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RESEARCH ARTICLE

Materials and techniques of Islamic manuscripts

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Abstract

Over 50 works on paper from Egypt, Iraq, Iran and Central Asia dated from the 13th to 19th centuries were examined and analyzed at the Straus Center for Conservation and Technical Studies. Forty-six of these were detached folios, some of which had been removed from the same dispersed manuscript. Paintings and illuminations from five intact manuscripts were also examined and analyzed, although not all of the individual works were included. The study was undertaken to better understand the materials and techniques used to create paintings and illuminations from the Islamic World, with particular attention paid to the diversity of greens, blues and yellows present. The research aimed to determine the full range of colorants, the extent of pigment mixing and the various preparatory drawing materials. The issue of binding materials was also addressed, albeit in a preliminary way.

Keywords: Islamic manuscript, Islamic painting, XRF, Raman, FTIR, Imaging, Hyperspectral imaging

Introduction

An ongoing interdisciplinary study at the Harvard Art Museums is investigating the materials and techniques used to embellish folios[1] from Islamic manuscripts and albums created from the 13th through the 19th centuries. Despite a number of studies from the 1970s onwards, technical study of Islamic manuscripts is limited compared to those of European manufacture. Synthesis of results is complicated by references to data from old and/or unpublished reports, which often require re-examination and reassessment. The current project is hence aimed at clarifying our understanding of the materials and techniques used in Islamic art works and relating these to the period and/or place of manufacture. As part of this, data from a number of unpublished student projects were incorporated where possible.

The artworks in this study are listed chronologically in table form (see Table 1) and are referred to by their chart number in this essay. The works draw from the collections of the Harvard Art Museums and from the Villa I Tatti, The Harvard Center for Italian Renaissance Studies in Florence, Italy. Many of the works included in the project have been assigned to a particular town or region and/or are well dated. Examination of folios from a single manuscript, including those now detached and dispersed as well as those still bound as a volume, enabled investigation of the variations between related works. Although the manuscripts and folios studied in the overall project span the Middle East, in this paper we focus on works from Egypt, Iraq, Iran and Central Asia. The chronological range spans the development of early "industrial" pigments in Europe and hence allows assessment of the extent to which these replaced or augmented traditional pigments in the 18th and 19th century. Works from Ottoman Turkey and Mughal India will be considered separately in further publications.

Methodology

The initial phase of the study consisted of thorough visual examination, including raking and transmitted light examination, and photography with visible, ultraviolet (UV) and infrared (IR) radiation, followed by photography of areas of interest at high magnification, ranging from 8 to 40 times, through a Nikon camera attached to a stereo microscope

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[1] The authors are using the term folio to correspond to the cataloguing of the collections. The term does not adhere to the strictest understanding of folio as a paper folded in half, which is one folio, two leaves and four pages.

Table 1 Pigments and underdrawing identified in the Islamic manuscripts studied

Number	Objects	Red/pink	Yellow	Blue	White	Green	Brown	Metals	Other	Underdrawing[b]
1	1971.95.1 1224, iraq Struchnos plants, illustrated folio from the *khawass al-ashjar*	Vermilion; organic red	Pararealgar, realgar	Indigo on plant side, ultramarine on land-scape side	n/a	Orpiment, indigo	Brown-red: ultramarine, organic red Dark brown-red: realgar, pararealgar	n/a	Calligraphy in brown ink, vermilion	Red ink
2	1965.478 1224, iraq Verbascum plants, illustrated folio from the *khawass al-ashjar*	Vermilion; vermilion and orpiment	Vermilion, orpi-ment (bird and plant side) Pararealgar (plant side)	Ultramarine	n/a	Orpiment, indigo	Brown-red: hematite, pararealgar (plant side) Light brown–pararealgar, vermilion (plant side)	n/a	Calligraphy in brown ink, vermilion Purple-red: organic red[a]	Red ink
3	1960.193 1224, iraq Warrior and physician with the plant kestron, illustrated folio from the *khawass al-ashjar*	Vermilion; red lead, organic red	Pararealgar	Ultramarine;ultramarine with copper green	Lead white	Orpiment, indigo Copper green	n/a	Gold	Calligraphy in brown ink, vermilion Purple: unknown with some vermilion	Red ink
4	2002.50.140 Early 13th cen-tury, iraq Fineleaf fumitory, illustrated folio from the *khawass al-ashjar*	Vermilion	n/a	n/a	n/a	Lighter green–orpiment, indigo Darker green–orpiment, indigo, unknown	Red-brown–vermilion, orpiment	n/a	Calligraphy in brown ink, vermilion Purple: unknown, some vermil-ion	Red ink (minor)
5	1960.204 1300s, maraga, iran Mule, illustrated folio from the manafi al-hayawan by ibn bakhtishu	Vermilion; red lead	n/a	n/a	n/a	Orpiment, indigo	Unknown with some vermilion	Gold	n/a	n/a

Table 1 (continued)

Number	Objects	Red/pink	Yellow	Blue	White	Green	Brown	Metals	Other	Underdrawing[b]
6	1960.205 1300s, maraga, iran Falcon, illustrated folio from the manafi al-hayawan by ibn bakhtishu	Vermilion	Pararealgar	Azurite, some with malachite inclusions	n/a	Orpiment, indigo	Pararealgar Probably with organic red, carbon black Light brown: pararealgar, vermilion, azurite[a]	Gold	Orange/yellow: pararealgar, vermilion Purple: vermillion, azurite, organic red[a] Gray: carbon black	n/a
7	1919.130, 1955.167, 1956.212, 1957.193, 1958.288, 1960.190 1330s, tabriz, iran Illustrated folios (6) great ilkhanid shahnama by firdawsi	Vermilion; organic red Pink: red lead, lead white orpiment	n/a[a]	Ultramarine	Lead white	Orpiment and indigo	Hematite, vermilion	Gold; silver-rich gold Silver	Orange: vermilion, orpiment; red lead, vermilion; red lead, hematite	
8	Villa i tatti 1330s, tabriz, iran Prince isfandiyar reproaches his father, gushtasp, illustrated folio from the great iklhanid shahnama by firdawsi	Vermilion; Vermilion, red lead; Red lead; Organic red Pink: vermilion, lead white, red lead, organic red[a]	n/a	Ultramarine, sometimes mixed with lead white	Lead white	Orpiment and indigo; copper green, lead white	Hematite, vermilion Hematite, vermilion, red lead Hematite, vermilion, orpiment	Gold Silver	Orange: red lead, vermilion, orpiment Purple: organic red, ultramarine, red lead; organic red, ultramarine, indigo; organic red, ultramarine; organic red, ultramarine, lead white gray: lead white, carbon black	Black ink

Table 1 (continued)

Number	Objects	Red/pink	Yellow	Blue	White	Green	Brown	Metals	Other	Underdrawing[b]
9	1960.96.1 C. 1330s, baghdad, iraq Mihras offers nushirvan the treasure of rum, illustrated folio from the shahnama by firdawsi	Vermilion Pink: lead white, vermilion	Orpiment	Indigo, ultramarine	Lead white	Rare copper green	Brown: orpiment, vermilion	Gold Silver	Orange: red lead, vermilion	
10	1960.186.1 1341, isfahan, iran Illustrated folio from the mu'nis al-ahrar fi daqa 'iq al-ash'ar (recto) 1960.186.2 1341, isfahan, iran Illustrated folio from the mu'nis al-ahrar fi daqa 'iq al-ash'ar (verso)	Vermilion; organic red Pink: vermillion, lead white, sometimes mixed with red lead Pink: vermilion, orpiment, lead white	Orpiment Orpiment	Azurite, sometimes with lead white Ultramarine Azurite; light blue: azurite, lead white	Lead white Lead white	Copper green; orpiment, indigo Orpiment, indigo	Hematite Red-brown: hematite, vermillion	Gold	Orange: orpiment, vermillion Purple[a]: vermilion, orpiment, indigo	
11	1965.476 1354, cairo, egypt Pump for raising water, illustrated folio from the kitab fi ma 'rifat al-hiyal al-handasiya by al-jazari	Vermilion Pink: vermilion, lead white	Orpiment (some vermilion)	Ultramarine; ultramarine, azurite; light blue: ultramarine, lead white	n/a	Orpiment, indigo	Vermilion, carbon black	n/a	Orange: vermilion, orpiment	n/a

Table 1 (continued)

Number	Objects	Red/pink	Yellow	Blue	White	Green	Brown	Metals	Other	Underdrawing[b]
12	1919.138 1354, cairo, egypt Female cupbearer, illustrated folio from the kitab fi ma 'rifat al-hiyal al-handasiya by al-jazari	Vermilion Pink: lead white, vermilion	Orpiment (some vermilion)	Indigo	n/a	Orpiment, indigo	n/a	Gold		Orange ink
13	Villa i tatti 1427, herat, afghanistan Anthology for prince baysunghur Manuscript: 4 illuminations, 7 paintings examined	Vermilion, red lead Pink: organic red, lead white, red lead	Orpiment	Ultramarine, sometimes mixed with lead white	Lead white	Orpiment and indigo; copper chloride, lead white	Hematite, vermilion, red lead Hematite, vermilion, red lead, orpiment Hematite, red lead, orpiment	Gold; silver-rich gold Silver	Orange: red lead Red lead, orpiment Red lead, vermilion Purple: organic red, ultramarine, lead white Red lead, indigo, lead white Grey: lead white and carbon black	Red ink
14	Villa i tatti 1436, shiraz, iran Timur receives guests, illustrated folio from the zafarnama by sharaf al-din 'ali yazdi	Vermilion Vermilion, red lead Red lead; organic red Pink: organic red, lead white, red lead	Orpiment	Ultramarine, sometimes mixed with lead white	Lead white	Orpiment and indigo; copper green, lead white	Hematite, vermilion Hematite, vermilion, red lead Hematite, vermilion Red lead, orpiment	Gold	Orange: red lead Purple: organic red, ultramarine, red lead Organic red, ultramarine, indigo, lead white Organic red, indigo Organic red, ultramarine, lead white	Orange or black ink[a]

Table 1 (continued)

Number	Objects	Red/pink	Yellow	Blue	White	Green	Brown	Metals	Other	Underdrawing[b]
15	1960.198 1436, shiraz, iran Timur celebrates his conquest of delhi, illustrated folio from the zafarnama by sharaf al-din ‘ali yazdi	Red lead, vermilion; vermilion; organic red	Orpiment	Ultramarine, lead white	Lead white	Orpiment, indigo; copper green	Lead white, red lead, vermilion, orpiment; red lead, indigo; orpiment; red lead, carbon black	Gold Silver	Orange: red lead, vermilion; orpiment, vermilion, lead white	Dry black carbonaceous material Brown-orange ink Same manuscript as i tatti
16	1936.29 C. 1440, iran Iskandar’s mother weeping over his bier, illustrated folio from the shahnama by firdawsi	Vermilion	Orpiment	Ultramarine	Lead white	Green: copper green Other green: orpiment, indigo	Hematite	Gold	Orange: red lead	
17	1960.199 C. 1475–1500 Herat, afghanistan Assault on a castle	Vermilion, red lead Pink: lead white, organic red, vermilion	Orpiment	Ultramarine, lead white	Lead white	Orpiment, indigo Atacamite Atacamite, malachite and ultramarine antlerite, ultramarine	Hematite, vermilion, red lead, sometimes mixed with lead white and/or orpiment	Gold; silver-rich gold Silver	Orange: red lead; red lead, vermilion; red lead, orpiment Purple: vermilion, ultramarine, organic red; lead white, organic red, ultramarine Gray: lead white, carbon black	Dry black carbonaceous material Black ink (very dry brush)

Table 1 (continued)

Number	Objects	Red/pink	Yellow	Blue	White	Green	Brown	Metals	Other	Underdrawing[b]
18	57.1965 C. 1477, shiraz, iran Rescue of sa`d vaqqas and dalafruz, illustrated folio from the khavaran-nama by muhammad ibn husam	Vermilion	Orpiment	Ultramarine	Lead white	Ultramarine, lead white, copper green; orpiment, indigo	Hematite	Gold	Orange: red lead Purple: ultramarine, organic red	
19	1956.23 C. 1477, shiraz, iran Qanbar delivers `ali's letter to mir sayyaf, illustrated folio from the khavaran-nama by muhammad ibn husam	Vermilion	Orpiment	Ultramarine	Lead white	Ultramarine, lead white, copper green; orpiment, indigo	Hematite, orpiment, ultramarine[a] Dark brown, hematite, vermilion	Gold	Orange: orpiment, vermilion Purple: ultramarine, organic red	
20	Villa i tatti 1480s, shiraz, iran Kay kavus crowns his grandson kay khusraw, illustrated folio from the shahnama by firdawsi	Vermilion; Vermilion, red lead, organic red; red lead Pink: organic red, lead white, red lead	Orpiment	Ultramarine, sometimes mixed with lead white Ultramarine, indigo	Lead white	Orpiment and indigo; copper green, lead white	Hematite Hematite, vermilion, red lead	Gold	Orange: red lead Purple: organic red, ultramarine Organic red, ultramarine, vermilion Organic red	Black ink
21	1957.148 1493–1494, gilan, iran Afrasiyab consulting his son shideh, illustrated folio from the shahnama by firdawsi	Vermilion	Orpiment	Ultramarine	Lead white	Copper chloride, lead white; orpiment, indigo	n/a	Gold; silver-rich gold Silver	Orange: red lead Purple: lead white, ultramarine, organic red	

Table 1 (continued)

Number	Objects	Red/pink	Yellow	Blue	White	Green	Brown	Metals	Other	Underdrawing[b]
22	1965.481 15th century, shiraz, iran Battle of the clans, illustrated folio from a manuscript of sufi poetry	Vermilion Pink: organic red[a], lead white	Orpiment	Ultramarine, sometimes mixed with lead white	Lead white	Copper chloride; orpiment, indigo	Hematite; orpiment, lead phase	Gold Silver	Orange: red lead Purple: ultramarine, organic red	
23	Villa i tatti 1520–30s, shiraz, iran Illustrated manuscript of the shahnama by firdawsi	Vermilion Pink: organic red, lead white	Orpiment	Illuminations: dark: ultramarine, light: ultramarine, lead white Paintings: ultramarine, azurite, lead white	Lead white	Copper chloride , lead white; orpiment, indigo	Hematite, red lead, sometimes mixed with orpiment	Gold; silver-rich gold Silver	Orange: red lead Purple: organic red, red lead, ultramarine, lead white Grey: lead white, carbon black	Black ink
24	2002.50.13 1520–1540, tabriz, iran Afrasiyab and siyavush embrace, illustrated folio from the shahnama by firdawsi	Vermilion Pink: lead white, vermilion	Orpiment; orpiment, vermilion, lead white	Ultramarine	Lead white	Green: antlerite, azurite; malachite, atacamite; malachite, calcite; malachite, atacamite, calcite; ultramarine, malachite, calcite Orpiment, indigo	Hematite	Gold Silver	Orange: vermilion, hematite, lead white; orpiment, lead white Purple: organic red, lead white, ultramarine	
25	1984.750.1 and .2 C. 1530, tabriz, iran Folios (2) from the divan of hafiz	Red lead, Vermilion	Orpiment	Indigo Ultramarine, sometimes mixed with lead white	Lead white	Copper green, sometimes mixed with ultramarine	n/a	Gold	Orange: red lead	Dry-hematite, carbon black, red lead

Table 1 (continued)

