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MEDIEVAL GLASS FROM *ROCCA DI ASOLO* (NORTHERN ITALY): AN ARCHAEOMETRIC STUDY*

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An archaeometric study was performed on 33 medieval glass samples from Rocca di Asolo *(northern Italy), in order to study the raw materials employed in their production, identify analogies with medieval glass from the Mediterranean area and possible relationships between chemical composition and type and/or production technique, contextualize the various phases of the site and extend data on Italian medieval glass. The samples are soda–lime–silica in composition, with natron as flux for early medieval glasses and soda ash for the high and late medieval ones. Compositional groups were identified, consistent with the major compositional groups identified in the western Mediterranean during the first millennium* AD. *In particular, Asolo natron glass is consistent with the HIMT group and recycled Roman glass; soda ash glass was produced with the same type of flux (Levantine ash) but a different silica source (siliceous pebbles, and more or less pure sand). Cobalt was the colouring agent used to obtain blue glass; analytical data indicate that at least two different sources of Co were exploited during the late medieval period. Some data, analytical and historical, suggest a Venetian provenance for the high/late medieval glass and a relationship between type of object (beaker or bottle) and chemical composition.*

KEYWORDS: ROCCA DI ASOLO, ITALY, GLASS, MIDDLE AGES, NATRON, SODA ASH, COMPOSITIONAL GROUPS, EPMA, SEM

INTRODUCTION

The Middle Ages represent a period of fundamental importance in the history of glass production. Indeed, after AD 800, a change in the use of raw materials occurred both in the Islamic world and in the West (Newton and Davison 1996; Henderson 2002). In both areas, natron, the source of alkali used from the middle of the first millennium BC, was replaced by plant ash. In the West, wood ash had become the main flux agent, whereas in the Middle East and southern Europe, the alkali source is generally believed to be ash from marsh plants such as *Salicornia* spp., which grow on Mediterranean and Atlantic coasts (Henderson 2002; Tite *et al.* 2006).

In this context, compositional and structural characterization was carried out on early to late Middle Ages glass finds excavated from the *Rocca di Asolo* (province of Treviso, northern Italy), in order to expand current knowledge of the compositional and technological features of Italian medieval glass (Mirti *et al.* 1993; Mirti *et al.* 2000; Uboldi and Verità 2003; Salviulo *et al.* 2004; Casellato *et al.* 2005; Silvestri *et al.* 2005; Cagno *et al.* 2008, 2010; Genga *et al.* 2008).

The *Rocca di Asolo* is a fort on the summit of Montericco, in north-east Italy, dominating the town of Asolo (Fig. 1), an ancient settlement of the Veneti (*c.* ninth century BC) and later a Roman *municipium*. Archaeological excavations have revealed several phases at the Montericco site. The

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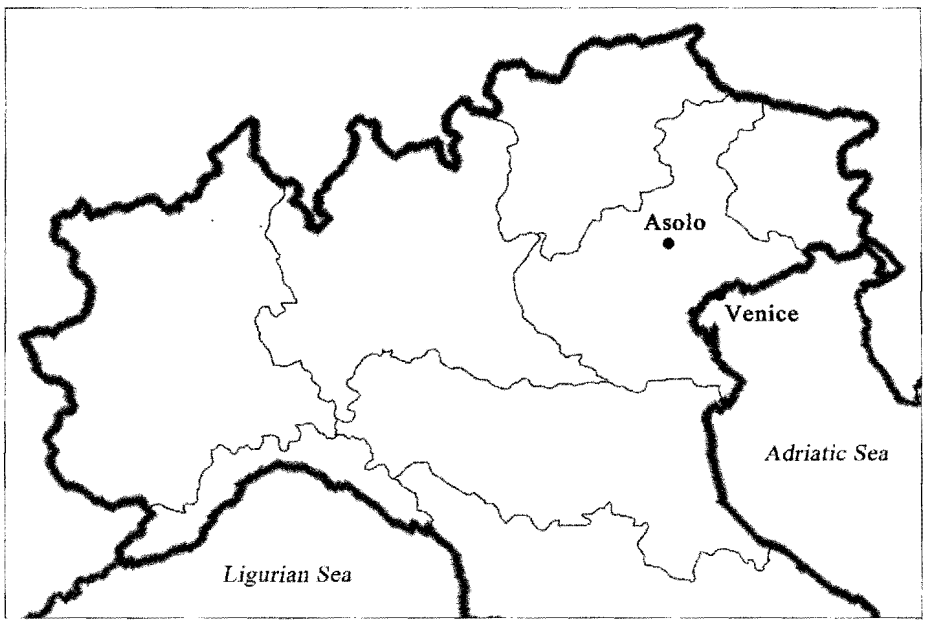

Figure 1 *The geographical location of Asolo (province of Treviso, northern Italy).*

first evidence of occupation dates to about the sixth century AD and was connected with the presence of a small church. Later (7th–10th centuries AD), the church was transformed into a monument, but already at the end of the 10th century it had decayed and was replaced by a burial area. Subsequently (10th–12th centuries), a settlement arose in the area, destroyed at the end of the 12th century when the military fortification (*Rocca*) was built. The *Rocca di Asolo* passed through various hands—including the Bishopric of Treviso, the Carraresi family from near Padova and the Republic of Venice—until its decay, at the end of the 16th century (Rosada 1989, 66–9; Bonetto 1993).

About 7000 glass fragments have been found at this site. There are a few finds—only about 100, including window panes—dating to the early Middle Ages; otherwise, high/late Middle Ages finds are more abundant and comprise various kinds of objects (beakers, bottles, lamps) (Rigoni 1986, 39–69). Analyses were performed on glass samples dating both to the early/high and late Middle Ages, for the following purposes: (1) to determine the raw materials used to produce the glass; (2) to classify samples according to the compositional groups reported in the literature for medieval glass; (3) to help set the various phases of the site in their proper context; (4) to identify possible correlations between chemical composition and type and/or manufacturing technique; and (5) to extend data on Italian glass of the Middle Ages.

MATERIALS AND EXPERIMENTAL

Thirty-three samples were analysed: 12 fragments of window panes, eight dating to the early Middle Ages (7th–10th centuries AD) and four to the late Middle Ages (15th century AD), and 21 fragments of objects dating to the high/late Middle Ages (12th–15th centuries). For five samples decorated with blue rims, both the colourless body and the blue decoration were analysed

separately, for a total of 38 samples, listed in Table 2 below. The ages and features of the samples are listed in Table 1.

The glass window panes are pale blue, greenish, yellowish and pale brown in colour; four are fragments of pieces with regular shape: one circular (*ruo*) and three triangular (*crosetta*) (Table 1). Two production techniques were identified: crown process (two samples) and cylinder process (10 samples) (Table 1). In these processes, a molten glass bubble was blown and then rolled on a smooth surface to obtain a disc-shaped crown or cylinder. In the former case, the crown was removed and cut; in the latter, the cylinder was cut lengthwise to obtain a flat sheet. The glass produced with these techniques is thinner than that produced by casting and has two smooth sides (Wolf *et al.* 2005; Arletti *et al.* 2010). The analysed objects are all for common use and include beakers and bottles. The first are of two types: beakers decorated with drops (*nuppenbecher*) (Stiaffini 1991, 1999) and flat-based beakers with blue rims (Fig. 2). The second are also of two types: the so-called *anghistere* (or *inghistere*) (Moretti 2001), bottles with a long neck and small body, and the *kropfflaschen* (Stiaffini 1991), characterized by a swelling at the base of the neck (Fig. 2). Optical microscopy, both stereoscopic and polarizing, was carried out on whole fragments and polished sections, respectively. For polished sections, the glass artefacts were cut perpendicularly to their surfaces with a diamond saw, mounted in epoxy resin, and then polished with a series of diamond pastes from 6 to 1 μm.

