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Dyes of late Bronze Age textile clothes and accessories from the Yanghai archaeological site, Turfan, China: Determination of the fibers, color analysis and dating

Annemarie Kramell [a], Xiao Li [b], René Csuk [a, *], Mayke Wagner [c], Tomasz Goslar [d, e], Pavel E. Tarasov [f], Nicole Kreusel [c, g], Ralph Kluge [a], Christian-Heinrich Wunderlich [h]

[a] Martin-Luther Universität Halle-Wittenberg, Bereich Organische Chemie, Kurt-Mothes-Str. 2, D-06120 Halle, Saale, Germany
[b] School of Chinese Classics, Renmin University of China, 59 Zhongguancun St., Haidian District, Beijing 100872, PR China
[c] Eurasia Department / Beijing Branch Office, German Archaeological Institute, Im Dol 2-6, D-14195 Berlin, Germany
[d] Faculty of Physics, Adam Mickiewicz University, Umultowska 85, 61-614 Poznan, Poland
[e] Poznan Radiocarbon Laboratory, Foundation of the A. Mickiewicz University, Rubież 46, 61-612 Poznan, Poland
[f] Institute of Geological Sciences, Palaeontology, Free University Berlin, Malteserstraße 74-100, Building D, D-12249 Berlin, Germany
[g] Department for Art History, East Asian Studies, Free University Berlin, Koserstr. 20, D-14195 Berlin, Germany
[h] Landesamt für Denkmalpflege und Archäologie Sachsen-Anhalt – Landesmuseum für Vorgeschichte, Richard-Wagner-Str. 9, D-06114 Halle, Saale, Germany

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ABSTRACT

A number of well-preserved garments from the ancient cemetery of Yanghai located in the Turfan Depression have been excavated from zone I and III of the burial ground of Yanghai and analyzed in this study. All textiles investigated in this study consist of keratinic fibers from animals. For selected samples an unambiguous dating has been performed by the radiocarbon method. The oldest textile object (wool trousers) from the tomb M157 is dated to 1261–1041 cal. BC with a 95% confidence and for several textile objects from the tomb M21 calendar ages are likely between 1074 and 926 cal. BC. Thus, they are among the oldest textile finds from the Xinjiang Region. The youngest analyzed textile piece from the tomb M18 is dated to 398–202 cal. BC with ~95% confidence. Analyses of the fibers revealed the use of the colorants alizarin, purpurin, rubiadin, quinizarin, indigo, and indirubin. Thus, for the creation of red shaded textiles most likely locally grown madder has been used, whereas for the production of blue shaded textiles indigo might have been to be imported. This represents one of the oldest scientifically proved evidence for utilization of madder and indigo for textile dyeing in the Xinjiang Region.

1. Introduction

The world we live in was never neither clearly black and white nor grey, but colorful since its beginning. At a time when our ancestors were seeking to customize their surroundings, this probably included the design and chromatic quality of their clothes. The history of textile technology and its related crafts (Good, 2001) is long, and the prehistoric production of textiles probably consumed more hours of labor per year (Barber, 1991) than pottery and food production put together. Clothing can no longer be seen just as an external wrapping but also as a means of communication and representation. However, like any organic material, textiles decompose rapidly in the archeological context. Among other factors, a dry climate is a prerequisite for the conservation of textiles; such conditions can be found in areas known as the Turfan Depression and the Tarim Basin. Analyses of dyes and fibers from prehistoric finds of textiles excavated in these regions in the Xinjiang Uyghur Autonomous Region from the Zaghunluq site (probable period of use correlating with phase C in Yanghai site, i.e. 7th–3rd century BC), from Sampula (probable period of use: 100 BC–300 AD (Wagner et al., 2009)), and from Yingpan (probable period of use: Han to Jin dynasty, i.e. 206 BC–420 AD) are well known. Analyses of red shaded textile finds from these locations revealed the presence of the anthraquinones alizarin and purpurin, thus indicating the use of plants from the family of Rubiaceae (Xie et al., 2001; Liu et al., 2011). A combination of red colorants and yellow dyes of the luteolin-type – finally resulting in an orange

* Corresponding author.
E-mail address: rene.csuk@chemie.uni-halle.de (R. Csuk).

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shade – has been reported for finds from Zaghunluq (Zhang et al., 2008). For these locations the use of indigo for obtaining blue colored textiles has already been described (Chen et al., 2000).

Written documents mention cultivation and use of indigo and several plants of the Rubiaceae family in Asia; for example, the "The Book of Songs" mentions their use during the Shang dynasty, ca. 1600–1046 BC (Cheng, 1992). However, to date there are no archaeological finds able to verify this text, compiled during 11th to 7th century BC.

In Europe, alizarin, purpurin and indigo have been detected in prehistoric finds of textiles, for example in samples from Danish peat bogs (covering a period of 4th century BC–1st century AD (Vanden Berghe et al., 2009)) and in samples from the salt-mining complex located at the Dürrnberg near Hallein and Hallstatt (Austria, 800–400 BC (Hofmann-de Keijzer et al., 2005; Stöllner, 2005)). The Danish finds from of Skærsø (350 BC–90 AD) are regarded as the earliest evidence for the use of madder in Scandinavia, and it was assumed that the dye must have been traded over a long distance, as madder was not cultivated in Scandinavia during the Early Iron Age (Vanden Berghe et al., 2009). Dyeing with indigo has been accomplished in early Europe, probably using widespread woad (*Isatis tinctoria* L.).

Finds of clothes from Xinjiang are documented from the archaeological graveyard sites of Sampula, Niya and Zaghunluq located in the southern Tarim Basin (e.g. Kuang, 2012) as well as from Yanghai and Subeixi archaeological sites located in the Turfan Depression. The periods of use for the graveyards vary greatly (Jiang et al., 2006, 2007, 2009; Ghosh et al., 2008; Wagner et al., 2009; Li et al., 2013). It is currently assumed that the finding sites cover the period from the end of the 2nd millennium BC until the beginning of the 1st millennium AD and show traces of a variety of different cultures. The very good preservation of organic materials over a period of 2000–4000 years is a special feature and is due to the extremely dry Xinjiang climate (Wagner et al., 2009 and references therein).

In this study, textile finds from the burial zones I and III from Yanghai have been investigated to determine what kind of fibers and colorants have been used, and what kind of dyeing technology has been applied. This information should allow some clues concerning questions of cultural exchanges and trading channels. They are also a prerequisite for detailed reconstructions.