Number	Objects	Red/pink	Yellow	Blue	White	Green	Brown	Metals	Other	Underdrawing[b]
26	1958.76 C. 1540s., tabriz, iran Nighttime in a city, folio from an album	Vermilion Pink: red lead, lead white, vermilion; orpiment, red lead, lead white; vermil-ion, red lead, orpiment	Orpiment; orpiment, vermilion; orpiment, lead white	Ultramarine	Lead white	Antlerite, with some azurite; antlerite, atacamite, malachite, azurite in vari-ous mixtures Malachite, cal-cite; orpiment, indigo	Orpiment, red lead, lead white; orpiment, hematite, lead white;	Gold; silver-rich gold Silver	Orange: red lead	Black ink
27	1958.75 C. 1540s, tabriz, iran Nomadic encampment, folio from an album	Hematite, lead white Vermilion; Orpiment, hematite Pink: lead white, organic red	Orpiment; pararealgar with some vermilion	Ultramarine	Lead white	Orpiment, indigo; cop-per green (with some ultramarine)	Orpiment, red lead, lead white; pararealgar, orpiment, lead white; vermilion, pararealgar	Gold; silver-rich gold Silver	Orange: red lead; lead white, orpiment, vermilion Grey: lead white, carbon black	Black ink
28	1979.20 1531–1532 (text), c. 1540 (paintings) uzbekistan and c. 1620 (paintings repainted), india Manuscript of the bustan (orchard) by sa`di	Hematite	Orpiment Folio 119 – indian yellow; possible lead tin yellow	Ultramarine	Lead white	Copper chloride Orpiment, indigo Folio 119-cop-per chloride Unknown lead-tin green (low copper)	Hematite, orpi-ment	Silver Gold, silver-rich gold	Orange: red lead	
29	1957.140 1551, bukhara, uzbekistan Manuscript of the timur-nama by hatifi	Vermilion Hematite, red lead Hematite	Orpiment	Dark: ultramarine Light: ultramarine, lead wnite	Lead white	Copper chloride Light: copper chloride, lead white Orpiment, indigo	Hematite, orpi-ment	Two folios: tin One folio: silver All: gold, silver-rich gold	Orange: orpiment, hematite	

Table 1 (continued)

Number	Objects	Red/pink	Yellow	Blue	White	Green	Brown	Metals	Other	Underdrawing[b]
30	1958.62 C. 1560, mashhad, iran Horseman and running page, illustrated folio from an album	Vermilion Pink: red lead, lead white; red lead, vermilion, lead white	Orpiment with some vermilion	Ultramarine	Lead white	Atacamite, lead white	Hematite, carbon black	Gold Silver	Orange: red lead Gray: lead white, carbon black	Red ink
31	2002.50.28 1562, shiraz The marriage of zal and rudaba, illustrated folio from the shahnama by firdawsi	Red lead Vermilion	Orpiment	Ultramarine	Lead white	Light green: verdigris, lead white Dark green: verdigris, lead white, prussian blue (later), ultramarine, gold, titanium (later)	NT	Gold	Purple: lead white, gypsum, vermilion	Red ink and dry black carbonaceous material
32	2002.50.34 and .35 1575–1590, shiraz, iran Illustrated folios (2) from the shahnama by firdawsi	Red lead Vermilion Organic red	Orpiment, sometimes mixed with other colors	Ultramarine Indigo	Lead white	Orpiment, indigo	Hematite Hematite, lead white, orpiment, bone black, red lead, carbon black, vermilion (branches)	Gold Silver	Flesh: red lead, lead white Purple: lead white, ultramarine, organic red; lead white, ultramarine, orpiment, vermilion, organic red	Red ink, black ink
33	2002.50.26 1600–1650, isfahan, iran Siyavush enthroned in a garden pavilion with attendants, illustrated folio from the shahnama by firdawsi	Vermilion Pink: red lead, lead white	Orpiment	Ultramarine	Lead white	Copper green; orpiment, indigo	Vermilion, hematite, red lead	Gold	Orange: red lead Purple: ultramarine, organic red	Black ink

Table 1 (continued)

Number	Objects	Red/pink	Yellow	Blue	White	Green	Brown	Metals	Other	Underdrawing[b]
34	Villa i tatti, C. 1600, isfahan, iran Illustrated manuscript of farhad and shirin by vashi	Red lead Pink: organic red, red lead, lead white	Orpiment	Ultramarine, sometimes mixed with lead white	Lead white	Copper green, lead white; orpiment, indigo	Hematite, vermilion, red lead; hematite, red lead, orpiment	Gold; silver-rich gold Silver	Orange: red lead, orpiment Purple: ultramarine, organic red, sometimes mixed with lead white Gray: lead white, carbon black	Black ink
35	Villa i tatti, 16th century, bukhara, uzbekistan and 1605–1627, india The assembly of sages (with added right panel)	Vermilion; red lead; red lead, vermilion; red lead, hematite Pink: organic red, lead white	Orpiment Added panel: indian yellow	Ultramarine, sometimes mixed with lead white; ultramarine, azurite Verso: ultramarine	Lead white	Orpiment, indigo; copper-tin–lead green; copper green, sometimes mixed with lead white; copper green, ultramarine; copper green, red lead, vermilion Verso: copper green Added panel: copper green, indian yellow	Hematite, red lead	Gold	Orange: red lead Purple: organic red, ultramarine, red lead Added panel: organic red, azurite, red lead Gray: lead white, carbon black	Black ink
36	1984.463 C. 1669–1670, iran, illustrated manuscript of a compendium of knowledge (jung), made for shah sulayman	Organic red (illumination & text) Red lead (illumination & text) Vermilion (illumination only)	Indian yellow (illumination & text) Orpiment (illumination only)	Ultramarine (illumination & text) Azurite (illumination & text)	Lead white (illumination & text)	Malachite (illumination) Copper green (text only) Copper blue-green (text only)	NT	Gold (illumination & text) Silver (text)	Orange: red lead (illumination & text) Realgar-pararealgar (text only)	

Table 1 (continued)

Number	Objects	Red/pink	Yellow	Blue	White	Green	Brown	Metals	Other	Underdrawing[b]
37	1960.76 C. 1750s, iran Young woman with flower, folio from an album[a]	Organic red Organic red, lead white	Indian yellow	Ultramarine	Lead white	Dark green: indigo and indian yellow Light green: indigo and orpiment	n/a	Gold	Orange: red lead Purple: indigo, lead white, organic red	Traces of dry black carbonaceous media
38	1960.77 C. 1750s, iran Youth with wine cup and ewer and hookah, folio from an album[a]	Organic red	Indian yellow	Azurite (lead white) Ultramarine	Lead white	Indigo and indian yellow	NT	Gold	Orange: red lead	Traces of dry black carbonaceous media
39	1958.143 1859, iran Portrait of five seated clerics, folio from an album	Red lead	Unid.	Prussian blue, lead white Indigo and ultramarine	Lead white	Orpiment and indigo	NT	Gold	Purple: unid., lead white	Graphite
40	1958.234 1894–1895, iran Imam ali with hasan and husayn, folio from an album	Red lead Organic red	n/a	Ultramarine	Lead white	Emerald green; indigo, barium sulfate Emerald green, barium sulfate	n/a	Gold	Orange: red lead	Dry black carbonaceous material
41	1959.207 19th century, iran Nuts and flowers, folio from an album	Red lead, barium sulfate, organic red	Orpiment	Ultramarine	Lead white	Orpiment, indigo	Orpiment, hematite	Gold	Purple: lead white, unid.	Graphite
42	1960.161.47 Mid to late 19th century, iran Qajar youth seated in a chair with waterpipe and sword, folio from an album	Red lead, barium sulfate, organic red	Orpiment, zinc phase present	n/a	Lead white, barium sulfate Calcite	Emerald green	Red lead, hematite	Grey: tin	Orange: red lead	Charcoal

Table 1 (continued)

Number	Objects	Red/pink	Yellow	Blue	White	Green	Brown	Metals	Other	Underdrawing[b]
43	2002.50.9 Mid-19th century, iran Portrait of a youth	Vermilion, lead chromate	Lead chromate, minor vermilion	Lead white, unid.	Lead white	Green (robe trim): chrome green, emerald green Green sleeve: ultramarine, lead chromate, lead white, emerald green	n/a	n/a	Orange: lead chromate, vermilion	Graphite
44	1958.137 Late 19th century, iran Imam 'ali with hasan and husayn	Red lead; vermilion	Orpiment	Ultramarine	Lead white	Emerald green	Red lead, hematite	Brass	n/a	Graphite
45	1958.250.120 Late 19th century, iran Ghazals beauty of the beloved, illustrated folio from the divan of hafiz with two 19th-century miniatures added	Red lead Vermilion Organic red	Orpiment	Ultramarine	Lead white	Orpiment and indigo Copper green	Hematite, red lead	Gold	Purple: unid	

Villa I Tatti: 7 works belong to the Villa I Tatti, The Harvard University Center for Italian Renaissance Studies in Florence. All other works belong to the Harvard Art Museums

NT not tested, *n/a* not applicable (i.e., not present), *Unid* unidentified

[a] Possible colorant. Refers to the last colorant listed if in a list

[b] Usually pale, dilute inks

(Leica Wild M10). Areas of interest included, for example, where pigments were overlapped, where gold was punched, and losses where underdrawing was visible. The authors focused on any area that provided clues to manufacture, order of execution and materials used and this varied from work to work. Non-destructive X-ray fluorescence (XRF) and Raman spectroscopy were then undertaken in multiple areas of each folio using the IR, visible and UV images as a guide to the different pigment mixtures present. The selection of sites was customized to each art work to ensure all colors and mixtures were analyzed non-destructively to assess the full range of materials used. In rare instances, selected areas were sampled where possible, following discussion with the curator, if the material identification remained ambiguous after non-destructive analysis. This was particularly relevant where organic pigments or dyes may have been used. Samples were analyzed by Raman spectroscopy, Fourier transform infrared (FTIR) spectroscopy and scanning electron microscopy with energy dispersive microanalysis (SEM–EDX). In many art works sampling was not possible and not all minor or organic phases were identified. Hyperspectral imaging was undertaken on select manuscripts to compare the results with those from the spectroscopic and other imaging techniques.

The Video Spectral Comparator 8000 from Foster + Freeman (VSC8000) provided an array of techniques for imaging of works of art, including infrared (IR) (from 645 to 925 nm at set intervals), ultraviolet (254, 312, and 365 nm), transmitted UV (365 nm), transmitted and raking light. The use of the VSC allows for a relatively fast examination of features such as underdrawing, changes to composition and pigment fluorescence. Using the VSC in concert with the stereo microscope and with analytical work can provide, for instance, further confirmation of pigment ID, such as when a pigment appears opaque with IR illumination. It is another tool used by the authors to ensure a thorough examination and a particularly useful feature is the ease with which visible, IR and UV images can be captured and compared side by side. Most paintings were only examined on the recto as many are mounted and some could not safely be placed face-down. Similarly, it was not possible to undertake examination with transmitted light on mounted art works.

The XRF system employed is a Bruker Artax XRF spectrometer with a Silicon Drift Detector (SDD) and a rhodium anode X-ray tube. The primary X-ray beam is collimated to give a spot size of 0.65 mm. Spectra were acquired for 100 s live time at 50 kV and 600 μA. A helium flux was used to increase the detection efficiency for light elements (atomic number of potassium and lower). The lowest element detected in any of the manuscripts analyzed was aluminum in the blue pigment ultramarine, which consists of the mineral lazurite ($3Na_2O{\cdot}3Al_2O_3{\cdot}6SiO_2{\cdot}2Na_2S$).

The Raman spectrometer utilized is a Bruker Optics Senterra dispersive Raman microscope with an Olympus BX51M microscope equipped with 20× and 50× long working distance objectives. The Raman spectrometer has three laser sources, 532 nm, 633 nm and 785 nm. The optimum laser source depends on the pigment analyzed but in general, blue and green pigments were predominantly analyzed with the 532 nm laser at a setting of 2 mW or 5 mW power and other colors analyzed with the 785 nm laser at a setting of 10mW power. In some instances, the 633 nm laser was used at a setting of 2mW but did not normally result in improved spectra and in general the other lasers were preferred. The power settings chosen were used since the lowest power settings 0.2 mW and 1 mW failed to give recognizable spectra from any materials. It should be noted, however, that these instrument settings may not be the actual values on the sample, which cannot be measured with the available equipment. Spectra were compared with reference libraries, particularly the RRUFF database, using the Opus software.

FTIR analysis was carried out using a Bruker Vertex 70 infrared bench coupled to a Bruker Hyperion 3000 infrared microscope. The sample was compressed on to a diamond cell (2 × 2 mm) with a stainless steel roller and the sample area defined by double shutters contained in the microscope. An absorbance spectrum was measured and subtracted against a blank background. The spectrum was compared with the Infrared and Raman Users Group (IRUG) database of artist's materials.

The SEM system used is a JEOL JSM-6460LV SEM with an Oxford Instruments x-Max^N (80 mm^2) silicon drift detector (SDD) operated by Oxford INCA software. The samples were run under standard SEM conditions of 20 kV, ca. 1 nA beam current and operated in low-vacuum mode with a chamber pressure of 35 Pa.

Hyperspectral imaging was carried out using a Resonon Pika II pushbroom camera system in which the object was focused onto an imaging slit and dispersed by a grating onto a sensor. The image cube was built up line by line as the camera was translated across the object using an automated stage. The wavelength range of this system is 400–900 nm with a spectral resolution of 2 nm. The manuscript pages were illuminated using tungsten-halogen lamps placed at 45° to the object normal. A Lambertian reflector with uniform response across the spectral range (Epson UltraSmooth Fine Art paper) was used to record the reflectance response of the light source and to convert the image cube into diffuse reflectance via normalization.

Results

The results of the analytical and technical study are discussed below and summarized in Table 1, which also gives the full details for the works studied.

Likely pigment identifications were based on the chemical composition from XRF analysis and were then confirmed by Raman spectroscopy. The in situ XRF analysis was considerably faster and positioning was easier than for the in situ Raman analysis and hence many more points were analyzed by XRF than by Raman. In general, if all points of a given color had identical XRF spectra, only one or two points were analyzed by Raman spectroscopy. The presence of organic colorants (such as red lake pigments, organic yellow pigments or indigo) were inferred from a lack of elements characteristic of the inorganic pigments of the corresponding color and from the Raman spectra for indigo. Raman spectra from red, pink or purple areas colored with an organic red showed a highly florescence background with no identifiable peaks regardless of operating conditions. The behavior in UV illumination also provided an indication of the presence of organic red pigments and in particular for the organic yellow pigment Indian yellow.

Historical sources

The materials used in Islamic paintings are discussed in a variety of historical sources, including Islamic treatises and travelers accounts. Recipes for the manufacture and use of artists' materials in Islamic treatises have been extensively studied [1–9]. The Persian palette was divided into six primary colors, white, yellow, red, green, blue, black, and six metal-based pigments, gold, silver, brass, copper, tin, mica [5–7]. Of these, the inorganic materials can be separated into naturally occurring minerals—orpiment (arsenic sulfide) for yellow, cinnabar (mercuric sulfide), realgar (arsenic sulfide), and various ochres (iron oxides) for red, and lapis lazuli (ultramarine) for blue—and synthetic compounds—lead white (basic lead carbonate) for white, red lead (lead tetroxide) and vermilion/cinnabar (mercuric sulfide) for red, and verdigris (copper acetate) for green. Several organic materials were also used as pigments and include those derived from plants such as rhubarb and saffron for yellow, safflower and brazil wood for red, and indigo for blue, and those produced from insects such as kermes, cochineal and lac for red. Many of the recipes mentioned mixing the pigments with gums for preparation or use. One 16th century Iranian paint box contained lead white, red lead, an organic red, yellow orpiment, organic yellow pigments derived from rhubarb and saffron, copper-based green, ultramarine, indigo and red and brown earth pigments [5, 7].

European visitors to Iran during the reign of the Qajar dynasty (c.1785–1925) published accounts of the local industries, but rarely focused on painting. The French scientist, Olmer, described European watercolors being mixed with gum tragacanth and used alongside traditional pigments such as buckthorn (yellow), cochineal (red), indigo (blue), lamp black and powdered gold, as well as a discussion of dyes used for textiles [10]. Textile dying and dyestuffs were also described by a 20th century German visitor, Wulff, who listed a wide range of dyestuffs derived from plants and insects [11].

