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RESEARCH PAPER

Novel implementation of the INFN-CHNet X-ray fluorescence scanner for the study of ancient photographs, archaeological pottery, and rock art

F. Taccetti[1] · L. Castelli[1] · C. Czelusniak[1] · F. Giambi[1,2] · M. Manetti[1] · M. Massi[1] · A. Mazzinghi[1,2] · C. Ruberto[1,2] · F. Arneodo[3] · R. Torres[2,3] · F. Castellá[4,5] · L. Gheco[4,6] · N. Mastrangelo[4,5] · D. Gallegos[4,7] · A. Morales[4,7] · M. Tascon[4,8] · F. Marte[4] · L. Giuntini[1,2]

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Abstract
INFN-CHNet is the cultural heritage network of the Italian National Institute of Nuclear Physics (INFN) and is constituted by units from Italy and from outside Europe, one of them at Universidad Nacional de San Martín (UNSAM) in Buenos Aires, Argentina. As a result of the initiative carried out during 2015 by the *Accademia dei Lincei* for the year of the Italian culture in Latin America, an INFN-CHNet laboratory was set at CEPyA-UNSAM with the collaboration of INFN and the Restoration Workshop Centro Tarea. Noteworthy, this laboratory is conceived as a multidisciplinary research facility with complementary skills, both scientific and humanistic. In this context, the first instrument jointly set up, optimised, and applied to Cultural Heritage was an X-ray fluorescence scanner. In this manuscript, we describe the instrument and its main features together with a set of representative yet novel applications in the field of cultural heritage, namely, the experimental study of hidden rock art through laboratory replicas that imitates the problems found in the archaeological sites (hematite drawings hidden below carbon deposition); the study and chemical characterisation of archaeological decorated pottery; and finally, the application of the XRF scanner to ancient photography, for quick and accurate identification of materials and techniques employed. Beyond these specific results, the primary output of this initiative has been the conception of a future network of scientific laboratories in South America, coordinated by CEPyA at UNSAM.

Keywords XRF scanner · Portable instrumentation · XRF imaging · Non-invasive analysis · In situ analysis · Cultural heritage

✉ M. Tascon
mtascon@unsam.edu.ar

✉ F. Marte
fmarte@unsam.edu.ar

1 Istituto Nazionale di Fisica Nucleare, Sezione di Firenze, 50019 Sesto Fiorentino, Italy

2 Dipartimento di Fisica e Astronomia, University of Florence, 50019 Sesto Fiorentino, Italy

3 Division of Science, New York University Abu Dhabi, Saadiyat Island, 129188 Abu Dhabi, United Arab Emirates

4 Centro de Estudios sobre Patrimonios y Ambiente (CEPyA), Escuela de Arte y Patrimonio, y Escuela de Habitat y Sostenibilidad, Universidad Nacional de San Martin, Av. 25 de mayo y Francia, Buenos Aires, Argentina

5 Consejo Nacional de Investigaciones Científicas y Técnicas (CONICET), Godoy Cruz 2290, Ciudad Autonoma de Buenos Aires, Argentina

6 Instituto Regional de Estudios Socioculturales (CONICET-UNCa), Prado 366, Catamarca, Argentina

7 Centro Tarea, Escuela de Arte y Patrimonio, Universidad Nacional de San Martin, B. Quinquela Martín1784, Ciudad Autonoma de Buenos Aires, Argentina

8 Instituto de Investigación e Ingeniería Ambiental (IIIA-CONICET-UNSAM), Av. 25 de mayo y Francia, Buenos Aires, Argentina

