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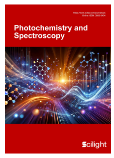

Article

Combined Raman and X-ray Fluorescence Spectroscopy for Garnet Provenance in Longobard Fibulae †

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† This article is dedicated to Prof. Giuseppe Zerbi in recognition of his outstanding scientific contributions to Spectroscopy.

How To Cite: Dalceggio, M.; Possenti, E.; Zanatta, M.; et al. Combined Raman and X-ray Fluorescence Spectroscopy for Garnet Provenance in Longobard Fibulae. *Photochemistry and Spectroscopy* **2026**, *2*(1), 7. https://doi.org/10.53941/ps.2026.100007

Received: 5 November 2025
Revised: 12 December 2025
Accepted: 12 December 2025
Published: 28 February 2026

Abstract: Archaeological research is increasingly relying on materials characterization protocols to provide information on artefacts and remains. Indeed, analytical results are fundamental to draw meaningful conclusions as concerns: raw materials procurement and provenance; technological advancement levels; commercial exchanges and societal aspects. Experimental measurements are done both *ex-situ*, by extracting small portions of material from the analyzed object, and *in-situ*, exploiting non-destructive techniques, that can probe the material without sampling or alteration. This approach is also interesting for *ex-situ* analyses, as it opens the possibility of combining different experimental techniques on the very same sample, even if a limited amount of material is available. In the present paper, we highlight the possibilities offered by the combined use of Raman and X-ray fluorescence (XRF) spectroscopy of Archaeological specimens. These two techniques are non-destructive and provide information on the elemental composition as well as on the structure of the investigated materials. The combined analytical and structural information is particularly important when dealing with natural materials, like garnets in the present study, commonly featuring intrinsic fluctuations in the chemical composition. Moreover, the approach can smoothly assess the actual origin of materials that can be of natural, like minerals, or anthropogenic, like glasses, origin. As a case study, we critically discuss the results obtained on two brooches, decorated with garnets and glasses, belonging to the Longobard culture in the North-Eastern Italian regions. The combination of the analytical data, from XRF spectroscopy, and of the structural ones, from Raman spectroscopy, provide indications on the composition of the base alloys and provenance of the mentioned stones.

Keywords: garnet; cloisonné technique; Raman spectroscopy; X-ray fluorescence spectroscopy; Longobards

1. Introduction

The archaeological research and, more broadly, the field of cultural heritage is benefiting to an increasing extent from the possibilities afforded by materials characterization techniques. They provide fundamental support to archaeological studies, like provenance of raw materials, manufacturing processes, technological evolution in production and relevant value chains.

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Scientific investigations are also very helpful in diagnostics and identification of degradation mechanisms of artistic and historical items, so that remediation strategies, restoration and conservation protocols can better achieve the expected results.

However, some limitations and precautions are to be considered when tackling any kind of scientific investigations in the mentioned fields. Very often, the experimental tests are conducted on single, unique, specimens or objects, an aspect which may raise questions about the statistical significance of the tests. Moreover, the investigated items on some occasion would tolerate not even micro-sampling, so that non-destructive protocols are required. Eventually, artefacts belonging to museum collections, may be difficult to be taken to the lab premises, mostly for insurance and handling issues, so that *in-situ* tests may be preferable.

In view of these aspects, an approach that is nowadays becoming a standard one implies the usage of combined experimental and methodological protocols, that through the selection of an appropriate set of techniques address the specific request of the research.

The materials characterization techniques used in this context on many occasions will have to transportable to out-of-laboratory environments, like excavations, museums, etc.

A case study is presented herewith, concerning the characterization of two early medieval brooches, made of noble metal alloys, decorated with stone insets. These latter have been provisionally identified as garnets, possibly from Far East procurement sites. Some of the insets turned out to be made of artificial glass.

One of the aims of the study is to evaluate the base alloy composition, including that of gilding layers. The fibulae are decorated with stones, preliminarily identified as garnets, using the cloisonné technique [1–3]. The aspects of some of the insets suggest, the deliberate usage of artificial glass. Therefore, the second main scope of the research concerns the assessment of these elements of the decoration patterns. In case the hypothesis of garnets is confirmed, a comparison has been made with literature on this subject, to identify the provenance sites of the garnets (vide infra).