Pigments collected in Persia in 1935 by Margaret Forbes Schroeder and by Katherine and Myron Smith, now part of the collection of the Straus Center for Conservation and Technical Studies at the Harvard Art Museums, provide a further source of information. Analysis of these indicated yellow ochre, gamboge and orpiment amongst the yellow pigments, various red to brown ochres, red lead for use as an orange-red, lead white, ultramarine for blue and emerald green as the only green. Several samples contained some barium sulfate, presumably added as a filler, and one sample of gum-arabic was present. Barium sulfate was also found in a 19th century paint box and 20th century paint palette [5, 7].

Paper

The paper supports used for these works were not a focus of this study. However, a few general remarks can be made and a handful of the papers have been studied in the past. The materials employed in Islamic papers have been described in detail by other authors [5, 12].

The papers seen in this study were generally tan in color and burnished. Although many Islamic papers are dyed, this study did not address the materials used for dying papers, which are known from historic recipes [8]. The papers appear to be somewhat thin and finely-made but, as most are mounted and many are nearly fully painted over, a complete examination was not possible. In past studies at the Harvard Art Museums, fibers were taken from one folio of the two 1575–1590 *Shāhnāma* paintings (Table 1, #32), as well as from the 1562 *Shāhnāma* painting (Table 1, #31). Both folios were made from well-beaten bast fibers, as is typical for Persian papers from this period. Both papers were light tan in color with a relatively even fiber distribution and exhibited the characteristic sheen indicative of burnishing. Some of the other works included here were also sampled for an unpublished study available in the Straus Center for Conservation and Technical Analysis (see Table 2). A paper used in 1540s Tabriz contained linen (Table 1, #27) and one from a folio produced around 1560 in Mashad (Table 1, #30) had hemp. As a comparison, in the 23 Persian and Egyptian works in the Vever study, most papers were made

Table 2 Summary of results from previous unpublished projects on Islamic manuscripts at the Straus Center for Conservation and Technical Studies—note that a more limited range of colors or paper fibers was sampled in these projects

14th century

1957.193, *Bahram Gur Hunts with Azada*, Tabriz, 1330–1340, paper–linen, hemp [Alexiou 1989]

1960.186.1&2, *Mu'nis al-ahrar fi daqa 'iq al-ash'ar*, Isfahan, 1341—lead white, azurite, arsenic yellow (probably orpiment), copper green, dark green (probably orpiment + indigo), vermilion, red lead, lead white and red lake, gold [Ali 1984]

1960.192, *Shapur Discovers Mihrak's Daughter at the Well*, Shriaz, 1341, paper–cotton [Alexiou 1989]

1960.195, *Rustam and the Iranian Army Besiege the Fortress of Kafur the Cannibal,* Iran, 1341—lead white, azurite, arsenic yellow (probably orpiment), red lead, red lake [Ali 1984]

15th century

1952.5, *Prince Baysunghur (1399–1433) Slays a Wolf*, Herat, c. 1425–1430, paper–linen, hemp [Alexiou 1989]

1939.225, *Tahmina comes to Rustam's chamber*, Herat, c. 1434, paper–linen, hemp [Alexiou 1989]

1956.23, *Qanbar Delivers `Ali's Letter to Mir Sayyaf*, Iran, c. 1477—lead white, arsenic yellow (probably orpiment) [White 1982]

1972.299, *Two Seated Men*, Herat, c. 1480–1490, paper-hemp [Alexiou 1989]

1960.199, *Assault on a Castle*, Herat, 1475–1500, paper-hemp [Alexiou 1989]

1965.481, *Battle of the clans*, Shiraz, 15th century—lead white, arsenic yellow (probably orpiment) [White 1982]

16th century

1958.79, *Manuscript of the Guy u Chawgan*, Herat, 1523—lead white, ultramarine, arsenic yellow (probably orpiment) [St Laurent Lockwood 1981]; paper–linen, hemp [Alexiou 1989]

1964.149, *Illustrated Manuscript of a Divan of Hafiz*, Tabriz, c. 1530, paper–linen, hemp [Alexiou 1989]

1958.75, *Nomadic Encampment*, Tabriz, c. 1540, paper–linen [Alexiou 1989]

1958.60, *Seated Princess with a spray of flowers*, Tabriz, c. 1540—lead white, ultramarine, copper green?, arsenic yellow (probably orpiment), vermilion, red lake, red lead, red lead + lead white, carbon black, gold [St Laurent Lockwood 1981, Khoury 1985]

1922.75, *Layla and Majnun at School,* Shiraz, c. 1560—lead white, arsenic yellow (probably orpiment) [White 1982]

1958.62, *Horseman and Running Page*, Mashhad, c. 1560, paper-hemp [Alexiou 1989]

1965.482, *Joseph Entertained by Zulaykha's Maids,* Shiraz, c. 1580—lead white, arsenic yellow (probably orpiment) [White 1982]

1936.27, *Young Man in a Blue Cloak*, Qazvin, c. 1587—lead white, ultramarine, red lead, red lake, red lead + lead white carbon black, carbon black + ultramarine + red lake, gold [Khoury 1985]

1936.28, *Sufis by a Mountain Spring*, Isfahan, c. 1590—lead white, arsenic yellow (probably orpiment) [White 1982]

1961.57, *A Man and a Lion in a Rocky Landscape*, Qazvin, 16th century, paper–linen, unidentified [Alexiou 1989]

1960.201, *Woman in a Green Robe, Holding a Spray of Flowers*, Iran, 16th century—lead white, ultramarine, copper green, arsenic yellow (probably orpiment) [St Laurent Lockwood 1981]; paper–linen, hemp [Alexiou 1989]

1955.12, *Isfayndirs Third Course: He Slays the Dragon*, Iran, 16th century—lead white, ultramarine, arsenic yellow (probably orpiment) [St Laurent Lockwood 1981]; paper–linen, hemp [Alexiou 1989]

1958.69, *Three Youths on a Garden Terrace*, Bukhara, mid 16th century—lead white, ultramarine, arsenic green (probably orpiment + indigo), copper green, arsenic yellow (probably orpiment), silver [St Laurent Lockwood 1981, White 1982]; paper–linen, hemp [Alexiou 1989]

1958.74, *Two Youths Under a Tree*, Bukhara, mid 16th century—lead white, arsenic yellow (probably orpiment) [White 1982]

1919.135, *Scene in a Palace*, Iran, late 16th century—lead white, arsenic yellow (probably orpiment) [White 1982]

1919.136, *Battle Scene*, Iran, late 16th century—lead white, arsenic yellow (probably orpiment) [White 1982]

17th century

1966.6, *The Return from the Flight into Egypt,* Isfahan, 1689—lead white, ultramarine, copper green (likely copper chloride), arsenic yellow (probably orpiment), red lake, red lead, vermilion, red ochre, carbon black (soot), gold [St Laurent Lockwood 1981, Khoury 1985]; paper-hemp [Alexiou 1989]

1941.294, *The Return of Zal, Rustam and Gudarz*, Isfahan, 17th century—paper–linen [Alexiou 1989]

18th century

1958.80, *Lovers*, Iran, c. 1700, paper–linen [Alexiou 1989]

Alexiou, Maria "An analysis of paper in Persian royal court and provincial workshop miniatures, 14th-17th C." Unpublished paper, Center for Conservation and Technical Studies Certificate, Harvard University Art Museums, 1989

Ali, Zakaria, "A preliminary report on an analysis of 14 colors from three Islamic miniature paintings" Unpublished paper, Center for Conservation and Technical Studies Certificate, Harvard University Art Museums, 1984

Khoury, Nuha NN, "Technical analysis of pigments in royal Persian miniatures. "Unpublished paper, Center for Conservation and Technical Studies Certificate, Harvard University Art Museums, 1985

St. Laurent-Lockwood, Beatrice, "Comparative examination of sixteenth century Persian and Indian miniatures: pigment analysis" Unpublished paper, Center for Conservation and Technical Studies Certificate, Harvard University Art Museums, 1981

White, Susan M., "Technical examination of yellow pigments and adjacent lead-white deterioration as seen on 16th century Persian miniatures" Unpublished paper, Center for Conservation and Technical Studies Certificate, Harvard University Art Museums, 1982

Pigment analysis was undertaken by polarized light microscopy (PLM) and by scanning electron microscopy with energy dispersive microanalysis (SEM–EDS) and fiber analysis by PLM. Attribution and titles of the art works are those currently given in the database of the Harvard Art Museums and may differ from those in the reports from linen or linen and hemp, with one from Bukhara c. 1600 made from an unidentified grass [13]. Cotton mixed with linen and hemp was found in two folios from Shiraz dated to 1341 [13]. Both raw materials and recycled textiles were identified, consistent with the literature on papermaking from these regions [13]. A more recent study identified 9 examples of 16th to 17th century Iranian paper as mainly made from fibers of flax from rags with the occasional addition of hemp, cotton and jute [5]. Another research project found that 15 samples of 18th to 20th century Iranian paper included hemp, flax, cotton, hardwood and softwood, with the wood fibers potentially from imported papers [12]. In our study, the 19th century papers tended to be either wove or laid, where structure was visible, and most were burnished. In summary, most papers analyzed thus far were made from bast fibers, using a combination of raw and recycled materials and they were burnished before being painted.

It was not part of this study to identify paper sizing. However, one study of the materials used to size Persian paper showed that historical treatises included recipes using fish glue and many vegetable-based options including rice or wheat starch, gum-arabic, gum-tragacanth, mucilage from plants and seeds (including cucumber seeds), fruit juice and sugar syrup [5, 7]. Analysis of 16th century miniatures and 16th and 17th century illuminated manuscripts found only one instance for starch sizing but 8 examples of sizing with cucumber seeds (in one case combined with gum-tragacanth) [5, 7]. Nearly half of the 228 papers examined for the project on 18th to 20th century papers were starch-sized [12], but these papers were not tested for some of the other sizing agents described above.

Artists techniques: underdrawing

The presence of underdrawing was revealed by both infrared imaging and examination of areas of missing pigment under magnification using optical and digital microscopes. Infrared imaging showed underdrawing executed in a carbon-based medium where the overlying pigments were transparent to infrared radiation but could not provide information where infrared opaque pigments were present and/or the underdrawing was executed in an infrared transparent medium—in both these instances underdrawing was sometimes visible in areas of loss. The presence and type of underdrawing is summarized in Table 1. In most cases, underdrawing was executed in either dilute black or red ink, as seen in Fig. 1a, b respectively, although dry black carbon-based underdrawing also occurs, albeit infrequently. In some instances, for example in detached folio from a later 16th century manuscript, also from Shiraz (Table 1, #32), two types of underdrawing were present—a brush-applied red ink and a dry black carbonaceous drawing material. Where analyzed, the red ink underdrawing contained vermilion and was similar to the red vermilion-based paint and inks used elsewhere. Previous studies also noted that colored underdrawing was executed with pigments found elsewhere on the art work [14, 15]. By the 19th century, most of the works studied were drawn first in graphite.

Comparison of the underdrawing with the final image showed that alteration to original compositions was normally minor, being restricted to slight changes to clothing, foliage and/or position of figures. This may reveal the skill of the artists, who did not need to adjust the initial drawing to produce the detailed final works. The fact that changes to the design are infrequent may also reflect a prescribed workshop practice and a relatively set vocabulary of images. In some instances, the underdrawing shows the order in which components were executed, for example placement of a tree over the initial outline of the hills in a painting from Shiraz dated to the 1480s, (Table 1, #20), (Fig. 2a, b).

However, in one folio of a manuscript produced in Isfahan around 1600, (Table 1, #34), infrared imaging revealed more extensive alteration. The lines of the underdrawing in the painting are darker and thicker than normal and many areas have multiple rather sketchy lines suggesting the position and size of features such as the weapons and horses' legs were altered several times, perhaps due to production by a less skilled or more experimental artist than what is usually encountered (Fig. 3a–c). In contrast, the other two paintings in this manuscript have only minor alteration with fine, light underdrawing, as is more typical (for example in Fig. 4a, b). Differences in the pigments used further support the possibility that one of the paintings may have been executed by a different artist (see below).

Artists techniques: layering and mixing

Examination with high magnification optical and digital microscopy provided information on layering or mixing of pigments. Mixing of pigments seems to be more common and the mixtures used more complicated in the 16th to 17th century Safavid art works than in earlier or later examples. The complexity of mixtures used in 16th to 17th century Iran has been commented on by other authors [16, 17]. At all periods, a wide range of shades was created with a relatively limited palette, as is evident from Table 1. In some works, the artists clearly layered pigments to create different hues or produce highlights on broader areas of color. The background color or sky was normally painted first, with areas to contain the other elements left blank. However, in the case of light colored backgrounds the whole area was sometimes filled in and

Fig. 1 **a** Black ink underdrawing in face from an early 14th century manuscript from Tabriz (Table 1, #8). **b** Red ink underdrawing visible in a loss in a painting from 16th century Shiraz (Table 1, #32)

Fig. 2 **a**, **b** Comparison of visible (**a**) and infrared (**b**) images shows the outline of the hills below the tree in a painting from 15th century Shiraz (Table 1, #20)

figures or other details painted over this. White details in thick lead white were normally the last to be executed. Details were applied to faces using red or orange-red ink and/or a carbon-based black ink at a late stage of production. Purple was often created with a mix of blue and red pigments, normally ultramarine and organic red with lead white used to lighten the shade as desired, as observed in other studies [17, 18]. Mixtures of ultramarine and vermilion could also be used for purple [16]. In some instances, the different pigments were simply mixed together as seen in Fig. 5a (Table 1, #13), whilst in others a layer of organic red was covered with a mixture of ultramarine with organic red, as seen in Fig. 5b (from another painting in Table 1, #13). A transparent organic red pigment was often used as a highlight on top of other colors, particularly for details of flowers, leaves or robes, as shown in Fig. 6 (Table 1, #14). Opaque copper greens (sometimes mixed with or on lead white) were often used

Fig. 3 **a**, **b** Comparison of visible (**a**) and infrared (**b**, **c**) images shows the unusual extent of alteration of the initial design, for example in the weapons and horse's legs (shown in detail in **c**), in a painting from an early 17th century manuscript made in Isfahan (Table 1, #34)

Fig. 4 **a**, **b** Comparison of visible (**a**) and infrared (**b**, **c**) images shows the more normal minimal alteration of the initial design in another painting from the same manuscript (Table 1, #34)

as highlights on more transparent duller green colors. Dark blue indigo was also used as a highlight color, applied in thin lines over other colors, often over mixtures of indigo with another color. The painting sequence is clearly shown by the flower in Fig. 7, with indigo lines over the orpiment–indigo green, granular ultramarine over the red lead and lead white, and translucent organic red on top of the other colors. One author noted that ultramarine is difficult to paint over and hence most artists painted this around details [19]. In some areas, a thin layer of wax was used over lead white–ultramarine mixture before gold details were applied to increase adhesion and prevent loss [19].