1 Introduction

INFN-CHNet is the cultural heritage network of the Italian National Institute of Nuclear Physics (INFN) and is constituted by INFN laboratories devoted to the analysis of Cultural Heritage (CH), Italian partners with complementary skills, and research institutions from abroad. During the period 2018–2020, researchers from INFN-CHNet and from Centro de Estudios sobre Patrimonios y Ambiente (CEPyA), with the collaboration of the Restoration Workshop Centro Tarea at UNSAM have created a multidisciplinary research facility with complementary skills, both scientific and humanistic. The main goal is to develop innovative instrumentation applied to CH research. In this regard, the new scientific laboratory established in connection with INFN-CHNet at CEPyA-UNSAM is the result of initiatives carried out during 2015 by the *Accademia dei Lincei* for the year of Italian culture in Latin America. The first instrument jointly set up, optimised and applied to CH is an X-ray fluorescence scanner. This instrument is mainly based on the equipment recently developed by INFN-CHNet (Taccetti et al. 2019) for applications in Cultural Heritage (Macková et al. 2016; Ruberto et al. 2016; Gasanova and Hermon 2017; Lazic et al. 2018; Striova et al. 2018; Giuntini et al. 2021; Sottili et al. 2021). The instrument has proven to be suitable for in situ, non-invasive and non-destructive characterisation of a wide variety of art objects, such as paintings (Albertin et al. 2021; Mazzinghi et al. 2021a) or ancient manuscripts (Mazzinghi et al. 2021b). The experiments can be carried out in situ in almost every environment, since the instrument is extremely compact and battery powered. Hence, measurements can be performed with no need of transportation of the pieces under study, which is of crucial importance in cases where the object cannot be moved to the laboratory, such as mural paintings or rock art. In this paper, the XRF scanner developed by INFN-CHNet at UNSAM is presented along with some preliminary results illustrating the potential of this instrument for a wide variety of applications in CH and archaeometry. It is worth mentioning that this XRF scanner is the first tangible result of the activity of INFN-CHNet at UNSAM.

2 Materials and methods

2.1 Instrumental

This instrument is largely based on the XRF scanner previously developed by INFN-CHNet (Taccetti et al. 2019), as can be observed in Fig. 1. The main features and differences with respect to the already published configuration are summarised below.

The main strong points of this setup are the low weight, low footprint, small form factor and low power consumption, which result in high portability. The scanner can be easily customised, depending on applications, thanks to the use of CAD-designed 3D-printed parts, directly produced on site. The latter is of great help when dealing with a great variety of applications, as it normally happens at CEPyA. For instance, for some of the experiments herein reported, it has been necessary to increase the height of the measuring head and to change both the position and the angle of

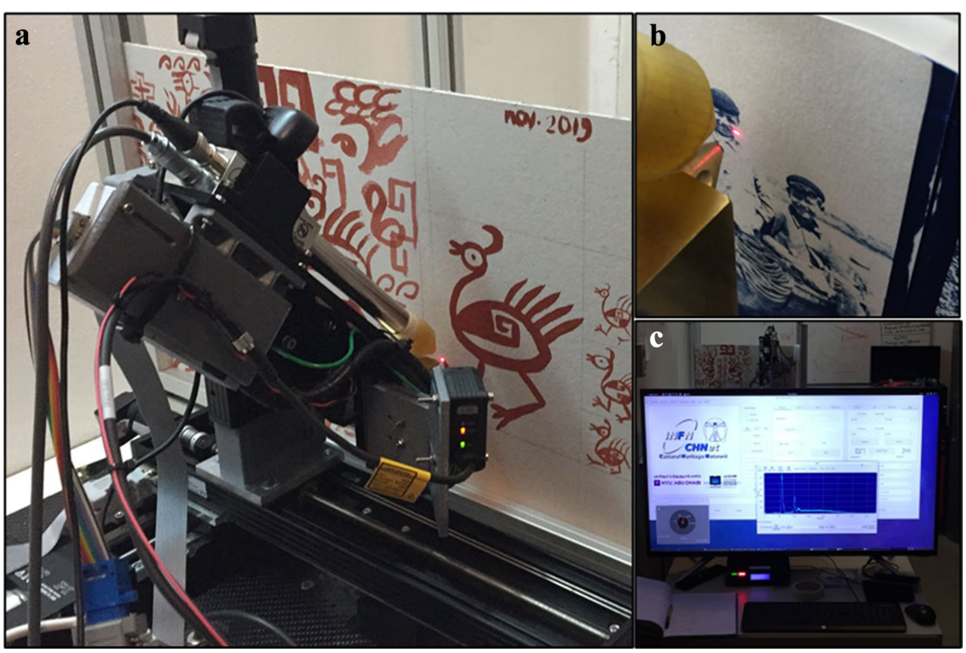

Fig. 1 The INFN-CHNet XRF scanner at CEPyA during a scan of (**a**) a mock-up and **b** a scan of a photograph. **c** The acquisition software interface