To pursue these tasks, the selected analytical and structural techniques selected for this study are: X-ray Fluorescence (XRF) and Raman (RS) spectroscopy. In fact, the composition of the base alloys has been measured with XRF tests, so that a straightforward classification of these materials could be achieved. The chemical composition of the gems decorating the selected artefacts has been also measured via XRF. In addition to this, the structural information obtained from RS has assessed the natural (garnet mineral) or anthropogenic (artificial glasses) of these features of the investigated jewels.

In this way, the chemical composition of both base alloys and decoration elements can be obtained straightaway. The test campaign has been conducted using transportable instruments. In this way the measurements have been carried out directly of the selected artefacts in a fully non-destructive mode in the premises of the exhibition sites.

2. The Archaeological Context

The results contained in this manuscript are part of a broader research carried within a national project (MiLongA—Migration, social organization and material culture of the Longobards in Italy: Archaeology, Archaeogenetics, Archaeometry, involving as partners: Università Cattolica of Milano and University of Trento) aiming at understanding the role of migration of Longobards in the Northern part of Italy in the introduction and development of manufacturing techniques, with particular focus on jewelry, with regard to the finds coming from different funeral trousseaus. In this way, by cross linking the results of the materials analyses and those from genetic investigations, a better understanding of the complex events associated with the mentioned migratory flux can be assessed.

For this article two so-called “S” shaped fibulae have been selected. These finds come from two different funerary contexts in Cividale del Friuli (Italy), that is Cella [4] and San Mauro hill [5,6], and are representative of early medieval Italic and Pannonian goldsmithing. They were characteristic of traditional Longobard-Pannonian women’s dressing and were worn at chest height, often in pairs, to close a shirt or other light garments [7]. Their sinuous form reproduces two opposing birds of prey with hooked beaks, often soldered to the body of the fibula, giving it its characteristic S-shape. Numerous examples are known of silver, bronze, and gilded copper, demonstrating that the choice of materials depended on the availability of the craftsman and the client. The fibulae were selected from a larger batch of materials due to the high number of garnets set in their cells. This decoration, in fact, harks back to the well-known and ancient cloisonné technique (or polychrome style) that was popular between the 5th and 7th centuries AD reached its peak on the European continent.

In general terms, cloisonné can be divided into two main types [3]:

- Jointed cloisonné (also called clasped cloisonné) is characterized by thin cells, arranged very close together, soldered together and often also soldered to the base plate. The base plate forms the base on which the individual garnets rest. They are set in the cells and blocked there by the riveted upper bent parts of the cell walls. In these types of jewelry, the garnets are cut into relatively thick sheets (1.5–2.0 mm).
- suspended cloisonné varies depending on the type of filling of the cells used for fastening. In this case, the cells are joined together and to the frame of the artifact and rest on a layer of filling material, like: clay, sandy stucco, or other mixtures. Thanks to this filling, the object looks thicker, although the garnets are not in contact with the base, and can be cut thinner than in the previous case, up to 1.5 mm.

To enhance the garnet brightness, thin gold sheets were placed at the bottom of each cell. The success of the cloisonné style led to a sort of mass production during the 6th century, which declined towards the end of the same century due to shortages or interruptions in the supply chains of garnets from Southeast Asia [8]. Several archaeometric studies have in fact demonstrated the widespread presence of almandine garnets from India and Sri Lanka in artefacts from the 5th and 6th centuries, while those decorating jewelry from the 7th and 8th centuries are mainly of Bohemian and Portuguese origin [9–12]. These garnets are typically pyropes, much smaller in size than the South Asian almandines used in the preceding period and are less easily worked. This justifies the decision to import higher-quality garnets from Southeast Asia and to rely on closer sources when these trade routes were interrupted.

