67.19	17.80	6.54	2.47	1.50	0.12	0.02	2.00	0.79	0.15	0.09	0.12	0.07	0.32	0.88	0.00	0.11	0.02	0.13	0.64	0.01	n.d	100.94
13	71.44	15.08	5.69	2.58	1.30	0.10	0.06	1.48	0.74	0.09	0.07	0.02	0.04	0.46	0.86	0.06	0.09	0.01	0.11	0.45	0.00	n.d	100.71
14	70.09	15.32	5.87	2.53	1.23	0.00	0.01	1.44	0.69	0.20	0.10	0.00	0.00	0.49	0.92	0.14	0.05	0.01	0.12	0.41	0.00	n.d	99.60
15	71.13	15.51	6.07	2.46	1.24	0.08	0.03	1.46	0.60	0.14	0.10	0.03	0.05	0.38	0.95	0.03	0.08	0.02	0.16	0.42	0.05	n.d	100.97
17	64.20	14.16	8.70	1.62	4.23	0.02	0.02	2.81	1.10	0.05	0.14	0.10	0.02	0.41	0.87	0.02	0.14	0.02	0.20	0.88	0.02	n.d	99.73
19	64.98	15.48	7.30	2.13	1.68	0.04	0.07	2.01	0.89	0.20	0.10	0.06	0.13	0.36	0.89	0.04	0.12	0.03	0.19	1.06	0.01	n.d	97.77
20	69.15	15.78	6.84	2.31	1.60	0.06	0.01	2.41	0.98	0.06	0.09	0.14	0.02	0.35	0.89	0.02	0.17	0.03	0.08	0.41	0.01	n.d	101.39
21	62.41	20.10	6.46	1.26	1.75	0.19	2.30	2.10	1.13	0.17	0.27	0.09	0.09	0.61	1.04	0.07	0.21	0.02	0.08	0.87	0.06	n.d	101.27
22	61.28	14.71	7.31	1.45	1.29	4.90	0.77	3.37	2.54	0.40	0.28	0.20	0.04	0.36	0.60	0.02	0.18	0.04	0.06	0.94	0.08	n.d	100.81
23	57.57	14.69	7.27	1.42	1.56	8.23	2.04	2.45	2.12	0.42	0.23	0.24	0.00	0.55	0.78	0.00	0.15	0.00	0.12	0.54	0.04	n.d	100.40
28	66.18	16.63	7.17	1.74	1.75	0.16	0.06	2.54	1.44	0.21	0.16	0.11	0.00	0.35	0.79	0.12	0.13	0.02	0.00	1.35	0.01	n.d	100.90
31	67.34	14.93	7.09	2.71	1.53	0.01	0.03	1.63	0.80	0.19	0.13	0.07	0.15	0.48	0.78	0.06	0.11	0.04	0.07	0.73	0.03	0.15	98.91
AVE.	66.69	15.66	7.00	1.95	2.03	0.08†	0.30	2.17	1.09	0.19	0.15	0.09	0.06	0.41	0.85	0.06	0.13	0.02	0.10	0.79	0.03	0.00	

†PbO average calculated after removing two outliers (samples 22 and 23).

Table 3
Mean chemical composition, standard deviation, and minimum and maximum values of the primary oxides for the various glass groups.

		SiO2	Na2O	CaO	K2O	MgO	Al2O3	Fe2O3	SO3	P2O5	Cl	TiO2	MnO
Primarily natron	Mean	67.25	17.73	6.41	0.93	0.95	2.55	0.96	0.29	0.18	0.84	0.17	0.86
	SD	1.39	0.56	0.52	0.27	0.26	0.33	0.30	0.056	0.073	0.03	0.045	0.23
	Min	66.11	17.05	5.71	0.58	0.52	1.98	0.38	0.19	0.1	0.8	0.1	0.4
	Max	69.92	18.72	7.15	1.24	1.2	2.87	1.23	0.35	0.26	0.78	0.23	1.05
Primarily plant-ash	Mean	65.75	15.32	7.65	2.28	2.73	2.62	1.31	0.14	0.49	0.82	0.15	1.18
	SD	2.59	2.23	1.61	0.32	0.59	1.05	0.81	0.03	0.12	0.13	0.11	0.45
	Min	60.37	13.65	4.59	1.99	2.05	1.44	0.65	0.08	0.35	0.64	0.08	0.43
	Max	67.69	20.1	8.94	2.95	3.66	4.7	3.07	0.18	0.67	1.00	0.40	1.62
Intermediate	Mean	66.75	15.63	7.02	1.95	2.07	2.17	1.09	0.15	0.40	0.85	0.13	0.79
	SD	3.64	1.40	0.89	0.44	0.93	0.55	0.49	0.07	0.10	0.10	0.04	0.27
	Min	57.57	14.16	5.69	1.26	1.23	1.44	0.6	0.06	0.23	0.6	0.05	0.41
	Max	71.44	20.1	9.14	2.71	4.27	3.37	2.54	0.28	0.4	1.04	0.21	0.79