2. Material and methods

2.1. Materials

This study presents results from the analysis of fibers and colorants from finds of textiles and accessories from the ancient cemetery of Yanghai located in the Turfan Depression. This graveyard is well known for a number of well-preserved garments (Xinjiang, 2011), including trousers excavated from tombs number M21 and M157 (Beck et al., 2014), which are also analyzed in our paper. The graveyard is assigned to the Subeixi (Subeshi) culture (e.g. Jiang et al., 2006, 2009), which is conventionally dated to the first millennium BC (Chen, 2002; Han, 2007; Xinjiang, 2011). The culture is associated with the Cheshi (Chü-shih) state known from Chinese historical sources (Sinor, 1990). Archaeological and historical data attest it as a developed agro-pastoral society (e.g. Jiang et al., 2006; Ghosh et al., 2008; Li et al., 2013) and suggest wide-ranging contacts between the Subeixi culture domain and other regions of Asia (see Li et al., 2013 for details and references).

2.2. Site setting and archaeological context

The Yanghai graveyard archaeological site (42°48′–42°49′N, 89°39′–89°40′E) conventionally assigned to the Subexi culture is situated in a gravel desert, about 43 km southeast of modern Turfan city (Fig. 1 and Jiang et al., 2006). The area experiences cold and dry winters, with the mean January temperatures about −9.5 °C recorded in Turfan, and extremely hot summers with the mean July temperature of +32.7 °C (Domrös and Peng, 1988). Although the maximum summer temperatures may reach 50 °C in the Turfan Depression, the Bogda Shan and Tian Shan mountain slopes and valleys to the north provide more comfortable environments for people and their livestock (Beck et al., 2014). The annual precipitation values recorded at the Turfan meteorological observatory are about 16 mm (Domrös and Peng, 1988).

The Yanghai graveyard was discovered by local villagers in the early 1970s (see Beck et al., 2014 for more details and references). It occupies an area of about 54,000 m^2 and consists of three distinct burial zones where approximately 3000 burials have been constructed from ca. 1200 cal. BC to 200 cal. AD, of which 501 have been excavated to date (Xinjiang, 2011). The full excavation report has not been published yet. Recently, detailed results of archaeological and archaeobotanical research performed on several individual graves representing the middle occupation phase (ca. 820-240 cal. BC) have been published internationally (Jiang et al., 2006, 2007, 2009) and demonstrated the high potential of such in-depth studies for better understanding of the past environments and subsistence strategies in the region.

In the current paper, textile samples representing tombs M21, M157 (both from burial zone I), M18 (burial zone III) and surface finds without allocation to a particular grave have been analyzed (Table 1). Tomb M21 consisted of two layers and hosted remains of three human bodies. The samples discussed here belong to one mummified body (body C) of a presumably 40 year-old male individual, whose skin, hair and garments (including trousers and poncho) were outstandingly well preserved. The man held a wooden whip rod wrapped in a bronze coil in his right hand. In his left hand, he held a bronze axe. Two leather bags lay beside his waist (one on each side). One bag contained a bronze knife, the other a bronze awl. A leather bridle hung from a wooden stick near the man's head. The bridle was fully strung with ring-shaped or cowrie-shaped bronze buttons and bronze plaques. A wooden horse-bit was attached to the bridle. Those grave goods bestowed on him characterized the deceased as mounted warrior. The same identification applies to the male individual of ca. 40 years of age at death in tomb M157 (Xinjiang, 2011). He was elaborately interred on a wooden bed in 2.64 m depth with a number of goods as whip, horse tail with colorful tassels, bow sheath, and bow beneath a wooden canopy and a cover layer of mud bricks. His trousers were less well preserved but made of colorful fabric. Both tombs, M21

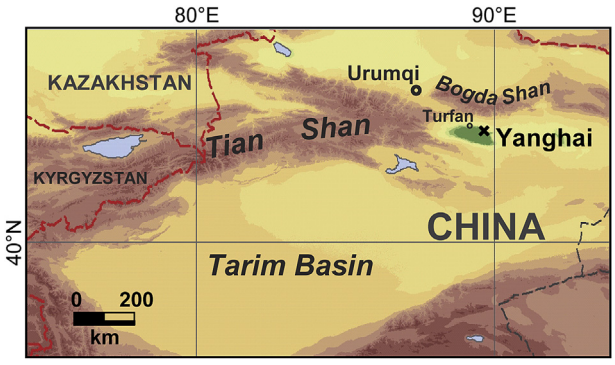

Fig. 1. Map showing major topographic features of the region and the location of Yanghai cemetery in the Turfan Depression (Xinjiang, China). Modern cities and political borders are shown for better orientation.

and M157, were constructed as straight vertical shafts, which the excavators interpret as indicative of an older period.

particular high headdress, and a lacquer plate mark him as person of rank (Xinjiang, 2011).

Table 1
Summary of the investigated objects from the Yanghai prehistoric graveyard and analyses performed on these objects. Notes: A detailed description of the objects 2003SYIM21:19 and 2003SYIM157:14 (object numbers after Xinjiang, 2011) indicated with an asterisk (*) can be found in Beck et al. (2013). In the object 2003SYIIIM18:17 from the tomb M18 the differentiation between warps and wefts is problematic as this object consists of a single fragment without a selvage. The yarn showing the tighter twist is assigned as the warp thread.

