Gilt Leather Threads in 11th-15th Century Textiles

Cristina Scibè, Caroline Solazzo, Isetta Tosini, Thomas Lam, Edward Vicenzi, Maria José González López

Abstract

For thousands of years metal threads have been used for the decoration of textiles and clothes to create luxurious objects. Between the 11th and the 15th centuries, gilt and silvered organic strips (made with animal skin, animal gut or paper) were among the metal threads most commonly used, either as flat strips or wound around a fibrous core. These types of metal threads were very popular due to their flexibility and reduced cost; indeed, the metal coating was applied in one or multiple thin layers on the organic substrate that made up most of the thickness of the thread.

While the use of paper as substrate was restricted to East Asia, animal substrates were widely used across Europe and Asia resulting in what has been called "membrane threads". The metal-coated organic threads have a layered and heterogeneous structure, of which the metal layer has mostly been the target of investigation due to the complexity of studying the organic component. In the present work, metal-coated skin threads from medieval Spanish, Sicilian, Middle Eastern, Central and East Asian textiles were investigated by a multi-analytical approach, combining for the first time Optical Microscopy, Scanning Electron Microscopy/Energy Dispersive X-Ray Spectroscopy and Proteomics.

Keywords

gilt leather, metal-coated organic threads, medieval textiles, proteomics, SEM-EDX, optical microscopy

Introduction

The use of gold and other precious metals for the decoration of clothes and textiles to create objects of luxury and power can be traced back as early as the Bronze Age.

Between the 11th and the 15th centuries, special type of metal threads largely used in weavings and rarely in embroideries, was historically known as "membrane threads". These threads were made by gilding/silver-plating an organic material, such as leather, parchment (vellum), membrane or paper, and then cutting the gilded material into narrow strips. These strips were used either flat (**Figure 1**) or wound around a fibrous core (silk, linen, cotton or other yarns) in the production of composite threads (**Figure 2**).

The introduction of the "metal-coated organic threads" represents a very important achievement in the development of metal threads technology. They were very popular and preferred to the earlier pure gold threads due to the flexibility of the wrapping materials and the reduced cost; indeed, the metal coating was applied in one or multiple thin layers on the organic substrate that made up most of the thickness of the thread. These features led to their extensive use in fabric decoration for a variety of textures and visual effects.

All the studies conducted until now dealing with "organic threads" show a lack of a unified nomenclature and classification. Historical literature often refers to "membrane threads" (made from membranous tissues) as "Cypriot gold", "Byzantine gold" but also as "skin gold", while some authors consider as "membrane threads" all types of gilt/silvered organic strips, generating a terminological ambiguity around them. Therefore, in the present paper, the authors will refer to this type of metal threads as "metal-coated organic threads", subdividing the category in three main groups according to the organic substrate: "metal coated skin threads" (made by leather or parchment strips), "metal-coated membrane threads" (made by membranous strips), and "metal-coated paper threads" (made by paper strips).

Figure 1: Magnified view. Gilded flat strip. Central Asia, 14th c.; Stralsund Museum, 1862: 16. Image acquired by HIROX KH-8700 3D digital microscope (Hirox-USA, Inc., NJ) by Cristina Scibè © Museum Conservation Institute, Smithsonian Institution

Figure 2: Magnified view. Gilt wrapped-skin strips. Italy?, possibly 13th c.; Cooper Hewitt, Smithsonian Design Museum, 1902-1385. Image by Cristina Scibè © Museum Conservation Institute, Smithsonian Institution

Metal-coated skin threads

It is unclear how early the practice of making metal-coated organic threads began. It is generally assumed that they were probably first manufactured in Asia. The finding in the Famen Temple (Fufeng County, Shaanxi Province) of a 6th century AD gilded organic thread, on which the substrate had completely degraded, represents the first evidence of the "organic" thread production in China (Karatzani, 2009). The spread to Southern Europe began probably from Byzantium, western Asian or African regions by the Levantine trade, through the ports of Cyprus, hence the name "Cyprus gold" or "Byzantine threads", as early as in the 10th-11th century.