Scanning electron microscopy (SEM) analysis was performed to evaluate the homogeneity and state of conservation of the findings. A CamScan MX2500 scanning electron microscope was used, with a LaB_6 cathode, four quadrant solid state BSE detectors and an EDAX EDS system with a 'Sapphire' detector for microanalysis. The analytical conditions were as follows: 20 kV accelerating voltage, 90 μA filament emission and about 30 mm working distance.

Bulk chemistry was determined by electron probe microanalysis (EPMA). Ten analytical points were made along a line crossing the thickness of the polished sections of each sample, and means and standard deviations were calculated. The standard deviations range from about 0.01% to 1.41%, thus proving the homogeneity of the glass fragments; only means are reported in the tables. The electron microprobe used for quantitative analysis was a CAMECA-SX50, equipped with four wavelength-dispersive spectrometers (WDS). The analysed elements were Na, Mg, Al, Si, P, S, Cl, K, Ca, Ti, Mn, Fe, Sb, Co, Ni, Cu, Zn, Sn and Pb. The following standards were employed: synthetic pure oxides for Mg, Al, Fe and Sn, a synthetic MnTi oxide for Mn and Ti, albite for Na, diopside for Si and Ca, apatite for P, sphalerite for Zn and S, vanadinite for Cl, orthoclase for K, Sb_2S for Sb, PbS for Pb, and pure elements for Co, Ni and Cu. The operating conditions were 20 kV and 2nA sample current, with the beam defocused at not less than 10 μm for Na, K, Si and Al, in order to minimize the loss of alkali elements and better evaluate Si contents, and 20 kV and 30 nA for other elements. X-ray counts were converted to oxide weight percentages with the PAP (CAMECA) correction program. The detailed analytical conditions used, and the precision, accuracy and detection limits of EPMA are given in Silvestri and Marcante (2011), as the present samples were subjected to the same analytical protocol. It is stressed here that the precision and accuracy of data were calculated by comparisons with measurements on the Corning glass B (Brill 1999, 541) international reference standard under the same analytical conditions as the Asolo glass. The precision of EPMA data was generally between 0.5% and 10% for major and minor elements, respectively. Accuracy was better than 1% for SiO_2, Na_2O and FeO, better than 5% for CaO, K_2O, P_2O_5 and Sb_2O_3, and not worse than 12% for other major and minor elements.

Table 1 *A table of the samples analysed: note that the dating of the Asolo glass is stylistic*

Sample	*Type*	*Part preserved*	*Colour*	*Age (century AD)*
ASL-01	Glass pane, cylinder process	Fragment of wall	Light blue	7th–10th
ASL-02	Glass pane, cylinder process	Fragment of wall	Yellowish-brown	7th–10th
ASL-03	Glass pane, cylinder process	Fragment of wall	Green	7th–10th
ASL-04	Glass pane, cylinder process	Fragment of edge	Yellowish-brown	7th–10th
ASL-05	Glass pane, cylinder process	Fragment of wall	Yellowish-brown	7th–10th
ASL-06	Glass pane, cylinder process	Fragment of wall	Yellowish-brown	7th–10th
ASL-07	Glass pane, cylinder process	Fragment of wall	Green	7th–10th
ASL-08	Glass pane, cylinder process	Fragment of wall	Light blue	7th–10th
ASL-09	Glass pane with circular shape (*ruo*), crown process	Fragment of edge	Yellowish-brown	15th
ASL-10	Glass pane with triangular shape (*crosetta*), cylinder process	Whole shape	Yellowish-brown	15th
ASL-11	Glass pane with triangular shape (*crosetta*), crown process	Whole shape	Yellowish-brown	15th
ASL-12	Glass pane with triangular shape (*crosetta*), cylinder process	Whole shape	Yellowish-brown	15th
ASO-01	Beaker (*nuppenbecher*)	Drop	Colourless	13th–14th
ASO-02	Beaker (*nuppenbecher*)	Drop	Light blue	13th–14th
ASO-03	Beaker (*nuppenbecher*)	Drop	Yellowish	13th–14th
ASO-04	Beaker (*nuppenbecher*)	Drop	Yellowish-green	13th–14th
ASO-05	Beaker (*nuppenbecher*)	Drop	Colourless	13th–14th
ASO-06	Bottle (*kropfflasche*)	Neck	Green	13th–14th
ASO-07	Bottle (*kropfflasche*)	Fragment of the swelling at the base of the neck	Green	13th–14th
ASO-08	Bottle (*kropfflasche*)	Fragment of rim	Light blue	12th
ASO-09	Bottle (*kropfflasche*)	Fragment of the swelling at the base of the neck	Green	13th–15th
ASO-10	Bottle (*kropfflasche*)	Fragment of the swelling at the base of the neck	Green	13th–15th
ASO-11	Bottle (*kropfflasche*)	Fragment of the swelling at the base of the neck	Green	13th–15th
ASO-12	Bottle (*anghistera*)	Base	Light blue	12th–15th
ASO-13	Bottle (*anghistera*)	Base	Yellowish-green	12th–15th
ASO-14	Bottle (*anghistera*)	Base	Yellowish-green	15th–16th
ASO-15	Bottle (*anghistera*)	Base	Yellowish-green	13th–15th
ASO-16	Bottle (*anghistera*)	Base	Green	13th–15th
ASO-17	Beaker	Fragment of rim	Colourless with blue rim	14th–15th
ASO-18	Beaker	Fragment of rim	Colourless with blue rim	14th–15th
ASO-19	Beaker	Fragment of rim	Colourless with blue rim	14th–15th
ASO-20	Beaker	Fragment of rim	Colourless with blue rim	14th–15th
ASO-21	Beaker	Fragment of rim	Yellowish with blue rim	14th–15th