The glass samples were embedded in acrylic resin and then polished to obtain a smooth surface of glass unaltered by corrosion or other chemical surface changes. Wavelength-dispersive (WDS) measurements were performed using a JEOL JX 8100 SUPERPROBE, using an acceleration voltage of 15 kV and a beam current of 3nA, with counting times of 20 s for peak and 10 s for background measurements. A defocused beam with a diameter of 20 µm was used to prevent volatilization of the alkalis. Verification of measurement accuracy was performed using analysis of natural and synthetic mineral standards (see e.g. Arletti et al. 2008; De Francesco et al., 2019). Four analyses, taken at different locations in the glasses, were obtained for each sample to quantify the concentrations of (mineral standards used are given in parenthesis) SiO_2 (orthoclase), Sb_2O_5 (Sb metal), PbO (Pb-glass), SO_3 (barite), ZnO (gahnite), Na_2O (jadeite), CaO (diopside), Cl (atacamite), ZrO_2 (zircon), CuO (atacamite), MgO (periclase), La_2O_3 (La-phosphate), K_2O (orthoclase), P_2O_5 (apatite), FeO (almandine), Al_2O_3 (corundum), TiO_2 (rutile), ThO_2 (Th-REE glass), SrO (strontianite), MnO (rhodonite) and Cr_2O_3 (chromite). Bulk concentration wt.% can be skewed by heterogeneity in glass samples, but most of the San Giuliano glass samples were relatively homogeneous: standard deviations were typically$<$1.5 wt%. Mean wt.% measurements were used in the analysis and reported in Table 2. Unaveraged wt.% measures can be found in the Supplemental Data 2.

4. Results

The subset of 32 glass shards represents 12.3 % of the total assemblage analyzed in the field by pXRF. pXRF data collected on these 32 samples reflect the distribution of MnO, Fe_2O_3, TiO_2, and ZnO concentrations in the total assemblage, and thus for these components the subset is a representative sample (Supplemental Table S1 and Fig. S1). The subset overrepresents samples with high CuO, PbO, NiO, and As_2O_3 content (Supplemental Table S1; Fig. S2) due to the preferential selection of colored glass shards for EPMA analysis.

All samples in the subset were silica-soda-lime glasses, with SiO_2 ranging between 57.57 and 71.44 wt%, Na_2O between 13.65 and 20.10 wt%, and CaO between 4.59 and 9.14 wt%, based on EPMA analysis (Table 2). Variation in their content of minor and trace elements are described in the following sections and interpreted as indicative of differences in the fluxes and decolorants used during the manufacturing process. These data provide evidence for widespread recycling and mixing of natron and plant-ash glasses.

4.1. Flux

EPMA analysis demonstrates that San Giuliano glasses range considerably in terms of potassium and magnesium: K_2O from 0.58 to 2.95 wt% and MgO from 0.52 to 4.265 wt% (Table 2). Six samples (#s 6, 7, 11, 27, 30 and 32) contained over 2 wt% of both K_2O and MgO, typical of plant ash glass (Sayre 1963; Lilyquist and Brill 1995; Henderson 2000). Among these plant ash glass samples, $K_2O = 2.47 \pm 0.48$ wt% and $MgO = 2.85 \pm 0.8$ wt% (Table 3). Graphing the San Giuliano glasses on a ternary plot comparing normalized Na_2O, CaO and MgO + K_2O content indicates that they are consistent with vegetable silica-soda-lime glasses produced from the 8th-9th centuries onward (Fig. 5).