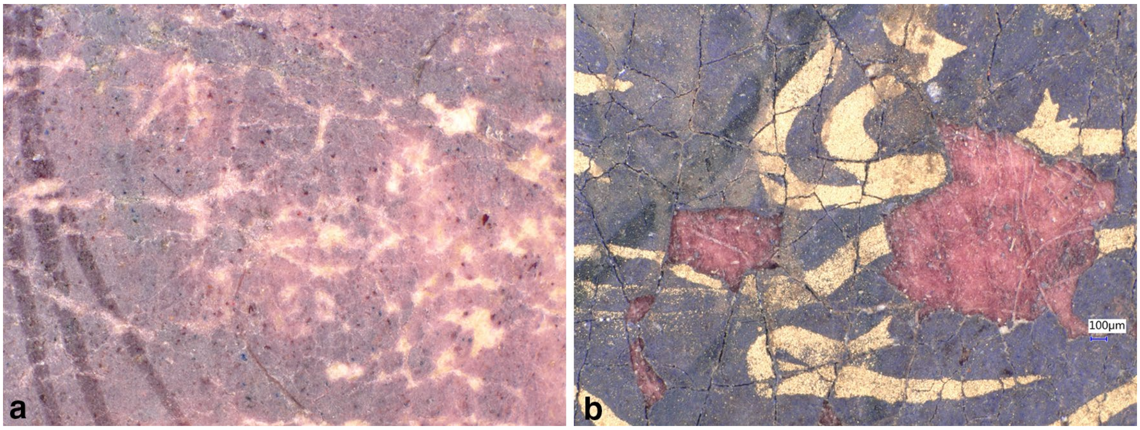

Fig. 5 **a**, **b**: Differences in the production of purple can be seen in paintings in a manuscript from Herat, (Table 1, #13) with organic red and blue simply mixed together (**a**) versus a layer of organic red covered with a mixture of organic red and ultramarine (**b**)

Fig. 6 Organic red pigment applied as transparent highlight over other colors on flowers in a painting from 15th century Shiraz (Table 1, #14)

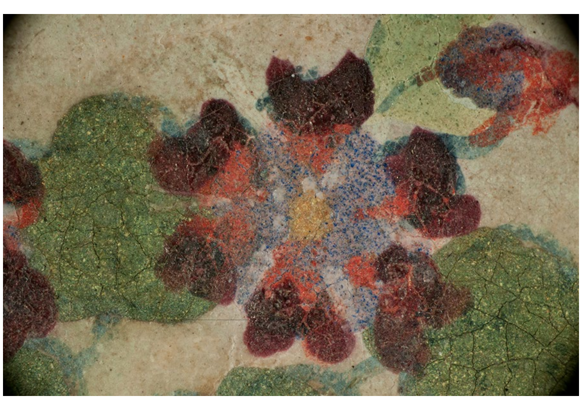

Fig. 7 Detail of flower showing indigo lines over the orpiment-indigo green, granular ultramarine over the red lead and lead white, and translucent organic red on top of the other colors

One commonly suggested workshop model of production for the art works involves specific artists being in charge of distinct assigned elements, which would be undertaken in stages before other elements were executed. Hence, for example, all horses might be done together, followed by people, or architecture, or clothing. In some instances, parts of these were left unfinished, showing that a particular element was executed towards the end of the commission—for example unfinished rocks in folios from a late 16th century manuscript from Shiraz (Table 1, #32), seen in Fig. 8. However, a different production mode can be found on an unfinished painting attributed to the renowned artist Bihzad working in Herat from 1475 to 1500, (Table 1, #17). The work is executed with great precision and shows careful selection of pigments and mixing of these. However, several elements, including the faces of horses and men, shields, and articles of clothing, are unfinished in some areas but finished with exquisite detail in others (Fig. 9a–c). In some instances, the unfinished element is completely blank, in others a first basic wash of color has been applied but no features or details have yet been added. The reason for this apparently unique style is unclear as this does not seem to be characteristic of other works attributed to Bihzad.

Artists' palette

Differences in the infrared behavior of areas of similar color allowed the use of different pigments to be assessed via infrared imaging. This was invaluable for informing the choice of sites for non-destructive analysis. In many folios, a contrast was seen between green foliage (infrared transparent) and green robes (infrared opaque), as shown in Fig. 10a, b (Table 1, #15). Detailed examination and analysis revealed that in most instances the infrared transparent foliage was a mixture of yellow orpiment

Fig. 8 Detail of unfinished rock showing the positioning gridlines in a painting from a late 16th century manuscript from Shiraz (Table 1, #32)

and blue indigo, whilst copper greens were used for the infrared opaque clothing. For the blues, infrared imaging revealed the rare occurrences of infrared opaque azurite in contrast to infrared transparent (and more frequently used) ultramarine and indigo. Similarly, a differentiation was made within the reds between infrared transparent vermilion and red lead versus infrared opaque hematite. Infrared imaging also enabled rapid identification of areas of silver (infrared opaque) from surrounding infrared transparent or reflective pigments where darkening of the original silver had obscured the boundary between the metal and the surrounding details in visible light, for example Table 1, #35 (Fig. 11a, b). Ultraviolet imaging distinguished areas with distinct florescence due to damage or the use of organic materials. In most works, limited florescence was seen despite the presence of organic red pigment layers, which often show some florescence. Ultraviolet imaging was most useful in revealing the presence of Indian yellow in some art works, particularly those with Mughal influence (for example the yellow florescence in Fig. 11c), and of retouching with modern pigments such as zinc white, which allowed these areas to be avoided during analysis.

The materials used showed considerable variation for the blue, yellow and green pigments, with less range amongst the red, orange and brown. The pigments found are documented in Table 1 and discussed below. In addition, art works studied in previous student projects at the Straus Center for Conservation and Technical Studies are summarized in Table 2. A more limited range of colors were analyzed in these early studies. Discussion of the

Fig. 9 **a**–**c** Contrasting finished and unfinished elements in a work by Bihzad, Herat, circa 1475–1500, (Table 1, #17)

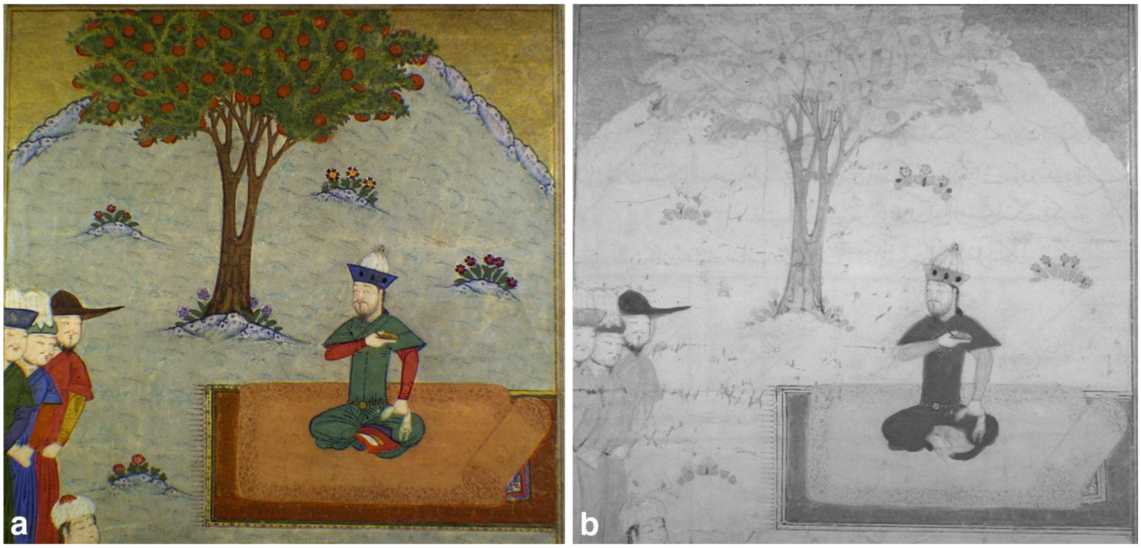

Fig. 10 **a**, **b** Visible (**a**) and infrared (**b**) images showing contrast between green foliage (infrared transparent) and green robes (infrared opaque) (Table 1, #31)

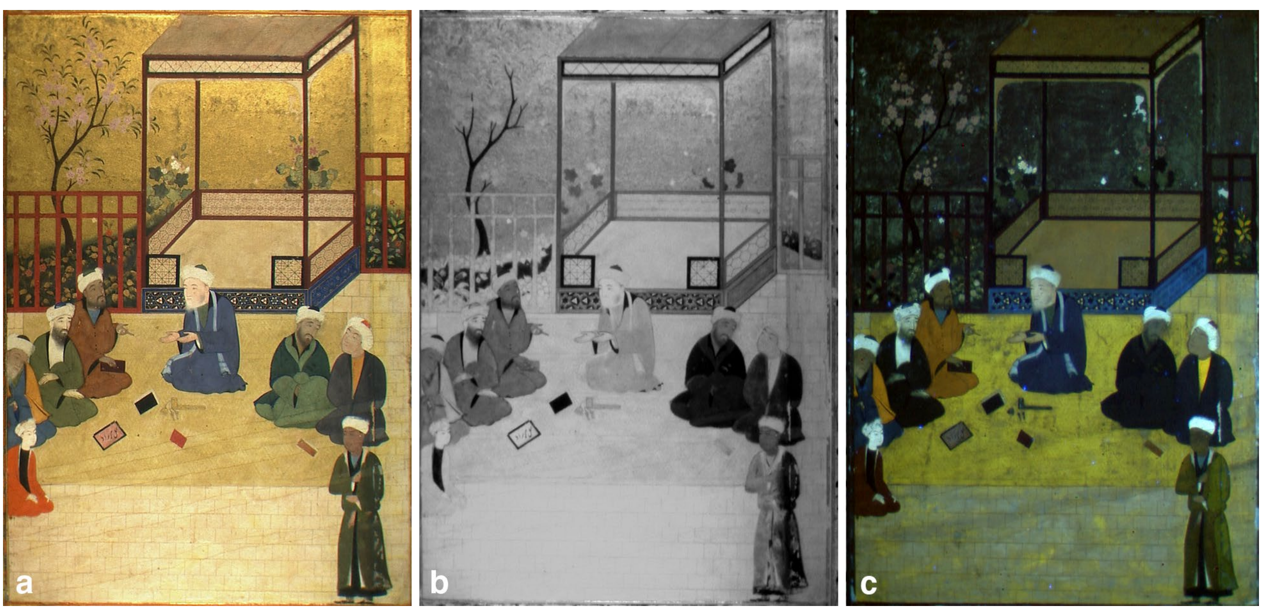

Fig. 11 **a**–**c** Comparison of visible (**a**), infrared (**b**) and ultraviolet (**c**) images from a 16th century folio altered in India in the 17th century (Table 1, #35). The effect of darkening can be seen in the top left where the river is clear in the infrared image but obscured in the visible image. The yellow florescence in ultraviolet light is characteristic of Indian yellow

results and comparison with those from published studies [5, 7, 14–25] is presented later in this paper.

Inks

Most text was executed in black carbon-based (infrared opaque) ink, sometimes with red vermilion-based ink text and accents. However, a brown ink was employed for the text on all the folios examined from a manuscript produced in Iraq in 1224 (Table 1, #1–4). This ink was transparent in infrared and lacked distinctive elements, suggesting used of a natural organic compound. XRF analysis did show slightly more iron in the inked areas but not enough to confirm iron-gall ink. As the ink lacks the characteristic haloing or cellulose degradation

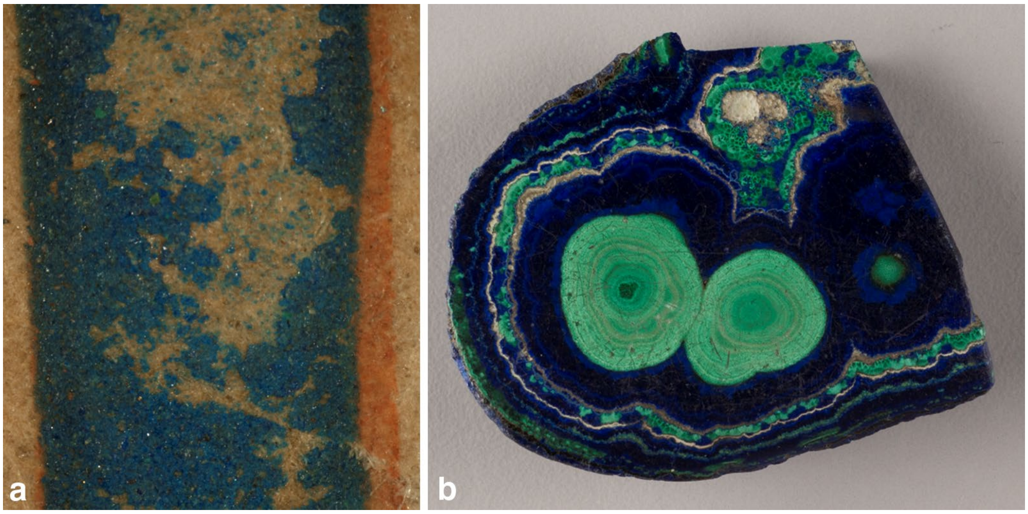

Fig. 12 **a**, **b** Mixtures of azurite and malachite in (**a**) the pigment on a painting from Maraga (Table 1, #6) and (**b**) a natural mineral sample

associated with iron-gall ink, it may be another type of brown organic ink. Historical recipes for the manufacture of Persian inks show that these consisted of multi-component mixtures, many of which contained soot and/or iron gall [26, 27]. Distinction between inks produced with different recipes relied on the use of false color infra-red imaging [26], which was not undertaken in this project. Colored inks were rare, although multiple colors of ink were identified in a manuscript currently attributed to late 17th century Iran, Table 1, #36, and are discussed below. Other studies have found colored inks on Islamic manuscripts from the 15th to 17th centuries to contain malachite, iron gall, red lead, vermilion, ultramarine and a range of yellow and red arsenic sulfides [24, 25].

White: lead white

In all art works examined in the current project, the only white used was lead white—both alone and mixed with other colors to adjust the shade. Zinc white was occasionally found in areas of restoration, for example on one early 15th century work from Shiraz (Table 1, #15) and is clearly revealed by ultraviolet examination.

Black: carbon black

All black pigment was carbon-based but the form (charcoal versus lamp black) was not investigated in detail. However, visual examination and other publications suggest use of lamp black is most likely.

Blue: ultramarine, indigo, azurite, Prussian blue

The dominant blue identified on folios from all periods was ultramarine (lazurite or lapis lazuli). Although the plant-based dye stuff indigo was also used extensively, this rarely occurred as a blue pigment. Instead, indigo was used chiefly in mixtures—with yellow orpiment to create a dull or yellow green or with ultramarine to modify the shade of blue—or for highlights over other colors—particularly indigo lines on clothing and foliage. However, there were some examples of indigo and ultramarine being used alone, for example indigo was mixed with lead white in the sky while ultramarine was used for dark blue areas in Safavid folios from Shiraz (Table 1, #32). Unusually, indigo is the only blue used on one folio from Cairo, 1354, (Table 1, #12) despite the use of ultramarine in a companion folio from the same manuscript, (Table 1, #11), indicating deliberate artistic choice rather than lack of availability of the more commonly used ultramarine.

The copper-based blue pigment azurite was rare, occurring only in 5 of about 50 art works analyzed. Two of these are from 14th century Iran (Table 1, #6 and 10). In the first of these (Table 1, #6), from Maraga and dated to the 1300s, a significant percentage of malachite is present amongst the coarse azurite, as shown in Fig. 12a, suggesting use of a natural mixed ore, similar to that shown in Fig. 12b. The companion folio from the same manuscript, (Table 1, #5), lacks blue areas but both folios contain indigo mixed with orpiment in the darkish green. The other contemporaneous folio, (Table 1, #10) was produced in Isfahan in 1341. The use of azurite rather than ultramarine was probably a local choice specific to the artist or workshop since ultramarine was readily available in 14th century Iran. All folios produced in Tabriz in the 1330s to 1340s as part of the Great Ilkhanid *Shāhnāma*, a lavish royal commission, contain abundant ultramarine with no azurite found on the examples studied here

Fig. 13 **a**, **b** Comparison of visible (**a**) and infrared (**b**) images shows the use of infrared opaque azurite and infrared transparent ultramarine on an 18th century Iranian folio (Table 1, #38)

(Table 1, #7–8). Azurite may be present in other folios from the Great Ilkhanid *Shanama* but is sometimes associated with retouching [14, 18].

Two other works in which azurite was identified have been altered later in their history and the azurite may be associated with this alteration rather than with the original execution. Both date to the 16th century, a manuscript from Shiraz produced in the 1520–1530s (Table 1, #23), and a folio produced in Bukhara and altered in the early 17th century in Agra, India (Table 1, #35). The manuscript from Shiraz (Table 1, #23) contains a number of paintings whose placement in the text suggest these may be later insertions. Comparison of pigments in the original illuminations and in the paintings shows the former contain only ultramarine, whilst the latter have a mixture of ultramarine and azurite. This may reflect either a chronological variation or different palette preferences amongst the illuminators and illustrators.