Description of the object/object ID	Sample ID	Description of the sample	Analyses
Tomb M21			
Brown trousers* 2003SYIM21:19	20110614/1	Dark brown braid for serging the warps (two-fold warps, S-twist) from the waist region; warps are interwoven by themselves	Fiber and dyestuff analysis
	20110614/2	Twill 2/2 S with dark brown warps (single yarn, S-twist) and light brown wefts (single yarn, S-twist) from the area above leg-appendages	Fiber and dyestuff analysis for warps and wefts; warps and wefts analysed separately.
	20110614/7b	Fragment with felty surface and light weave pattern (light brown and dark brown fibers)	Fiber analysis
Poncho 2003SYIM21:22	20110614/3	Attached plaited ribbon (blended yarn made from red and blue-black fibers, S-twist) with light brown sewing threads (sZ-twisted two-ply yarn)	Fiber analysis covered the ribbon as well as the sewing threads (analysed separately); dyestuff analysis of the ribbon
	20110614/4	Loose yellow-brown residues of fibers	Fiber and dyestuff analysis
	20110614/5	Red single yarn, S-twist	Fiber and dyestuff analysis
	20110616/9	Blue-black sZ-twisted two-ply yarn	Fiber and dyestuff analysis
Tassel with cowrie snail 2003SYIM21:18	20110614/14	Turquoise single yarn, S-twist	Fiber and dyestuff analysis
	20110614/15	Red single yarn, S-twist	Fiber and dyestuff analysis
Tassel from ribbon; area above left knee 2003SYIM21:20	20110615/3	Blue sZ-twisted two-ply yarn	Fiber and dyestuff analysis
	20110615/4	Red sZ-twisted three-ply yarn	Fiber and dyestuff analysis
Leather coat 2003SYIM21:25	20110616/4	Twill S with brown warps (single yarn, S-twist) and brown-black wefts (blended yarn made from brown and black fibers, scarcely twisted) from the inner surface of the coat, float over 2 warps	Fiber and dyestuff analysis for the textile, no separate analysis of warps and wefts
Band covering boot, right lower leg 2003SYIM21:24	20110616/6	Made from reddish and dark brown sZ-twisted two-ply yarns braided band	Fiber and dyestuff analysis
	20110616/7	Yellowish single yarn, S-twist	Fiber and dyestuff analysis
Tomb M157			
Fragment of trousers* 2003SYIM157:14	20110614/8	Dark brown weft (single yarn, S-twist) from superimposed gusset, tabby weave	Fiber and dyestuff analysis
	20110614/9	Yellowish warp (single yarn, S-twist), broken twill	Fiber analysis
	20110614/10	Red weft (single yarn, S-twist)	Fiber and dyestuff analysis
	20110614/11	Dark brown warp (single yarn, Z-twist) and turquoise weft (single yarn, S-twist), broken twill	Fiber and dyestuff analysis
	20110615/2	Yellowish warps (single yarn, S-twist) and blue wefts (single yarn, S-twist), broken twill	Fiber analysis for the warps and wefts (analysed separately) and dyestuff analysis for the wefts
Tassel on horse's tail 2003SYIM157:6	20110614/13	Red single yarns (S-twist) and blue two-ply yarns (sZ-twisted)	Fiber and dyestuff analysis, separate analysis of red and blue yarns
Tomb M18			
Textile fragment from inside a leather boot 2003SYIIIM18:17	20110615/5	Twill with yellowish warps (blended yarn with blue-black fibers, Z-twist) and wefts (single yarn, Z-twist), from the inner side of the leg warmers, 2:2 float	Fiber and dyestuff analysis, separate analysis of warps and wefts
Surface finds without assignment to specific graves			
Samples without object number	20110615/OF8-A	Twill with red warps (single yarn, Z-twist) and wefts (blended yarn with blue fibers, Z-twist), 2:2 float	Fiber analysis on separated warps and wefts and dyestuff analysis on warps
	20110615/OF8-B	Blue wefts (single yarn, scarcely twisted) of a plain weave	Fiber and dyestuff analysis
	20110615/OF11-A	Half-panama fabric (plain weave) containing doubled red warp and a simple red weft (zS-twisted, three-ply yarns, blended with blue fibers) and turquoise incorporated (woven) stripes (zS-twisted two-ply yarn)	Fiber and dyestuff analysis, separate analysis of turquoise strip and of red warps and wefts
	20110615/OF11-B	Twill with blue warps and wefts (single yarn, Z-twist), 2:2 float	Fiber and dyestuff analysis, no separate analysis of warps and wefts

Tomb M18 in burial zone III, in contrast, consists of a vertical shaft and a horizontal side chamber, regarded as a method of construction used towards the end of the burial activity in Yanghai. In this tomb three individuals were interred side by side, a ca. 45 years old male, a ca. 25 years old male and a ca. 12–15 years old female. The oldest male was dressed in high leather boots padded with wool fleece and a textile fabric which was preserved only in fragments. Gold ornaments, a leather cushion beneath his head, a

2.3. Radiocarbon dating

All together, we dated ten samples from Yanghai (see Table 2 for details). This number includes one sample from grave M157 (14/12 – fragment of trousers); four samples from grave M21, including 14/6 – poncho; 14/7a – brown trousers; 16/1 – woven band covering boot, left lower leg (no sample of this object has been used for the analyses of fibers or colorants); and 16/2 – woven band

covering boot, right lower leg; one textile fragment from grave M18; and four samples from surface finds, including 20110615/OF 11-A; 20110615/OF 11-B; 20110615/OF 8-A and 20110615/OF 8-B. No systematic age determination for a statistically significant number of objects from this cemetery has been reported so far. Hence, an unambiguous age determination by radiocarbon dating has been performed for selected samples of the textile finds. All samples were dated in the Poznan Radiocarbon Laboratory, Poland. The accelerator mass spectrometry (AMS) radiocarbon dates provided in Table 2 were then calibrated against the IntCal09 calibration curve (Reimer et al., 2009) using OxCal v4.1.5 calibration software (Bronk Ramsey et al., 2010). Table 2 provides intervals of calendar age, where the true ages of the samples lie within probabilities of ~68% and ~95%.

Table 2
AMS radiocarbon dates processed on samples from the Yanghai ancient graveyard. All dates were generated in the Poznan Radiocarbon Laboratory, Poland. The dates expressed in ^{14}C BP (radiocarbon years before 1950 AD, conventionally taken as the 'present') were converted to calendar years before Christ (cal. BC) using OxCal v4.1.5 calibration software (Bronk Ramsey et al., 2010); r:5, and atmospheric data from Reimer et al. (2009). Given are intervals of calendar age, where the true ages of the samples encompass with the probability of ca. 68% and ca. 95%.

Sample number	Laboratory number	Grave number	Dated material/ archaeological context	Radiocarbon date, ^{14}C BP (±1-sigma)	Calibrated individual dates, cal. BC (68.2% probability)		Calibrated individual dates, cal. BC (95.4% probability)		Calibrated related dates, cal. BC (95.4% probability)	
					From	To	From	To	From	To
20110614/12	Poz-43696	M157	Wool/trousers	2935 ± 30	1212	1056	1261	1041		
20110614/6	Poz-43694	M21	Wool/poncho	2870 ± 30	1114	1003	1188	931	1103	938
20110614/7a	Poz-43695	M21	Wool/trousers	2855 ± 30	1056	940	1122	926	1074	935
20110616/1	Poz-43708	M21	Wool/band 1 (left leg)	2825 ± 35	1016	922	1113	900	1038	926
20110616/2	Poz-43709	M21	Wool/band 2 (right leg)	2810 ± 40	1009	912	1110	843	1038	926
20110615/OF11-A	Poz-57394	Surface find	Textile	2565 ± 30	800	671	806	556		
20110615/OF11-B	Poz-57395	Surface find	Textile	2610 ± 35	812	780	839	601		
20110615/OF8-A	Poz-57397	Surface find	Textile	2525 ± 30	784	567	795	540		
20110615/OF8-C	Poz-57398	Surface find	Textile	2635 ± 30	819	794	888	774		
20110615/5	Poz-59925	M18	Textile	2250 ± 30	389	211	398	202		

Taking into account archaeological background information, we assumed that among four samples from tomb M21, the two samples representing bands (wrapped around the left and right lower leg) have the same age, while samples representing poncho and trousers could be 1–30 years older. The first pair of dates was COMBINED while the second one was grouped in PHASE of the SEQUENCE deposition model in the Bayesian software OxCal (Bronk Ramsey et al., 2010; https://c14.arch.ox.ac.uk/oxcal/OxCal.html) defined above. The calibration results for the tomb M21 obtained with such assumptions are presented in Fig. 2. In this figure, the light silhouette shows age distribution of calibrated ^{14}C date treated as purely individual (i.e. independent of dates of other samples), and the dark silhouette shows modelled age distribution constrained by the conditions of SEQUENCE as described above. The SEQUENCE model allows narrowing the age interval for the textile objects from the tomb M21 (Table 2), so calendar ages are likely between 1074 and 926 cal. BC (~95% confidence interval).