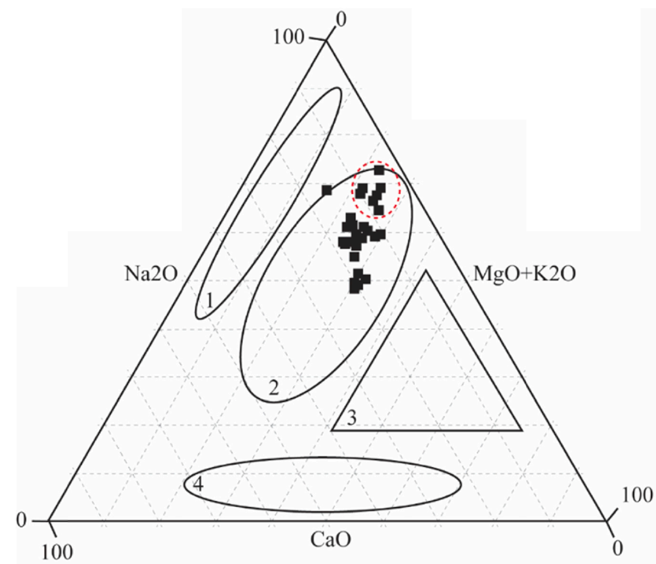


Fig. 5. Ternary plot of the normalized Na_2O, $MgO + K_2O$, and CaO concentrations in the San Giuliano glass finds. The four groups indicated by the ellipses and triangle are: 1) natron silica-soda-lime glass (800 BCE to AD 800–1000); 2) plant ash silica-soda-lime glasses dating to the 8th century CE onwards; 3) mixed alkali glass of the Late Bronze Age and post-medieval period; and 4) potash glass of the medieval period (ternary plot and groupings follow Barca and Papparella 2020; Cagno et al. 2012). The dashed red ellipse indicates the glasses thought to contain higher proportions of natron glass.

While none of the San Giuliano glass fragments are within the pure natron silica-soda-lime ellipse in Fig. 5, the low concentrations of K_2O and MgO (both 0.9 ± 0.3 wt%) of at least six samples—5, 18, 24, 25, 26, and 29 (see dashed ellipse in Fig. 5)—suggest that those glass batches predominantly comprised recycled natron glass from earlier periods (Fig. 6). The use of natron as flux is supported by comparing K_2O versus P_2O_5 content (Fig. 7). Phosphorous is found in very low concentrations in natron glasses; for example, in 85 samples of colorless natron glass found abord the 3rd century CE shipwreck of the *Iulia Felix,* the average P_2O_5 content was 0.05 wt% and with a maximum of 0.24 wt% (Silvestri et al. 2008). Similarly, the six San Giuliano shards of primarily natron glass contain $<$ 0.26 wt% P_2O_5.

Although a handful of glass shards may have been primarily natron glass or primarily plant ash glass, almost 60 % of the samples (N = 19)