the telemeter. For such purposes, new parts of the interface between X and Y axis and a new support for the telemeter were designed and printed. Furthermore, when compared to commercial XRF scanners, this instrument has a very low cost but equivalent performance. Exploiting a single battery, the scanner can run for 12 h under continuous use. Regarding radio-safety, the instrument has no restriction for use in public areas; thus, it can be used even in the presence of public (i.e. persons that are not under radiation protection surveillance) such as students, restorers and conservators, see for example INFN-CHNet training camps [https://chnet.infn.it/it/divulgazioneformazione]. These features make the scanner perfectly suited for applications of interest in CH, especially for in situ measurements, even in harsh environments (e.g. study of rock art in caves and shelters at remote places).

From a schematic point of view, the scanner is composed of two main sections (Fig. 1a): the measuring head and the carbon-fibre box supporting the X–Y–Z linear stage assembly.

The measuring head, as previously presented in detail by Taccetti et al. (Taccetti et al. 2019), is constituted of an X-ray tube and PS (Mo anode Moxtek MAGNUM® X-ray tube) with a 127 µm Be exit window, 40 kV maximum voltage and 0.1 mA maximum anode current (MOXTEK 2018). Beam diameter is defined by a collimator (with user-definable diameter, ranging from 0.1 up to 2.0 mm in steps of 0.1 mm), with 0.8 mm being the most typical choice. An X-ray detector and preamplifier (SDD Amptek XR100 SDD, 25 mm^2 active area, collimated to 17 mm^2, and 500 µm thick), equipped with a nozzle, allows for saturating with helium the X-ray path. A Keyence laser telemetry sensor (Keyence 2018) allows for keeping the scanner-to-specimen distance constant during the measurements within a ~ 50 mm range and a live camera (Ethernet controlled). The carbon-fibre box supports the measuring head via the X–Y–Z linear stage assembly (three axes from Physik Instrumente), the CAEN DT5780 (CAEN) signal digitizer and all the auxiliary equipment (https://www.caen.it/products/dt5780/). The scanner control/acquisition/analysis software installed in an external PC unit has been developed by our group (Taccetti et al. 2019) using open-source software.

2.2 Samples

As a proof of applicability and versatility of the new setup, three different samples were analysed:

1. Mock-ups of rock art were performed in-house. A piece of wood was used as support, and haematite (Fe_2O_3) and charcoal (carbon black) as red and black pigments, respectively (Tascon et al. 2016). The pigments were bound with glue prior to their application. Once the paint was applied, the mock-up was dried for at least 4 h at room temperature.
2. Ceramic pottery fragments, discovered at an archaeological site located at El Alto Ancasti's mountains, were studied. The fragment was attributed to Aguada Portezuelo style, corresponding to the prehispanic Middle period of Northwest Argentina, between 600 and 900 A.D.
3. Photographs from the beginning of the twentieth century from an archival work of Centro Tarea were analysed. An iron-based photography (cyanotype) was chosen to prove the applicability of the XRF scanner in this field.

3 Results and discussions

As mentioned above, three different types of samples have been studied, as they are representative of the problems we are facing in the activities carried out at CEPyA and Centro Tarea and allow showing how helpful the XRF can be within this wide context.

3.1 Recovering hidden rock art paintings

The study of rock art is a complex branch of CH due to some critical factors. (a) The work of art has been exposed to diverse and, sometimes, extreme environmental conditions for long periods of time (in our case, e.g. from 500 years *b.p.* to thousands of years *b.p.*). (b) Despite that the number of pigments employed might be limited, the similarity with degradation products, environmental contaminants and rock substrates can lead to wrong conclusions and biased results. (c) Usually, the archaeological sites are in isolated and hard-to-access areas, where no electric power is available and environmental conditions can be very harsh for on-site analysis. (d) Samples are fragile, hampering the sampling process and the correct preparation of cross sections for analysis. In this regard, along with the enormous cultural and historical importance of these paintings, the use of non-invasive analytical techniques performed with ultra-portable, robust, and low power-absorption (our instrument can even operate without being connected to the mains) instrumentation emerges as the most logical and often the only possible alternative. In this context, an archaeological campaign to study rock art at the site of Oyola, Catamarca, Argentina, has found a great diversity of painting styles and numerous overlapped motifs having heterogeneous pigmentary mixtures (Tascon et al. 2016; Gheco et al. 2017, 2019b). Recent studies have proposed the existence of contributions of different painting episodes in the same site throughout its history (Gheco et al. 2013, 2019a; Quesada and Gheco 2015).