2.4. Analytical techniques and instrumentation

The analytical techniques included optical microscopy (OM), scanning electron microscopy (SEM), the combination of SEM with energy dispersive X-ray spectroscopy (SEM-EDX), attenuated total reflection – infrared spectroscopy (ATR-IR), high performance liquid chromatography-diode array detection (HPLC-DAD) and the combination liquid-chromatography-mass spectrometry (LC-MS/MS). Thus, for fiber analysis a CX41- (transmitted light microscope, from Olympus) and a SZX7- optical microscope (stereomicroscope, from Olympus) was used. Scanning electron microscopy was performed using a LEO 1550VP microscope (from Zeiss) equipped with an energy dispersive X-ray spectrometer (Oxford INCA X-sight, from Oxford Instruments). The system operated at an accelerating voltage of 20 keV.

For the ATR-IR-measurements (range: 200–4000 cm^{-1}) a Tensor 27 FT-IR spectrometer (from Bruker Optik), equipped with a diamond crystal ATR-unit (from Bruker Optik) was used. For the HPLC-DAD investigations a LaChrom D-7000 HPLC-system (from MERCK HITACHI) was used at an operating temperature of 25 °C (Table 3). The LC-MS/MS spectra were taken using an Agilent HPLC 1100 (from Agilent Technologies) coupled with an API 2000 mass spectrometer (from Applied Biosystem, ion source: ESI, temperature: 400 °C, curtain gas: 30 kPa (N_2), ion source gas 1: 60 kPa (N_2), ion source gas 2: 40 kPa (N_2), collision gas: 10 kPa (N_2)). The selective detection of compounds was performed using the MRM mode; prior the measurement the substance-specific parameters were determined by direct inlet of defined reference compounds using an automatized optimization analysis (reference compounds: alizarin, apigenin, brazilein, brazilin, carminic acid, ellagic acid, flavokermesic acid, gallic acid, genistein, indigo, indirubin, isatin, isoindigo, kaempferol, kermesic acid, lawson, luteolin, purpurin, quercetin, quinizarin, rubiadin, rutin; solvents ACN:MeOH (1:1 v/v). A RP-18 Superspher column (125-2 mm, 4 μm) was used as the stationary phase at an operating temperature of 25 °C (Table 4).

Table 3
Experimental conditions used for HPLC-DAD investigations. System 2 is optimized for the investigation of indigoids.

Conditions	System 1	System 2
Stationary phase	RP-18 Kinetex column (100-3 mm, 2.6 μm)	RP-18 ec Nucleodur 100-5 (250–4.6 mm; 5 μm)
Mobile phase	A: ACN (0.1% (v/v) HCOOH) B: H_2O (0.1% (v/v) HCOOH)	A: ACN/THF (9:1 v/v) B: H_2O/ACN/THF (50:45:5 v/v)
Sequence of elution	0–0.5 min 5% A, 0.5–10 min linear gradient until 40% A, 10–15 min linear gradient until 70% A, 15–16 min linear gradient until 100% A, 16–21 min 100% A	0–9 min 20% B; 9–15 min linear gradient until 100% B, 15–25 min 100% B
Flow rate	1 mL/min	0.7 mL/min
Injection volume	10 μL	15 μL
Detector	Diode Array Detector (wavelength range: λ = 200–700 nm)	Diode Array Detector (wavelength range: λ = 200–700 nm)

Table 4
Experimental conditions used for LC-MS/MS investigations. System 3 has optimized conditions for the following compounds: apigenin, genistein, luteolin, quercetin, carminic acid, rutin, kermesinic acid, flavokermesinic acid, brazilin/brazilein, kaempferol, ellagic acid, isatin, lawson, gallic acid (anion sensitive mode); indigo, indirubin, and isoindigo (cation sensitive mode). System 4 is optimized for the anthraquinones alizarin, purpurin, quinizarin, and rubiadin.

Conditions	System 3	System 4
Mobile phase	A: ACN (0.6% (v/v) HCOOH) B: H_2O (0.6% (v/v) HCOOH)	A: MeOH (0.6% (v/v) HCOOH) B: H_2O (0.6% (v/v) HCOOH)
Sequence of elution	0–2 min 20% A, 2–3 min linear gradient until 40% A, 3–10 min linear gradient until 75% A, 10–11 min linear gradient until 100% A 11–21 min 100% A	0–2 min 60% A, 2–7 min linear gradient until 80% A, 7–10 min linear gradient until 100% A, 10–19 min 100% A
Flow rate	0.3 mL/min	0.3 mL/min
Injection volume	5–7 μL	5–7 μL

2.5. Samples and their description

In this study, textile finds from the burial zones I and III have been investigated (Table 1). Samples of the characteristic shades from grave M21 and from fragments of the trousers from grave M157 have been investigated (burial zone I). In addition, a textile fragment from grave M18 (burial zone III) has been analyzed, and some material (samples without object numbering) without definite burial context. Warps and wefts of different color were investigated separately. A separation of fibers of different colors out of one yarn was not possible due to the fragility of the material.

2.6. Methods for the extraction

Depending on the used amount of fibers to be analyzed, the samples were treated with varying amounts of reagents.

2.6.1. Extraction with HCOOH-EDTA (modified according to Valianou et al., 2009)

The samples (0.4–9 mg) were extracted with MeOH/H_2O (400 μL or 200 μL, 1:1 v/v) in the presence of HCOOH (400 μL or 200 μL, 5 M) for 5 min at 100 °C under reflux. An aqueous solution of EDTA (400 μL or 200 μL, 0.5 mM) was added, and heating under reflux continued for another 5 min at 100 °C. After cooling to room temperature, the supernatant was taken off, and the extraction process was repeated. The remnants of the fiber were rinsed with MeOH; this solution and the supernatants were combined, filtered (0.2 μm or 0.45 μm, PTFE-filter), and the solvents were removed under a gentle flow of argon at 65 °C. The residue was dissolved in ACN/MeOH (1:1 v/v), and an aliquot of this solution was directly injected into the HPLC-DAD- or into the LC-MS/MS-system.

2.6.2. Extraction with HCl (modified according to Novotná et al., 1999)

The samples (0.5–31 mg) were extracted with MeOH/H_2O (400 μL or 200 μL, 1:1 v/v) and concentrated aqueous HCl (400 μL or 200 μL) for 15 min at 100 °C under reflux. The extract was dried under argon at 65 °C, the residue was dissolved in MeOH, filtered (0.2 μm or 0.45 μm, PTFE-Filter), and the solvent was removed again. The residue was dissolved in ACN/MeOH (1:1 v/v), and an aliquot of this solution was directly injected into the HPLC-DAD- or into the LC-MS-system system.