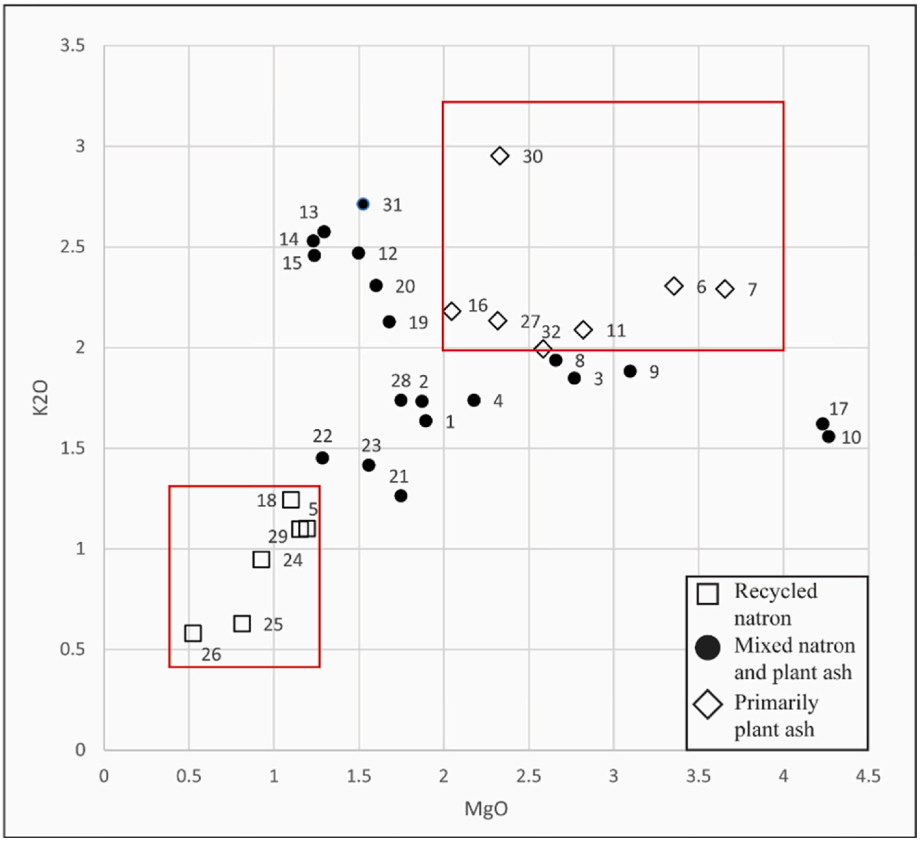


Fig. 6. Bi-plot of MgO and K_2O. The boxed areas indicate samples that are either primarily recycled natron glass (lower left) or primarily plant ash glass (upper right).

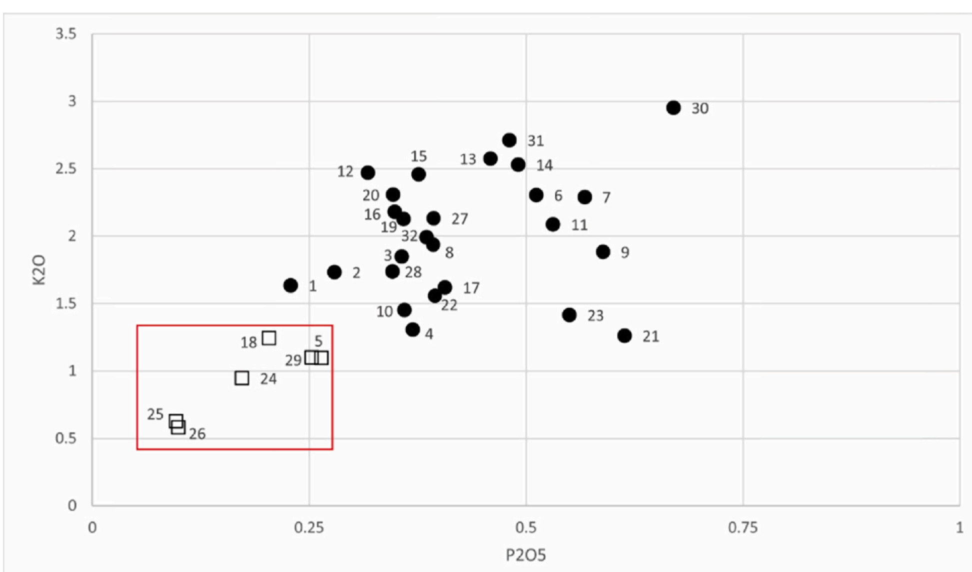


Fig. 7. Bi-plot comparing K_2O and P_2O_5 content of the San Giuliano glasses. The red box indicates the samples primarily composed of natron glass. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

yielded chemical compositions intermediate between these groups (see Figs. 6 and 7). Although it is currently uncertain where San Giuliano residents may have obtained their glass, it was produced by combining plant-ash glass—perhaps newly arriving from the eastern Mediterranean—and varying quantities of recycled natron glass cullet (see below), creating glass with a hybrid composition somewhere between the two.

4.2. *Decolorizers and other evidence of recycling*

Evidence for the continued incorporation of recycled Roman glass is provided by the presence of antimony as a decolorizer in most of the San Giuliano assemblage. Glassmakers used various decolorants to remedy the greenish tint imparted by iron in the silica source. From approximately the 1st through 4th centuries AD, natron glass was decolored

