



Haft rang or cuerda seca? Spectroscopic approaches to the study of overglaze polychrome tiles from seventeenth century Persia



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ABSTRACT

Seventeenth century polychrome overglaze *haft rang* tiles found in various regions of Iran (Isfahan, Qazvin, Mashhad, and Mazandaran) were investigated with ultraviolet–visible spectroscopy (UV–Vis), portable X-ray fluorescence (pXRF), energy dispersive X-ray spectroscopy (EDS), and micro-Raman spectroscopy. Two types of glazes were identified, namely low lead-alkali and high lead glazes, in which cobalt, copper, iron, and manganese were used as colourants. Tin oxide and lead-tin yellow were recognised as white and yellow opacifiers respectively. The black line, which was used for delineating the tiles' design, was mainly composed of manganese, iron, and aluminium oxides. The high maturing temperature of this line prevented the low-temperature coloured glazes run together during the firing. Finally, technological differences between *haft rang* and *cuerda seca* techniques are highlighted and *minai* overglazes are briefly compared with *haft rang* overglazes.

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1. Introduction

Polychrome overglaze ceramics have a nearly continuous line of history in Islamic Persia. In the twelve century AD, a technique of overglaze polychromic ceramic emerged in central Iran, Kashan, which was called *haft rang* (e.g. 'seven colours') (Abu'l Qasim, 1301), nowadays known as *minai* (Fehérvári, 1973). After going into oblivion, a similar technique of *haft rang*, i.e. *lajvardina*, was used in the thirteenth and fourteenth centuries (Ritter et al., 1935) whose extensive use is reported from Takht-i Sulayman, north-western Iran (Osete-Cortina et al., 2010). In the fifteenth century, overglaze polychrome ceramics were widely used in north-eastern Iran, under the Timurids (1370–1507 AD) (O'Kane, 2011). In the seventeenth century, the boom of the art of tile-making in the lands under the Safavids (1501–1736 AD), *haft rang* tiles extensively covered the facades of public and royal buildings in the Safavid headquarters (Savory, 1980) including Qazvin, Mashhad, and Isfahan. This tradition was never stopped and experienced another period of immense use in eighteenth and nineteenth century Persia, under the Qajars (1785–1925 AD), when it was called *haft rangi* (Ali Mohamed, 1888). These overglaze polychrome glazed tiles are

still in massive production in today's Iran under the name *haft rang* (Holakoei et al., 2012).

It is not clear when and how the Spanish term '*cuerda seca*' was used to describe the Persian technique of *haft rang*. Referring to the polychrome ceramics found in Samarra (Iraq) and Susa (Iran), Délerý (2009) believes that the Spanish technique of '*cuerda seca*' technique has its origin in the Near East. The fact is that the term '*cuerda seca*' is a new term which is recently used in the archaeological literature. Gestoso and Pérez in 1904, based on a document from the archives of the Cathedral of Seville, proposed the term '*cuerda seca*' which was referred to describe this decorative technique (Suárez, 2007). '*Cuerda seca*' literally means 'dry cord' or, as O'Kane (2011) suggested, 'colourless line' and refers to a black line which delineates polychrome designs (Zamora, 2007). The nomination of the Persian technique to '*cuerda seca*' is based on the fact that in both techniques a black line, mainly composed of manganese compounds, was used to keep separated different coloured glazes. The term '*cuerda seca*' is commonly used in the art and architecture literature to describe the polychrome glazed tiles used in Iran from the fifteenth century onwards. Despite this fact, there is only a report published by Tite and Salter (2011) which briefly highlights the technological differences between the overglaze Persian polychrome tiles and those Spanish polychromes. In this study, various approaches of ultraviolet–visible (UV–Vis) spectroscopy, portable X-ray fluorescence (pXRF), energy dispersive X-ray spectroscopy (EDS), and micro-Raman spectroscopy were used

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to shed light on the technological features of seventeenth century Persian *haft rang* tiles. Finally, the technological characteristics of *haft rang* technique is compared to the already published data on the Spanish *cuerda seca* (Pérez-Arantegui et al., 1999; Chapoulie et al., 2005).