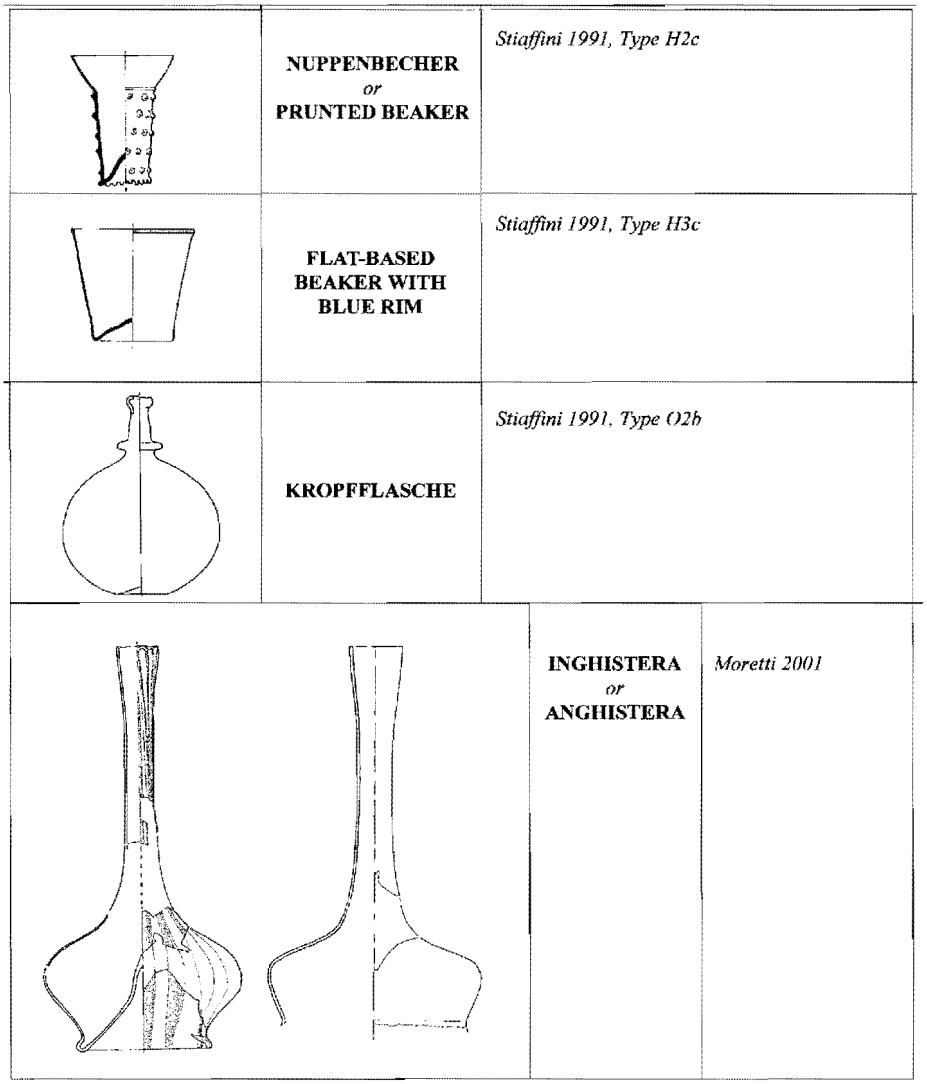

Figure 2 *Sketches of the analysed archaeological types (*nuppenbecher, *flat-based beaker with blue rim,* kropfflasche *and* anghistera*): references are also given (courtesy of A. Marcante).*

RESULTS AND DISCUSSION

OM and SEM analyses on polished sections revealed the homogeneity of all glass fragments: neither newly formed nor residual crystalline phases were identified, except for one blue glass sample, discussed below.

The chemical data are listed in Table 2: major and minor elements are expressed as weight per cent of oxides, and traces in parts per million (ppm). For the beakers decorated with blue rims, sample labels include the letters 't' to indicate the colourless body and 'b' for the blue glass. All samples are soda–lime-silica glass with SiO_2, Na_2O and CaO in the ranges of 61.8–70.9 wt%,

Table 2 *The chemical composition of all glass samples, expressed as weight per cent (wt%). Only means are reported: (a) less than 0.04 wt%; (b) less than 0.02 wt%; (c) less than 0.04 wt%; (d) less than 0.05 wt%; (e) less than 0.04 wt%; (f) less than 0.04 wt%; (g) less than 0.08 wt%; n.d. not detected. The type and compositional group are also reported for each sample*

Label	*Type*	*SiO_2*	*Na_2O*	*CaO*	*Al_2O_3*	*K_2O*	*MgO*	*Fe_2O_3*	*TiO_2*	*MnO*	*Sb_2O_3*	*P_2O_5*	*SO_3*	*Cl*	*CoO*	*CuO*	*NiO*	*ZnO*	*SnO_2*	*PbO*	*Group*
ASL-01	Glass window	68.92	17.74	7.01	2.44	0.64	0.94	0.89	0.19	1.22	0.15	0.10	0.29	1.26	(b)	0.08	n.d.	(c)	0.05	0.13	N/1
ASL-02	Glass window	65.47	19.12	8.34	2.35	0.64	1.33	0.89	0.17	1.86	(a)	0.10	0.47	1.16	(b)	(c)	n.d.	(e)	(f)	(g)	N/2
ASL-03	Glass window	64.31	18.68	8.63	2.59	0.71	1.33	0.97	0.16	2.07	(a)	0.11	0.37	1.10	(b)	(c)	(d)	(c)	(f)	(g)	N/2
ASL-04	Glass window	66.65	17.15	7.27	2.67	0.89	1.28	1.04	0.18	1.83	(a)	0.14	0.43	1.06	0.04	(c)	(d)	(e)	0.07	(g)	N/2
ASL-05	Glass window	65.92	19.01	7.82	2.56	0.66	1.21	1.00	0.14	1.80	(a)	0.11	0.44	1.27	(b)	(c)	n.d.	(e)	(f)	(g)	N/2
ASL-06	Glass window	64.21	18.84	8.25	2.59	0.78	1.44	1.04	0.17	2.12	(a)	0.15	0.46	1.18	(b)	(c)	(d)	(e)	(f)	0.11	N/2
ASL-07	Glass window	66.99	18.28	8.15	2.35	0.66	1.27	0.86	0.16	1.52	(a)	0.09	0.37	1.25	(b)	0.05	n.d.	(e)	(f)	(g)	N/2
ASL-08	Glass window	70.00	18.03	7.01	2.32	0.56	0.64	0.46	0.09	0.58	0.26	0.10	0.29	1.50	(b)	(c)	n.d.	(e)	(f)	(g)	N/1
ASL-09	Glass window	65.89	11.11	12.00	2.47	2.41	3.52	0.81	0.12	1.60	(a)	0.28	0.22	0.79	(b)	(c)	n.d.	(e)	(f)	(g)	A/1
ASL-10	Glass window	69.20	12.80	8.87	2.34	2.47	3.48	0.55	0.07	0.58	(a)	0.21	0.30	1.00	(b)	(c)	n.d.	(e)	(f)	(g)	A/1
ASL-11	Glass window	69.71	14.07	9.08	0.84	2.09	3.15	0.30	0.06	0.57	(a)	0.24	0.30	1.08	(b)	(c)	n.d.	(e)	(f)	(g)	A/1
ASL-12	Glass window	65.71	14.29	9.31	2.05	2.44	4.47	0.56	0.08	0.94	(a)	0.28	0.28	1.12	(b)	(c)	(d)	(e)	(f)	(g)	A/1
ASO-01	Beaker (*nuppenbecher*)	68.78	12.51	9.10	1.87	2.74	3.22	0.49	0.10	0.75	(a)	0.35	0.22	1.15	(b)	(c)	(d)	(e)	(f)	(g)	A/1
ASO-02	Beaker (*nuppenbecher*)	67.33	13.51	10.34	1.35	2.25	3.81	0.55	0.08	0.71	(a)	0.28	0.23	1.17	(b)	(c)	(d)	(e)	(f)	0.11	A/1
ASO-03	Beaker (*nuppenbecher*)	65.92	10.65	12.35	3.06	2.71	2.86	0.47	0.09	1.27	(a)	0.30	0.22	0.94	(b)	(c)	(d)	(e)	(f)	0.12	A/2
ASO-04	Beaker (*nuppenbecher*)	66.97	12.64	10.03	1.41	2.38	4.19	0.38	0.07	0.94	(a)	0.23	0.29	0.98	(b)	(c)	(d)	(e)	(f)	(g)	A/1
ASO-05	Beaker (*nuppenbecher*)	67.29	13.11	10.31	1.46	2.45	3.60	0.40	0.07	0.75	(a)	0.28	0.22	1.10	(b)	(c)	(d)	(e)	(f)	(g)	A/1
ASO-06	Bottle (*kropfflasche*)	67.51	10.51	10.05	3.39	2.52	1.79	0.92	0.15	2.49	(a)	0.30	0.09	1.08	(b)	(c)	(d)	(e)	(f)	(g)	A/3
ASO-07	Bottle (*kropfflasche*)	65.87	13.12	8.54	4.09	2.29	1.83	1.58	0.23	2.52	(a)	0.46	0.10	1.33	(b)	(c)	(d)	(e)	(f)	(g)	A/3
ASO-08	Bottle (*kropfflasche*)	69.22	12.53	8.64	1.58	2.88	3.33	0.54	0.10	1.47	(a)	0.35	0.18	0.93	(b)	0.05	(d)	(e)	(f)	(g)	A/1
ASO-09	Bottle (*kropfflasche*)	70.90	14.74	3.70	2.84	2.10	1.83	1.46	0.36	2.16	(a)	0.42	0.08	1.36	(b)	(c)	n.d.	(e)	0.08	0.17	A/3
ASO-10	Bottle (*kropfflasche*)	66.37	12.96	8.22	3.71	2.31	2.01	1.30	0.21	2.32	(a)	0.43	0.10	1.21	0.03	(c)	n.d.	(e)	(f)	(g)	A/3
ASO-11	Bottle (*kropfflasche*)	70.57	13.26	4.95	2.75	2.47	2.15	1.29	0.32	1.97	(a)	0.39	0.07	1.07	(b)	(c)	n.d.	(e)	0.05	0.14	A/3
ASO-12	Bottle (*anghistera*)	68.45	9.70	11.97	2.73	2.29	2.82	0.45	0.08	1.34	(a)	0.20	0.13	0.96	(b)	(c)	n.d.	(e)	(f)	(g)	A/2
ASO-13	Bottle (*anghistera*)	67.55	9.96	11.79	2.67	2.30	2.86	0.46	0.08	1.44	(a)	0.21	0.24	0.81	(b)	(c)	n.d.	(e)	(f)	(g)	A/2
ASO-14	Bottle (*anghistera*)	68.19	12.00	10.98	1.17	2.24	3.99	0.44	0.07	0.75	(a)	0.25	0.27	1.11	(b)	0.05	n.d.	(e)	(f)	(g)	A/1
ASO-15	Bottle (*anghistera*)	67.03	9.58	12.83	2.67	2.29	2.96	0.43	0.07	0.82	(a)	0.18	0.29	0.91	(b)	(c)	n.d.	(e)	(f)	(g)	A/2
ASO-16	Bottle (*anghistera*)	66.99	13.10	8.32	3.43	2.73	2.29	1.01	0.14	1.68	(a)	0.35	0.11	1.07	(b)	(c)	n.d.	(e)	(f)	(g)	A/3
ASO-17t	Beaker, colourless body	67.79	11.64	11.01	1.59	2.31	3.78	0.52	0.07	0.96	(a)	0.28	0.25	0.97	(b)	0.05	(d)	(e)	(f)	(g)	A/1
ASO-17b	Beaker, blue rim	65.19	11.11	10.55	1.72	2.34	3.66	2.84	0.08	0.92	(a)	0.30	0.24	0.75	0.38	0.66	(d)	0.25	0.05	0.15	–
ASO-18t	Beaker, colourless body	65.49	11.32	11.31	1.68	2.58	3.93	0.69	0.10	2.38	(a)	0.32	0.22	0.85	(b)	0.06	(d)	(e)	(f)	0.11	A/1
ASO-18b	Beaker, blue rim	64.19	10.91	11.18	1.75	2.55	3.89	1.66	0.09	2.24	(a)	0.32	0.20	0.71	0.19	0.49	(d)	0.22	0.05	0.18	–
ASO-19t	Beaker, colourless body	65.53	11.80	10.46	1.58	2.58	4.16	0.73	0.07	2.49	(a)	0.28	0.25	0.85	0.04	0.08	(d)	(e)	(f)	0.13	A/1
ASO-19b	Beaker, blue rim	61.84	10.82	9.75	1.74	2.56	3.83	4.76	0.09	2.23	(a)	0.29	0.21	0.59	0.78	1.22	(d)	0.50	0.07	0.23	–
ASO-20t	Beaker, colourless body	65.56	13.16	10.21	1.43	2.12	4.49	0.87	0.10	1.04	(a)	0.24	0.32	1.08	(b)	(c)	(d)	(e)	(f)	(g)	A/1
ASO-20b	Beaker, blue rim	63.74	12.66	9.72	1.53	2.12	4.27	2.04	0.09	1.03	(a)	0.24	0.32	1.00	0.49	0.81	0.15	(e)	(f)	(g)	–
ASO-21t	Beaker, colourless body	67.77	10.90	9.83	1.10	2.17	3.28	1.15	0.06	2.68	(a)	0.24	0.25	0.93	(b)	(e)	(d)	(e)	(f)	0.12	A/1
ASO-21b	Beaker, blue rim	64.02	10.11	9.32	1.34	2.15	3.11	4.36	0.08	2.66	(a)	0.25	0.21	0.68	0.72	0.75	(d)	0.50	0.07	0.20	–