According to the textile and scientific literature[1], and on the strength of archaeological evidences, while the use of paper substrate was restricted to the Far East (China and Japan), animal substrates were widely used across both European and Asian textile productions. Braun-Ronsdorf (1961) asserts that the presence of a gilt-leather thread in a fabric is a useful indication of its Far Eastern origin. According to Braun-Ronsdorf, gilt and silvered leather strips were first used for the decoration of silk brocades in the Far East as untwisted wefts. Then, the Mongol conquest of Persia in the 13th century led to the introduction of the gilt-leather technique into Persian brocade weaving and consequently in the Middle East. From there, beginning in the 14th century, these Chinese fabrics made their appearance in Western Europe and became very popular. No mention is made regarding the Arabian production of the Northern Africa, Iberian Peninsula and Sicily under the Muslim domination, which instead had developed its own production of gilt skin threads during the medieval times. To the best of our knowledge, the earliest evidence of gilt skin threads in Europe dates back to the 10th-11th century in al-Andalus; the 10th century "Pyrenees" Peacock tapestry and the early 11th century veil of almaizar Hisham II, both belonging to the Cordoba *Tiraz* manufacture (Borrego et al., 2017), and the 11th century shroud of St Lazarus from Autun, assigned to the Andalusí manufacture of Almeria (Rinuy, 1989).

Meanwhile, in European workshops (especially Italy and Germany) threads were mostly made with very thin animal membranes, usually reported as animal gut, but stomach or bladder membranes might have been used as well. The production started in the 13th and 14th centuries as a less expensive imitation of the "Cyprus Gold". If in Europe, from the early 15th century onwards, solid metal threads gradually replaced organic metal threads with the advent of the velvet weaving, in the Far East gilt paper and leather were still in use in the 20th century (Járó, 2003; Solazzo, unpublished data).

Not only their origin, but also their manufacturing techniques still raise several questions. Metal-coated skin threads have a layered and heterogeneous structure: the metal layer (in the form of powder or thin leaves) was applied on skin tissues (leather, parchment, vellum) by an adhesive medium (animal glue, egg or bole), before

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[1] On the historical development of metal threads and their geographical spread: Braun-Ronsdorf (1961), Wardwell (1989), Indictor et al. (1989), Járó et al. (1990).

cutting the skin into narrow strips. The metal surface has mostly been the target of investigation; conversely, the distinction among the substrate materials (base layer and adhesive) has often been clouded in the literature, probably due to analytical limits. Until now, the investigation of the organic component has been mainly conducted by morphological analysis, which has proven to be a useful preliminary examination, yet a subjective one. Indeed, since it is based on visual identification, observations are influenced by variations along the threads and the appearance of the substrate that can be altered from its original. De Reyer's DNA amplification study (2002) has been one of the few studies conducted on the organic component by means of more advanced techniques beyond microscopic ones. However, on account of the extensive fragmentation of the DNA molecules and the presence of others protein-based materials in the complex structure of these threads, the 2002 study was inconclusive at the time. The resulting lack of a unified investigation method of these materials led to their confusing and incomplete classification. The recent proteomics application to the study of ancient animal membranes (Popowich et al., 2018), has opened new perspectives both in species identification of the base layer and in the characterization of the protein adhesive.

The present work shows some of the results of parallel research projects that the authors are carrying on with the aim to organize the knowledge acquired until now on these materials, providing at the same time new insights by a multi-disciplinary and multi-analytical approach to the subject. Correlating our data with textile research on medieval textiles, such investigation forms the hypothesis that it would be possible to relate materials and manufacturing techniques of a metal-coated organic thread to a specific period and workshop.

Materials and Methods

A series of 12th-14th century textiles of Central and East Asian, Middle Eastern, Spanish and Italian origin were selected from various European and American museums, such as the Brandenburg Textile Treasury, the Stralsund Museum, the Terrassa Textile Museum and Documentation Center, the Modena Museum of Civic Art, the Prato Textile Museum, and the Cooper Hewitt Smithsonian Design Museum of New York.

We collected 76 samples of metal-coated organic threads, in detail 32 membrane-type threads and 44 samples of skin-type threads, trying to reach a representative number of samples for each period and textile center in order to determine and compare European (Spanish and Italian) and Oriental (Near East and Far East) patterns of fabrication.