2.6.3. Extraction with THF

The samples (0.1–6.8 mg) were extracted with THF (300 μL) and concentrated aqueous HCl (2.5 μL) for 20 min at 80 °C under reflux. After cooling to room temperature, the supernatant was taken off, filtered, and an aliquot of this solution was directly injected into the HPLC-DAD- or into the LC-MS-system.

2.7. Dyeing procedure (modified according to Schweppe, 1992)

2.7.1. Mordant dying with madder (Rubia tinctorum L.)

The woolen fleece was treated for 2 h at 30–40 °C with an aqueous solution of $KAl(SO_4)_2 \cdot 12H_2O$ (20% m/v), the fleece was wrung out and neutralized by application of an aqueous solution of Na_2CO_3 (5% m/v). After having rinsed the fleece with water, the dyeing process was performed.

For the dyeing of the stained fleece, dried rhizomes (free from any cuticles and fibriles) from madder were used. The crushed rhizomes (10 g) were digested in H_2O (200 mL); this mixture was slowly heated to a final temperature of 60 °C. The stained fleece was inserted, turned over a couple of times, rinsed with water, and dried.

2.7.2. Vat dyeing with woad (I. tinctoria L.)

Leaves and stems of *I. tinctoria* L. plants were collected, mashed, pressed into bales, allowed to ferment at room temperature for two weeks, and finally dried. Dried material (20 g) was suspended in H_2O (100 mL) and fermented at room temperature for another three days. Finely grounded $Ca(OH)_2$ (3 g) and water (200 mL) were added, and the mixture was warmed to 90 °C. As soon as an iridescent surface had formed (and some traces of blue pigment have been deposited in the beaker), $Na_2S_2O_4$ (0.5–1 g) was added, until the color of the reaction's foam turned yellowish-green. Wool was inserted, turned over a couple of times, air-dried, and after having turned blue rinsed with water and dried.

2.8. Chemicals and materials

Luteolin (≥90%) and genistein (≥98%) were purchased from Wako Chemicals, quercetin (≥98%), apigenin (≥95%), kaempferol (≥97%), gallic acid (97.5–102.5%) and hydrochloric acid (p. A., 37%) from Sigma–Aldrich, quinizarin (≥98%) from Fluka Analytical, acetonitrile (HPLC gradient grade) from Fischer Scientific, methanol (HPLC gradient grade) and formic acid (99–100%) from VWR, ethylenediaminetetraacetic acid (EDTA) (p. A., ≥99%) from Roth, tetrahydrofuran (HPLC grade) from Applichem, alizarin (97%), rutin (≥97%), 2-Hydroxy-1,4-naphthoquinone (lawson) (99%) and indigo from ACROS Organics, isatin (≥98%) from TCI, ellagic acid from Minakem. Purpurin and karmesinic acid have been obtained from Aldrich's collection of rare chemicals. A mixture consisting of kermesinic acid and flavokermesinic acid has been obtained from the collection of historical colorants (Technical University of Dresden, Prof. Dr. H. Hartmann). Brazilin has been extracted from brazilwood (*Caesalpinia* trees) bought from Kremer Pigmente GmbH & Co KG (Batubara et al., 2010; Wiese, 2013). Rubiadin, indirubin and isoindigo have been synthesized according to procedures of M. Puchalska et al. (2004) and T. Takano et al. (2006).

Sheep wool was purchased from Galerie Smend (100% merino wool), syringe filters (0.2 or 0.45 μm, PTFE) were obtained from Roth, and double distilled water was produced in a distillation apparatus obtained from Gebr. Rettberg GmbH. Madder (*R. tinctorum* L., 2–3 year old plants, sunny habitat rich in humus, clay and sand, collected in autumn), and woad (*I. tinctoria* L., 1–2-year old

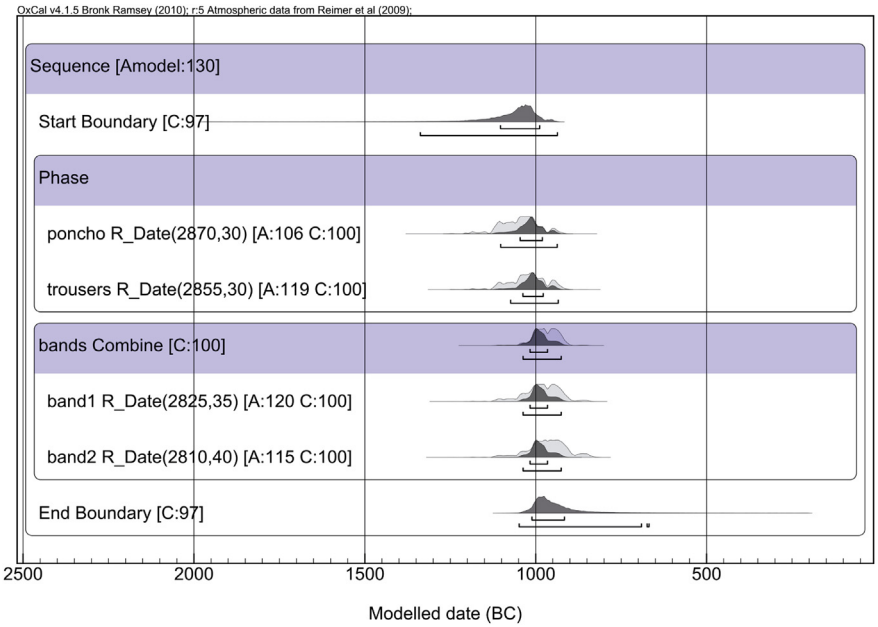

Fig. 2. Results of calibration of ^{14}C dates of the samples from the tomb M21. Light silhouettes present calibration of independent ^{14}C dates, dark silhouettes present calibration of related dates (details in the text). The 1-sigma (68%) and 2-sigma (95%) ranges are given by the horizontal brackets beneath the probability densities.

plants, sunny habitat rich in humus, clay and sand, collected in June) have been grown by one of the authors (C.-H. W.) in his garden.

3. Results and discussion

3.1. Fiber analysis

Methods including optical microscopy, SEM (or SEM-EDX) as well as ATR-FTIR spectroscopy have been used for the analysis of the fibers. The advantage of using these non-invasive techniques is the ability to perform analyses without destruction of material. Thus, the same material could be used for additional analyses of the colorants.

From optical microscopy as well as from scanning electron microscopic (SEM) studies, valuable results could be obtained concerning the fiber diameter, the presence of flakes and their distribution as well as the presence of growing nodes for plant derived fibers. In addition, the application of these technologies allowed an unambiguous documentation of the state of preservation of the fibers.