In France, thanks to decades of research conducted on various finds decorated with the cloisonné technique and originating from Merovingian areas, at least six types of garnets have been proposed, based on their chemical-physical composition. The data were compared with mineralogical samples from deposits in Southeast Asia, which allowed us to trace their provenance in ancient times with good precision (Table 1). These reference values have been widely used for the study of Late Antique and Early Medieval garnets and have substantially confirmed the picture outlined by previous research works [13–17]. However, it cannot be ruled out that in between the 5th and 6th centuries AD, Portuguese and Bohemian garnets also largely circulated, together with other types of garnets present on the continent and not yet identified and classified by scientific research.

Table 1. Chemical compositions of the six identified types of garnets, according to the classification proposed in [18].

Provenance	Garnet Classification											
	Type I		Type II		Type III [a]		Type III [b]		Type IV		Type V	
	India Rajasthan		India ?		Central Sri Lanka		South Sri Lanka		Portugal Monte Suimo		Czec Rep. Bohemia	
Oxide (%)	**Mean**	**Std. Dev**	**Mean**	**Std. Dev.**	**Mean**	**Std. Dev.**	**Mean**	**Std. Dev.**	**Mean**	**Std. Dev.**	**Mean**	**Std. Dev.**
SiO_2	36.0	1.2	37.3	0.8	40.3	1.1	38.2	1.1	41.2	0.8	41.5	0.7
TiO_2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.4	0.04	0.45	0.16
Al_2O_3	20.8	1.2	21.5	0.7	22.4	0.6	21.3	0.6	23.1	0.4	21.6	0.6
Cr_2O_3	0.0	0.0	0.06	0.04	0.0	0.0	0.0	0.0	0.0	0.0	2.2	0.7
FeO	37.5	2.2	32.1	1.5	19.7	2.6	25.7	2.6	12.7	1.6	8.9	0.5
MnO	0.4	0.5	1.2	0.9	0.3	0.4	0.5	0.4	0.4	0.03	0.3	0.03
MgO	4.4	0.7	6.2	0.9	12.7	2.3	12.3	2.3	16.3	0.9	19.8	0.5
CaO	0.7	0.3	1.4	0.6	3.0	1.5	1.3	1.5	5.4	0.2	4.3	0.28

The [a] and [b] come from the original classification proposed in Ref. [18] and identify two separate kinds of Type III granates. Concerning "?" in Type II column, indicates the uncertainty as regards the region in India from which Type II comes.

3. Materials

The brooches (fibulae) investigated in the present study are shown by Figures 1 and 2 and are codenamed: Sample 735 and Sample 24678, in agreement with the inventory numbers assigned to the two objects, part of the collection of the National Archaeological Museum in Cividale (Friuli Venezia Giulia region—Italy).

The two artifacts were selected for their good state of preservation, allowing us to appreciate their technical and stylistic characteristics: the cloisonné technique with garnets of various origins, the use of colored glass, and surface fire gilding. Artifact no. 24678 also features the use of a thin gold leaf that enhanced the garnet's brilliance. The two jewels are significant examples of early medieval goldsmithing, widespread in Longobard Central-Northern Italy between the last third and the end of the 6th century AD.

The find 735 (Figure 1) comes from the necropolis of Cella and must certainly have been part of a rich female funerary trousseau. The item was probably worn in pairs [19,20].

The fibula features a very sinuous shape, representing two opposing birds of prey with hooked beaks welded to the body of the object. The eye contour is accentuated by an angular shape decorated in niello with opposing triangles. The body of the fibula features a honeycomb structure composed of eight cells set with flat garnets (see “Results and Discussion” section) resting on a thin, partially visible, milled gold leaf. Moreover, two semicircular cells are also present, featuring opaque blue glass. The back of the fibula no longer exhibits the fastening pin. The fibula features an animalistic so called Second Style decoration and was produced in Italic context, perhaps in Cividale itself, around 600 AD [20–22].