9.6–19.1 wt% and 3.7–12.8 wt%, respectively. Early medieval samples have lower potassium and magnesium contents (K_2O = 0.56–0.89 wt%; MgO = 0.64–1.44 wt%) than the others (K_2O = 2.09–2.88 wt%; MgO = 1.79–4.49 wt%) (Table 2). This suggests that the high and late medieval samples, including four window panes and 21 objects (beakers and bottles) were produced using soda-rich plant ash as a network modifier, whereas the early medieval ones, comprising eight window panes, were produced with natron as flux.

Natron glass

Some interesting observations may be made about the Asolo natron glass, in spite of the low number of samples. As shown in the plots in Figure 3, they fall into two groups with differing chemical characteristics, called for convenience groups N/1 and N/2. Group N/1 contains only two pale blue panes; group N/2 contains six panes, yellowish-green in colour. With respect to group N/2, group N/1 has higher SiO_2 contents (69.46 ± 0.77 wt% versus 65.59 ± 1.16 wt%; Table 3) and lower MgO and MnO (MgO = 0.79 ± 0.21 wt% versus 1.31 ± 0.08 wt%, MnO = 0.90 ± 0.45 wt% versus 1.87 ± 0.21 wt%; Table 3—see also Figs 3 (a), 3 (b) and 3 (d)). Both groups are consistent with the major compositional groups of natron glasses identified in the first millennium AD in the western Mediterranean (Table 3). Group N/1 is both similar to 'Group 3' of Foy *et al.* (2003) and 'Group A2/1' of Silvestri *et al.* (2005) (Fig. 3 and Table 3), including Roman and early medieval glasses found in the West; glasses of these groups are thought to have been made from coastal sands of the Syro-Palestinian region, probably near the mouth of the Belus river (Foy *et al.* 2003). Unlike reference Groups 3 and A2/1, group N/1 has higher Sb_2O_3 (0.21 ± 0.8 wt%), which is below the EPMA detection limit in group N/2 (Table 3 and Fig. 3 (c)). Antimony, together with manganese, was the main decolouring agent used in Roman times but, from the end of the third century AD, it was gradually replaced by manganese (Jackson 1996; Silvestri *et al.* 2008). Therefore, taking into account the chronological pattern of Group N/1 samples (dated to the 7th–10th centuries AD), the presence of Sb_2O_3 in Asolo early Middle Ages coloured glass is mostly suggestive of recycling of Roman colourless glass, and not of its intentional addition as a decolourizer. The practice of recycling of earlier glass has already been observed for early Middle Ages glass from other Italian sites (Mirti *et al.* 2000; Verità *et al.* 2002). Following Silvestri (2008), a recycling index (RI) was calculated, with a value of about 24%, indicating that the group N/1 samples were probably produced by recycling about 24% of colourless antimony glass in the batch.