During this research work, one of the main problems to identify and characterise rock paintings is the presence of heavy carbon, calcium oxalate or anthropic depositions (typically in the range of 10- to 250-µm thickness range) covering the main motifs. The most common and extended depositions are soot layers, resulting from fires lighted in the hearths inside the caves, which cover the oldest episodes of the painting history at the sites (Tascon et al. 2016). Noteworthy, this phenomenon was not just evidenced in this archaeological site, but is a very general issue reported in many other sites (Bonneau et al. 2012). The presence of these hidden paintings has been identified by micro-stratigraphic samples, but, until now, the shape of the figures remains unknown (Gheco et al. 2019b). Following this line, XRF imaging is expected to be able to recover the hidden figures by recording the elemental maps of the elements contained in the pigments, because the absorption in the soot layers of the X-rays emitted by the pigment is quite low. To demonstrate the potential of this approach, the mock-ups consisting of Fe-based pigment drawings covered in carbon black (see materials and methods section) were prepared. For these samples, X-ray transmission for Fe excitation K_α transition (6.4 keV) is thus very high through the carbon layers. Just to give an example, transmission is > 65% for C thickness up to 200 µm, the thicker measured carbon deposition in our archaeological samples, and still > 15% for depositions up to 1 mm. Therefore, we have carried out determinations on mock-ups prepared using a red pigment (hematite, Fe_2O_3), with similar or thinner stroke thicknesses than those expected for rock paintings (Tascon et al. 2016; Gheco et al. 2019b). In Fig. 2a, d, the drawn figures representing a *Suri* (*Rhea pennata*), a large flightless bird, are shown, while in Fig. 2b/e and c/f, the elemental mappings of iron and calcium, respectively, are reported. In the first case (2-a), the stroke width is similar to the one found in rock art, being in the range within 5–10 mm; on the other hand, Fig. 2d shows the same motif painted with a stroke line of 0.5–2 mm width. In both cases, the mapping spatial resolution is well adequate for the drawings to study, demonstrating the capabilities of this setup for rock art applications.

From the X-ray spectrum, we found that essentially only Fe and Ca are present in the sample, corresponding to the drawing and the support cardboard, respectively. Ca maps in Figs. 2c, f) show Suri shapes in negative, since Ca K_α X-rays coming from the support are absorbed by the pigment of the drawings. Then, the drawings have been covered with a thick carbon deposition (thickness variable from a few to some hundreds of microns). As Fig. 3a, b, c shows, the upper part of the picture was covered with a layer of carbon black of 50 µm ± 12 thickness ($n = 3$, 25 measurements per sample). It is important to highlight that the carbon black layer made the drawing invisible. In Fig. 3d, e, the XRF scanner shows the capability to scan and reconstruct the hidden image based on the Fe X-ray fluorescence line mapping. Noteworthy, the fact that Ca was also detected proves that calcium-based pigments such as gypsum or calcite can be detected even in images under the soot layer, as X-ray transmission for Ca K_α X-rays (3.7 keV) through measured