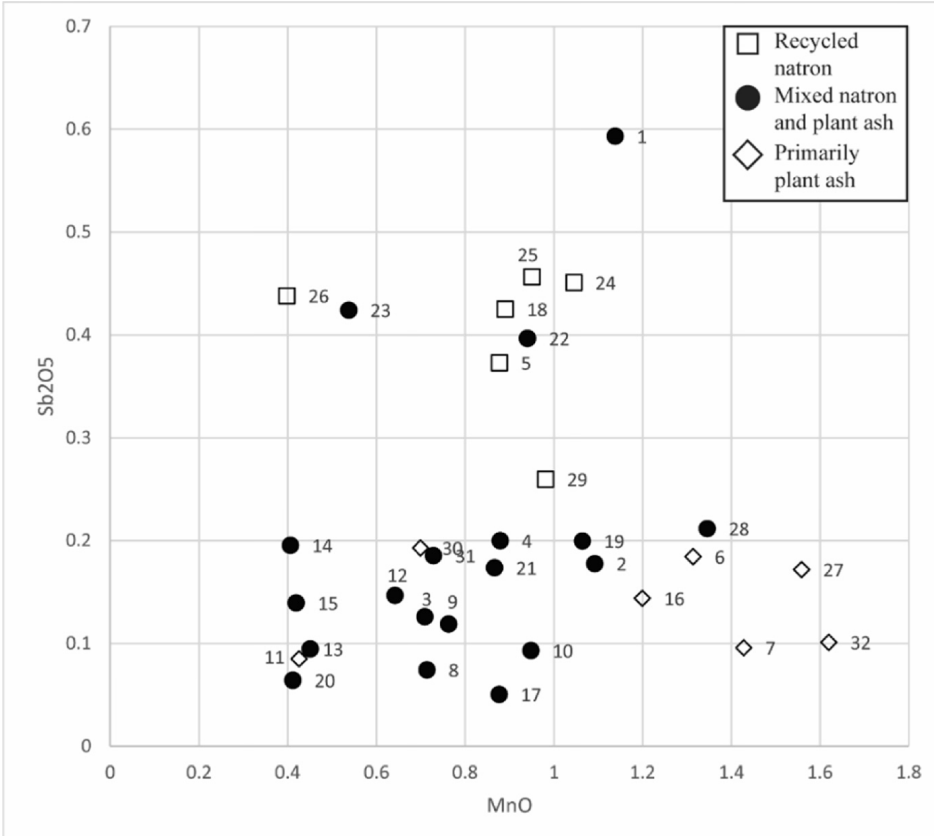


Fig. 8. Bi-plot comparing Sb_2O_5 and MnO, decolorants used in the San Giuliano samples.

using antimony, probably added as stibnite (Sb_2S_3; Jackson 2005). Antimony is a strong decolorizer and additionally acts as a finer, removing dissolved gasses that would otherwise leave behind bubbles in the glass (Bamford 1977). Because antimony is not found above a few ppm in geological materials used in glassmaking, its presence above 0.1–0.2 wt% is considered intentional (Paynter and Jackson 2016; Uboldi and Verità 2003).

Around the 4th century, it appears that antimony became unavailable to glassmakers in the eastern Mediterranean, who began to incorporate manganese oxide as a decolorant (Sayre and Smith 1967; Whitehouse 2002). Manganese oxide is generally seen as a less effective decolorizer than antimony, as it can interact in complex ways with iron oxides depending on the furnace conditions (Brill 1988; Freestone 2015). While present in small quantities in some silica sources, concentrations of more than 0.5 wt% MnO indicate intentional addition (Jackson 2005). Drawing on primary glassmaking sites and raw glass from shipwrecks in the eastern Mediterranean, Freestone (2015:31) argues that there is "no evidence of the addition of both manganese and antimony to the same batch at the primary stage". This means that the co-occurrence of antimony and manganese in medieval Italian glass samples almost certainly indicates mixing by recycling, with antimony only present if the cullet included in the batch contained remnant antimony from Roman glass predating the 4th century CE. This type of recycling was already taking place in the Roman period, as indicated by analysis of glass cullet from the 3rd century CE shipwreck of the *Iulia Felix*: while most of the glass had been decolorized either by antimony or manganese, a subsample contained both decolorizers and was thus taken as evidence that these shards were from vessels produced from recycled glass (Freestone 2015; Silvestri et al. 2008; see also Jackson and Paynter 2015; Paynter and Jackson 2016).

Among the San Giuliano assemblage, almost 80 % of the samples contain more than 0.1 wt% Sb_2O_5, suggesting the widespread incorporation of antimony-containing cullet (Fig. 8). Ten of the samples contain greater than 0.2 wt% Sb_2O_5, including all five fragments of the recycled natron glass, consistent with the glass originally being formed in the Roman period. It is notable that eight of the higher Sb_2O_5 group contain >0.5 wt% manganese, which means that most of the primarily antimony-decolored glass had been previously combined with manganese-decolored glasses post-dating the 4th century. There is generally a good correspondence between the Fe_2O_3 content and the amount of MnO that had been added (Fig. 9, $r2 = 0.65$, when excluding the three outliers 22, 23, and 32; see below), suggesting that manganese had been intentionally included to counteract the greening effect of the iron. Nonetheless, truly colorless glass was only infrequently achieved by the glassmakers.