Group N/2 shows a good match with 'Group A2/2' of Silvestri *et al.* (2005) (Figs 3 (a), 3 (b), 3 (c) and 3 (d); see also Table 3), composed of HIMT glass from north-east Italy and dated to the fifth to eighth centuries AD. HIMT glass (high iron, manganese and titanium) is defined by high levels of iron (≥0.7 wt%), manganese (usually ~1–2 wt%), magnesium (usually ≥0.8 wt%) and titanium (≥0.1 wt%), with a positive correlation between Fe and Al, and its yellow–green colour is due to levels of iron, suggestive of a relatively impure sand source (Foster and Jackson 2009). These are also the key characteristics of group N/2, which does have high contents of Fe_2O_3 (0.97 ± 0.07 wt%), MnO (1.87 ± 0.21 wt%) and MgO (1.31 ± 0.08 wt%), together with positive correlations between Fe_2O_3 and Al_2O_3 (R^2 = 0.89). The acronym HIMT was first used by Freestone (1994) for raw glass from Carthage and glass vessels from Cyprus (Freestone *et al.* 2002), although a glass with high contents of iron, manganese and titanium was first identified by Sanderson *et al.* (1984). This kind of glass is also common in Britain, the western Mediterranean and Egypt (Foster and Jackson 2009), France (Foy *et al.* 2003) and Italy (Mirti *et al.* 1993; Silvestri *et al.* 2005; Arletti *et al.* 2010).

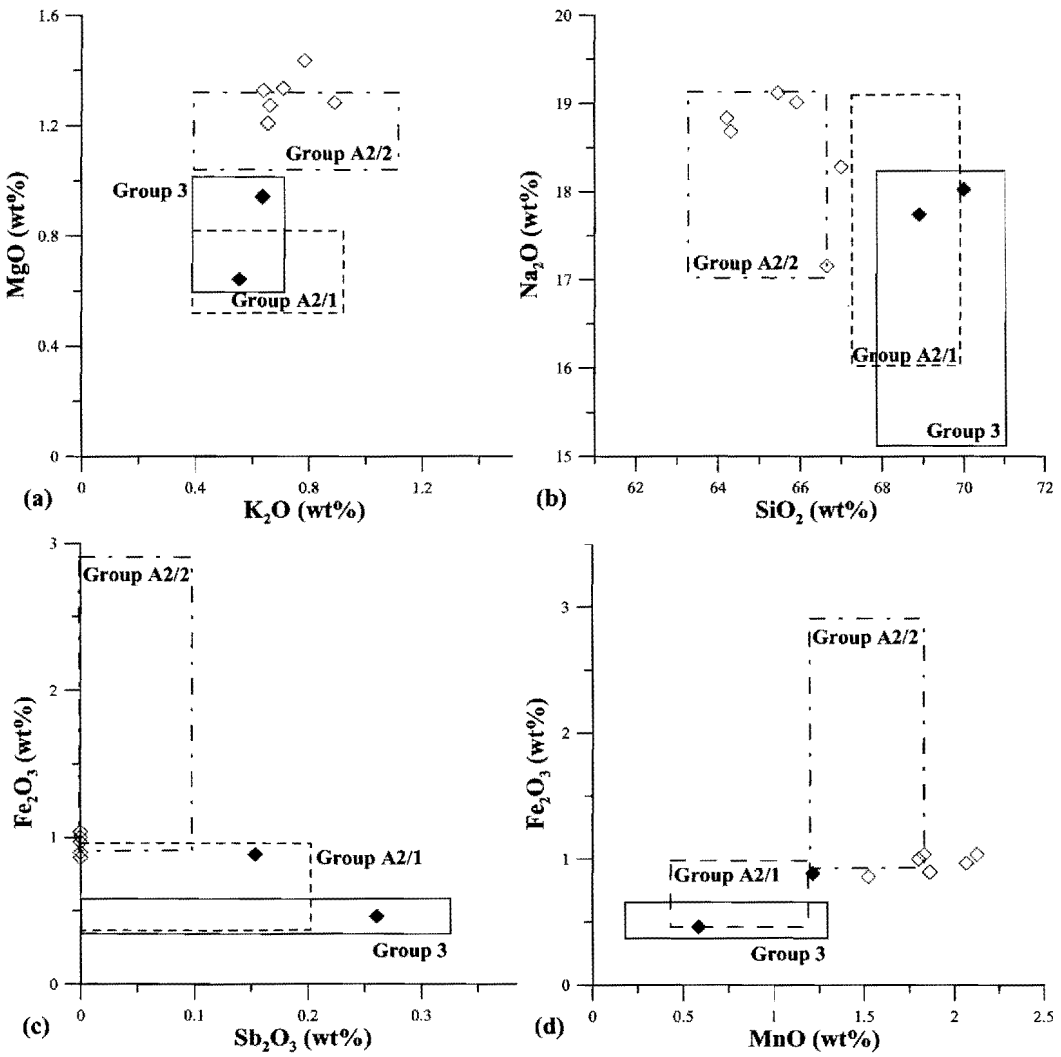

Figure 3 *Plots of samples of groups N/1 (◆) and N/2 (◇): (a) MgO versus K_2O; (b) Na_2O versus SiO_2; (c) Fe_2O_3 versus Sb_2O_3; (d) Fe_2O_3 versus MnO. Plotted areas refer to Group 3 (continuous line), according to Foy* et al. *(2003), and to Groups A2/1 and A2/2 (dotted line), according to Silvestri* et al. *(2005).*

Soda ash glass

Ash glass from Asolo was obtained with ash from coastal plants, which introduces high levels of Na_2O (9.58–14.29 wt%; Table 2) and low levels of K_2O (2.09–2.88 wt%; Table 2) when compared with wood ash ($Na_2O = 0.89 \pm 0.99$ wt%, $K_2O = 13 \pm 5$ wt%) (Wedepohl *et al.* 2011). The high level of CaO (3.70–12.83 wt%; Table 2) is also due to plant ash and not to the carbonatic fraction of sand, as confirmed by analyses of Levantine plant ash, which typically have high CaO (Brill 1970; Ashtor and Cevidalli 1983; Verità 1985). During the Middle Ages,

Table 3 *Mean chemical compositions in weight per cent (element oxides) and standard deviations for identified groups. Also reported: comparisons between the chemical composition of identified groups (bold) and those of natron and plant ash glass identified in the western Mediterranean from the mid-first millennium AD to the mid-second millennium AD (italic) (Group 3 from Foy* et al. *2003; Groups A2/1 and A2/2 from Silvestri* et al. *2005; Venetian glass 'low-Al' and 'high-Al' from Verità and Zecchin 2009)*