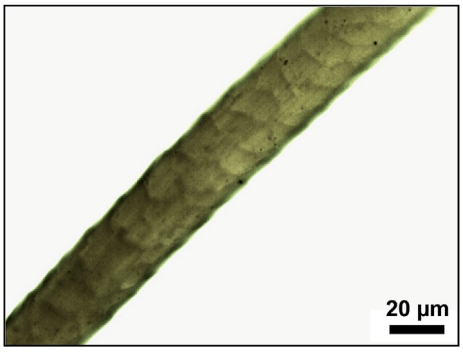

Fig. 3. Transmitted light microscopy (using a polarizing filter) of a fiber from a red welft (sample ID 20110614/10, object: fragment of trousers).

Optical microscopy as well as SEM clearly indicated a degradation of the fibers. To some extent, a scale structure could be determined, and scales typical for wool are depicted in Fig. 3. In some samples, scales could not be detected and the fibers showed cracks. These techniques also demonstrate that many of the yarns have been dyed in a homogeneous manner but they also document the use of raw material possessing different pigmentation. The use of this manufacturing technique seems to be intended to achieve best color results. This is supported by the fact that for samples 20110614/3, 20110615/OF8a and 20110615/OF11a blue fibers have been incorporated into red yarns, and in addition in sample 20110615/5 the warps contain some bulky blue-black colored fibers (diameter of the fiber > 50 µm, indigo has been found as the colorant). This yielded effects of striation.

Elemental analysis of fibers and adhesions was performed by SEM-EDX. Use of this technique allowed some indication concerning the mordant (albeit most natural dyes are mordant dyes) and the presence of metal ions. These metal ions may be capable of altering the colorfulness of the fibers as well as to catalyse aging processes (Joosten et al., 2006). The presence of certain chemical elements like aluminum or iron might originate from the process of dyeing. Their presence, however, could also be the result of a subsequent contamination during the use of the clothes or originate from the surrounding of the grave. Hence, we investigated small areas being free of particles (or adhesives) compared to inorganic particles having been separated from the fibers. SEM-EDX pictures showed (for the fibers free of adhesives) the presence of the elements carbon, oxygen and sulfur (clearly indicating the presence of a keratin fiber), and the presence of Na, Cl, Mg, K and Ca: this is not astonishing, as these elements are ubiquitous. The elements Al, Si and Fe were found in the inorganic particles. Fe and Al were detected in brown, turquoise, yellow, and red colored samples (Fig. 4). There is no connection between the presence of these elements and the process of dyeing. Iron salts are able to

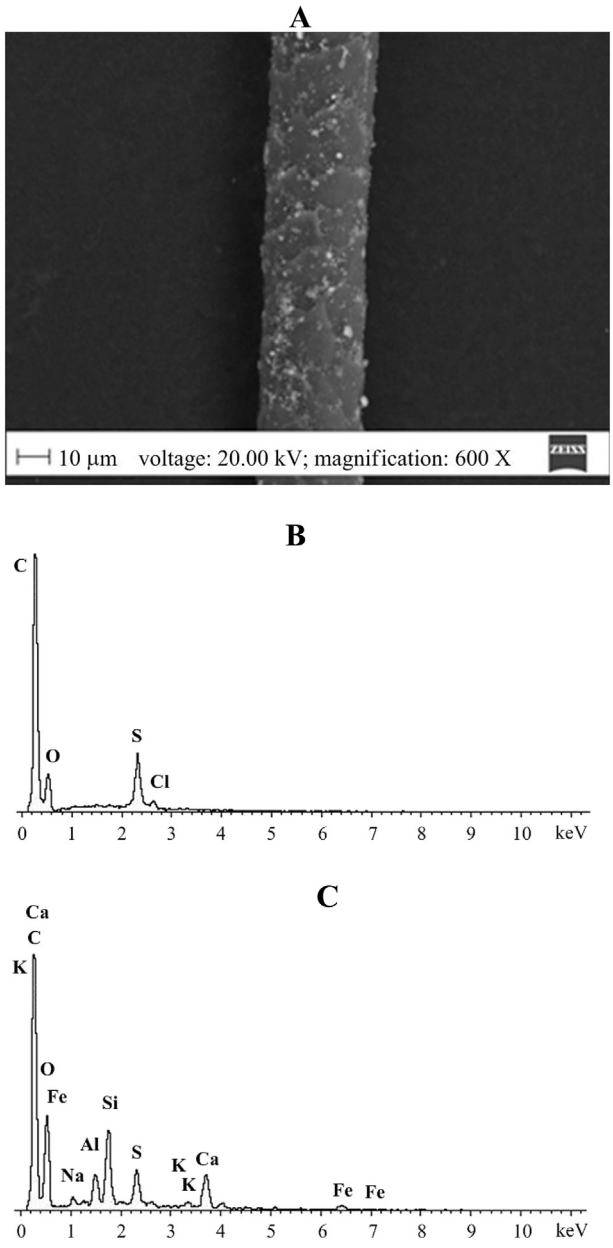

Fig. 4. SEM picture and corresponding SEM-EDX spectra ((B): small areas (free of particles), (C): attached particle) of a fiber taken from the blue two-ply yarn (sample ID 20110614/13; object: tassel on horse's tail), the presence of flakes can be seen to some extent.

alter the colorfulness of red mordant dyes. This "post mordanting effect" results in a darkening of red mordant dyes (Joosten et al., 2006).

The use of ATR-FTIR allowed an easy distinction between plant and animal derived fibers. The fibers consisted of protein, and they show the characteristic absorption bands assigned to the peptide bonds, i.e. characteristic amide I and II vibrational bands. Typically, vibrational bands were detected between $\tilde{\nu} = 1600–1690\ cm^{-1}$ (amide I, C=O stretching vibration, and to a smaller extend CN stretching and C–CN-deformation vibrations) and $\tilde{\nu} = 1490–1560\ cm^{-1}$ (amide II, assigned to CN stretching and NH-deformation vibrations). This is unambiguous proof for the presence of protein fibers.

Thus, using these technologies allows the clear distinction between animal or plant-derived fibers. In addition, these methods foster the differentiation between the fibrous proteins keratin and fibroin – hence an explicit assignment (either wool/hairs or silk). All of the samples showed the presence of keratin fibers (cf. Table 1).

3.2. Dyestuff analysis

Invasive methods (HPLC-DAD and LC-MS/MS, Karadag et al., 2010; Lech and Jarosz, 2011; Yurdun et al., 2011) have been applied for the identification of dyes and their biological sources. Thus, databases have been created containing specific data of known reference compounds; this included retention times, UV/Vis spectra and mass spectra. Reference materials were either purchased from companies, brazilin was isolated from plant material, and indirubin, isoindigo, as well as rubiadin were synthesized; wool fibers were treated with extracts from the dye plants or dye insects, e.g. wool fibers were dyed with madder (*R. tinctorum* L.) or woad (*I. tinctoria* L.) according to traditional recipes.