The samples were investigated by a multi-analytical approach, combining traditional techniques such as Optical Microscopy (OM) and Scanning Electron Microscopy (SEM) /Energy Dispersive X-ray Spectroscopy (EDS) with proteomics. Here, we present selected results obtained on metal-coated skin threads. The present investigation can be considered as the first application of proteomics analysis, microscopy and metal analysis to the morphological and compositional characterization of the diverse layers of gilt skin strips.

SEM-EDS: was performed using an Hitachi S3700N scanning electron microscope (SEM) equipped with a Bruker 6|60 silicon drift X-ray detector and Esprit V2 analysis software for compositional imaging and analysis. Samples were analyzed at a working distance of 10 mm with a15 kV electron beam and < 1 nA of current. A large solid angle 5 segment backscattered electron detector was also used for average atomic number imaging. Hyperspectral X-ray imaging of the specimens allowed for extraction of spectra from compositional images. P/B ZAF matrix corrections were applied to raw X-ray data for quantification; these results were then normalized and reported in elemental mass per cent.

Optical microscopy: a cross-section of each sample was prepared by embedding a micro-fragment perpendicularly in polyester resin and polishing with silicon carbide discs (grit from p120 to p1200). The cross-sections were observed by Optical Microscopy in UV reflected light using a Zeiss Axio Immager A1 microscope, equipped with a mercury vapor lamp HBO100 with the Filter set 49 (excitation G - 365 nm, beam splitter FT – 395 nm, emission BP – 445/450).

Proteomics: starting with samples as small as 1×1 mm, the proteomes of each sample were characterized by nanoLC-tandem mass spectrometry (separation of peptides followed by mass spectrometry analysis on a Thermo Scientific Dionex Ultimate 3000 UHPLC system coupled to a Thermo Scientific LTQ Velos Dual Pressure Linear Ion Trap mass spectrometer), following a protocol for extraction and trypsin digestion of the proteins present in the whole samples adapted from (Solazzo et al., 2017). Data files were imported into PEAKS studio 8.5 (Bioinformatics Solutions Inc.) for searching against protein sequence information available in public databases (Uniprot for general searches against mammals and birds, and NCBI for searching collagen proteins for mammals and fish). In addition, because sheep (*Ovis aries*), goat (*Capra hircus*) and cow (*Bos taurus*) were found to be the most common species identified, the identification was validated through a series of distinctive markers from collagen type I and type III chains.

Results and Discussion

Preliminary examination:

The first step of the investigation was the examination of gilt skin threads directly on the textile, in order to take note of the variability of some technical features that describe the thread technology and that may be lost in the sampling. For example, in the same textile, we may find the presence of more than one type of metal thread. Then, a careful examination of the sample under a stereo binocular microscope was conducted to define the main morphological characteristics of the thread, such as the type of thread, its diameter, the strip width, and the wrapping direction (S or Z twist) of both the strip and the fibrous core. The samples belonging to the Asian textiles are typically of the flat-strip type. All the other samples are metal-wrapped threads. The threads belonging to European textiles are mostly Z-twisted around a silk core **(Figure 3)**, while the Middle Eastern skin strips are S-twisted around a linen core **(Figure 4)**.

SEM-EDS Analysis:

Qualitative and semi-quantitative analyses were performed on the metal surface by using an energy dispersive spectrometer (EDS) system attached to the scanning electron microscope. By the micro-morphological examination of the metal coating, it was possible to distinguish mainly two gilding media: powder **(Figure 5)** and leaves **(Figure 6)**.

The accuracy of the quantitative analysis of the metal layer is highly dependent on its thickness and homogeneity. Gilt/silvered skin threads are a complex target of samples due to their uneven and thin metallic surface. Therefore, the values obtained for gold and silver must be considered as indicative of the original metal surface composition instead of absolute values. Consequently, the results of the metal layer composition will only be suitable for purposes of comparison with other published data, since most studies conducted on metal threads have been using SEM-EDS.