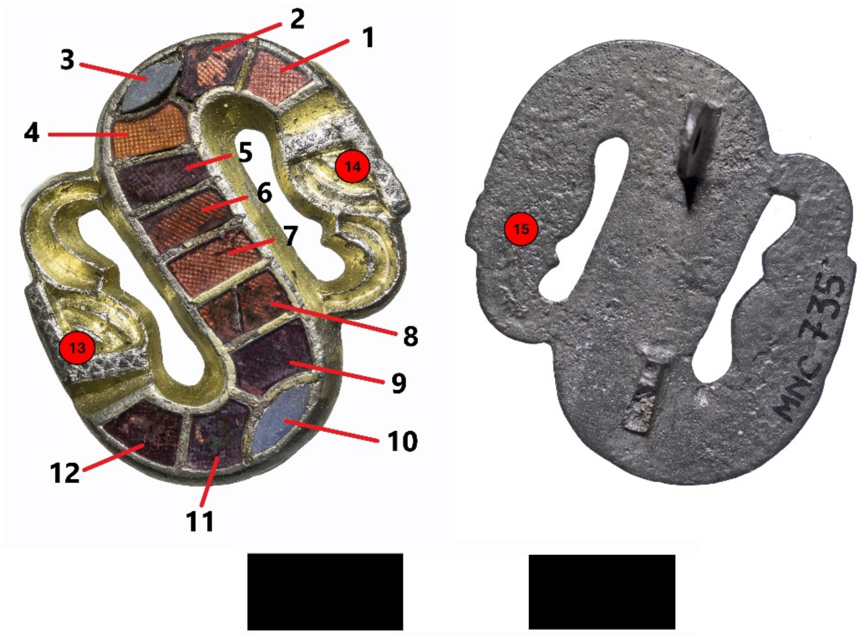

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Figure 1. Front and rear side of ‘S’ shaped fibula—Sample n.: 735 (from Cella—Cividale). The numbers indicate the spots where the XRF (black on red background) and Raman (just black) spectra have been acquired. © Museo Archeologico Nazionale Cividale.

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Figure 2. Front and rear side of ‘S’ shaped fibula—Sample n.: 24678 (from San Mauro hill, Cividale. Tomb n.:51). The numbers indicate the spots where the XRF (black on red background) and Raman (just black) spectra have been acquired. © Museo Archeologico Nazionale Cividale.

The second “S” shaped fibula (24678) (Figure 2) is apparently made of the same base alloy and decorating coating as item n. 735. It comes from the set of goods found in the rich tomb n. 51 of the necropolis of the hill of San Mauro, north of Cividale and, at the time of its discovery, it was found together with a similar item. The subject of this fibula is the same as the previous one. The eyes are formed by two round cells, of which only one still retains the garnet. The alveolar structure along the body is composed of eight cells filled with flat garnets. In cell 10, a fragment of thin textured gold sheet on which the circular garnet originally rested is preserved. On the back of the fibula, the fastening system is still present, except for the needle. The fibula can be classified as belonging to the type of Rácalmás tomb 2/tomb 20-Cividale Gallo and it is dated to the second half of the 6th century AD [21,22].

4. Experimental Methods

For the XRF analyses a hand-held Hitachi X-MET8000 spectrometer has been employed. This instrument is equipped with a 4 W X-ray tube, featuring a Rh anode, operated at a maximum voltage of 50 kV, and a silicon drift detector (SDD) with a Be window (Oxford Instruments). The spectrometer has also a camera used to record the surface spots of the analyzed sample, from which the X-rays emitted from the samples are collected. In all tests, an inner collimator has been used, thus restricting the investigated spot to 3 mm diam. For all tests an acquisition time of 60 s has been used and the regions of interest have been considering specific features, surface details and topography, both on the front and back face of the artefacts.

For the quantification of the analytical data, two calibrations have been used: Mining for the glass and garnet insets; Alloy for the metallic parts of the relevant item. All data have been normalized in the end and each single spectrum has been checked for the presence of any external contaminant.

The Raman measurements were carried out using a portable i-Raman (B&W Tek) spectrometer equipped with a thermoelectrically cooled 2048-pixel CCD detector and operating with a 785 nm laser excitation. The instrument provides a spectral resolution of 5 cm^{-1} within the 175–3200 cm^{-1} spectral range. The Raman system was connected to a video-microscope sampling unit (BAC151A) equipped with an integrated camera for real-time tracking of the laser beam position and focusing. Measurements were performed in backscattering geometry. The portable configuration of the instrument allowed *in-situ* and non-invasive analyses directly on the archaeological artefacts, without the need for sampling or surface preparation. During the experiments, the artworks were positioned horizontally under the portable microscope, allowing stable focusing and accurate alignment of the laser spot on the surface. The laser power was carefully maintained well below the damage threshold of the analysed materials, as verified by preliminary tests, in order to prevent any thermal or photochemical alteration of the artefacts. For each object, several measurement points (see Figures 1 and 2) were selected along different surface areas of the investigated gemstones, by manually adjusting the microscope's movable stage. The integration time and number of accumulations were optimized to achieve a satisfactory signal-to-noise ratio while avoiding fluorescence saturation.