	Group N/1 (N = 2)	*Group 3*	*Group A2/1*	*Group N/2 (N = 6)*	*Group A2/2*	*Group A/1 (N = 15)*	*Venetian glass, 'low Al'*	*Group B/1*	*Group A/2 (N = 4)*	*Group A/3 (N = 6)*	*Venetian glass, 'high Al'*
SiO_2	**69.46 ± 0.77**	*69.36 ± 1.64*	*68.53 ± 1.38*	**65.59 ± 1.16**	*65.03 ± 1.59*	**67.36 ± 1.48**	*67.26 ± 1.58*	*68.25 ± 1.15*	**67.24 ± 1.05**	**68.04 ± 2.17**	*66.68 ± 1.54*
Na_2O	**17.88 ± 0.20**	*16.77 ± 1.55*	*17.42 ± 1.49*	**18.51 ± 0.73**	*18.17 ± 0.97*	**12.49 ± 1.03**	*12.75 ± 1.40*	*12.00 ± 0.91*	**9.97 ± 0.48**	**12.95 ± 1.37**	*11.39 ± 2.43*
CaO	**7.01 ± 0.00**	*7.81 ± 0.94*	*7.21 ± 0.77*	**8.07 ± 0.48**	*7.73 ± 1.10*	**10.10 ± 0.98**	*8.97 ± 1.53*	*9.51 ± 0.91*	**12.24 ± 0.46**	**7.30 ± 2.43**	*10.25 ± 2.80*
Al_2O_3	**2.38 ± 0.09**	*2.53 ± 0.35*	*2.53 ± 0.31*	**2.52 ± 0.14**	*2.78 ± 0.17*	**1.59 ± 0.44**	*1.47 ± 0.58*	*1.66 ± 0.20*	**2.78 ± 0.19**	**3.37 ± 0.51**	*3.17 ± 0.68*
K_2O	**0.60 ± 0.06**	*0.55 ± 0.14*	*0.67 ± 0.26*	**0.72 ± 0.10**	*0.79 ± 0.39*	**2.41 ± 0.22**	*2.32 ± 0.50*	*2.26 ± 0.16*	**2.40 ± 0.21**	**2.40 ± 0.22**	*2.37 ± 0.28*
MgO	**0.79 ± 0.21**	*0.62 ± 0.14*	*0.62 ± 0.14*	**1.31 ± 0.08**	*1.18 ± 0.14*	**3.76 ± 0.44**	*3.28 ± 0.57*	*3.32 ± 0.25*	**2.88 ± 0.06**	**1.98 ± 0.20**	*2.71 ± 0.50*
Fe_2O_3	**0.67 ± 0.30**	*0.51 ± 0.15*	*0.67 ± 0.23*	**0.97 ± 0.07**	*1.92 ± 0.99*	**0.60 ± 0.22**	*0.51 ± 0.21*	*0.65 ± 0.24*	**0.45 ± 0.02**	**1.26 ± 0.25**	*0.74 ± 0.45*
TiO_2	**0.14 ± 0.07**	*0.07 ± 0.02*	*0.11 ± 0.04*	**0.16 ± 0.01**	*0.28 ± 0.09*	**0.08 ± 0.02**	*0.11 ± 0.06*	*0.10 ± 0.02*	**0.08 ± 0.01**	**0.23 ± 0.09**	*0.13 ± 0.08*
MnO	**0.90 ± 0.45**	*0.73 ± 0.58*	*0.81 ± 0.37*	**1.87 ± 0.21**	*1.51 ± 0.30*	**1.24 ± 0.72**	*0.97 ± 0.52*	*1.33 ± 1.05*	**1.22 ± 0.27**	**2.19 ± 0.32**	*1.31 ± 0.44*
P_2O_5	**0.10 ± 0.00**	*0.13 ± 0.10*	*0.11 ± 0.07*	**0.12 ± 0.02**	*0.13 ± 0.09*	**0.27 ± 0.04**	*0.34 ± 0.09*	*0.21 ± 0.08*	**0.22 ± 0.05**	**0.39 ± 0.06**	*0.35 ± 0.13*
Sb_2O_3	**0.21 ± 0.08**	*0.09 ± 0.24*	*0.06 ± 0.13*	**<0.06**	*0.03 ± 0.07*	**<0.06**	*n.r.*	*0.00 ± 0.01*	**<0.06**	**<0.06**	*n.r.*

N, number; n.r., not reported.

coastal plant ash was reported to have been imported into Italy from the eastern Mediterranean (Levantine ash) (Verità and Zecchin 2009) or from near Alicante in Spain (Frank 1982, 2–10). Because of the different nature of the soil and of the plants used, these two types of ash produce different glass compositions: Spanish ash yields glass with a Na_2O/K_2O ratio of about 2, and Levantine ash glass in which the Na_2O/K_2O ratio is about 5 (Cagno *et al.* 2008, 2010). In the Asolo ash glass samples, the Na_2O/K_2O ratio varies from 4.2 to 6.7, suggesting that Levantine ash was used in their production. These data may support the hypothesis of Venetian provenance: from the end of the 14th century, Asolo was under the influence of Venice, the most important Italian glass manufacturer and the main importer of Levantine ash in this period. In Venice, the use of Levantine ash had become mandatory by the early 14th century, because the government was determined to ensure the high quality of Venetian glassware (Jacoby 1993).

On the basis of their chemical characteristics, the Asolo soda ash glass is subdivided into three groups: group A/1, composed of 15 samples, A/2 (four samples) and A/3 (six samples). A first distinction can be made between group A/1 and groups A/2 and A/3 in view of their Al_2O_3 contents: group A/1 has lower Al_2O_3 (1.59 ± 0.44 wt%) with respect to groups A/2 and A/3 (2.91 ± 0.33 and 3.35 ± 0.57 wt%, respectively) (Table 3). As already observed by other authors (Cagno *et al.* 2008, 2010; Verità and Zecchin 2009), this evidence suggests the use of different silica sources to produce Asolo soda ash glass: a purer silica source, such as siliceous pebbles, for the samples of group A/1, and sands richer in feldspars for those of groups A/2 and A/3. The separation into three groups is well illustrated in Figure 4 (a): groups A/2 and A/3, as already mentioned, have higher Al_2O_3 contents with respect to group A/1, whereas the distinction between groups A/2 and A/3 is given by the Fe_2O_3 contents, higher in group A/3 (1.26 ± 0.25 versus 0.45 ± 0.02 wt% in group A/2; Table 3). In addition, group A/3 has lower MgO and higher TiO_2 (Fig. 3 (a)) and MnO (Table 3) than the other groups. These data, particularly the higher contents of Fe_2O_3 and TiO_2, may indicate the presence of greater amounts of heavy minerals in the sand used to produce the samples of group A/3. As shown in Figure 4 and Table 2, group A/1 is mainly composed of window panes and beakers (both *nuppenbecher* and flat-based beakers) and groups A/2 and A/3 comprise the vast majority of the bottles. This evidence is suggestive of a relationship between raw materials and type: a purer sand source, probably originally siliceous pebbles, was employed to produce the more precious products, such as window panes and beakers, and a lower-quality sand was used to produce bottles. The same subdivision into 'low-Al' and 'high-Al' glasses, recognized in the Asolo soda ash samples, has been observed in some Venetian glass, dating to the 11th–14th centuries (Verità and Zecchin 2009) (Fig. 4 (a)), suggesting the possible provenance of Asolo findings from Venice. In particular, groups A/2 and A/3 show a good match with the chemical composition of 'high-Al' Venetian glass, and group A/1 has composition similar to the 'low-Al' group (Table 3). Group A/1 is also similar to group B/1 of Silvestri *et al.* (2005), including medieval glass from Grado (province of Gorizia, north-east Italy) and Vicenza (Table 3), with composition comparable to the 'Islamic' glass found in Israel and Syria and dated to the ninth to 10th centuries AD (Henderson 2002). It is worth noting that the history of Venetian glass-making is closely related to the Levantine (Byzantine and Islamic) tradition, for the importation of both raw glass and raw materials, such as soda ash (Verità and Zecchin 2009). In this context, the compositional homogeneity between ninth- and 10th-century Islamic glass and Venetian glass of the 11th–14th centuries is suggestive not only of a relationship between Islamic and Venetian glass-making, but also of a technological continuity from the early to high/late Middle Ages.