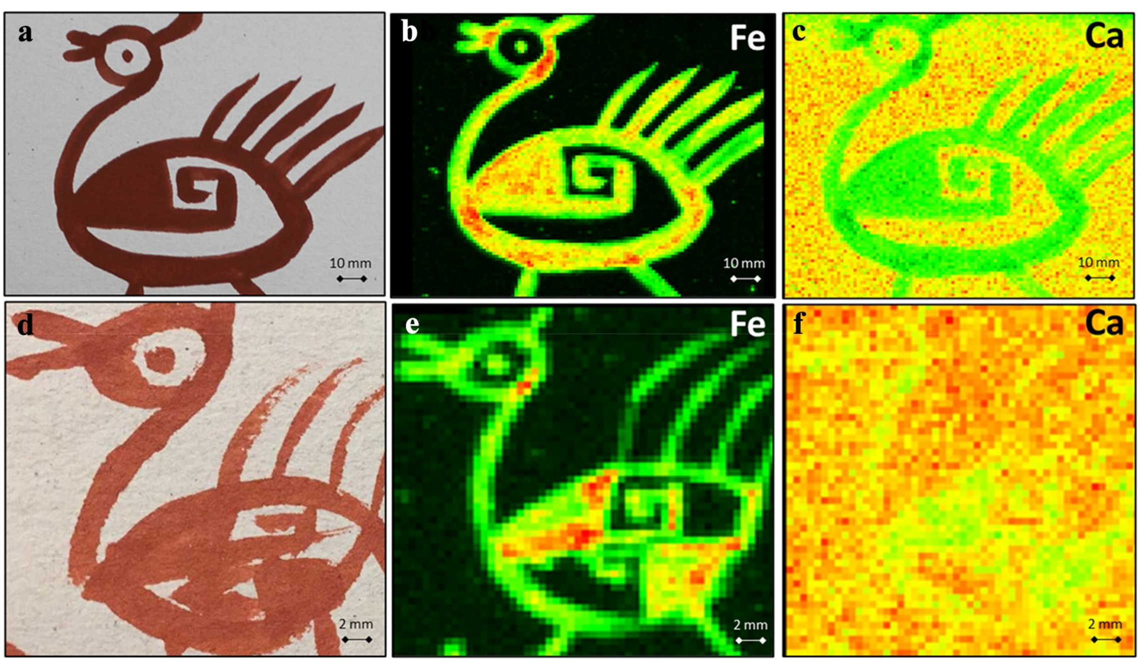

Fig. 2 (**a** and **d**) Photographs of the motifs before being covered with a carbon layer with thickness of some hundreds of micrometres; (**b** and **e**) Fe K_α X-ray map of the areas shown in **a** and **d**; finally, **c** and **f** maps of the Ca K_α X-rays of the areas shown in a) and d). Tube settings: 100 µA, 20 kV, 0.8 mm collimator. Scanning parameters: scanning speed of 1 mm/s for a) and 0.5 mm/s for d), pixel size 0.5 mm

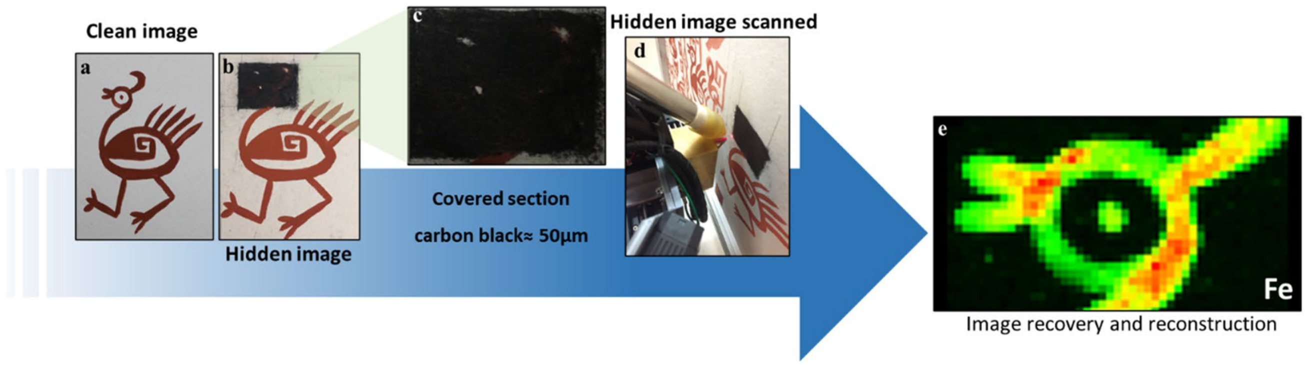

Fig. 3 **a** Uncovered motif mock-up made by iron red (Fe_2O_3) on wood. **b** Partially covered motif with carbon black and **c** detailed image of the covered area. **d** Scanner performing measurements on the covered surface. **e** Elemental map of Fe K_α (6.4 keV) collected at the following conditions: tube settings at 100 µA, 20 kV, 0.8 mm collimator, scanning speed of 1 mm/s and a step size of 1 mm

carbon layers is still acceptable (> 10% for C thickness up to 200 µm). Thus, there is a good hope to recover the lost images using XRF imaging.