Significant alteration has occurred in the folio from 16th century Bukhara (Table 1, #35). This has a later insert of paper along the right side and some of the pigments cross over this junction. In one figure with a distinct purple robe, the part on the original folio contains ultramarine whilst the portion on the insertion to the right contains azurite. This difference is clearly revealed by microscope examination, infrared imaging and analysis. Whilst all analyzed blue areas on the unaltered verso contain only ultramarine, a number of areas on the painting, particularly towards the right side and the right side border, contain both azurite and ultramarine. Other blue areas, such as clothing and the borders on the unaltered left, also have ultramarine alone. The azurite in the figures, architecture and right border is hence likely to date from the extension of the folio.

Both azurite and ultramarine were used on an 18th century Iranian folio, (Table 1, #38), but in different areas—ultramarine for the decoration on a blue and white ceramic vessel, azurite for flowers in a glass vessel and the cord attached to this. This is clearly seen in the infrared image which shows the contrast between the infrared opaque azurite and infrared transparent ultramarine in Fig. 13a, b. Representative Raman spectra from the blue animal on the ceramic vessel and the blue braid attached to the glass vessel are shown in Fig. 14. This deliberate pigment choice by the artist may reflect the difference in hue between the darker ultramarine and lighter azurite.

In the 19th century, the synthetic pigment Prussian blue, developed in Berlin in 1704, entered the available palette. Prussian blue was not found on the two 18th century works examined here (Table 1, #37–38) but was identified on one Iranian painting dated to 1859, (Table 1, #39). In this, Prussian blue was used with lead white for

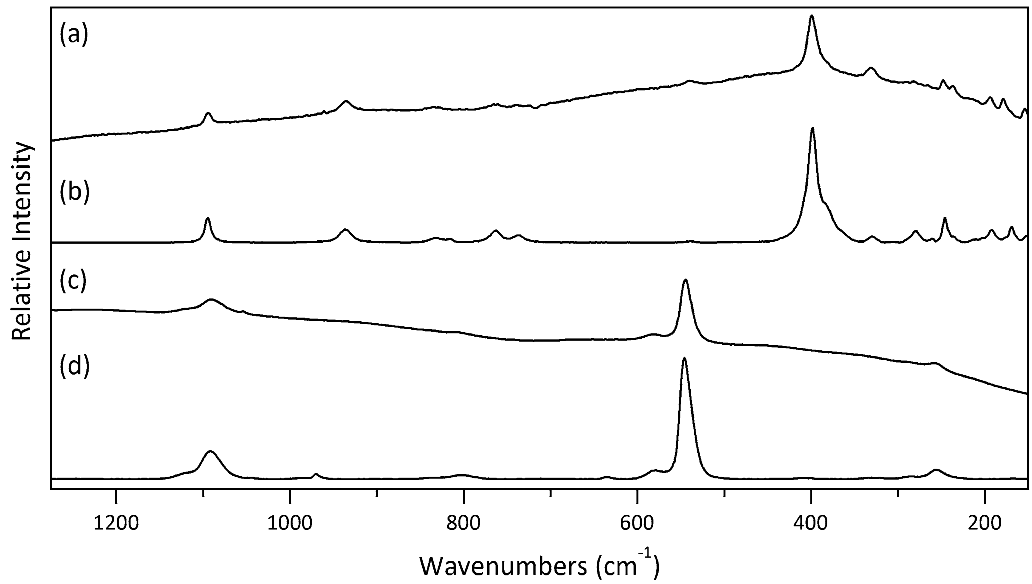

Fig. 14 Raman spectra of blue used for the blue braid of the glass vessel (a) and the blue animal on the ceramic vessel (c) on an 18th century Iranian folio (Table 1, #38), compared to the reference spectra for azurite (b) and ultramarine (d). All reference spectra from RRUFF database http://rruff.info/

the sky, whilst ultramarine and indigo were used in other areas.

Green: orpiment + indigo, copper green (unknown), copper carbonate (malachite), copper chloride (atacamite), copper sulfate (anterlite, brochantite), copper-tin–lead green, Indian yellow + indigo, emerald green, viridian

The greatest range of materials occurs within the green pigments. From the 13th to the 14th century, most green areas were painted with a mixture of orpiment and indigo, producing a dull or yellow green. This mixed green continued to be widely used from the 14th century onwards, particularly for foliage or backgrounds, but is joined by copper greens in the later works. Initially, use of bright copper green was fairly rare and often restricted to details, for example on costly robes or architectural features (Table 1, #3, #7–8, #10). Bright copper greens became more abundant in art works from the 15th century onwards, although orpiment–indigo mixtures remained important. Thus, in two folios from a manuscript produced in Shiraz, 1436, (Table 1, #14–15), copper green is used for the entire robe of the ruler and for solid features such as sleeves and boots with orpiment-indigo used for foliage (as seen previously in Fig. 10a, b). XRF analyses of the 14th and 15th century copper greens indicate the presence of high levels of copper, often with lead from lead white mixed with or possibly underlying the copper green. In-situ Raman spectroscopy of these areas produced only broad florescence with no distinct peaks or a small peak for lead white. FTIR of a small number of samples was inconclusive in identifying the specific copper compound as samples showed some similarity to a variety of copper pigments including copper chloride (atacamite) but did not match any reference spectra closely.

Copper greens are widely used in the later 15th and 16th century, and greatly improved analytical data was obtained from these, partly due the use of purer pigments without admixtures of lead white and other materials. Thus, a variety of copper compounds were identified, including malachite $Cu_2CO_3(OH)_2$, atacamite $Cu_2Cl(OH)_3$, antlerite $Cu_3SO_4(OH)_4$ and brochantite $Cu_4SO_4(OH)_6$. Figure 15 shows representative Raman spectra for antlerite, malachite and atacamite from various folios. In some folios different copper greens were used to create subtle variations in shade, for example in the unfinished 15th century folio from Herat and in two folios from 16th century Tabriz. In the former, (Table 1, #17), a variety of mixtures of copper greens were used to produce robes with subtle variations in color, including atacamite alone, mixtures of atacamite, malachite and ultramarine, and mixtures of antlerite and ultramarine. In the 16th century folios from Tabriz, probably from royal workshops, different mixtures were used for specific features. Thus, in a folio dated to 1520–1530, (Table 1, #24) the main image contains mixtures of antlerite with azurite, the illumination contains a mixture of malachite and coarse calcite and the border contains a mixture of

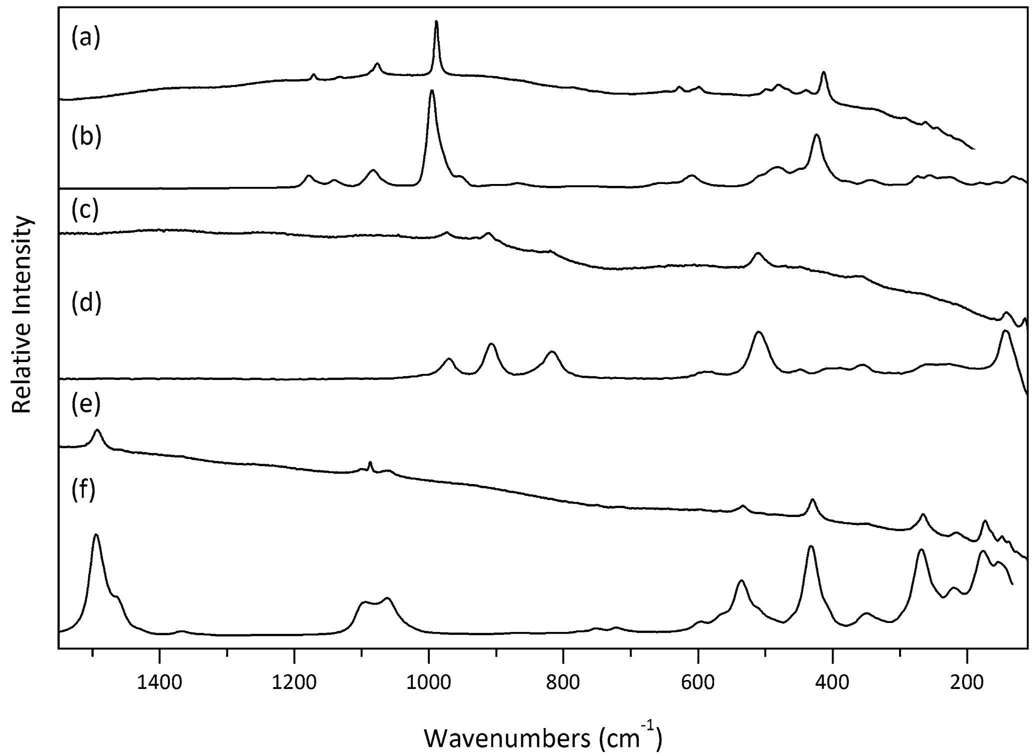

Fig. 15 Raman spectra for green areas from a variety of folios showing the some of the different copper greens identified—a: green from folio Table 1, #24 matched to b: reference spectrum of antlerite; c: green from folio Table 1, #17 matched to d: reference spectrum of atacamite; green from folio Table 1, #26 matched to reference spectrum of malachite. All reference spectra from RRUFF database http://rruff.info/

atacamite and malachite. In contrast, in a detached folio dated to 1540, (Table 1, #26), the copper greens are dominated by antlerite with intermingled azurite, but various mixtures of antlerite, atacamite and/or malachite, and of malachite plus coarse calcite are also found. The malachite mixture is used mainly for floor tiles and architectural features, whilst the antlerite and antlerite mixtures are used for clothing. As with earlier examples, orpiment and indigo mixtures are used for the background as well as for some robes. The specific nature of the green (the chemistry) was likely unimportant to the artists and these different greens were probably selected for specific color properties and are either natural mixtures resulting from use of different ores or the different batches of artificially produced copper corrosion products which varied in the specific phases formed. This rich use of diverse copper greens contrasts with slightly later works from Shiraz of somewhat lesser status than these examples from Tabriz. For example, one folio dated to 1562 (Table 1, #31) contains only green verdigris, whilst others from a less important manuscript dated to 1575–1590 (Table 1, #32) lack copper green and only have mixtures of orpiment and indigo.

A mixed green containing Indian yellow and indigo occurs on the 16th century folio altered at the Mughal court, (Table 1, #35)—where it is used on the insert—and in two 18th century Iranian works (Table 1, #37–38). In all three of these, Indian yellow also occurs as the pure yellow pigment and this use of Indian yellow can be attributed to links with Mughal India (see below). In one of the 18th century works, (Table 1, #37), the Indian yellow and indigo combination was used for dark green details whilst a mixture of orpiment and indigo was used for light green details on the same robe.

The 16th century folio (Table 1, #35) is also unusual in having a green robe which XRF revealed to contain significant copper, lead and tin. A similar green with lead, tin and some copper occurs in another painting produced in Bukhara in 1531–1532 and altered in Mughal India (Table 1, #28). It is possible that this is a synthetic copper green pigment is derived from corrosion of bronze (the copper tin alloy, which can also contain lead) but this has yet to be determined. No peaks were seen in the Raman spectra obtained from these areas. This painting also contains a yellow with high levels of lead and tin, which has yet to be conclusively identified. Two other paintings in this manuscript (Table 1, #28) contain copper chloride and orpiment–indigo green.

Emerald green–copper acetoarsenite, $Cu(C_2H_3O_2)2.3Cu(AsO_2)_2$—was found in several 19th century works, (for example Table 1, #40, 42, 43 and 44). This synthetic pigment was first manufactured commercially in Germany in 1814 and marketed rapidly following this. Despite the popularity of the new green, the traditional mixture of orpiment and indigo was still used for green in some 19th century Iranian works (for example Table 1, #41).

Green areas on one folio from 19th century Iran, (Table 1, #43), had high levels of chromium. In the main robe and sleeves, this occurs with high levels of lead, and there appears to be a mixture of ultramarine, lead chromate, lead white, and possibly emerald green. In contrast, the green decoration on trim to the red robe has high levels of chromium without lead, suggesting the use of viridian—chromium oxide dehydrate—patented in Paris in 1859, again with possible emerald green. The use of viridian, like that of emerald green, confirms an active import market for European pigments in the 19th century.

Fig. 16 Large yellow crystals of orpiment are visible in the mixture of indigo and orpiment used for the majority of the green in this manuscript from 14th century Tabriz (Table 1, #8)

Yellow: orpiment, pararealgar, Indian yellow, chrome yellow

By far the most common yellow pigment identified on works from all locations and time periods was orpiment (arsenic trisulfide, As_2S_3). This was employed both as a pure yellow, often mixed with small amounts of vermilion to adjust the shade, and mixed with indigo to produce green. In most art works, the orpiment used alone had a much finer crystal size than that mixed with indigo, which often contains fairly large rectangular crystals as seen in Fig. 16 (Table 1, #8). Orpiment was also the most common yellow identified in the unpublished studies from the Straus Center for Conservation (Table 2).

Yellow pararealgar (arsenic sulfide, AsS) was found in a number of disparate works - three early folios from Iraq dated to 1224 (Table 1, #1–3), one from Maraga dated to the early 1300s (Table 1, #6), a folio attributed to Tabriz around 1540 (Table 1, #27) and in some yellow text of a 17th century manuscript. Since yellow pararealgar is formed by the light-induced alteration of red realgar (arsenic disulfide, As_2S_2), it is debatable whether the original pigment used was yellow, red or orange (with both phases present originally). Some historical Persian artists' treatises refer to realgar as "red orpiment" showing this was available as an original pigment [6, 7]. Since the preparation methods include drying in the sun, it is likely that some alteration to pararegalgar would occur. In some instances, the yellow pararealgar is seen to be intermixed with red realgar, for example a yellow robe with distinct patches of red realgar on a folio from Tabriz (Table 1, #27). Similarly, unusual colored manuscript text (Table 1, #36) ranges in color from yellow to orange and contains varying proportions of the two phases. Raman spectroscopy shows a greater proportion of the red realgar is present in orange-red patches, and a higher proportion of yellow pararelagar is present in more yellow areas. Pararelagar was often used with other yellows, for example in the manuscript some visually similar yellow text is written with an organic yellow and the illuminations only contain orpiment and Indian yellow, without any pararealgar. In one 13th century folio (Table 1, #2) pararealgar occurs in yellow areas on one side, with orpiment on the other, as seen in the Raman shown in Fig. 17. The pararealgar is finer grained than the coarser, more clearly crystalline orpiment, and is currently a lighter, almost tan color. In the miniatures, pararealgar is used as the yellow and in mixtures (for example to create a tan color) whilst orpiment occurs with indigo in the dull green.

It is worth noting that the distinction between the different arsenic sulfides (orpiment, pararelagar and realgar) requires spectroscopic analysis (in this case Raman spectroscopy) or polarized light microscopy (PLM) in addition to elemental analysis (XRF). Since PLM was used in previous projects at the Straus Center for Conservation, it is likely that the identification of orpiment in these is accurate (see Table 2). However, Raman spectroscopy would be required to confirm this for certain.