Additional factors affecting the color and transparency of the glass, as well as providing further evidence for recycling of glass cullet, are trace amounts of elements added for coloring, such as copper or lead (Silvestre and Marcante, 2011; Uboldi and Verità 2003; Verità and Zecchin 2007); these are known as recycling markers. This likely occurred when cullet with colored glass, perhaps as trails or other decorative elements, found its way into batches of transparent glass. The San Giuliano total glass assemblage (n = 261) included 50 shards (19 %) that had anomalously high outlier CuO concentrations and 42 shards (16 %) with outlier PbO concentrations (Table S1, Fig. S2). We interpret the outliers from the normal distribution as evidence for the addition of trace elements as colorants. The subsample analyzed by EPMA included 30 of 32 shards with quantifiable copper and lead, including seven outliers for CuO and four outliers for PbO (Table S1). Among the 26 clear or weakly colored glass shards from the subsample, all 26 samples contained traces of copper, ranging from 0.001 to 0.275 wt% (average 0.05 ± 0.06 wt%; Table 2). Lead was found in 24 of these samples, at levels between 0.007 and 0.68 wt% (average 0.1 ± 0.12 wt%). The prevalence of antimony, its appearance in combination with manganese,

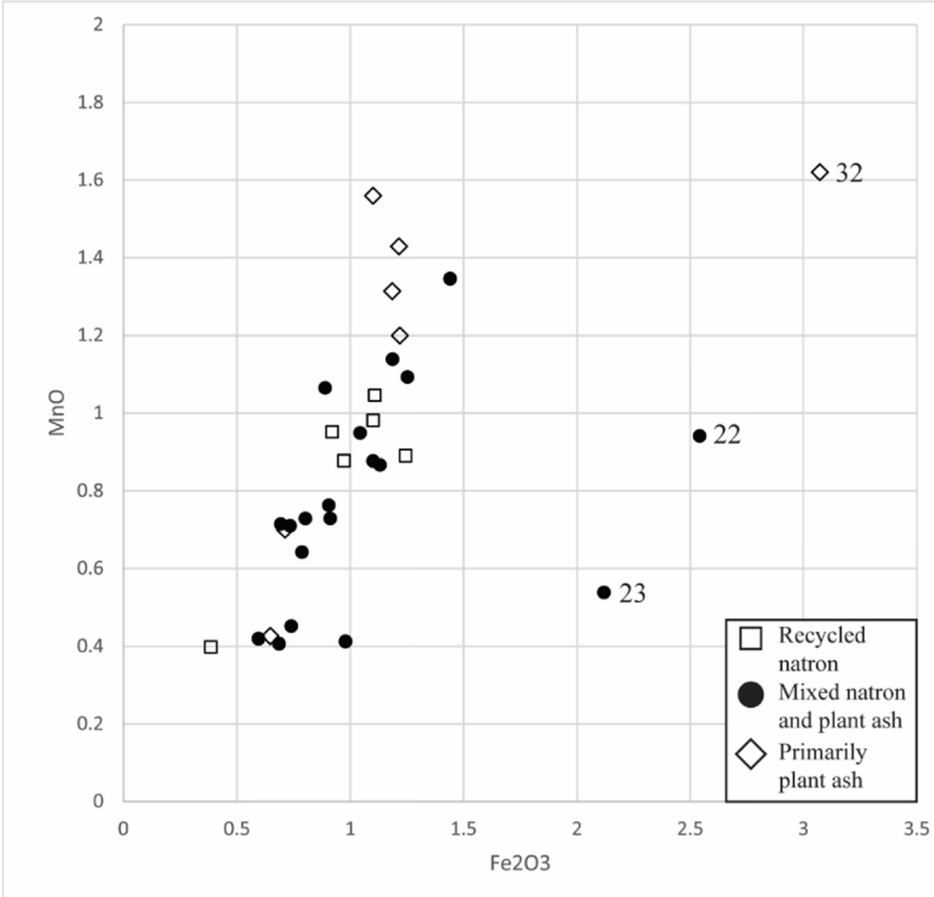


Fig. 9. Bi-plot of MnO vs Fe_2O_3, indicating a good correlation between iron content and manganese used to decolorize the glass.

and the presence of trace elements such as copper and lead further reinforce the conclusion that inhabitants of San Giuliano had access to glass that had been extensively recycled, some of it dating to the 4th century CE or earlier.