3.2 Archaeological study of pottery

The archaeometric research of pottery decoration is a widespread issue. The diversity of materials and techniques employed for their manufacture raises new challenges to explore ancient technological developments. Aiming to identify a model problem in this matter, we have performed a scan over a pottery sherd attributed to Aguada Portezuelo style, corresponding to the prehispanic Middle period of Northwest Argentina, between 600 and 900 a.C. This style stands out by an extraordinary polychromy (red, purple-red, black, white, brown, yellow), complexity of figures and iconographic richness. Prior studies carried out using SEM–EDS and µ-Raman spectroscopy described the elemental and molecular composition of pre- and post-firing paintings from Aguada Portezuelo pottery style (De La Fuente et al. 2005; De la Fuente and Pérez Martínez 2018). However, these analyses were performed by point-mode studies that do not show the spatial distribution of pigmentary mixtures on the vessel decoration. In this context, the XRF scanner plays an important role as it can put into evidence the spatial distributions of the different elements in the pigments. In this manner, it allows for highlighting correlation between the pottery decorations and their elemental composition, thus simplifying the pigment identification process and minimising the possibility of wrong assignments due to local interferences originating from elements present both in the painting and in the supporting clay. Besides, portability makes it perfect for on-site measurements without the necessity of a massive collection and transportation of fragments. As an example, Fig. 4-a shows a fragment recovered from El Taco 19 village, representative of the decorated pottery of an archaeological

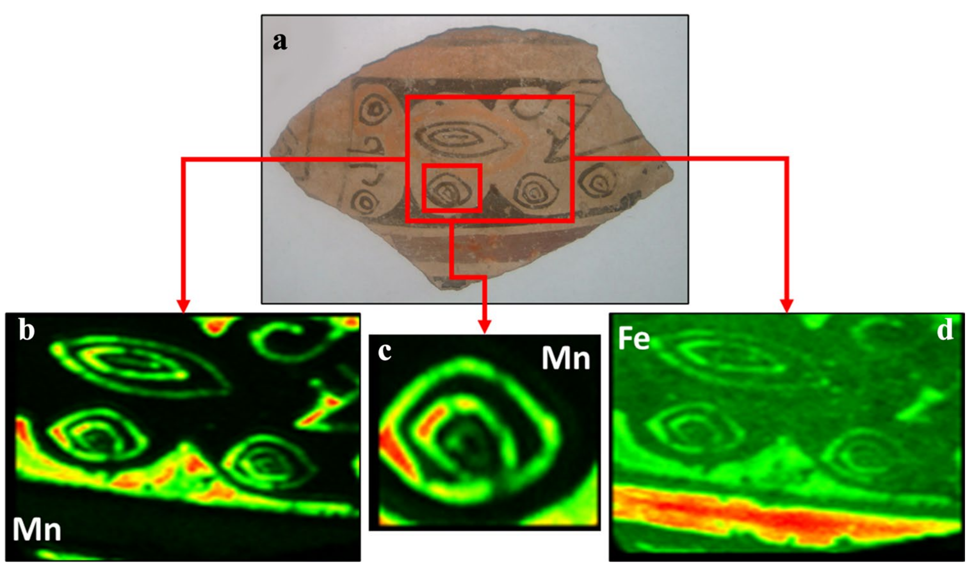

Fig. 4 **a** Photograph of the pottery fragment attributed to La Aguada Culture. **b** Elemental mapping of Mn K_α line, with a scanning speed of 1 mm/s and a step size of 1 mm. **c** Elemental mapping of Mn K_α with a scanning speed of 0.5 mm/s and a step size of 0.5 mm. This figure is compressed along the horizontal, as it is clearly different from the optical figure and from fig b and d. **d** Elemental mapping of Fe K_α with a scanning speed of 1 mm/s and a step size of 1 mm. Tube settings at 100 µA, 20 kV with a 0.8 mm collimator