5. Results and Discussion

The XRF analyses have been conducted both on metal alloy parts and on the decorating insets. The sampled spots are indicated in Figures 1 and 2. In Table 2, the average overall compositions of the metallic parts of the fibulae are indicated.

Table 2. Average compositions (wt.%) of the base alloys and surface coatings of fibula n. 735 and n. 24678 as obtained from XRF data.

Overall Composition										
Sample Code	**Fe**	**Cu**	**Zn**	**As**	**Ag**	**Sn**	**Pb**	**Au**	**Hg**	**Bi**
735	3.88	11.93	0.98	0.02	58.99	1.95	1.99	17.78	2.45	0.04
24678	6.59	16.21	1.63	0.02	61.79	1.39	2.51	9.09	0.72	0.03

Both samples exhibit on the front surface a coating which is certainly contributing to the measured compositions in Table 2. To evaluate separately the composition of the base alloy from that of the surface coating, it was assumed that those elements present in the spectra acquired on the back side of the two fibulae (spot 15, Figure 1; spot 13, Figure 2) were exclusively present in the base alloy. Under this assumption, the compositional results listed in Table 3 have been obtained, considering both compositional data coming from both the back and front side of each brooch.

The alloy used for the manufacturing of the two fibulae is a silver-base alloy with a significant concentration of copper, in addition to other main elements, like: lead, tin and zinc.

The two S-shaped fibulae, although different in typology and chronology, were both produced through a mold casting probably obtained from metal models produced using the lost-wax casting technique [23].

For the surface gilding coating the established technique based on the use of the amalgam alloy is proved by the residual concentration of quicksilver (Hg, see Table 2) [24,25]. This coating is still covering all the surface of sample n.735, whereas just residual patches are visible on the surface of the sample n. 24678.

Table 3. Average compositions (wt.%) of the base alloys of fibula n. 735 and n. 24678 as obtained from XRF data.

Alloy Composition					
Code	**Cu**	**Zn**	**Ag**	**Sn**	**Pb**
735	14.83	1.19	78.86	2.60	2.53
24678	20.03	1.97	73.29	1.72	2.99

The XRF data, in particular, the spectrum acquired in the spot 10 of fibula n. 24678, corresponding to a cell from which the decorating stone came off, have confirmed that a gold textured reflecting foil is used to enhance the brightness of the decorating stones.

The characterization of the decorating gems has been conducted with the combination of Raman and XRF spectroscopies.

Typical Raman spectra of garnet and glass insets obtained in this study are presented in Figure 3. They refer to the fibula 735, cell n. 2 and 10. In the Raman spectrum exhibits the typical lines ascribable to the garnet phase. Similar spectra have been acquired from other cells of both this item and also the other one which is under investigation in the present study. Not all cells of the investigated fibula n. 735 resulted to be hosting garnet pieces, indeed shows the Raman spectrum from sample n. 735 spots: 10 (see Figure 1), exhibiting broad lines, indicating the amorphous character of the relevant specimen.

For a complete characterization of these parts, XRF spectra have been acquired in the same spots as the Raman ones. In Table 4, the results of these combined analyses are given. As concerns the garnets, following the classification proposed in [18], it turns out that most of the can be classified as types 2, 3a and 3b considering both their average concentration values and their compositional variation range. In fibula 735, six garnets are type 3b, i.e., from southern Sri Lanka, and one is type 2, i.e., from India. The remaining three garnets, however, do not fall within the recalled classification. We are revising these data to make clear whether any precision issues there, or if another class of garnets, not classified as yet has been really used in the manufacturing of this item.