Figure 3: Gilt skin thread. The strip is wound around a silk core in Z-twist. Spain, 13th c.; Cooper Hewitt, Smithsonian Design Museum, 1902-1-977. Image acquired by HIROX KH-8700 3D digital microscope (Hirox-USA, Inc., NJ) by Cristina Scibè © Museum Conservation Institute, Smithsonian Institution

Figure 4: Gilt skin thread. The strip is wound around a linen core in S-twist. Italy? Persia?, 14th-15th c.; Cooper Hewitt, Smithsonian Design Museum, 1902-1-271a. Image acquired by HIROX KH-8700 3D digital microscope (Hirox-USA, Inc., NJ) by Cristina Scibè © Museum Conservation Institute, Smithsonian Institution

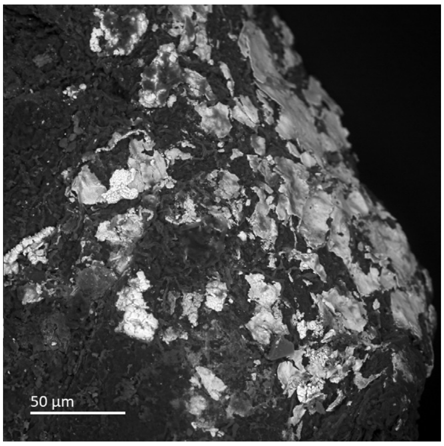


Figure 5: Scanning electron micrograph of a silver powder coating on a skin strip. Spain?, 13th c., Cooper Hewitt, Smithsonian Design Museum, 1902-1-229b. Image by Thomas Lam © Museum Conservation Institute, Smithsonian Institution

In this study, EDS analysis of the surface showed that the metal coating is mostly made of gold and silver, often with a higher amount of gold than silver in the Spanish and Sicilian samples (except for one sample that showed the presence of silver with traces of gold (<2.60 wt%)). In 14th century Middle Eastern, Central Asian and East Asian samples, the coating is made of well-refined gold. Our results agree with former scientific and historical investigations aimed at provenancing threads based on their metal composition. For those samples belonging to textiles of uncertain provenance, a tentative assignment will be made once the evaluation of all the analytical data has been carried out.

In order to understand whether the metal coating is made of a single or multiple metal layers, the EDS elemental mapping appears to be a representative method **(Figure 7)**. In the samples shown below, the homogeneous distribution of both the main elements, gold and silver, might lead to the conclusion that a single layer of gold with silver was used (A). While, the homogeneous distribution of silver and the discontinuous presence of gold might indicate the use of two metal layers (B), as in gilt-silver coatings.

Comparison of the OM and Proteomics analysis:

The optical examination of the stratigraphy of gilt skin strips is necessary to understand their layered structure and to establish further analytical steps, to determine the thickness of the strip, and to identify the fibrous core. All the skin-type samples present the following layered sequence: metal coating, adhesive layer and base layer **(Figure 8)**. Few samples show the presence of a "lacquer" coat above the metal, already detected by the visual examination of the textile itself. Such "lacquer" might have been used as a varnish to give a specific shade to the gold leaves; further investigations will be needed to identify its nature.

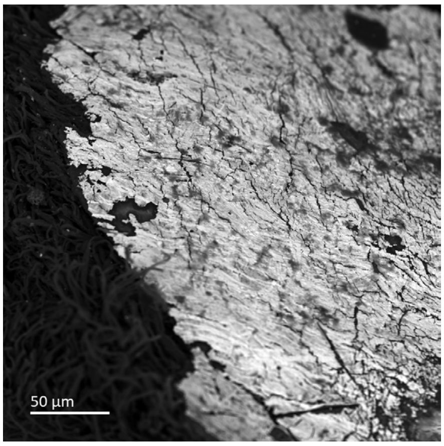


Figure 6: Scanning electron micrograph of a gold leaf coating on a skin strip. Italy, 14th c., Cooper Hewitt, Smithsonian Design Museum, 1902-1-292a. Image by Thomas Lam © Museum Conservation Institute, Smithsonian Institution