site excavated by our group on the top of El Alto Ancasti Mountain (Catamarca province, Argentina). In Figs. 4b, c the Mn elemental maps at low and high spatial resolution are shown. When compared with Fig. 4a, they evidence the presence of Mn in the areas painted in black, thus suggesting the use of the MnO pigment. Finally, the iron elemental map displayed in Fig. 4d confirms the presence of iron in the red band of the bottom, indicating the presence of hematite as the main pigment. Interestingly, significant concentration of iron was also found in the black areas, suggesting the use of a black pigmentary mixture composed of manganese and iron oxides. To conclude, having this example in mind, the potential of this new setup in the context of CEPyA activities is enormous, not only for the portable features for on field measurements, but also for the capacity to understand the technical and technological processes involved in pottery production.

3.3 Ancient photographs

Until the appearance of digital photography, image construction of photographs was essentially a chemical process. Since its inception in 1839, more than 150 different photographic processes have been developed with different degrees of success and in diverse kinds of applications. Photographs are complex and composite objects, made of different materials. In their structure, one can distinguish different parts such as the primary support, the sensitive layer and organic coatings (Shugar and Mass 2015; Modica et al. 2017). In this regard, photography material characterisation requires a 3D conception of the sample instead of the classical bidimensional interpretation. Currently, most of these chemical photographs are stored in museums, archives, libraries and private collections. Therefore, the study of the technique and materials involved in the photograph manufacture plays a crucial role in the restoration and preventive conservation of this cultural heritage. Also, the thorough study of the photograph technology throughout time will allow not only the development of suitable strategies for long term preservation, storage, exhibition and conservation but also will boost the possibilities of photograph authentication (Castellá et al. 2020). The latter is of great concern in the growing market of vintage photographs which can be easily confused with modern prints (Stulik and Kaplan 2008; Shugar and Mass 2015).

Aiming to obtain information about photographic materials and techniques, several spectroscopic techniques were applied, such as Fourier-transform infrared spectroscopy FTIR (Stulik et al. 2013), XRF and 2-D XRF microscopy (Stulik and Kaplan 2008; Shugar and Mass 2015; Kozachuk et al. 2018), synchrotron-based X-ray absorption near edge structure (μ-XANES) (Kozachuk et al. 2018), laser-induced breakdown spectroscopy (LIBS) (Anglos et al. 2002), laser ablation mass spectrometry (Feigerle et al. 1999) and scanning electron microscopy with energy-dispersive X-ray detector (SEM–EDX) (Kozachuk et al. 2018). In these works, critical elemental information about photographs is obtained. Therefore, the combination of XRF and macro elemental mappings emerges as a suitable alternative for photograph characterisation. Hence, in the framework of the new university chair on photograph conservation in the Conservation and Restoration of Cultural Heritage degree at UNSAM, a set of ancient photographs performed with different techniques was analysed. In Fig. 5 an ancient cyanotype can be seen, together with two insets: on the left side inset there is a detailed image of the picture while, on the right side, there is the Fe K_α mapping of the area. In the latter, there is a clear correlation between the iron concentration with the blue colour of the picture. In other words, the higher the iron concentration (Fe) the more saturated is the blue colour (Prussian blue). This correlation is proper of a cyanotype technique. Noteworthy, in this kind of samples, the resolution is essential and, as gathered in Fig. 5, the use of a collimator of 0.8 mm in this setup allows the construction of higher-resolution images, adequate for studying these particular objects. In this specific case, the elemental map is 10 mm × 20 mm.

4 Conclusions

Following up the initiatives carried out during 2015 by the *Accademia dei Lincei* for the year of Italian culture in Latin America, INFN has contributed to create the first unit of INFN-CHNet in South America at CEPyA of UNSAM for common development of instrumentation and applications to CH research and conservation. The CEPyA-UNSAM unit of CHNet has started its experimental activity in 2018 with the implementation of an XRF scanner for CH applications. The scanner has been used for the study of representative samples of the main activities carried out at CEPyA (*i.e.,* rock art, archaeological pottery, and ancient photographs). The instrument has demonstrated to be a suitable tool for this kind of activities, being highly portable (low weight, low footprint, small form factor, low power consumption) and customizable due to the extensive use of CAD-designed 3D-printed parts. Also, beyond portability, the possibility of being used in public areas is another feature to highlight. In this sense, results obtained from a set of representative applications have been very promising, particularly:

– The study of haematite drawings hidden below carbon deposition has been successful and demonstrated to be a very promising approach to recover hidden images in rock art sites. It has been easily possible to fully recover the lost drawings for depositions up to 50 μm.