Indian yellow, a magnesium euxanthate salt, is another yellow pigment that was looked for within these art works. Indian yellow contains light elements that cannot be detected by XRF and often gives a poor Raman spectrum, so identification is normally based on the characteristic bright yellow florescence in ultraviolet light. Samples, if available, can be identified by PLM or FTIR analysis. Indian yellow occurs in Indian works from the

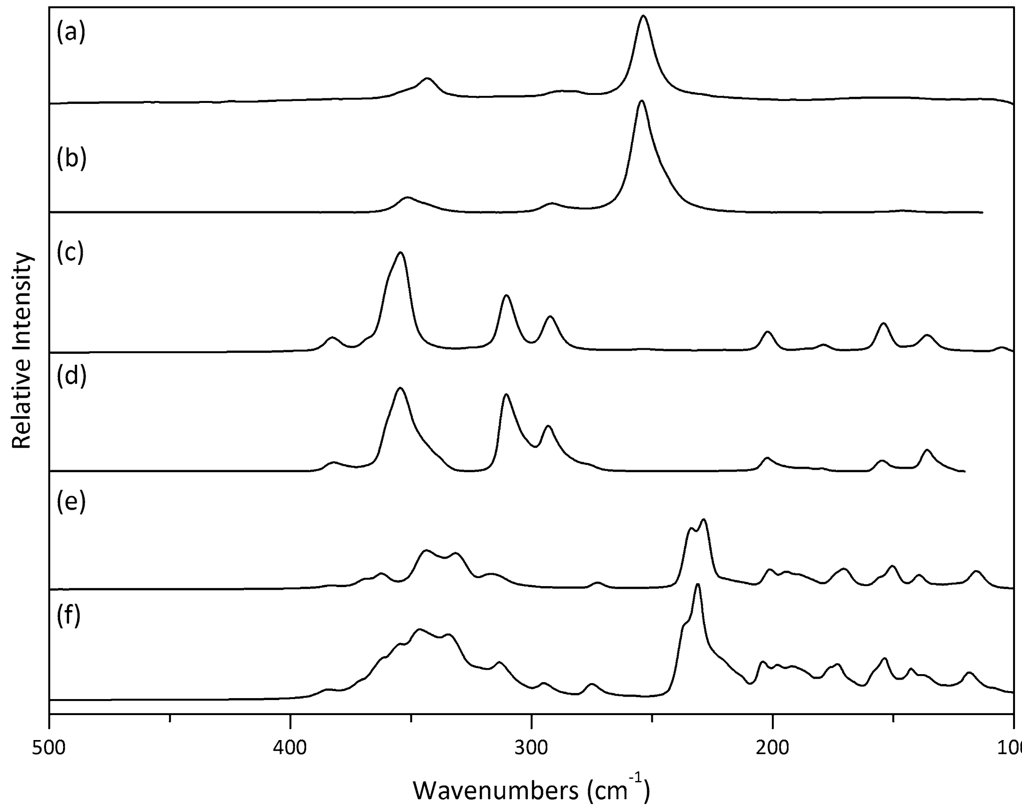

Fig. 17 Raman spectra for yellow within a 13th century manuscript from Iraq (Table 1, #2) showing the use of orpiment with vermilion on one side and of pararealgar the other side—a: red particle in yellow on bird side, b: reference spectrum for vermilion, c: yellow from bird side, d: reference spectrum for orpiment, e: yellow from plant side, f: reference spectrum for pararealgar. All reference spectra from RRUFF database http://rruff.info/

16th through 19th century and has not previously been found on works firmly ascribed to Iran [5, 14, 18, 28, 29]. The presence of Indian yellow on a small number of definitively Iranian works in the current study marks the first known identification in Persian rather than Indian art works, see Table 1. The most important of these is a high quality manuscript believed to have been produced at the end of the 17th century, probably in Isfahan (Table 1, #36). This manuscript contains several illuminations in which Indian yellow is used in addition to orpiment. The Indian yellow was used mainly for solid fields of yellow with orpiment used for fine flowers, as shown in Fig. 18a, b. Identification of Indian yellow is based on the characteristic florescence in UV and by FTIR of a small sample, as shown in Fig. 19. Indian yellow was identified from its characteristic florescence in the only two works examined in detail from 18th century Iran, (Table 1, #37–38). In these, Indian yellow occurs alone in the bright yellow clothing of the figures (Fig. 20a, b, Table 1, #37), and mixed with indigo in the green vegetation.

Indian yellow also occurs in at least two paintings believed to have been produced in Bukhara in the 16th century but later altered in Mughal India (Table 1, #28, #35). In the latter, (Table 1, #35), Indian yellow occurs as a wash over the floor area, on orange and yellow clothing and in an insert on the right hand side, seen previously in the yellow florescence of Fig. 11c. Details painted in an orpiment–indigo mixture on the original, left hand side, are mimicked in an Indian yellow-indigo mixture in the later insert, for example the leaves in Fig. 21a compared to the leaves in Fig. 21b. The Indian yellow is thus demonstrably late in this illustration and likely applied at the Mughal court in the 17th century—its application over the floor probably aimed to blend in the alterations. In the other example, (Table 1, #28) Indian yellow occurs in most yellow to orange robes as well as in patchy alteration to the floor in one page and in touched-up areas of foliage on another page.

An unidentified yellow pigment was found in a single yellow robe on one painting in the manuscript produced in Bukhara but altered in India, Table 1, #28. XRF analysis showed this yellow contains high levels of lead and tin. FTIR analysis of a small sample did not produce a match for any yellow pigment, including the historic pigment lead–tin yellow. Despite widespread use in European painting from the 15th to 17th centuries [30], lead–tin

Fig. 18 a, **b** Comparison of visible (**a**) and ultraviolet (**b**) images shows that the Indian yellow (fluorescing bright yellow in ultraviolet) and in a 17th century Iranian manuscript (Table 1, #36)

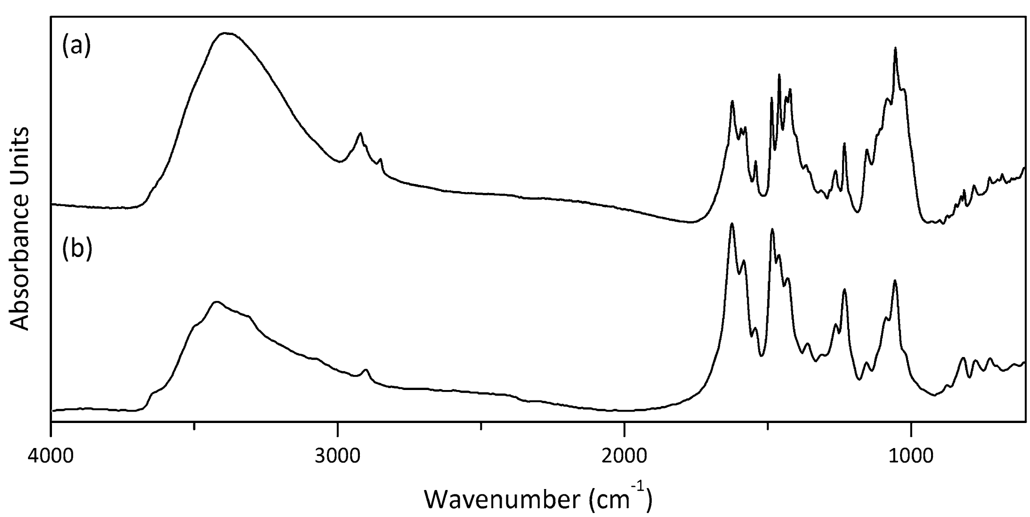

Fig. 19 Comparison of FTIR spectra from a: sample of yellow from a 17th century manuscript from Iran (Table 1, #36) and b: reference spectrum of Indian yellow from IRRUG database

Fig. 20 **a**, **b** Visible (**a**) and ultraviolet (**b**) images showing bright yellow Indian yellow fluorescing in an 18th century Iranian painting (Table 1, #37)

Fig. 21 **a**, **b** Comparison of original leaves (**a**) and leaves on the insert (**b**) in a 16th century folio altered in India in the 17th century (Table 1, #35)

yellow has not been reported in any Indian or Islamic work. An alternative possibility might be use of a yellow lead compound such as litharge or massicot mixed with a tin white. Further work is required to identify this yellow pigment and determine whether this was a Mughal alteration.

The traditional yellow palette was expanded in the 19th century with the addition of the synthetic pigment, chrome yellow (lead chromate), which was marketed in Europe from the early 1800s. This occurs as the main yellow in a mid-19th century Iranian folio (Table 1, #43). Chrome yellow was also found (often with zinc white) in areas of restoration, for example on a folio from 15th century Shiraz (Table 1, #15).

Red: vermilion, hematite, red lead, organic red

The red palette appears to have been the most traditional with vermilion, hematite and red lead widespread and present in most works examined from all periods and locations. These materials were frequently mixed with each other or other colors to adjust the tone. Organic reds were probably present in many works but can normally only be inferred from the lack of detectable elements such as lead, iron or mercury by XRF analysis and from careful visual and, sometimes, ultraviolet examination. Visually, organic reds tended to be added on top of other colors and they appear more transparent and glassier. Higher percentages of red lead give a more orange color, vermilion and organic reds give a darker or pinker red, and hematite gives a browner tint. Other pigments such as lead white, orpiment or pararealgar were mixed in with the reds to give a range of orange or pink hues, particularly in the 15th and 16th centuries, (for example Table 1, #17 and #26).

It has not yet been possible to identify the specific organic red used, although these generally show minimal UV florescence, possibly suggesting insect-based reds such as kermes or cochineal rather than plant-based reds such as madder. Sampling is required for firm identification of the organic reds and even FTIR analysis has proved poor at differentiating the possibilities.

Metallic pigments: gold, silver, tin

Silver was frequently used for arms and armor, water features, such as rivers and fountains, and architectural features, such as doorways or windows, at all periods. The silver was powdered, mixed with binder and used as a paint. The silver is now darkened due to reaction with atmospheric gases resulting in black silver chloride and/or silver sulfide on the surface, making it hard to discern original details drawn on the surface with dark inks or pigments. One of the most impressive and extensive examples of the use of silver is the now darkened silver cavalry on a folio from the Great Ilkhanid *Shāhnāma* (Table 1, #7) shown in Fig. 22.

Fig. 22 Darkened silver cavalry and two compositions of gold, silver-rich and pure gold, on a folio from the Great Ilkhanid *Shāhnāma* (Table 1, #7)

Although most rivers were executed in silver, occasional examples occur where other materials were employed for these such as tin (Table 1, #29, Bukhara, 1551), gold (Table 1, #34, Isfahan, 1600) and a grey mixture of lead white and carbon black (Table 1, #35, Bukhara c.1600). In the latter case, the dark river can only be clearly distinguished from the surrounding darkened vegetation in the infrared image. Although two of these exceptions come from 16th century Bukhara, other 16th century paintings from Bukhara show the more normal use of silver for water (Table 1, #28). Tin was also found on a 19th century drawing from an artists' album, where it was used depict a grey seat/throne (Table 1, #42).

Gold was used extensively on the manuscripts, both for the background or sky, and for details such as vessels, arms and armor, and some architectural features. In most cases, the gold is of high purity but in some paintings bright high purity gold was deliberately alternated with lighter silver–rich gold to create color differences, for example in armor on a folio from the Great Ilkhanid *Shāhnāma* (Table 1, #7) shown in Fig. 22 and in detail in Fig. 23. In some cases, the gold or silver was decorated with fine punchwork to create texture. In all art works studied, microscopic and transmitted light examination shows use of shell gold—finely powdered gold mixed with a binder—rather than gold leaf. Historical treatises propose the use of glue as a binder for gold and either glue or (preferred) gum-arabic as a binder for silver [6].

Imaging

Hyperspectral imaging was used to produce distribution maps of different pigments within selected folios. Although hyperspectral imaging is being increasingly applied to the imaging of western manuscripts [31, 32], it has not previously been applied to pigments on Islamic works but has instead been used for examination of Islamic papers [33].

The folios studied here include three 15th century Iranian works (Table 1, #13–14, #20). The pigment

Fig. 23 Detail of alternating pale, silver-rich gold and dark, purer gold in the armor of cavalry in the folio from the Great Ilkhanid *Shāhnāma* shown in Fig. 21 (Table 1, #7)

distributions from these are illustrated in Figs. 24, 25, 26 and show good agreement with the data from the other techniques used (IR imaging, XRF and Raman spectroscopy). For each manuscript eight pigments were selected for the preparation of the maps, based on the palette identified by other analysis. Organic red could not be included as there was no reference available for animal-based dyes. Comparison of the maps for red lead and vermilion from one painting (Table 1, #13) clearly shows the inpainting on the right hand side of the building (see Fig. 24).

Hyperspectral imaging was particularly useful in identifying mixtures of ultramarine and indigo, which were hard to detect with other methods. For example, in Table 1 #13 p26 hyperspectral imaging confirms that ultramarine is the principal blue used for the clothes and the decorative borders around the window and the door. Indigo occurs in the decorative strip above the windows and the terrace and a mixture of the two blue pigments seems to have been used in the darker blue areas of the decorative border of the window and the terrace (Fig. 24). Similarly, in another example (Table 1, #14) ultramarine was used alone in the king's robe, the blue hats and the background, and was mixed with indigo and an organic red to create purple in the tent and on the dress of the figure in the left (Fig. 25). Indigo was used mostly in mixtures with orpiment in green areas and with ultramarine in darker blue or purple areas. Use of indigo alone can

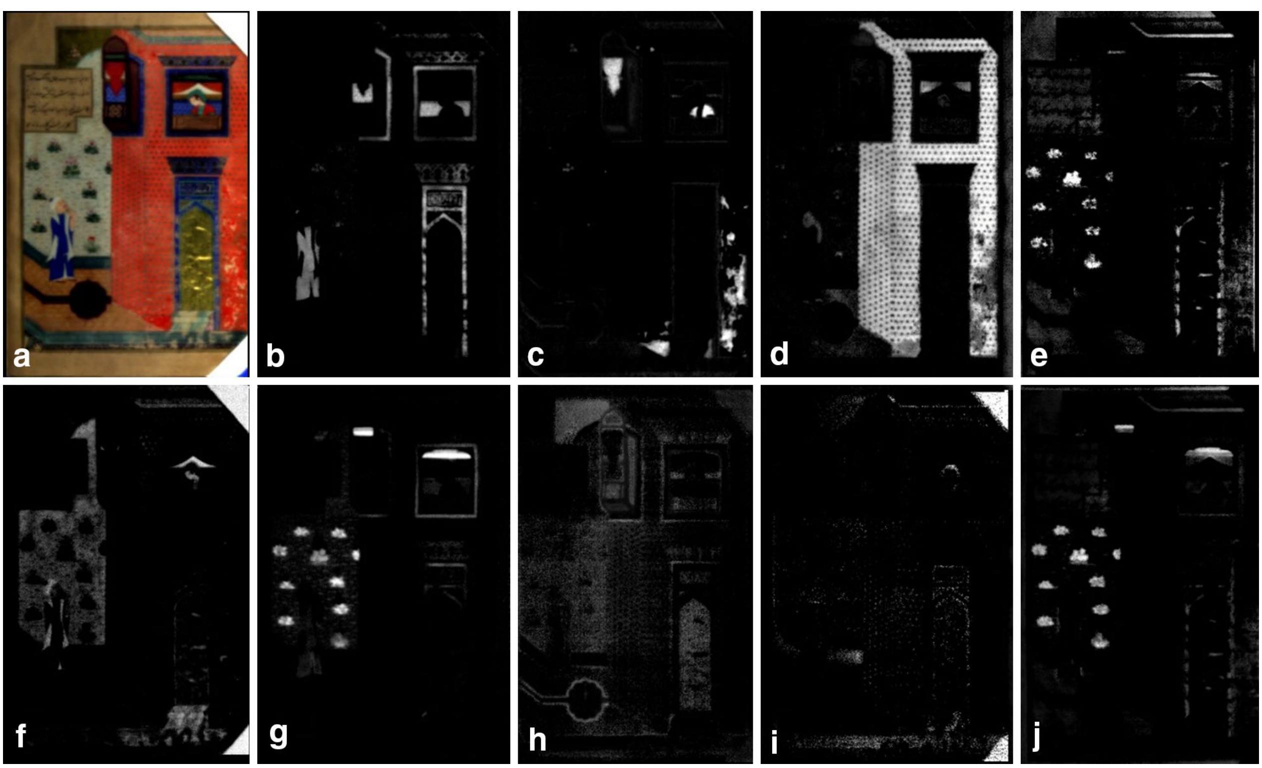

Fig. 24 **a** RGB image reconstructed from bands 118, 75, and 35 from the hyperspectral cube for a painting in a manuscript from Herat, 1427, (Table 1, #13 p26). Linear least squares un-mixing of the datacube using references from the Forbes collection provide the following images of pigment distribution: **b** ultramarine (Na_8–$10Al_6Si_6O_{24}S_{2-4}$), **c** vermillion (HgS), **d** red lead (Pb_3O_4), **e** orpiment (As_2S_3), **f** lead white ($PbCO_3$), **g** indigo, **h** hematite (Fe_2O_3) **i** copper green, **j** orpiment (As_2S_3) and indigo