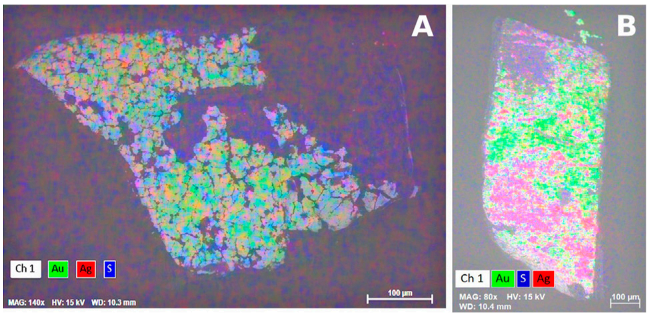


Figure 7: Results of the EDS surface mapping analysis. (A) Gold (higher amount) with silver can be detected on the surface of the strip. The spatial distribution of the elements is homogeneous. Spain, 13th c.; Cooper Hewitt, Smithsonian Design Museum, 1938-78-1. (B) Gold (lower amount) with silver can be detected on the surface of the strip. The spatial distribution of the silver is homogeneous while the presence of gold is detectable only in some areas. Spain, 14th c.; Cooper Hewitt, Smithsonian Design Museum, 1902-1-311. Image by Thomas Lam © Museum Conservation Institute, Smithsonian Institution

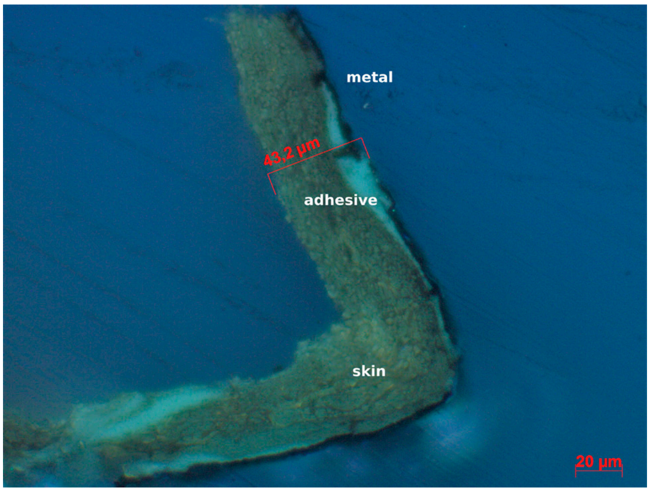


Figure 8: Cross section micrograph of a skin strip, UV reflected light, 20× magnification. Spain, 13th c., Textile Museum and Documentation Center, CDMT 6005. Image acquired by Axio Zeiss Imager A1, by Cristina Scibè, METHIT PhD thesis © Opificio delle Pietre Dure

By cross section analysis, we can establish different typologies and production technologies of gilt strips. For example, the whitish or yellowish color of the skin might reveal the use of a parchment or vellum strip while a dark brown or blackish tone could be indicative of a leather produced by a vegetable tanning process. Due to the stretching process they undergo, the collagen fibers in parchment and vellum are aligned while in leather their distribution tends to be more random. Moreover, the presence of depressions left from depilated hair follicles is more often observable in leathers than in parchment or vellum. Finally, differences in the skin strip thickness might be representative of different animal skins or

Table 1: proteomics results of selected samples

Sample	Layer	Database	Species Latin name	Species common name	Protein	Score -10lgP	% coverage	# peptides
Central Asian, 14th century	Skin base identification	NCBI Collagen	*Ovis aries*	Domestic sheep	COL1A1	166.19	43	59
					COL1A2	164.00	46	52
					COL3A1	123.67	28	33
	Adhesive identification	NCBI Collagen	*Acipenser schrenckii*	Amur sturgeon	COL1A1	118.02	23	39
					COL1A2	157.16	48	67
Hispano-Moorish, 13th century	Skin base identification	NCBI Collagen	*Capra hircus*	Domestic goat	COL1A1	224.01	61	110
					COL1A2	211.98	62	81
					COL3A1	159.14	42	50
	Adhesive identification	Uniprot Aves	*Gallus gallus*	Chicken	OVALBUMIN	85.28	33	11

different skin regions. However, the advanced state of deterioration and damage of the skin materials often make such observations challenging.

