

Influence of composition on some industrially relevant properties of traditional sanitary-ware glaze

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Abstract

Two series of glazes have been produced from different combinations of the same raw materials in the range of interest for sanitary-ware applications: they are designed to allow one to get insight into network-forming and network-modifying species. Fusibility tests and hot stage microscope observations show the influence of even low differences in the starting chemical compositions on the transformation temperatures. X-ray powder diffraction, wavelength dispersion spectrometry and scanning electron microscopy prove that: (i) zircon, the most abundant crystalline phase, is homogeneously distributed and decreases by a 3% from its starting value; (ii) the glass-phase of glaze has a quasi-uniform composition. X-ray synchrotron radiation micro-tomography shows that glaze porosity is 15% by volume, and voids are prevalently not interconnected and with size up to 50 μm . The linear thermal expansion of the glass phase of glaze ranges between 6 and $7 \times 10^{-6} \text{ }^\circ\text{C}^{-1}$, without apparent correlation with composition.

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1. Introduction

Sanitary-glazes are technological materials that play a crucial role in the aesthetic quality and durability of the final ceramic output [1,2]. Glaze is generally constituted by a dominant amorphous phase in which other crystalline phases (such as zircon, diopside, wollastonite and quartz) may be dispersed so as to provide opacity and give a thermal shrinkage tallying with that of the ceramic bulk [3–6]. The properties of glaze depend on a variety of aspects, and in such a view the studies reported in literature deal with many topics that can be roughly gathered into the following main classes: the effects of raw materials and process conditions on the glaze formation and its performances [7–15], the role of the glaze rheology as a function of technological parameters [16–18], the occurrence of defects and flaws degrading the glaze performances [19,20], the formation of complex micro-structures that influence the macroscopic

response of a body to external thermal–mechanical forces [21], and the glaze-support/glaze-environment interactions, along with the related phenomenology [22–24]. A recent and exhaustive review on glass-ceramic glazes used for covering and pavement ceramics is reported in [25].

The assessment of the quality of a glaze mainly relies on:

- (i) fusibility behaviour and rheological properties, which have an important part being related to the adhesion and diffusion of a raw glaze on the ceramic support upon firing. Such an aspect influences both durability and surface roughness of the final product;
- (ii) colour and opacity, which usually have to be homogeneous in a ceramic body and are crucial to confer appeal to a ceramic piece on market. It is therefore fundamental to characterize glaze in terms of spatial distribution of optically active species and voids, the latter influencing also mechanical properties and durability;
- (iii) thermal expansion that must be consistent with the one of the coated body to prevent surface versus bulk shrinking-mismatches upon cooling, giving rise to cracks and surface flaws which hamper the marketability of the product.

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In this light the results here discussed refer to a total of 42 different glazes, which mimic common industrial compositions in sanitary-ware and are produced using firing-time/firing-temperature formation conditions representative of the whole-sale production. Data redundancy is a key to bring to light correlations that are hard to be observed if variables lie on a narrow interval but condition and steer the practice of use of a glaze.

This study has been carried out using a multi-methodological approach including X-ray powder diffraction (phase quantification), flowability measurements, hot-stage microscopy (glaze evolution upon heating; sintering (T_{sin}), softening (T_{soft}), sphere (T_{sph}), half-sphere ($T_{\text{1/2sph}}$), melting (T_{melt}) temperatures), scanning electron microscopy (micro-structure characterization), wavelength-dispersion spectrometry (chemical composition and mapping) and thermal dilatometry.

Moreover, X-ray synchrotron radiation micro-tomography (SR micro-CT) has been successfully used to investigate glazes, providing also 3D-quantitative morphological analyses of sample volumes: total bulk porosity value, pore size distribution and morphological characterization of voids were measured. Such results, coupled with those from other analytical techniques, supply important pieces of information for better predicting the physical–chemical behaviour of the glazes during the industrial sanitary-ware production.

2. Experimental procedure

2.1. Materials and processing

Two sets of sanitary-ware glazes based on different initial mineralogical compositions were prepared combining the same raw materials. The first series (addressed to as **F**; 21 samples) is tailored in order to allow one to explore the role of the main network-formers, i.e. SiO_2 and Al_2O_3 ; the second one (**M**; 21 samples) has been designed paying more attention to the network modifiers, i.e. CaO , ZnO , MgO , Na_2O and K_2O . Table 1 shows the chemical molar compositions (calculated according to the Seger formula) of the **F**- and **M**-series on the basis of the mineral phases of the starting blends (quartz, feldspar, kaolinite, zircon, calcite, wollastonite, zinc oxide, talc). In each sample, a further 0.4 wt% bentonite has been added as a thickener agent.