4.3. *Strongly colored glasses*

4.3.1. *Opaque red glass*

Samples 22 and 23 are shards of opaque red glass. Opaque red glass can be divided into two groups: a high-copper high-lead variety containing typically 5–12 wt% CuO and 20–45 wt% PbO and usually produced in the pre-Roman period, and a low-copper low-lead group containing around 1–2 wt% and up to 5 wt% CuO and lead levels lower than 10–12 wt% produced in the Roman, Late Antique, and medieval periods (Freestone 1987; Freestone et al. 2003; Hendersen, 1991). In these low-copper low-lead glasses, the red color was obtained by nanoparticles of metallic copper that separate from the melt under reducing conditions, brought about by the addition of iron or tin oxide as reducing agents (Barber et al. 2009; Freestone et al. 2003). The lead oxide aids in the crystallization of the cuprite in ways that produce the desired red color (Barber et al. 2009).

Chemical analysis documents that Samples 22 and 23 are both low-copper low-lead red glasses, albeit distinct from one another and therefore likely deriving from different vessels. The lead content was 4.9 wt% and 8.23 wt% (assemblage average: <0.2 wt%), while copper levels were 0.77 wt% and 2.03 wt% respectively (assemblage average: 0.15 wt %).

The makeup of the red glass shards from San Giuliano suggests that they were produced using recycled Roman red glass. This glass may have derived from mosaic tesserae, as suggested by the similar chemical composition to dullish-red tesserae from 1st century CE mosaics from Milan and Pompeii (Boschetti et al. 2016: their Table 2). As with these Roman tesserae, the composition of the red shards from San Giuliano appear to have been produced on a base of plant ash glass: K_2O is 1.4–1.5 wt% and MgO ranges from 1.3 to 1.6 wt%, while phosphorous levels are higher than typical of natron glass, at 0.4–0.6 wt%. This is unsurprising, given that even when natron glasses were dominant during the Roman period, at least some opaque red glasses were produced using plant ash flux (Boschetti et al. 2016; Freestone et al. 2003; Henderson 2013; although see Andreescu-Treadgold and Henderson, 2006 for opaque red tesserae used in 11th century mosaics at the Cathedral of Santa Maria Assunta on the island of Torcello, near Venice, which are a mixture of plant ash and natron glass). The presence of 0.4 wt% Sb_2O_5 in the San Giuliano opaque red shards, however, suggests that there may have been some additional admixture with Roman glass decolorized using antimony. The challenges presented by working with red opaque glass, particularly in the need to maintain a reducing environment during both production and working (see e.g. Brill and Cahill 1988), suggests that red glass vessels would have been considered luxury wares.

4.3.2. *Blue glass*

Strongly colored blue glass in the San Giuliano sample included one fragment of blueish-green (Sample 21) and two clear glass vessels, most likely goblets, with dark blue rims (Samples 27 and 31; see Table 4). Sample 21 contains comparatively high levels of copper, at 2.3 wt% CuO. The blue color may derive from cupric ions dispersed in glass produced under oxidizing—rather than reducing—conditions (see Brill and Cahill 1988). EMPA analysis of the blue rims documents that while cobalt was absent from Sample 27, Sample 31 contained 0.15 wt% CoO. This blue glass may have been derived from reutilization of deep-blue

Table 4
Averaged wt.% of the most highly colored glasses from the San Giuliano sample, sampled using EPMA-WDS analysis.

Sample #	SiO_2	Na_2O	CaO	K_2O	MgO	PbO	CuO	Al_2O_3	Fe_2O_3	Sb_2O_5	SO_3	ZnO	P_2O_5	Cl	TiO_2	SrO	MnO	Total
26 (white reticella)	69.04	15.55	5.97	0.82	1.32	0.56	0.08	2.11	0.57	4.33	0.38	0.22	0.14	0.66	0.12	0.23	0.21	102.27
26 (yellow trail)	67.92	15.40	5.93	0.76	1.36	0.50	0.10	2.08	0.53	4.20	0.35	0.01	0.05	0.58	0.06	0.01	0.25	100.07
27 (blue rim)	68.12	15.25	7.28	2.17	2.24	0.15	0.01	2.56	0.95	0.27	0.16	0.12	0.44	0.76	0.14	0.02	1.46	102.07
30 (blue rim)	61.17	16.26	7.80	2.61	1.81	0.10	0.03	1.35	8.19	0.13	0.08	0.11	0.79	0.69	0.06	0.16	0.77	102.20

Roman mosaic tesserae, which contain CoO around 0.2 wt% (see e.g. Schibille and Freestone 2013).