Table 4. Composition (wt.%) of the materials used for the decoration insets. In the last column, for those insets identified as garnets by Raman spectroscopy, the relevant type attribution described in [18] (see Table 1) is indicated, the coloured raws refer to glassy gems.

Sample Code	Cell Number	Composition of the Garnet and Glass Decoration							Type
		SiO_2	TiO_2	Al_2O_3	FeO	MnO	MgO	CaO	
735	1	41.3	0.0	7.8	33.0	0.7	15.9	1.2	3b
	2	41.9	0.1	7.9	34.3	0.6	14.0	1.3	3b
	3	79.0	0.0	2.3	5.6	0.4	0.0	12.7	glass
	4	42.9	0.0	6.7	17.3	0.8	26.8	5.4	n.n.
	5	40.9	0.0	7.9	26.1	1.9	21.0	2.2	n.n.
	6	41.0	0.0	7.8	30.5	0.4	19.1	1.3	3b
	7	41.6	0.0	7.5	28.7	0.4	19.9	2.0	n.n.
	8	43.9	0.5	8.0	38.7	0.6	7.0	1.2	2
	9	40.7	0.0	8.2	32.1	0.5	17.5	1.0	3b
	10	72.0	0.0	3.2	9.8	0.3	5.8	8.9	glass
	11	40.7	0.1	7.7	29.2	0.6	20.9	0.8	3b
	12	42.6	0.0	8.1	36.3	0.1	12.0	1.0	3b
24678	1	36.0	0.0	6.0	42.3	0.3	13.6	1.7	3
	2	39.6	0.0	7.0	32.8	3.6	14.0	3.0	3a
	3	39.3	0.0	7.2	34.8	0.5	17.2	1.0	3b
	4	41.5	0.0	7.7	39.3	0.1	10.2	1.2	3b
	5	40.7	0.0	7.3	39.2	0.2	10.6	1.9	3
	6	40.0	0.0	7.1	38.6	0.3	12.9	1.2	3b
	7	39.7	0.0	7.4	39.5	0.3	12.0	1.1	3b
	8	37.2	0.0	6.1	40.1	0.1	15.0	1.5	3a
	9	39.1	0.0	5.5	38.6	0.1	15.6	1.0	3b

Cells 3 and 10 are filled with opaque blue glasses, as confirmed by the Raman spectra (Figure 3) and their composition (Table 4). The colour is to be ascribed mainly to the presence of iron oxide [26]. In the composition of lack of opacity may be due to deliberate intention to render these two insert glaze like, and differentiate them from the transparent gems.

In fibula 24678, two garnets are type 3a, meaning from central Sri Lanka, five are type 3b, meaning from southern Sri Lanka, and two garnets cannot be clearly distinguished between the subtypes 3a and 3b. In this case, no glasses are present.