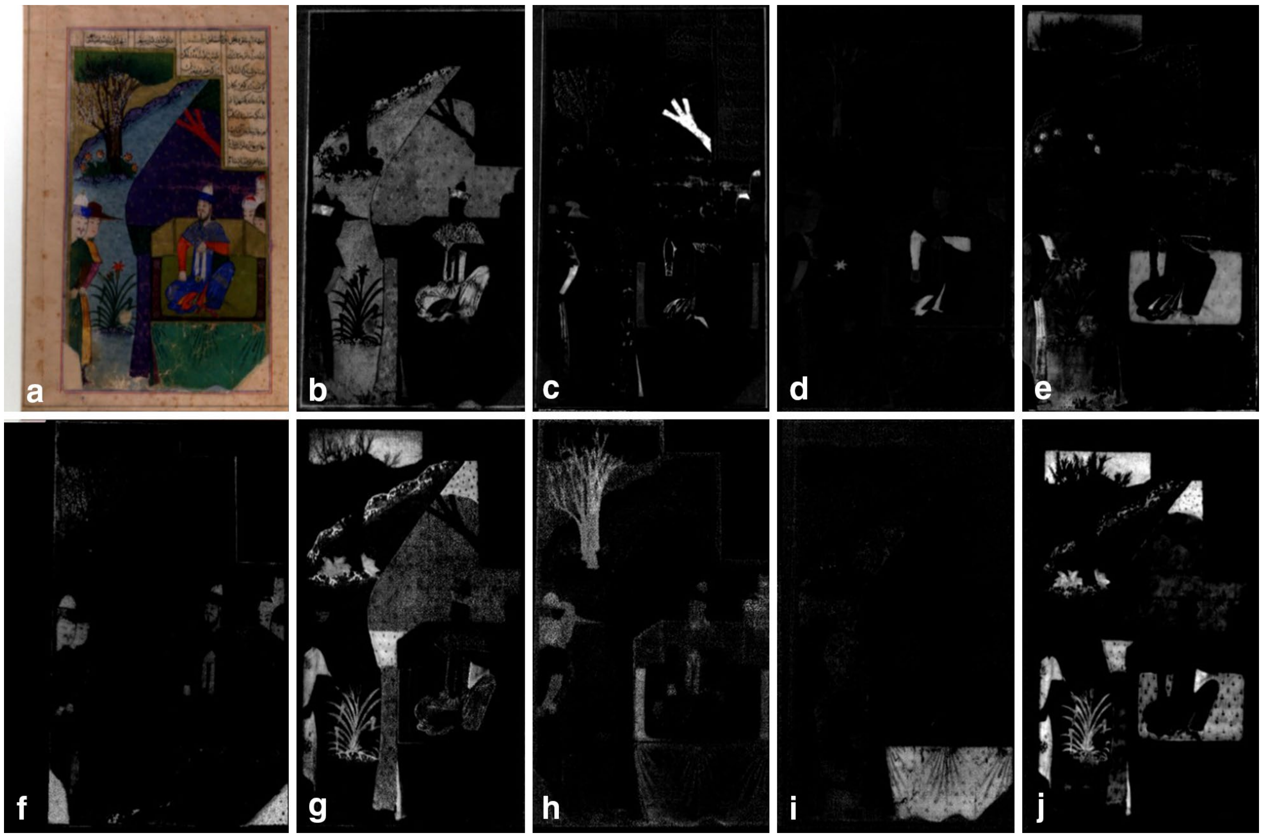

Fig. 25 **a** RGB image reconstructed from bands 118, 75, and 35 from the hyperspectral cube for a folio from Shiraz 1436 (Table 1, #14). Linear least squares un-mixing of the datacube using references from the Forbes collection provide the following images of pigment distribution: **b** ultramarine ($Na_{8-10}Al_6Si_6O_{24}S_{2-4}$), **c** vermillion (HgS), **d** red lead (Pb_3O_4), **e** orpiment (As_2S_3), **f** lead white ($PbCO_3$), **g** indigo, **h** hematite (Fe_2O_3) **i** copper green, **j** orpiment (As_2S_3) and indigo

be clearly seen in the outline of the hill and the creases of the dress of the royal figure in the center (Fig. 25). Figure 26, (from Table 1, #20), shows a somewhat different distribution of the two blues–ultramarine is mixed with lead white for the light blue area sky and mixed with indigo for dark blue robes.

Binders

The nature of the binder used with the pigments remains somewhat elusive. In part, this reflects the need to remove a small sample for FTIR analysis, and to follow this with additional analytical techniques such as gas chromatography with mass spectroscopy (GCMS) or matrix assisted laser desorbtion ionization (MALDI) for definitive identification. FTIR analysis of a small number of samples from works spanning the 14th to 17th century (from Table 1, #4, 10, 15, 19, 27, 31, 32, 33) indicated gum-based binder in all samples, with only the occasional presence of minor proteinaceous material. All identification is currently based on FTIR analysis alone but work is on-going to improve characterization of the binders from a wider range of folios. Although it has been proposed that use of gum-arabic only began at the end of the 16th century [2, 14, 18], 13th to 19th century recipes indicate mixing of pigment with gum-arabic for preparation and use, with only rare mention of egg or glue [6]. The exception is for metallic pigments for which glue was recommended [6]. This dominance of gum-arabic is consistent with the presence of gum in all samples analyzed to date. However, other studies have found lipidic binders in 16th century Persian manuscripts [17, 19].

Case studies

The project is fortunate in being able to include a number of manuscripts containing multiple paintings, as well as detached folios from the same dispersed manuscripts. This has enabled comparison of closely related works, although it is worth noting that the association of works in a single manuscript does not necessarily mean they were all produced within a single workshop or region. Examples of related works include three from a manuscript produced in Iraq in 1224 (Table 1, #1–3), seven

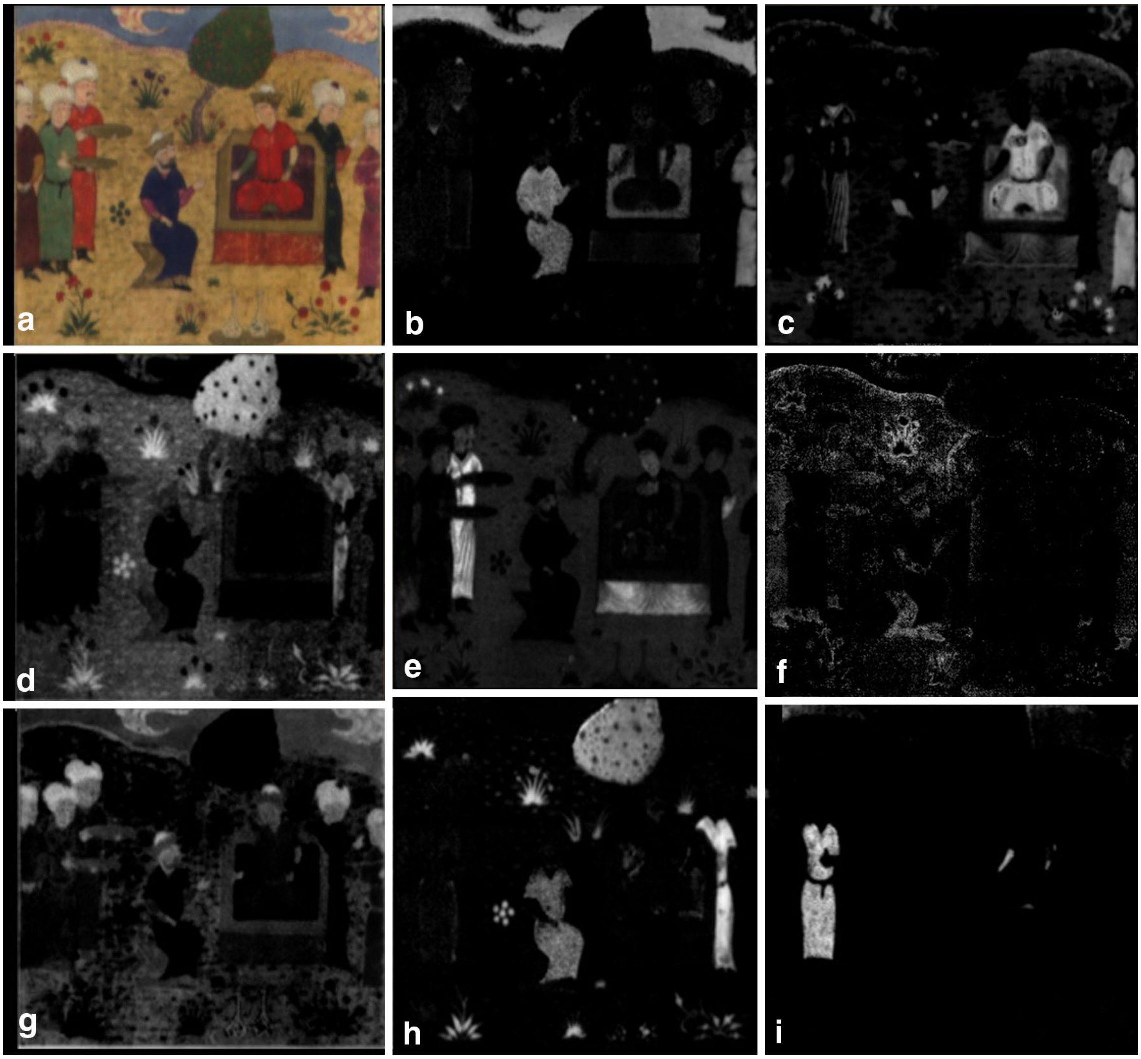

Fig. 26 **a** RGB image reconstructed from bands 118, 75, and 35 from the hyperspectral cube from a folio from Shiraz, 1480s, (Table 1, #20). Linear least squares un-mixing of the datacube using references from the Forbes collection provide the following images of pigment distribution: **b** ultramarine (Na_8–$10Al_6Si_6O_{24}S_{2-4}$), **c** vermillion (HgS), **d** orpiment (As_2S_3) and indigo, **e** red lead (Pb_3O_4), **f** hematite (Fe_2O_3), **g** lead white ($PbCO_3$), **h** indigo, **i** copper green

from Tabriz dated to the 1330s (Table 1, #7–8), and two from Shiraz dated to 1436 (Table 1, #14–15). Associated folios generally have very similar pigments and artistic techniques, with variations in palette reflecting different requirements of the scene—for example an abundance of silver and two compositions of gold in *Iskandar's Iron Cavalry Battles King Fur of Hind* but not in *Bahram Gur Hunts with Azada* from the Great Ilkhanid *Shāhnāma* (Table 1, #7).

However, in two folios attributed to Tabriz around 1540 and thought to have belonged together, the pigments used differ significantly (Table 1, #26–27). In one of these *Nighttime in the City*, (Table 1, #26), a range of copper greens—atacamite, antlerite, and malachite—were used in different areas, and orpiment was used for yellow. In the other folio *Nomadic Encampment*, (Table 1, #27), analysis indicated an unknown copper green that does not give an identifiable Raman spectrum and mixtures of orpiment and indigo. The yellow and orange-yellow areas contain pararealgar although orpiment was obviously available. Although both folios were formerly attributed to Mir Sayyid ʻAli, only *Nighttime in the City*, (Table 1, #26), remains confidently attributed to this artist. The difference in palette is consistent with production by a different artist and possibly within a different workshop and/or area.

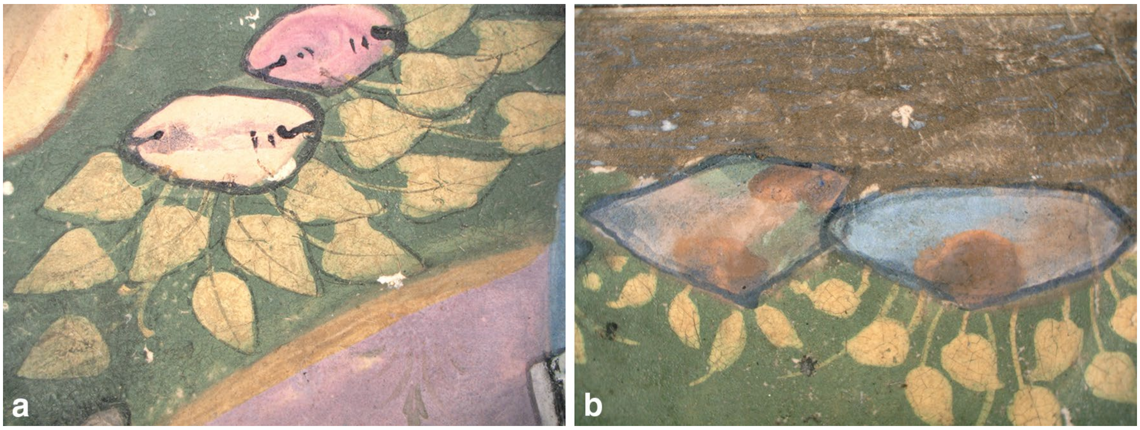

Fig. 27 **a**, **b** Differences in the treatment of rocks and leaves in paintings from a single manuscript, painted in Isfahan around 1600, (Table 1, #34), suggest a different artist may have worked on the pages

Perhaps the clearest variation occurs within one manuscript from Isfahan, 1600, (Table 1, #34). Two of the three paintings are very similar in style and pigments but differ significantly from the third paintings. Several key differences are apparent in comparison of the infrared images (previously shown in Figs. 3b and 4b). For example, all three paintings contain rocks throughout the background. However, the rocks in one painting are entirely transparent in infrared due to the use of a pale pink, probably organic, pigment throughout (Fig. 27a). In contrast, rocks in the other two paintings are more colorful and employ mixtures of copper green, orange, pink, blue and brown, resulting in opaque areas in infrared, due to the copper green and iron-based browns (Fig. 27b). The yellow to yellow green leaves in the river on one painting have thin outlines drawn in a carbon-based medium applied after the pigment, seen in Fig. 27a. The yellow leaves in the other two are not outlined in this way (Fig. 27b) and are indistinct in infrared. The execution of the clouds also differs—in two paintings the banded white–grey-bluish clouds were produced using a mixture of carbon-based pigments, indigo and lead white applied in bands that merge into each other. In the other painting, the clouds are more simply produced in a basic light grey color overlain by white lead and firm thin outlines and details in carbon-based pigment are clearly seen in the infrared image.

The two paintings also have a wider range of pigments, with two greens present– a mixture of orpiment and indigo for the foliage and a brighter copper green for the saddle cloths, robes and details on the rocks. In contrast, only the mixed orpiment–green was present on the other painting. It is noticeable that the tufts of grass decorating the background in this painting are much fainter than those on the others and are also a different shape, being taller and narrower. Another clear difference is the use of gold mixed with orpiment and lead pigments for the river on two paintings, with no silver found anywhere. Highlights of lead white were used to give the appearance of waves on the now dulled gold river, as seen in Fig. 28a. The use of gold as a pigment for a river is unique amongst the Persian manuscripts studied here and is not mentioned in other studies (for example [14, 18]). In contrast, the river on the other painting was depicted with the expected silver, with waves suggested by dark lines within this, shown in Fig. 28b. Overall, these differences suggest a higher quality and more confident work for two of the paintings, with no adjustment of the initial sketch required, and a more varied and complex range and use of pigments.

Discussion

Consideration of the results from the art works studied here and those presented in published studies [5, 7, 14–25], allows some general conclusions to be drawn for the Islamic artists' palette. These will be discussed in terms of the main colors identified from historical recipes [6]. It should be noted that chronological trends may be distorted due to the fact that there is a higher proportion of Safvaid, and to a lesser extent Timurid works, analyzed than those from either earlier or later periods. Similarly, the majority of works studied are attributed to Iran, and the specific place of manufacture is rarely discussed in the analytical publications. Hence, more data is required before commenting in detail on geographical variations.