4.3.3. *White reticella*

Sample 26 derives from a thin-walled colorless glass goblet with an opaque-white-and-colorless *reticella* swirl running parallel to the rim. EPMA analysis suggests that Roman tesserae were used in the production of this white reticella decoration. The white glass is significantly enriched in antimony, containing 4.33 wt% Sb_2O_5 (Table 4). Opaque white was achieved by the Romans using calcium antimonate, which manifests as elevated levels of Sb_2O_5 (Lahlil et al. 2008). In *reticella* rods documented by Schibille and Freestone (2013: their Table 1), wt.% Sb_2O_5 of white glass ranged from 2.61 to 5.81 wt%. Roman white glasses are chemically typical of natron glass but contain elevated levels of MgO (Schibille and Freestone 2013); this is also observed with the San Giuliano white glass (Table 3). This particular vessel is unique at San Giuliano for its very low levels of Fe_2O_3 (0.38 wt%), similar to *vitrum blanchum* produced in Venice (see e.g. Verità 2013, his Table 6.2.4), and attests to the selection of raw materials with little iron contamination. The absence of PbO and near-absence of CuO (0.02 wt%) suggest that this natron glass had been subject to little or very selective recycling.

5. Discussion and conclusions

The glass assemblage from San Giuliano supports the notion that recycling of earlier glass—including Roman mosaic tesserae— was prevalent during the Middle Ages: 80 % of the sample contained quantities of antimony suggestive of the incorporation of Roman glass predating the 4th century, supported by the co-occurrence of antimony and manganese (post-dating the 4th century) in a majority of the samples. While this degree of recycling may have resulted from reductions in *trans*-Mediterranean trade in glass, it is notable that the San Giuliano sample also contains evidence for the incorporation of plant-ash glasses into economic networks on the Italian peninsula. This is demonstrated by the presence of glasses that are predominantly plant-ash (N = 7). Perhaps more importantly, approximately 60 % of the sample comprised intermediate glass, formed by melting together new(er) plant-ash glass with recycled cullet of variable chemical composition.

Intermediate glass has been found at other contemporaneous Italian sites. In many cases, it comprises a small proportion of the sample. Glass assemblages from 10th-12th century Venice, for instance, contain only about 4 % intermediate glasses (Verità and Zecchin 2007; Verità 2013). Other sites contain slightly greater proportions: five out of 58 shards from the 9th-11th century site of Nogara (Silvestre and Marcante, 2011), and five out of a subsample of 19 shards from four 8th-12th century sites north of Milan (Uboldi and Verità 2003) present the intermediate composition. At the 7th-11th century site of Comacchio, 19 out of 89 samples are of an intermediate composition (Bertini et al. 2020). Mixing of natron and plant ash glass is more common at other sites: one example is a 13th-16th century assemblage from the San Severus monastery in Classe, where 7 out of 14 samples had an intermediate composition (Vandini et al. 2018). By contrast, samples from other locations in Italy, such as Tuscany (13th-16th century; Cagno et al. 2008, 2010), the northern Adriatic site of Grado (5th-14th century; Silvestri et al. 2005), and Apulian site of San Lorenzo di Carmignano (12th-14th century; Gliozzo et al. 2021) contain little to no evidence of the combining of natron and plant ash glasses. Among samples that include the 11th-13th centuries, San Giuliano stands out with a high proportion of glasses produced by mixing natron and plant ash glasses.

Inhabitants of San Giuliano primarily had access to glass vessels produced through recycling of older glasses, some of which entailed the incorporation of various trace elements, as well as the extensive mixing of natron and plant-ash glasses. This explains the dearth of perfectly clear glass at the site. Nonetheless, the residents of the medieval hall were aware of glass as a prestige item and directed resources toward obtaining glass drinking cups, goblets, lamps, and even high-status red glass vessels. This testifies to the importance of glasswares in signaling prestige for an aspiring medieval elite.

CRediT authorship contribution statement

Colleen Zori: Conceptualization, Investigation, Formal analysis, Methodology, Data curation, Visualization, Writing – original draft. **James Fulton:** Methodology, Visualization, Validation, Writing – review & editing. **Peter Tropper:** Methodology, Formal analysis, Visualization, Validation, Writing – review & editing. **Davide Zori:** Methodology, Funding acquisition, Project administration, Resources, Writing – review & editing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Excel sheets of the data will be included as supplemental materials.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.jasrep.2022.103731.

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