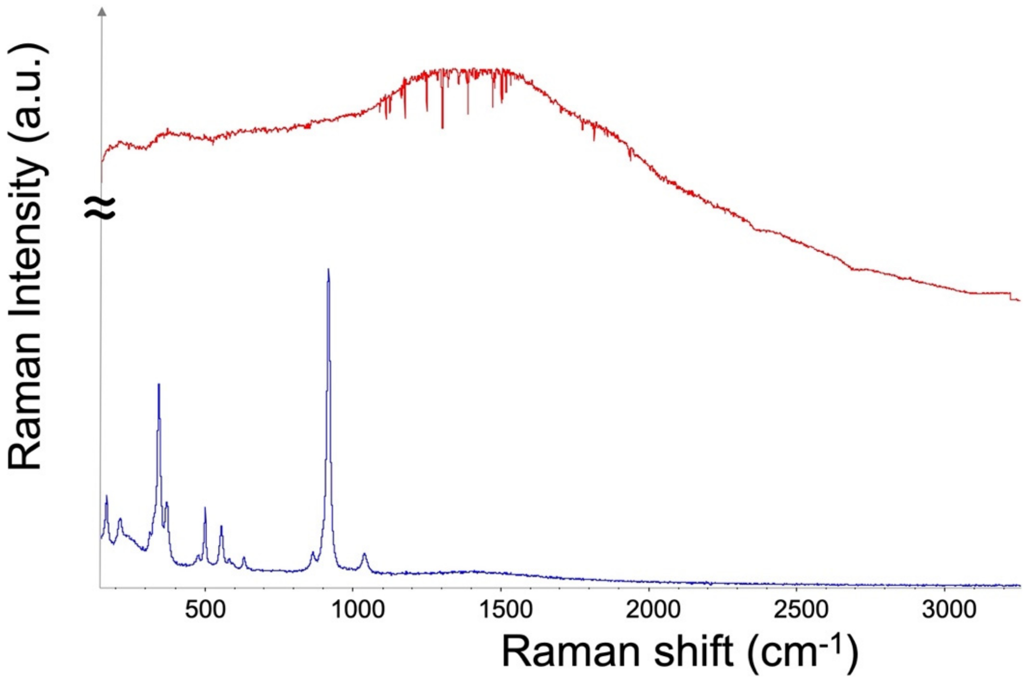

Figure 3. Raman spectra acquired from sample n.735 spots: n.2 garnet phase identified (blue); n.10 glass spectrum (red), respectively.

6. Final Comments

The variety of garnets in the two "S" shaped fibulae demonstrates that the craftsman used what was available at the time to decorate the artefacts with the cloisonné technique, although garnets of Sri Lankan origin, namely Pyraldines, featuring a low calcium content, are prevalent. Only one Indian almandine was identified among those analyzed. The presence of two glass inclusions in the fibula n. 735 may be due to the need of using alternative materials due to the scarcity of garnets that began in the early 7th century. On the other hand, it cannot be ruled out that the usage of glass was an intentional choice, aiming at creating an extremely colorful and lively visual effect, according to the tastes and preferences of the client.

On the basis of this positive early characterization campaign, the investigation will be extended to a more numerous set of finds to attain a better picture of the production and circulation of these items in the Northern regions of Italy, during the 5–7th centuries AD. In this context the trading of garnet from Far East regions poses the question of the main reasons for this.

As concerns the proposed materials characterization protocol, it turns out to be very effective for a fast, non-destructive characterization of the multi-materials objects. Furthermore, combining XRF and Raman spectroscopy, with the capability of acquiring both spectra from the exact same region of interest, significantly enhances the reliability of the experimental results. This is especially relevant when working with portable instruments, whose hardware constraints inevitably limit their precision and sensitivity compared to laboratory-based systems.

The research presented herewith is part of a wider project of national interest (PRIN) titled: MiLongA—Migration, social organization and material culture of the Longobards in Italy: Archaeology, Archaeogenetics, Archaeometry (n. 2022ZYTJLX, CUP E53D23022260006). The results obtained from the characterization of a larger number of items of Longbard production, including both cloisonné decorated jewels and dress accessories of different kinds, will be presented in a forthcoming publication. In this context, the key issue of the exotic provenance of the garnets decorating these items will be fully assessed.

Author Contributions

M.D.: has conducted the test campaigns, cured the experimental results, drafted and revised the manuscript; E.P.: has conceived and supervised the research, contributed to draw archaeological conclusions from the results of the materials characterization; M.Z.: supported data curation and the revision of the experimental results; A.B.: has made the investigated finds available to the materials analysis; A.L.: provided and expert support for Raman spectroscopy; S.G.: has contributed to the test campaigns, supported the discussion of the experimental results. All authors have read and agreed to the published version of the manuscript.

Funding

The research reported in this paper has been conducted within national project: MiLongA—Migration, social organization and material culture of the Longobards in Italy: Archaeology, Archaeogenetics, Archaeometry, (Project No. 2022ZYTJLX, Project code: E53D23022260006). Financed by the European Union under the scheme "NextGenerationEU", through the Italian Ministery for University and Research.

Institutional Review Board Statement

Not applicable.

Informed Consent Statement

Not applicable.

Data Availability Statement

The data that support the findings of this study are available on request from the corresponding author.

Acknowledgments

The authors would like to thank the Museo Archeologico Nazionale di Cividale del Friuli for making the investigated artefacts available for the materials characterization tests.

Conflicts of Interest

The authors declare no conflict of interest.

Use of AI and AI-Assisted Technologies

No AI tools were utilized for this paper.

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