Figure 4 *Plots areas refer to 'h has a character pane; star, blue*

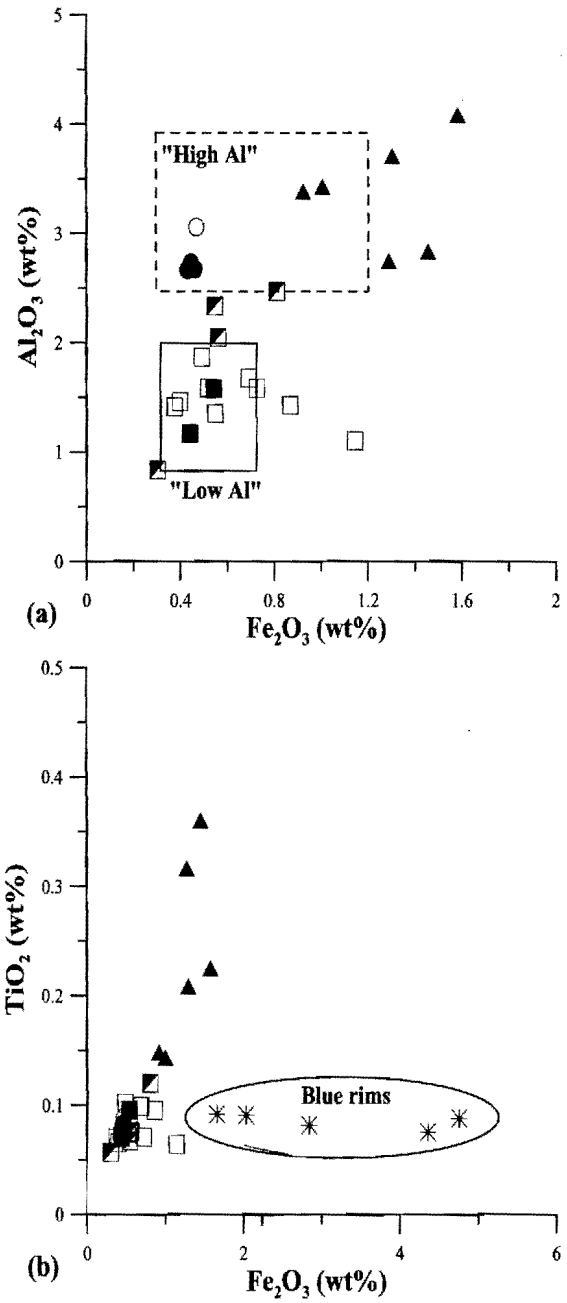

Figure 4 *Plots of samples of groups A/1 (squares), A/2 (circles) and A/3 (triangles): (a) Al_2O_3 versus Fe_2O_3, plotted areas refer to 'high-Al' and 'low-Al' groups, according to Verità and Zecchin (2009); (b) TiO_2 versus Fe_2O_3. Each symbol has a characteristic indicating the type of sample: full symbol, bottle; empty symbol, beaker; halved symbol, window pane; star, blue rim.*

Colouring and decolouring agents

The colour of the vast majority of glass findings varies from green to yellow and pale blue; five beakers have deep blue decorative rims, and were analysed separately (ASO-17b, ASO-18b, ASO-19b, ASO-20b and ASO-21b). Iron was probably the main colouring element and was introduced into the glass as an impurity. The TiO_2 versus Fe_2O_3 plot (Fig. 4 (b)) does show that the contents of these two elements are closely related in most of the samples, indicating that iron was added unintentionally, together with titanium, as mineral impurities in the sand. Manganese was the decolouring agent used deliberately, its contents varying from 0.58 to 2.68 wt% (Table 2). It was added to all samples, since contents above 0.5 wt% are considered intentional additions (Jackson 2005). Group A/3 has the highest percentage of Mn (2.29 ± 0.23 wt%; Table 3), to better contrast the colouring effect caused by high Fe (1.31 ± 0.28 wt%; Table 3). In two samples, ASL-01 and ASL-08, Sb_2O_3 is also present (0.15 and 0.26 wt%, respectively; Table 2): it was one of the main decolouring agents, together with manganese, used in Roman times. However, its contents are too low to be considered as an intentional addition so, as already mentioned, the presence of Sb_2O_3 in some Asolo natron samples indicates recycling of Roman glass.

The five high-Fe and low-Ti samples encircled in Figure 4 (b) are the deep blue decorative rims: in this case, the higher iron content is due to the raw materials added to colour the glass. Except for colouring agents which, according to Mirti *et al.* (1993), may have been added to the glass batch intentionally but are not related to the basic raw materials, blue rims have a chemical composition similar to that of the corresponding colourless body (Table 2). This indicates that the same base glass was used to produce both colourless and coloured (blue) glass, and that it was modified by adding colouring and/or decolouring agents. High percentages of lead (0.11–0.18%; Table 2) were found in three colourless beakers (ASO-18t, ASO-19t and ASO-21t) and are probably additions intended to give brilliance to the glass.

As already mentioned, analyses show that the blue glass was obtained by adding a Co-based colourant to the same glass employed for the colourless body (Table 2). In this context, the elements related to the colourant were quantified by subtracting the composition of the colourless glass from the coloured and possible correlations between them were investigated. In all five blue rims, cobalt correlates with copper and iron (Figs 5 (a) and 5 (b)), suggesting that these elements were associated in the ores exploited to produce the colourant. In four blue rims (ASO-17b, ASO-18b, ASO-19b and ASO-21b), the high cobalt content (0.19–0.78 wt% as CoO) is associated with high Cu (0.78–0.19 wt% as CuO), Fe (0.97–4.03 wt% as Fe_2O_3), Zn (0.22–0.5 wt% as ZnO) and Sn (0.05–0.7 wt% as SnO_2), with a strong correlation between these elements (Figs 5 (a)–5 (d)). Elevated amounts of Pb (0.15–0.23 wt%, as PbO) were also revealed, due to the addition of colourant. These data suggest a Co source linked to lead–zinc ores; as reported by Gratuze *et al.* (1992), the blue glass coloured with this type of raw material forms a homogeneous group dating to the 13th–15th centuries AD. Sample ASO-20b is different from the other blue glass samples: Zn and Sn are not present (Figs 5 (c) and 5 (d)) and Co (0.49 wt%, as CoO) is associated with Cu (0.81 wt% as CuO), Fe (1.17 wt% as Fe_2O_3) and Ni (0.15 wt%, as NiO) (Table 2), suggesting a different source. Three inclusions with irregular shape and rounded edges were observed in this sample (Fig. 6). Their quantitative chemical profiles (Fig. 7), along the black line in Figure 6, indicate that they are basically composed of an association of iron, cobalt and nickel, and are considered to be residues of raw materials added to colour the glass. The chemical composition of the colourless body of sample ASO-20t also differs from those of the other colourless beakers, due to its higher Na_2O, MgO, Fe_2O_3 and lower K_2O and MnO (Table 2), suggesting a different production technology.