2. Materials and methods

2.1. Samples

Forty three *haft rang* tile sherds were collected from nine sites in Isfahan, Mashhad, Qazvin, and Mazandaran zone (including Amol and Behshahr cities) all dated to the seventeenth century. More than 250 coloured glazes were applied on these tiles comprising of white, brown, yellow, black, green, turquoise, blue, and violet colours. Nine sites were subject of sampling: Jame' Abbassi Mosque, Bethlehem Church, Ali-Qapu Palace, Esfahanak collection storage, the collection storage of the Iran Cultural Heritage, Handcrafts, and Tourism Organization (ICHHTO) (all in Isfahan), *madrassa* of Abbas-Qoli Khan, Mossalla complex (all in Mashhad), Cheshmeh Emarat Palace in Behshahr, Mir-Bozorg tomb in Amol (all in Mazandaran zone), and Jame' Mosque of Qazvin. The samples were labelled according to the city and the site from which they were collected. The first letter in the samples' labels stands for the city, the second letter for the site of sampling, and the numerical part of the labels shows how many samples were collected from each site. The label of each sample can be found in a separate column in Table 1. The photos and labels of the samples are provided in Fig. 1.

2.2. UV–Vis spectroscopy

Over 460 *diffuse* and *total* reflectance measurements of clean surfaces of the coloured glazes were acquired in the range 360–740 nm using a Konica Minolta spectrophotometer CM-2600D

equipped with a barium sulphate integrating sphere with $d/8^\circ$ configuration. Colour coordinates were plotted in the CIEL*a*b* colour system, with reference to the illuminant D65 and spectroscopic data were processed with the SpectraMagicNX software. The spots where the UV–Vis spectra were acquired from are represented with small red (in the web version) and black circles in Fig. 1.

2.3. pXRF microanalysis

A portable ARTAX™ 200 XRF spectrometer from Bruker AXS Microanalysis GmbH was used for qualitative microanalysis of the glazes. The instrument was equipped with an X-ray tube with a Mo target placed at 6° , a 100 μm beryllium window, and a maximum voltage and current of 50 kV and 1500 μA respectively. A SSD Peltier-cooled detector (10 mm^2 active area and resolution of <155 eV at 10 kcps) was used for the detection of the secondary fluorescence X-rays. The X-ray beam on the surface of the sample had a diameter of 1 mm. The measurements were performed in air for about 120 s.

2.4. Micro-Raman spectroscopy

An Olympus BXM microscope coupled to a LabRam HR800 spectrometer (Horiba Jobin Yvon, France) was used to acquire Raman spectra using an air-cooled CCD detector (1024 \times 256 pixels) at -70°C . The spectrometer had a focal length of 80 mm and the acquisitions were performed with a 600 grooves/mm grating. A He–Ne laser (632.8 nm) was used as excitation source with the laser power between 0.2 and 4 mW and the exposure time of between 5 and 10 s with 10 accumulations. Spectra were recorded by placing the samples on the X–Y motorised stage and observing them with 50 \times and 100 \times objectives. The spectrometer was calibrated and checked with silicon at 520 cm^{-1} using LabSpec 5 software.

Table 1
The results of EDS microanalysis on the *haft rang* glazes (normalised to 100%).