In a first step, the dyestuff was extracted (according to the class of dyestuff) using one of three optimized methods developed for the extraction of colorants from animal fibers initially extraction of the colorant. For mordant dyes, the extractions were performed after having added acid to facilitate the extraction of the dye. To provide conditions as gentle as possible, many extractions were performed in the presence (Valianou et al., 2009) of diluted formic acid and EDTA (ethylenediaminetetraacetic acid). This kind of extraction preserves the glycosidic linkages of e.g. flavonoid or anthraquinone glycosides. Thus, more information about plant source and the dyeing process can be obtained. Dyestuffs occur in plants mostly both as aglycon and its corresponding glycoside.

Harsh acidic conditions, however, can be realized by an extraction in the presence of hydrochloric acid (Novotná et al., 1999). Therein, glycosidic bonds are hydrolyzed, and colorants such as brazilein, haematein or curcumin (Hofenk de Graaff, 2004, pp. 212–213) are either decomposed or a subject to changes in their structure. Thus, dehydrobrazilein and dehydrohaematein have been described as products resulting from a HCl treatment of samples containing brazilein or haematein, respectively (Mantzouris et al., 2011). This HCl based extraction, however, is known to provide the best results and the highest yields in the extractive process for colorants possessing an anthraquinone-type skeleton, such as alizarin and purpurin (Valianou et al., 2009). Indigoid dyes or more generally vat dyes are best extracted using THF in the presence of hydrochloric acid.

Sample material was available only in limited amounts, and hence only a single method of extraction could be performed. Thus, for red colored samples, extraction in the presence of HCl was performed, since it seemed most likely that the samples contain anthraquinone-type colorants. All glycosides were cleaved during the extraction; this resulted in a higher concentration of the corresponding aglyca. This facilitated their determination tremendously. For blue colored samples the amount of substance was <1 mg, and hence the determination of the colorant was performed using LC-MS/MS.

The results from these extractions as well as some data from the HPLC-DAD and LC-MS/MS investigations have been summarized in Table 5. Different eluents as well as elution programmes and stationary phases have been used. They have been optimized for the different types of dyes. For example, system no. 4 delivers better results for the analysis of certain anthaquinones (e.g. alizarin, purpurin, quinizarin and rubiadin) whereas system no 2 is suited best for the analysis of indigo-type compounds (e.g. indigo, indirubin, isoindigo and isatin).

Table 5
Colorants identified in the textile samples determined by HPLC-DAD and LC-MS/MS.

Sample ID	Color	Extraction method	Detected compounds
20110614/1	Dark brown	HCl HCOOH/EDTA THF/HCl	Gallic acid
20110614/2 warp	Dark brown	HCOOH/EDTA	No dyes detected
20110614/2 weft	Light brown	HCOOH/EDTA	No dyes detected
20110614/3 ribbon	Red and blue-black	HCl THF/HCl	Indigo, alizarin, quinizarin, rubiadin
20110614/4	Tawny	HCl THF/HCl	Gallic acid
20110614/5	Red	HCl	Alizarin and rubiadin
20110614/8	Dark brown	THF/HCl	Indigo
20110614/10	Red	HCl	Alizarin
20110614/11	Dark brown and turquoise	THF/HCl	Indigo
20110614/13	Blue	THF/HCl	Indigo and indirubin
20110614/13	Red	HCl	Alizarin, quinizarin, purpurin, rubiadin, gallic acid
20110614/14	Turquoise	THF/HCl	Indigo and indirubin
20110614/15	Red	HCl	Alizarin, purpurin, rubiadin
20110615/2 weft	Blue	THF/HCl	Indigo and indirubin, ellagic acid
20110615/3	Blue	THF/HCl	Indigo
20110615/4	Red	HCl	Alizarin, quinizarin, purpurin, rubiadin
20110615/5 weft	Yellowish	HCOOH/EDTA	No dyes detected
20110615/5 warp	Yellowish thread with single bluish fiber	THF/HCl	Indigo
20110616/4	Brown	HCl	No dyes detected
20110616/6	Red and dark brown	HCl THF/HCl	Alizarin, quinizarin, purpurin, rubiadin
20110616/7	Yellowish	HCOOH/EDTA	No dyes detected
20110616/9	Blue-black	THF/HCl	Indigo and indirubin
20110615/OF8a warp	Red	HCl	Alizarin, purpurin, rubiadin
20110615/OF8b	Blue	THF/HCl	Indigo
20110615/OF11a red warp and weft	Red thread with blue fibers	HCl THF/HCl	Indigo, indirubin, alizarin, quinizarin, purpurin, rubiadin
20110615/OF11a turquoise strip	Turquoise	THF/HCl	Indigo
20110615/OF11b	Blue	THF/HCl	Indigo and indirubin

3.3. Red dyes plants

The main coloring components of red-shaded samples belonged to the class of anthraquinones, with alizarin, purpurin, and rubiadin being the major components. In some cases quinizarin (Fig. 5) was detected. Hence, it seems most likely that members of Rubiaceae plant family, such as *R. tinctorum* L., *Rubia peregrina* L., *Rubia cordifolia* L. or *Galium verum* L. have been used as a source. The family of Rubiaceae is distributed worldwide, and the roots of these plants have been used for dyeing since antiquity. Early evidence has been reported (Bhardwaj and Jain, 1982) for textile finds from the archaeological site of Mohenja-daro dating to the 3rd millennium BC. In addition, the species *R. cordifolia* L. has a wide ecological range and grows in both in forest edges and as a scrub but also in grassland or open rocky areas (Cardon, 2007, pp. 129–130). Phytolith and palynological studies on ovi-caprid dung from the Yanghai site (Ghosh et al., 2008) suggest that climate conditions were suitable for the growth of plants from the Rubiaceae family in locally moist environments during the first half of the first millennium BC.

Determination of a single species is problematic because the ratio between alizarin and purpurin but also the concentration of minor components including quinizarin strongly depends on dyeing process but also on the growth conditions of the plants (Zhang et al., 2008). Ageing and environmental conditions usually trigger deterioration of major and minor components by many different pathways. Thus, the usage of a mixture of different plants for the dyeing procedure cannot be ruled out.

As far as the dyeing technology is concerned, mordant dyeing has been applied to produce the red shaded dyes. Thus, for the creation of red shaded textiles there was no necessity to import raw material, although this cannot be completely excluded.