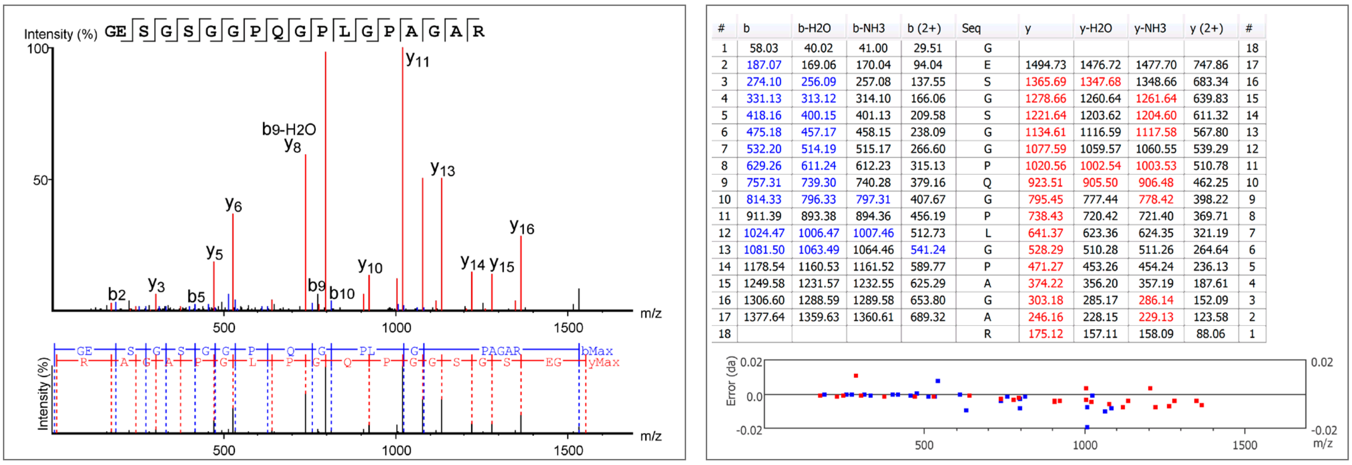
In this regard, proteomics analysis was conducted to determine the origin of the organic components and the species used. Using nanoLC-MS/MS, all proteins that can be extracted from the skin substrate and a proteinaceous adhesive are characterized in one single analysis, thus minimizing the amount of sample necessary per analysis. The relevance and novelty of the present study is represented by the comparison of the cross section's morphological information with proteomics data **(Table 1)**. For the first time, we will be able to identify the different organic layers and "reconstruct" the manufacturing techniques of gilt organic strips.

The proteomics results of a Central Asian thread **(Figure 1)** and a Spanish thread **(Figure 8)** are given in **Table 1**. In the Central Asian thread, the base layer was found to be made from sheep skin while the adhesive was identified as a fish glue with a best match found for Amur sturgeon. Due to limitations in fish sequences available (only two species of sturgeon have collagen sequences in NCBI), the exact species identification is still under review **(Figure 9** shows a species-specific peptide from sturgeon). In the Spanish thread, the base layer was found to be from goat skin, while the adhesive was identified as egg white from chicken due to the identification of ovalbumin.

Further research is under way to characterize the skin substrate from the adhesive separately; this would be particularly relevant in cases where animal glue from the same species as the skin substrate had been used in the adhesive.

Conclusion

This paper reports on an ongoing research project, which focuses on metal-coated organic threads. The outcomes of this research will considerably expand our knowledge in this field by answering some questions of great academic interest, and especially contribute to better define the different typologies of metal-coated organic threads by refining the organic substrate classification.