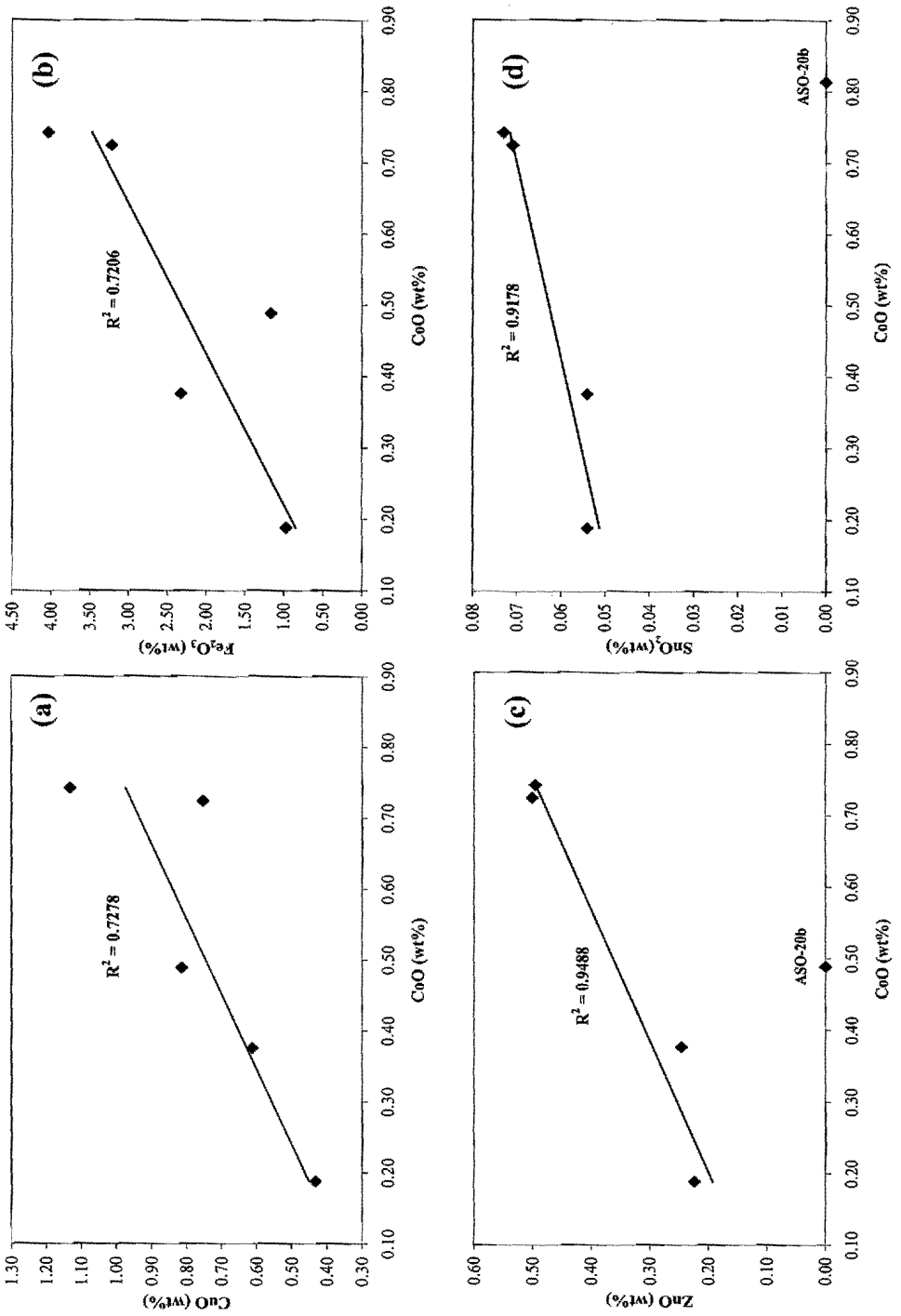

Figure 5 *Plots of (a) CuO versus CoO, (b) Fe_2O_3 versus CoO, (c) ZnO versus CoO and (d) SnO_2 versus CoO (wt%) for blue rims, obtained by subtracting the chemical composition of colourless glass from that of coloured glass (see text for details). The R^2 value is also reported in each plot.*

Figure 6 *A SEM–BSE image of a normal section of sample ASO-20b. The dark grey area is blue glass of a decorative rim. Two inclusions (paler grey) are embedded in glass matrix: the black line is the chemical profile shown in Figure 7.*

CONCLUSIONS

The compositional characterization of 33 samples from the *Rocca di Asolo* shows good matches with the glass production in western Europe in the period between the early and late Middle Ages. All samples are soda–lime–silica glass in composition, with natron as flux for early medieval glasses and plant ash for high and late medieval ones. In the case of soda ash glass, the analytical data indicate possible provenance of the ash from Syria or Egypt (Levantine ash).

The varying colours of the samples are mainly due to differing contents of iron and manganese, except for blue decorations in late medieval beakers, probably achieved by adding cobalt to the same base glass employed to produce the body of the beakers. Analytical evidence suggests that two different sources of Co were exploited.

The combined approach involving analytical and archaeological evidence allowed us to set the different phases of the site in their proper context. Eight flat, thin window panes are attributable to a church built in the area between the sixth and the 10th centuries. They are made with natron and their chemical composition is consistent with Roman and Late Antique glass, testifying to technological continuity from Roman times to the early medieval period. According to their dating, these samples show a predominance of HIMT glass (six samples) with respect to the recycled Roman glass (two samples). A change occurred in raw materials in the fourth century, and at least two new glass compositions, HIMT and Levantine, were introduced into the Roman world and continued in production until the late first millennium AD.

The four window panes of geometric shape and all tableware objects, dating to the high/late Middle Ages and produced with soda ash as flux, belong to the fortification that replaced the

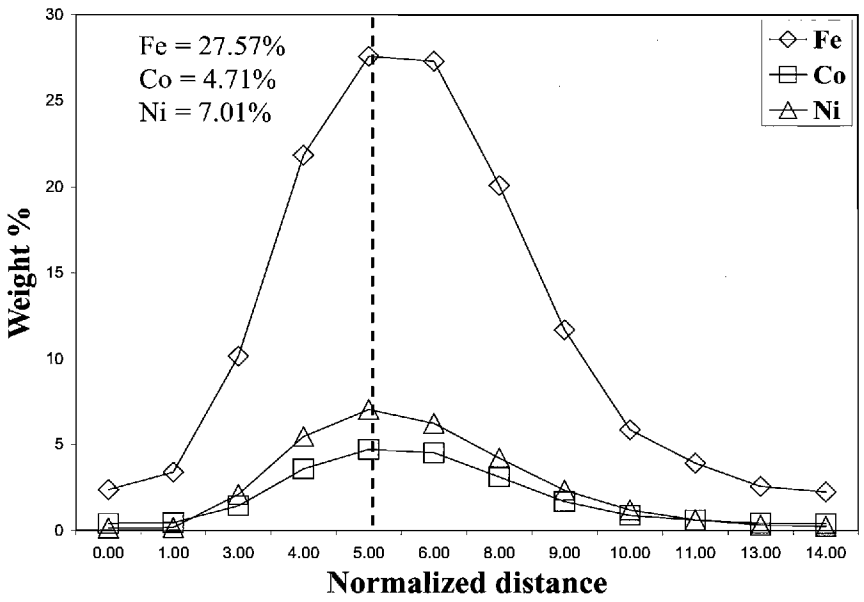

Figure 7 *The chemical profile (EPMA analysis) of one inclusion in sample ASO-20b. Data are expressed in weight per cent of elements. The dotted line shows the Fe, Co and Ni contents of the central point of inclusion, which is also reported.*

church in the 12th century. At least three different silica sources were employed in their production. A purer silica source, probably siliceous pebbles, was used for window panes and the vast majority of the beakers, whereas two types of sand, richer in feldspars, one particularly rich in heavy minerals, were mostly employed to produce bottles. This evidence suggests a correlation between chemical composition and type, although a larger number of data, based on both chemical and typological analysis, is necessary to support this hypothesis. For the window panes, no relationships were found between chemical composition and production technique (crown and cylinder process), either for natron or soda ash glass.

The similarity with Venetian glasses of the same period suggests a Venetian origin for the high/late medieval findings from the *Rocca di Asolo*. In addition, the good match between Asolo ash findings and ancient glass from Grado and Vicenza, of 'Islamic' composition, may be another indication of Venetian provenance, due to the well-attested relationship between Venetian and Islamic glass-making, and testifies to technological continuity from the early to high/late Middle Ages. This chemical evidence also matches the political situation of the *Rocca* which, from the end of the 14th century until its decay, was under the influence of the Venetian Republic, one of the most important centres of glass manufacture and trade in western Europe.

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