n.	Glaze	Label	Na ₂ O	MgO	Al ₂ O ₃	SiO ₂	K ₂ O	CaO	Fe ₂ O ₃	MnO	CoO	PbO	CuO	SnO ₂	ZnO	PbO/SnO ₂	Na ₂ O/K ₂ O	Maturing T (°C) ^a
1	Blue	I.C.5	18.6	1.9	1.7	65.9	4.7	4.2	2.1	n.d. ^b	0.2	0.7	n.d.	n.d.	n.d.	n.c. ^c	4.0	770
2	Blue	I.E.5	6.1	1.4	3.6	66.6	4.7	5.0	4.2	n.d.	0.8	6.3	1.4	n.d.	n.d.	n.c.	1.3	942
3	Blue	I.S.8	12.6	1.2	4.2	58.8	12.0	3.6	3.9	n.d.	0.4	3.4	n.d.	n.d.	n.c.	1.0	787	
4	Turquoise	I.A.2	15.0	2.1	7.2	54.4	4.1	6.5	2.4	n.d.	n.d.	5.4	3.0	n.d.	n.c.	3.7	819	
5	Turquoise	I.A.3	17.7	1.5	4.8	47.3	5.1	4.5	2.3	n.d.	n.d.	8.6	2.7	5.6	n.d.	1.5	3.4	708
6	Turquoise	I.A.7	20.9	3.5	4.2	52.3	3.2	3.5	n.d.	n.d.	n.d.	11.5	0.8	n.d.	n.c.	6.5	637	
7	Violet	I.A.3	15.1	1.4	3.9	46.1	5.4	4.9	1.6	2.2	n.d.	19.2	n.d.	n.d.	n.c.	2.8	613	
8	Violet	I.C.3	8.5	2.3	2.8	63.1	6.6	6.4	1.7	3.9	n.d.	4.6	n.d.	n.d.	n.c.	1.3	888	
9	Violet	I.E.5	15.4	1.6	5.9	44.0	4.9	5.2	1.3	2.2	n.d.	19.4	n.d.	n.d.	n.c.	3.2	615	
10	White	A.M.1	16.8	2.3	4.9	53.6	4.6	5.3	n.d.	n.d.	n.d.	9.7	n.d.	2.8	n.d.	3.5	3.6	843
11	White	I.C.2	11.7	2.1	2.8	57.4	6.1	6.0	2.1	n.d.	n.d.	7.9	n.d.	3.9	n.d.	2.0	1.9	902
12	White	I.C.7	11.9	1.9	3.6	51.4	8.2	4.9	0.8	n.d.	n.d.	9.3	n.d.	7.9	n.d.	1.2	1.5	844
13	Brown	I.A.7	4.8	1.0	3.0	28.6	2.1	1.9	1.2	0.4	n.d.	46.5	n.d.	10.4	n.d.	4.4	2.3	616
14	Brown	I.C.1	5.3	0.7	2.2	34.4	1.1	0.9	1.2	0.7	n.d.	44.4	n.d.	9.1	n.d.	4.9	4.9	627
15	Brown	I.E.2	6.2	0.4	2.1	33.4	2.0	1.8	1.0	n.d.	n.d.	46.3	n.d.	6.9	n.d.	6.7	3.0	545
16	Brown	I.E.4	2.9	0.7	3.6	35.2	3.5	2.0	1.5	0.2	n.d.	41.4	n.d.	7.3	1.7	5.7	0.8	664
17	Brown	M.A.2	7.8	1.6	6.7	34.3	3.4	2.5	5.3	n.d.	n.d.	38.5	n.d.	n.d.	n.c.	2.3	547	
18	Green	I.A.4	2.4	0.5	2.0	30.4	0.3	1.4	1.6	n.d.	n.d.	44.6	1.0	15.0	0.7	3.0	7.2	755
19	Green	I.A.7	3.8	0.4	2.4	29.0	1.5	2.2	2.7	n.d.	n.d.	49.5	0.5	7.4	0.6	6.7	2.5	551
20	Green	I.C.2	3.7	0.5	2.2	25.8	0.3	2.5	1.4	n.d.	n.d.	54.2	1.5	6.8	1.2	7.9	12.0	497
21	Yellow	I.C.6	3.8	0.5	3.8	29.8	1.3	1.1	n.d.	n.d.	n.d.	50.8	n.d.	9.0	n.d.	5.6	3.1	585
22	Yellow	I.C.7	2.2	0.8	3.1	40.9	2.1	1.9	1.8	n.d.	n.d.	37.0	n.d.	10.1	n.d.	3.7	1.1	783
23	Yellow	I.S.9	8.4	0.6	2.8	35.4	1.9	3.0	n.d.	n.d.	n.d.	40.1	n.d.	7.7	n.d.	5.2	4.5	596
24	Black line	I.A.4	1.4	1.8	7.7	33.3	2.5	7.4	9.9	1.8	n.d.	33.2	1.0	n.d.	n.d.	n.c.	0.6	1168
25	Black line	I.C.4	3.6	3.1	16.8	35.6	4.0	11.7	6.8	11.5	n.d.	7.1	n.d.	n.d.	n.d.	n.c.	0.9	1176
26	Black line	I.E.7	4.5	2.3	12.7	38.9	4.1	4.3	8.8	19.7	n.d.	4.7	n.d.	n.d.	n.c.	1.1	1159	

^a Temperature at which 10^4 P of viscosity is achieved. Calculations based on Fluegel et al. (2006).

^b Not detected.

^c Not calculable.

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