White

Lead white was the primary white phase identified in this and other studies and is the main white mentioned in historic sources, which give recipes for its production [5–7,

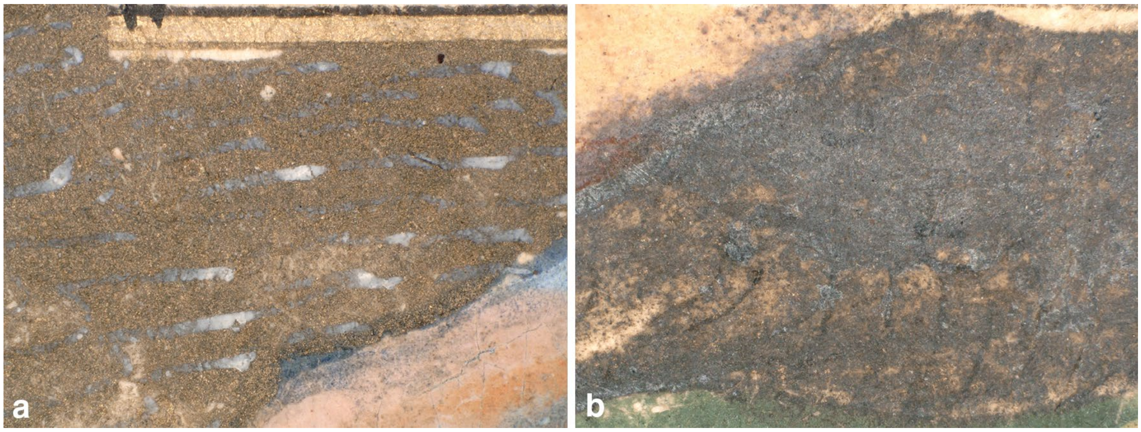

Fig. 28 **a**, **b** The use of gold and lead white (**a**) to depict a river in one painting and of silver and black ink (**b**) for a river in another painting from an Isfahan manuscript, (Table 1, #34), reinforces the idea that a different artist may have worked on the pages

9]. Although different historical recipes result in a range of associated materials [9], none of these have yet been identified in analyses of Persian pigments. Interestingly, in one study chalk rather than lead white was found on a 19th century paint box and 20th century paint palette and tin white rather than lead white occurred on a 19th century paint palette [7]. Although there are historical Persian recipes for production of tin white [6], this has not otherwise been found in Persian art works.

Blue

Ultramarine was the dominant blue identified on folios from all periods and locations in this and the other published studies and is the main blue discussed in the historical treatises [6, 7]. Ultramarine was sourced from quarries in Badakhshan, present day Afghanistan. The ubiquitous use of ultramarine indicates active trade routes from the source quarries across the Islamic world throughout the entire period studied. Azurite is not mentioned by historical treatises [6, 7] and was found only rarely in our study or other publications (see for example [14, 18]). The use of azurite appears to be somewhat scattered chronologically and geographically and may be opportunistic, perhaps used when azurite was available due to an association with other, more deliberately sourced or manufactured copper pigments, such as natural mixed deposits of malachite and azurite. Use of azurite may also reflect a desire for a particular hue—for example on an 18th century Iranian folio (Table 1, 38) the artist appears to have deliberately used both darker ultramarine and lighter azurite for different features. Similarly, different shades of blue of blue in both the illumination and text on a late 17th century manuscript (Table 1, 36) correspond to use of azurite for lighter blues and ultramarine for brighter, darker blues. Azurite also occurs with ultramarine in paintings in a 16th century *Shahnama* (Table 1, 23) whereas only ultramarine occurs in the illuminations. A similar feature was observed in a 16th century a manuscript from Shiraz [14]. This may reflect a chronological difference between the illuminations and paintings or use of a different palette by illuminators versus painters. Further comparison of the palette used for illuminations versus paintings would hence be interesting. The blue pigment indigo, the highest quality of which had been exported from India since antiquity, is used alone as a blue occasionally but is more commonly mixed with other colors [5, 7, 14, 18]. Historical texts describe production of artificial ultramarine by mixing indigo with lead white [5–7]. Finally, the identification of Prussian blue in 19th century works is consistent with its presence in 18th century Iranian book covers [24] and a 19th century paint palette [7], indicating import from Europe in the 19th century.

Green

Extensive use of orpiment–indigo mixtures to produce green was noted in this and other published studies, particularly for vegetation (for example [16, 17, 22]). An interesting related mixture, indigo and a yellow lead oxide phase (possibly litharge or massicot) was identified on a 16th century Persian manuscript [19] but was not found here. The use of copper-based greens, particularly for clothing, appears to increase from the late 14th century. In our study, copper-based green pigments from the 13th to 15th century proved the most difficult to firmly identify and are likely to be the product of varied sources, both natural and synthetic. In many publications, the exact copper species present is not definitely identified and the term “verdigris” is often used generically for green to blue copper corrosion products rather than referring strictly to the various copper acetates that comprise true verdigris [18]. Verdigris is the only green pigment described in the historical treatises and one of two main techniques to produce synthetic copper pigments involved use of vinegar, which would favor formation of various copper actetates [6, 7]. Alternative recipes involved use of sour sheep-milk, milk from sheep-yoghurt or water mixed with sheep's blood [6, 7]. Published studies have identified malachite [5, 7, 14, 16, 18, 20, 24, 25], brochantite [14] and atacamite [14, 19, 24], in addition to verdigris [3–5, 7, 14, 17, 18]. Verdigris was proposed but not confirmed for copper green in folios from an early astronomical and cartographic treatise dated to about 1200, which also had orpiment–indigo mixtures [22]. The identification of copper greens other than verdigris, for example malachite, indicates use of other recipes or, perhaps more likely, natural sources in addition to the known recipes that would produce verdigris. Copper chlorides, normally atacamite, are used extensively in the 15th and 16th century and malachite appears to be more common in the 16th to early 17th century. Use of a natural rather than synthetic source of atacamite, possibly from deposits near Isfahan, has been proposed based on the uniformity of copper to chloride ratios in a 16th century manuscript [19] and the greater overall use of copper chloride in this period may reflect discovery and exploitation of such a natural source. The presence of other copper-based phases mixed with or used instead of atacamite may be due to deliberate artists' selection of different natural or synthetic mixtures based on hue.

Verdigris was known to be corrosive to papers from historical times and historical treatises recommend adding saffron to buffer the verdigris and prevent damage [3–5]. The authors found 3 instances of verdigris mixed with saffron in 16th century Iranian art works without damage to the paper, whilst visible deterioration had occurred in 2 instances where verdigris was used [3]. Identification of saffron mixed with verdigris requires sampling and this mixture has not been identified in any of the manuscripts studied here. However, very few of papers show any signs of damage from the copper compounds and it is hence possible that some of the unidentified copper greens may be mixtures of verdigris and saffron. The lack of clear Raman spectra from these phases may be due to both poor crystallinity of the synthetic corrosion products produced and increased florescence due to the presence of saffron as well as an organic binder. No chlorine was detected in these with XRF, consistent with organic copper salts such as acetates rather than copper chloride, but the presence of high levels of lead and proximity of the lead M and chlorine K peaks increases the detection limit.

The presence of Emerald green in 19th century works is consistent with the identification of this pigment in Qajar lacquer art works from at least 1852–1853 onwards [34] and in the early 20th century pigments brought back from Iran and now part of the SCC pigment collection. This shows that European pigments such as Emerald green were imported to Iran from the mid-19th century onwards. The likely use of viridian in some 19th century paintings also reflects import of new, fashionable European pigments.

Yellow

Orpiment was the dominant yellow identified in Islamic paintings and manuscripts produced outside of Mughal India from all periods in all studies [5, 7, 14–24]. This indicates a ready supply of natural orpiment, which is not surprising given the presence of several deposits within Persia and nearby countries (including Iraq and Armenia).

Realgar often occurs with orpiment and is described as red orpiment in some Persian treatises [6]. One treatise mentions drying red orpiment in the sun after mixing it with gum-arabic [6]—since realgar alters to pararealgar with exposure to light, it is likely that the original pigment used contained a mixture of realgar and pararealgar. This suggests that the original color used may have been closer to red than yellow. Realgar and pararealgar are rarely identified in published studies on Islamic pigments (for example they are absent in [14, 17–19, 21]). In this study, these phases occur in in a set of folios from 1224, Iraq, in two Iranian folios, one from around 1300 and one from the early 16th century, and in an Iranian manuscript from the late 17th century. This somewhat sporadic use is interesting and suggests this was not a major contributor to the general palette—although a lack of published data may partially reflect the need for spectroscopic techniques to identify these phases. Realgar with pararealgar or another alteration product was found in four published 16th century manuscripts [16, 24, 35], only one of which is attributed to Iran [16]. Of the others, one is a Turkish work [35], one is attributed to an area outside Iran, such as Central Asia or a provincial Ottoman center [24], and one to the Ottoman empire [24]. In this last Ottoman example, the realgar–pararealgar mixture occurs in colored inks [24] similar to those seen in the 17th century manuscript analyzed here [Table 1, #36]. A mixture of realgar/pararealgar was also identified as the yellow of two 16th century Ottoman paintings at the Harvard Art Museums (not included in Table 1). Hence, Ottoman rather than Persian influence may be responsible for the 16th to 17th century use of realgar.

Yellow ochre was not conclusively identified in this study but may have been used in addition to red/red-brown hematite-rich ochres as this phase can be difficult to identify without sampling. However, other authors also noted a near absence of yellow ochre—finding it only in one folio from Iraq, 1224 and two early 15th to 16th century Iranian works [14, 18], whilst historical treatises mention only red and brown earths [6]. Similarly, sampling would be required to identify organic yellows, such as saffron or rhubarb which are described in historic treatises [6]. Rhubarb and saffron have been identified as yellow colorants in 16th century Iranian miniatures and 19th century paint boxes and palettes [7].

The identification of Indian yellow in some of the art works studied contrasts with published studies to date. The manufacture of Indian yellow has been the subject of a number of recent papers [28, 36]. Indian yellow occurs in Indian works from the 16th through 19th century [18, 28, 29] and was likely produced in India from the urine of cows fed exclusively on mango leaves [28]. Although there is one reference to Indian yellow being introduced to India from Persia in the 15th century [37], Indian yellow has not previously been found on works firmly ascribed to Iran [5, 7, 14, 18]. It is hence often considered diagnostic of Indian works—a view that must now be reconsidered in light of its identification in works of clear Persian rather than Indian production. However, use of Indian yellow within Persia does appear to be fairly rare and restricted to the late 17th to 18th century. This may reflect the culture of the time as well as the production site. The manuscript containing Indian yellow (Table 1, #36) is believed to have been made in late 17th century Isfahan, known for its cosmopolitan nature and extensive trade networks. The figures depicted in the two folios (Table 1, #37–38) have Afsharid clothes, named for the dynasty founded by Nader Shah Afshar in about 1736. Nader Shah Afshar invaded India in 1739 and although he did not remain long in India, the works were thus produced in a society that had recent close links to Mughal India with exchange of materials or local patronage of the arts likely. Work is on-going to assess the extent to which Indian yellow occurs within the Persian manuscript and painting tradition.

Chrome yellow is another pigment not traditionally considered part of the Islamic artists' palette but seen here occasionally in 19th century examples due to import from Europe, confirming the active trade routes.

Reds

The red palette identified in this and other studies shows little variation with place or date, consisting of red lead, vermilion/cinnabar, red to brown ochres and organic reds [7, 18, 20]. The production of red lead by heating lead white was known since antiquity and described by some Islamic authors [6, 9]. Natural cinnabar could be sourced from Soviet Turkestan or the synthetic form, vermilion, produced from several known recipes [6]. It is not possible to distinguish between these options without PLM of samples and this was not undertaken here. Ochres from natural clay deposits were widely available in the Islamic world, most famously brown Hormuz clay from the island in the Persian Gulf, mentioned in a 16th century recipe [6]. A further red may have been provided by realgar, which has now altered to give a more yellow to orange shade in all instances so far identified.

The most difficult pigments to identify were the organic reds, which was also noted by other studies and most authors merely report the presence of organic reds (for example in [14, 18]). A range of red dyes are mentioned by the historical sources [6, 11], and have been proposed as likely options [14, 18]. These include the plant-based dyes safflower red, madder and Brazil wood, and the insect-based dyes lac, kermes or cochineal. Little florescence occurred under UV examination, with the exception of pink page borders on a manuscript from 17th century Isfahan (Table 1, #36) where the orange florescence suggests use of madder. The few firm published identifications of organic reds include carmine as the organic red on a late 17th century Persian manuscript [16], a red insect-derived anthraquinone dye on a 16th century Persian manuscript [17] and lac as a bright burgundy red and safflower as a peach color in another 16th century manuscript [19]. Cochineal imported from America via Europe was available only after the 16th century. However, cochineal may have been produced locally in Armenia and northern Persia and used earlier than this [11]. Cochineal is the organic red present in Iranian lacquer from the 17th to 19th centuries [34]. Organic reds within pink and purple colors, often mixed with ultramarine in the latter case, were observed in this and other studies [16–18]. Historical recipes mention the use of lac dye for pink and purple (with ultramarine and cinnabar) [7].

Metallic pigments

Gold and silver were the main metallic pigments identified here, with two compositions of gold. These are consistent with those found in other studies and show little variation with place or time of manufacture. Historical recipes also record preparation of brass and copper pigments and ternary copper alloys chosen to mimic gold have been found in some Qajar manuscripts [38]. Historical treatises mention white tin, which has not been noted in any Islamic manuscript, but metallic tin instead of silver was found in a few instances in this study.

It is worth noting here that the most unusual and diverse results were obtained from works produced in Bukhara in the 16th century—two manuscripts with multiple illustrations (Table 1, #28–29) and a single folio (Table 1, #35). Since two of these (Table 1, #28 and #35) were unquestionably altered at the Mughal court subsequent to production, some of this may reflect the later history of the works. However, the use of lead white and carbon-based black for the river in one folio (Table 1, #35), of tin for the river in a double page frontispiece (Table 1, #29) and of silver for the rivers and water features (Table 1, #28) or armor (Table 1, #29) in other images are believed to represent original differences. In another study, a figural study from Bukhara dated to around 1600 had a fairly standard range of pigments and was most notable for the rare definitive identification of malachite [18]. Of three works from Bukhara published elsewhere [14]—one dated to 1615 is somewhat unusual in containing both ultramarine and azurite and three copper greens, verdigris, possible malachite and brochantite, whilst one dated to 1525–1550 contains both verdigris and atacamite. Silver is noted only for the folio from 1615 but the folio from 1525 to 1550 has a distinct grey river which does not appear to have been analyzed and would benefit from this. Analysis of additional works could help determine whether folios from Bukhara are as varied as would seem from the current data.

Conclusions

Careful study and analysis of Islamic manuscripts and folios at the Harvard Art Museums is revealing a wealth of detail about the production of these works and the range of materials used. It is hoped that this study can join others to deepen an understanding of Islamic works on paper collections worldwide. Some colors, such as red and brown, show little variation in the pigments used over the full chronological period studied. Other pigments, in particular the greens and yellows, vary considerably. Indian yellow was found in a few Islamic works, representing the first known such identification. Further study is required to understand the rare use of this vibrant yellow pigment outside of India. Introduction of "industrial" pigments occurs in some 19th century works but in others traditional pigments similar to those used in the preceding centuries were employed. The occasional and sporadic use of pigments such as azurite and realgar–pararealgar has been demonstrated but requires further exploration to more fully understand the variations seen. Continued study of art works from Bukhara is planned due to the limited number of such art works examined to date and the diversity of materials found in these. Further work is required for the identification of the organic components, such as organic reds and binders. Such identification may, however, be limited by the need for micro-sampling, which is only possible where existing damage is present and often does not allow for sampling of all, or even the most desired, areas and hence colors.

Authors' contributions

PK led the IR, UV and visible imaging and examination of the art works, including microscopy, and was responsible for conservation assessments. KE undertook the XRF, Raman and SEM–EDS analysis and interpretation and collaborated on the IR and UV imaging and microscope examination. MW and AB were responsible for the hyperspectral imaging and data processing. GR undertook the FTIR analysis and interpretation for binding media and selected pigments. All authors read and approved the final manuscript.

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Competing interests

The authors declare that they have no competing interests.

Availability of data and materials

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