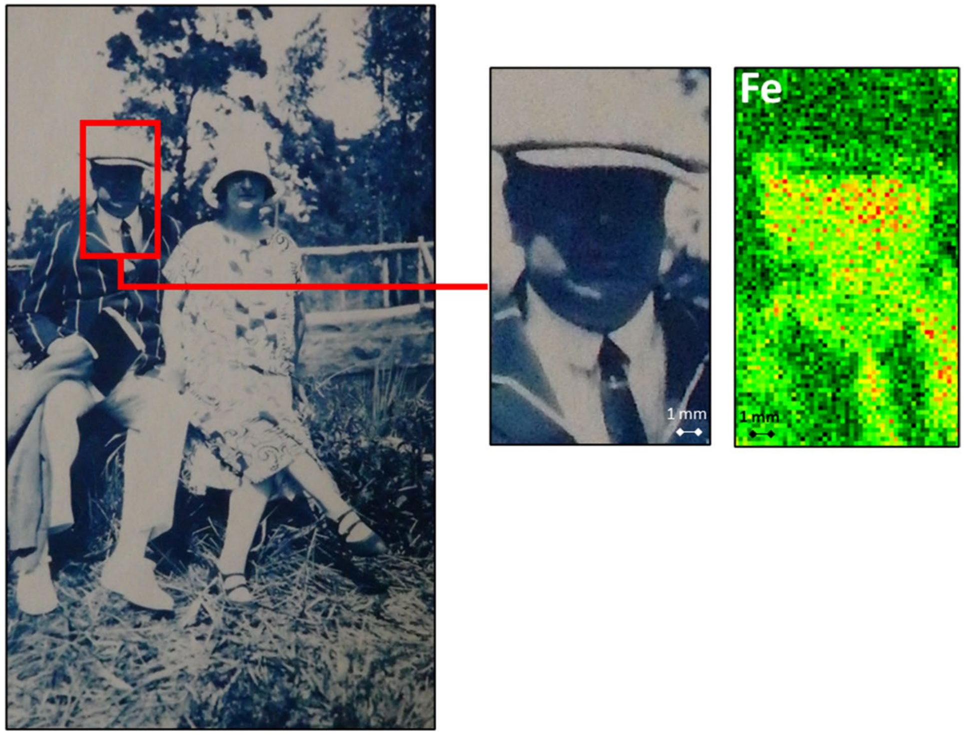

Fig. 5 On the left-hand side, image of the analysed ancient photograph; on the right-hand side, inset of the photograph and Fe Kα mapping. Scanning speed of 0.5 mm/s and a step size of 0.5 mm. Tube settings at 100 µA, 20 kV with a 0.8 mm collimator. Scanned area: 10 mm × 20 mm

- In the study of archaeological decorated pottery of the sites currently under study, elemental mapping has allowed for an easy characterisation of the main pigments and reconstruction of decorations. From these analyses, important conclusions, such as attribution to a specific culture, provenance, or trade routes, among others, can be drawn.
- In the application of the XRF scanner to ancient photography, it has been confirmed that Fe-based photos can be easily characterised, allowing for the quick identification of the employed materials and techniques. These preliminary measurements have shown successful characterisation of ancient Fe-based photographs. However, detector efficiency should be increased by adopting a wider detector area and primary X-ray beam intensity increased, by installing a new tube. These improvements will make it easier to study ancient photographs even when based on other materials.

To conclude, beyond these specific results, the primary success of this initiative has been the creation of the first network of scientific laboratories in South America devoted to CH, coordinated by CEPyA-UNSAM node, as part of INFN-CHNet, and the beginning of the activity of CHNet in Argentina. In fact, the network is planning to be extended to the whole country and to the rest of South America in the foreseeable future.

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Data availability The datasets generated during and/or analysed during the current study are available from the corresponding author on reasonable request.

Declarations

Ethical approval The authors have declared no conflict of interests.

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