After 45 min of grinding in a corundum jar-mill, about 10 g of each glaze precursor blend have used for flowability tests and hot stage microscopy observations. The remainder has been first treated in humid conditions with a flunger and then cast according to two shapes:

- cylinders, with length and diameter of 5 and 0.4 cm, respectively, for thermal dilatometry measurements and X-ray powder diffraction;
- 2D-slabs (1 mm thickness), deposited on an ordinary ceramic tile (50 wt% clay, 25 wt% feldspar and 25 wt% quartz) by means of an airbrush, for electron microprobe analyses, scanning electron microscope pictures and micro-tomography images.

Table 1
Starting molar composition in oxides of the raw glazes on study.

Sample	Chemical composition (mol normalized)							
	SiO_2	Al_2O_3	CaO	MgO	K_2O	Na_2O	ZnO	ZrO_2
F1	67.22	7.20	13.90	2.59	2.30	1.89	0.93	3.97
F2	68.16	7.20	12.90	2.61	2.31	1.90	0.93	3.99
F3	69.26	7.21	12.31	2.49	2.21	1.82	0.89	3.81
F4	70.32	7.22	11.71	2.37	2.10	1.75	0.85	3.66
F5	71.41	7.23	11.11	2.26	2.00	1.68	0.80	3.51
F6	72.51	7.25	10.52	2.14	1.89	1.61	0.76	3.32
F7	73.63	7.27	9.92	2.02	1.78	1.53	0.72	3.13
F8	74.73	7.28	9.33	1.91	1.68	1.46	0.67	2.94
F9	75.85	7.30	8.72	1.79	1.57	1.39	0.63	2.75
F10	76.97	7.31	8.12	1.67	1.46	1.31	0.59	2.56
F11	73.39	5.80	10.87	2.19	1.95	1.59	0.79	3.43
F12	75.62	5.81	9.68	1.96	1.74	1.44	0.70	3.05
F13	77.86	5.84	8.48	1.72	1.52	1.29	0.61	2.67
F14	66.70	7.02	13.73	2.77	2.46	1.99	0.99	4.34
F15	68.27	7.18	12.81	2.59	2.30	1.88	0.93	4.04
F16	69.59	7.33	12.03	2.44	2.16	1.80	0.87	3.80
F17	70.80	7.46	11.31	2.30	2.03	1.71	0.82	3.57
F18	68.89	6.56	12.83	2.59	2.30	1.86	0.93	4.05
F19	70.39	6.70	11.95	2.42	2.14	1.76	0.86	3.77
F20	71.67	6.82	11.21	2.27	2.01	1.67	0.81	3.54
F21	72.81	6.94	10.54	2.14	1.89	1.59	0.76	3.33
M1	66.69	7.02	13.87	3.61	1.94	2.00	0.52	4.37
M2	66.69	7.02	13.89	2.66	2.11	2.14	1.13	4.36
M3	66.68	7.02	14.11	2.03	2.40	2.39	1.00	4.37
M4	66.68	7.02	14.30	3.00	2.05	2.09	0.50	4.37
M5	66.71	7.02	14.34	2.31	2.05	2.08	1.15	4.34
M6	69.53	7.32	11.99	2.77	2.07	2.12	0.32	3.88
M7	69.56	7.32	12.05	1.99	2.13	2.17	0.93	3.84
M8	69.57	7.32	12.47	1.70	2.09	2.13	0.88	3.84
M9	69.61	7.33	12.44	2.30	2.11	2.15	0.27	3.79
M10	68.84	6.55	12.81	3.20	2.05	2.06	0.37	4.12
M11	68.84	6.55	12.82	2.90	2.20	2.19	0.38	4.12
M12	68.88	6.56	12.96	2.77	1.85	1.89	1.03	4.07
M13	68.80	6.55	13.31	2.31	1.92	1.95	1.00	4.15
M14	68.80	6.55	13.28	2.57	2.16	2.15	0.34	4.16
M15	68.81	6.55	13.25	2.88	2.05	2.07	0.24	4.14
M16	68.82	6.55	13.28	2.21	1.96	1.98	1.06	4.14
M17	71.63	6.82	11.30	2.61	1.86	1.91	0.28	3.60
M18	71.60	6.82	11.24	2.04	1.87	1.92	0.88	3.64
M19	71.60	6.82	11.21	2.71	1.83	1.89	0.31	3.63
M20	71.57	6.82	11.55	1.73	1.89	1.94	0.84	3.65
M21	71.61	6.82	11.57	2.20	1.90	1.94	0.34	3.62

The samples have been fired following the cycle shown in Fig. 1 that consists of a 5 °C/min heating ramp up to 1200 °C, a soaking time of about 50 min and a cooling rate of about 6 °C/min.

2.2. Fusibility behaviour

The fusibility behaviour has here been determined in terms of:

- flowability tests relating to the grade of viscosity of the melted glaze. Some 5 g green glaze are deposited on a 45° inclined ceramic tile and fired following the thermal cycle of Fig. 1. The distance the glaze stretches is measured in mm. This measurement is commonly used to get hints about the quality

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