#	b	b-H2O	b-NH3	b (2+)	Seq	y	y-H2O	y-NH3	y (2+)	#
1	58.03	40.02	41.00	29.51	G					18
2	187.07	169.06	170.04	94.04	E	1494.73	1476.72	1477.70	747.86	17
3	274.10	256.09	257.08	137.55	S	1365.69	1347.68	1348.66	683.34	16
4	331.13	313.12	314.10	166.06	G	1278.66	1260.64	1261.64	639.83	15
5	418.16	400.15	401.13	209.58	S	1221.64	1203.62	1204.60	611.32	14
6	475.18	457.17	458.15	238.09	G	1134.61	1116.59	1117.58	567.80	13
7	532.20	514.19	515.17	266.60	G	1077.59	1059.57	1060.55	539.29	12
8	629.26	611.24	612.23	315.13	P	1020.56	1002.54	1003.53	510.78	11
9	757.31	739.30	740.28	379.16	Q	923.51	905.50	906.48	462.25	10
10	814.33	796.33	797.31	407.67	G	795.45	777.44	778.42	398.22	9
11	911.39	893.38	894.36	456.19	P	738.43	720.42	721.40	369.71	8
12	1024.47	1006.47	1007.46	512.73	L	641.37	623.36	624.35	321.19	7
13	1081.50	1063.49	1064.46	541.24	G	528.29	510.28	511.26	264.64	6
14	1178.54	1160.53	1161.52	589.77	P	471.27	453.26	454.24	236.13	5
15	1249.58	1231.57	1232.55	625.29	A	374.22	356.20	357.19	187.61	4
16	1306.60	1288.59	1289.58	653.80	G	303.18	285.17	286.14	152.09	3
17	1377.64	1359.63	1360.61	689.32	A	246.16	228.15	229.13	123.58	2
18					R	175.12	157.11	158.09	88.06	1

Figure 9 a-b: Example of species-specific peptide from Amur sturgeon: COL1A2, GESGSGGPQGPLGPAGAR, m/z 776.3818, z=2

The data presented here are indicative of the variations on the morphological and technological characteristics of metal-coated skin-type threads. For the first time, the species identification of the base layer and the characterization of the protein adhesive was achieved in a single analysis, allowing the description of the threads beyond the sole analysis of the metal layer. The combination of proteomics with traditional microscopic techniques will contribute to uncovering different production technologies and to resolving the age and provenance of gilt organic threads. Indeed, the provenance study of medieval textiles has usually been done by comparing the patterns with other textiles of known age and origin. However, the extensive trade of raw materials (including metal threads) and movements of workshops and textile workers have somehow blurred the lines defining the origin of many medieval textiles. Not surprisingly, in the textile literature and in museum's inventories, fragments from similar textiles appear with varying geographical assignments. The ultimate goal of this project hopes to combine the analytical data from metal threads, along with those from fibers and dyes, with weaving techniques and patterns, thus revealing the complex nature and origin of the threads and their interworking towards the final textile.

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Biographies

Cristina Scibè holds an MA in Science and Technologies for Archaeological and Artistic Heritage from the University of Padua and is currently completing her PhD in the "Art and Heritage" program of the Faculty of Fine Arts, University of Seville. She is conducting her research on "Metal threads in 11th-15th century Hispano-Islamic and Italian textiles: methodological approach for the investigation of materials and manufacturing techniques".

Caroline Solazzo has been a research scientist at the Smithsonian's Museum Conservation Institute since 2017 and a research fellow since 2012. She has a PhD in analytical chemistry from the University of Lille 1. Her research is focused on the utilization of protein products in material culture and the development of proteomics methods for the analysis of ancient proteins in cultural heritage. She specializes in the characterization of keratin-based tissues and other textile fibers.

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Thomas Lam has a Ph.D. in Ceramics from Alfred University, NY. After earning his Ph.D., he completed postdoctoral employment at the National Institute of Standards and Technology. He is a physical scientist at the Museum Conservation Institute (MCI), where he applies his knowledge of material science and characterization skills (including SEM-EDS, CL, X-ray fluorescence, and microfade testing) as part of the MCI technical studies team.

Edward Vicenzi is a research scientist at the Smithsonian Institution's Museum Conservation Institute (MCI). He utilizes microbeam techniques to study of museum specimens to understand their history and origin. He served as the Director of the Analytical Laboratories in the National Museum of Natural History's (NMNH) Department of Mineral Sciences, and the co-manager of the Imaging and Analysis Facility at Princeton University. Additionally, he served as President and Director of the Microanalysis Society (MAS), organizer of MAS topical conferences, chair of the Smithsonian's Senate of Scientists, co-chair of the M&M conference, organizer for symposia at M&M annual meetings, AGU, and Goldschmidt conferences. He is the past President for the International Union of Microbeam Analysis Societies (IUMAS), and is an editorial board member for *Heritage Science*. He obtained a PhD from Rensselaer Polytechnic Institute, an MS from the University of Oregon, and a BSc from McGill University, all in Earth Sciences.

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