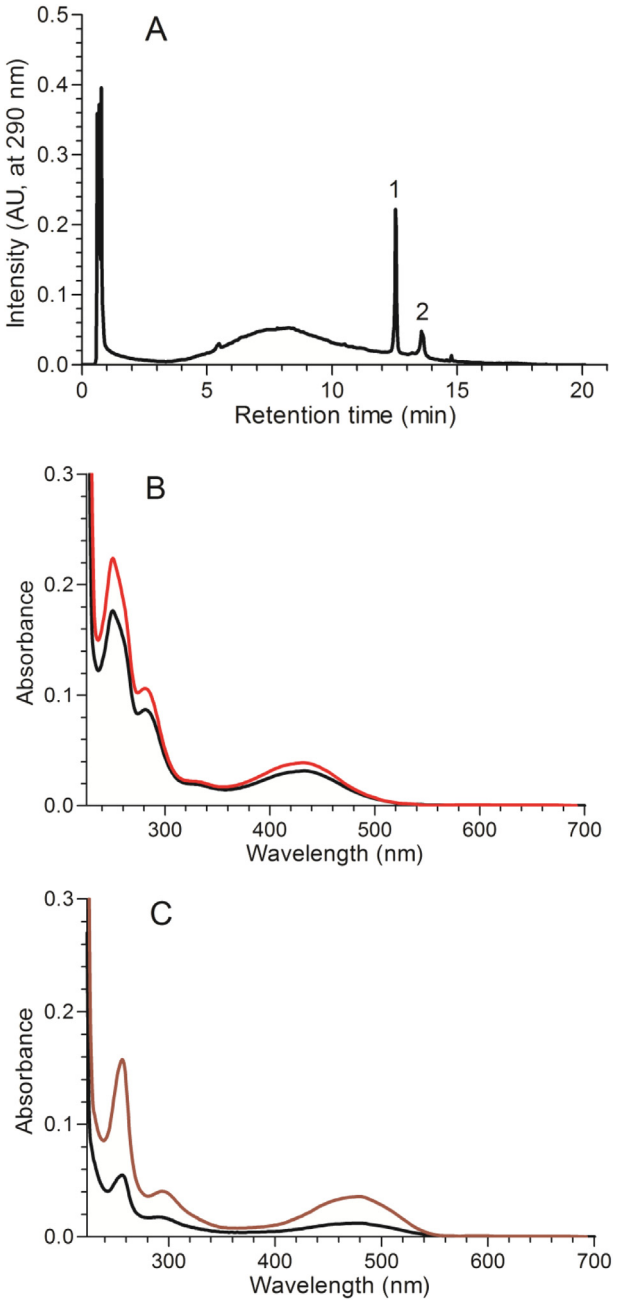

Fig. 5. (A) UV-chromatogram of a HCl-extract from red sample 20110615/OF8a; signal 1: alizarin; signal 2: purpurin; (B) UV/Vis-spectra black graph: signal 1; red graph: reference compound alizarin; (C) UV/Vis-spectra black graph: signal 2; brown graph: reference material purpurin. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

3.4. Blue dyes

The different shades of blue colors (blue, blue-black, turquoise) have been obtained by dyeing using the vat dye indigo. In-depth analysis of the sample revealed the presence of isomeric indirubin. As a source, different plants might have been used, e.g. true indigo (*Indigofera tinctoria* L.), dyer's knotweed (*Polygonum tinctorium* Ait.) or Chinese woad (*Isatis indigotica* Fortune ex Lindley). In the case of using dyer's knotweed, a plant that needs a hot, humid tropical climate (Cardon, 2007, pp. 379–386) for growth, and true indigo (needing warm climates with an average annual temperature of 23 °C (Cardon, 2007, pp. 354–355)) an import of raw material (plants, dyes or prefabricated threads) from southern regions would have been mandatory. The results from the analyses exclude the identification of a single species of plants. Even though all plants exhibit a different glycosylation pattern (Cardon, 2007, pp. 337–338) in their colorless precursors of indigo, but during work-up of the crude plant material the carbohydrates are cleaved off by enzymatic hydrolysis and indoxyl is formed. Indigo, the actual colorant, is obtained by oxidation of the indoxyl. Depending on differences in the extraction processes and during dyeing, different constituents of natural indigo can be formed. For example, the formation of indirubin depends on pH and temperature during the dyeing process (Cardon, 2007, pp. 337–339).

Different shades of blue can be realized by slight modifications of the basic dyeing process. Thus, the manufacture of these different shades can be regulated by choosing the number of times the fibers were immersed into the vat. In addition, shades of green can be gained by a process of "over-coloring" using a yellow colorant. The investigation of the turquoise colored samples, however, gave no evidence for the use of an additional yellowish dye. Hence, most likely the different shades of blue are the result of variations during the dyeing process.

3.5. Brownish and yellowish dyes

Shades of brown and black can be made using a variety of different plants, among them *Juglans regia* L. Here, gallotannins or condensed proanthocyanidines in the presence of iron pickling solutions have been used. These compounds belong to the class of hydrolzable and condensed tannins, respectively. They can be obtained from a variety of different shrubs and trees of the genus *Rhus*. Gallic acid and ellagic acid are products from the hydrolysis of metal-gallotannin complexes.

Gallic acid was found in a dark brown and a tawny colored wool sample. Gallic and ellagic acid can be found almost everywhere; these compounds are also formed during degradation processes of organic material. In addition, both compounds have been detected in a red and a blue sample. No additional colorant could be found in all of the brown and yellow samples of this study. Thus, the use of brown and/or white fleeces seems most probable. The shades of color seen today can be attributed to the natural pigmentation of the wool fibers as well as to ageing and degradation effects due to long-term storage in desert sands.

4. Conclusions

All textiles investigated in this study consist of keratinic fibers from animals. Red and blue colored textile samples have been investigated utilizing a broad variety of non-invasive as well as destructive analytical techniques. These textiles gained their color from using mordant as well as vat dyeing technology. Textiles colored red have been made by treating the wool fibers with extracts from plants of the Rubiaceae family: most probably these plants grew locally. The different shades of blue result from an indigo-dyeing process. As a source for this dyeing, different plants might have been used, but the determination of a single species was not possible. The use of Chinese woad (*I. indigotica* Fortune ex Lindley) as a dyeing plant cannot completely be ruled out. True indigo (*I. tinctoria* L.) or of dyer's knotweed (*P. tinctorium* Ait.), however, would have been imported, as both plants need warm to hot climates for growth. Under these circumstances, import of raw material (plants, dyes, or prefabricated threads) from southern regions would be probable.

The results from the radiocarbon dating of several carefully selected samples taken from the objects allow an unambiguous dating the objects; e.g. the sample 20110614/12 representing wool trousers from the tomb M157 has been radiocarbon-dated to an interval from 1261 to 1041 cal. BC with ~95% confidence. Hence, we could provide scientific proofs for the use of madder and indigo for textile dyeing in the Turfan region as early as 3200-3000 years ago, i.e. during the Bronze Age.

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