Characterization of Natural Pigments from the Archaeological Context of Traful Lake (Neuquén, Argentina)

Verónica Aldazabal, Maria Reinoso, Graciela Custo, Luciana Cerchetti, Emilia B. Halac, Griselda Polla, and Eleonora Freire

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In this paper we discuss the use of mineral pigments recovered in stratigraphic position from three excavated archaeological sites (Traful Lake area, Argentina) with the aim of providing information on selection and frequency of use of these raw materials. In this region, human occupation has been recorded since 4000 BP. In order to identify the chromophoric minerals, we applied wavelength dispersive X-ray fluorescence spectrometry, Raman spectroscopy, and X-ray diffraction techniques. Green, red, yellow ochre, and white pigments dated between 3490 and 590 BP were analyzed. The results show that different analytical techniques provide complementary information in order to identify the pigments. Green, red, and yellow samples are related to iron-based compounds. The green pigments can be associated with celadonite and the others with hematite. White samples revealed the presence of hydroxyapatite. Black dots, identified as carbon, were observed in several samples. Red pigments are always predominant, and their relative abundance increases in recent strata. Results are discussed in light of the context and previous reports of chroniclers.

Keywords: northwestern Patagonia; Late Holocene; pigments; WXRF; Raman spectroscopy; XRD

En este trabajo se discute el uso de pigmentos minerales recuperados en posición estratigráfica durante la excavación de tres sitios arqueológicos en el área del Lago Traful (provincia de Neuquén, Argentina) con el objeto de proveer información sobre la selección y frecuencia de uso de estas materias primas. En esta región las ocupaciones humanas presentan una cronología a partir de 4-000 años aP. Para la identificación de minerales cromóforos se aplicaron las técnicas de fluorescencia de rayos X, espectroscopia Raman y difracción de rayos X. Se seleccionaron muestras de color verde, rojo, amarillo ocre y blanco datadas entre 3490 y 590 aP. Los resultados muestran la complementariedad de las técnicas para el análisis y la identificación de pigmentos. Las muestras verdes, rojas y amarillas están relacionadas con compuestos basados en hierro. Las verdes pueden asociarse con celadonita y las restantes con hematita; las blancas revelaron la presencia de hidroxiapatita. En varias muestras se observaron también puntos negros identificados como carbón. Los pigmentos rojos son predominantes y su abundancia relativa aumenta en los estratos recientes. Los resultados se discuten a la luz del contexto arqueológico y de los reportes previos de cronistas.

Palabras clave: Patagonia noroccidental; Holoceno tardío; pigmentos; WXRF; espectroscopia Raman; XRD

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The use of color by hunter-gatherer societies is a topic that has been widely addressed in archaeological research. However, in the Patagonia region, archaeological studies were mainly focused on recognition of the pigments involved in rock art paintings, to identify the raw materials and their geographic provenance (Boschin 2009; Boschin et al. 2011; Masaferro et al. 2012; Wainwright et al. 2000). The aim of this work is to look over the totality of the raw coloring materials recovered from the excavations. Ochre is common in archaeological sites. Based on ethnographic information, it was used as a pigment in a variety of contexts and supports, including rock art, artifacts, body painting, medicine, and as a component of ritual and mortuary practices.

Silveira began archaeological research in the area of the Nahuel Huapi Lake-Traful National Park, Neuquén Province, Argentina, in the 1980s. Silveira's research was aimed at discussing the human use of natural forest resources. During this project, excavations and surveys were conducted at different locations (Silveira et al. 2013). The archaeological sites under study were placed between 300 and 3500 m away from the northern side of the Traful Lake and in the middle- and upper-valley of the Traful River (Figure 1). The cultural remains correspond to hunter-gatherer settlements, dating from 4000 BP. The landscape is characterized as an ecotonal environment between the rainforest and the steppe, presenting a high diversity and availability of mineral, faunal, and floral resources.

The Patagonian Andean region is characterized by a pronounced west-east geomorphological, lithostratigraphic, and bioclimatic gradient. Geomorphologically, the region encompasses part of the northern segment of the Cordillera Patagónica Austral, the southern end of the Cordillera Neuquina, and most of the Collon Cura formation. It presents three clearly differentiated areas: a higher western area along the Cordillera, dominated by a glacial landscape, a central volcanic area, and an eastern plateau-shaped area. Granodiorites, granites, tonalites, and diorites of the Los Machis Formation with outcrops of basalts can be observed at the west side of the lake; in the eastern sector, ignimbrites, dacitic and rhyolitic tuffs, as well as volcanic breccias, andesites, basalts, and trachyte from the Sañico Formation are present; tuffs and ignimbrites of the Collon Cura Formation, along with sandstones, conglomerates, and claystones typical of the Huitrera-Ventana Formations are also present. Finally, all over the landscape, glacial and glaciofluvial deposits are recorded: gravels, sands, and silts (Ecosteguy et al. 2013).

Considering previous archaeological research on the area, the use and supply of pigments is still poorly understood; only a preliminary study (Aldazabal et al. 2014) has been presented. Knowledge about the composition of these materials is scarce in comparison to other artifacts such as pottery, obsidian, or metals.

The use of natural pigments has already been reported in art from North Patagonia (e.g., Masaffero et al. 2012; Prates 2009; Rousaki et al. 2015; Sepúlveda 2011; Wainwright et al. 2000). However, documentary evidence of earliest interethnic contacts shows a wider scope in the use of these natural pigments. Vivante (1944) has described how early missionaries and travelers were impressed at the sight of the body painting of the Tehuelches groups in the area of San Julian, who covered their entire body and hair with pigments, primarily red and white but also yellow and black ones. Sánchez Labrador (1936 [1767]) and García (1972 [1819]) highlight the importance of body painting for human groups from southern Buenos Aires and, additionally, report that pigments were collected from the banks of the Colorado River, where white, red, black, yellow, blue, and green colored minerals can be found. Gallardo (1910) also reports that Ona groups obtained the red and yellow pigments from river banks, whereas white paint was obtained from soils or, if they preferred a pure white, by using burnt bones. Black color was obtained from burned pastures, usually mixed with grease in order to smear it easily. These inorganic pigments were used both for dyeing and body painting. Prichard (2003 [1900–1901]) reports that the Tehuelches of Santa Cruz used some kinds of soils for dyeing wool; however, the resulting colors were pale. Inhabitants of the *pampas* employed products derived from herbal tincture that produced more vivid colors.

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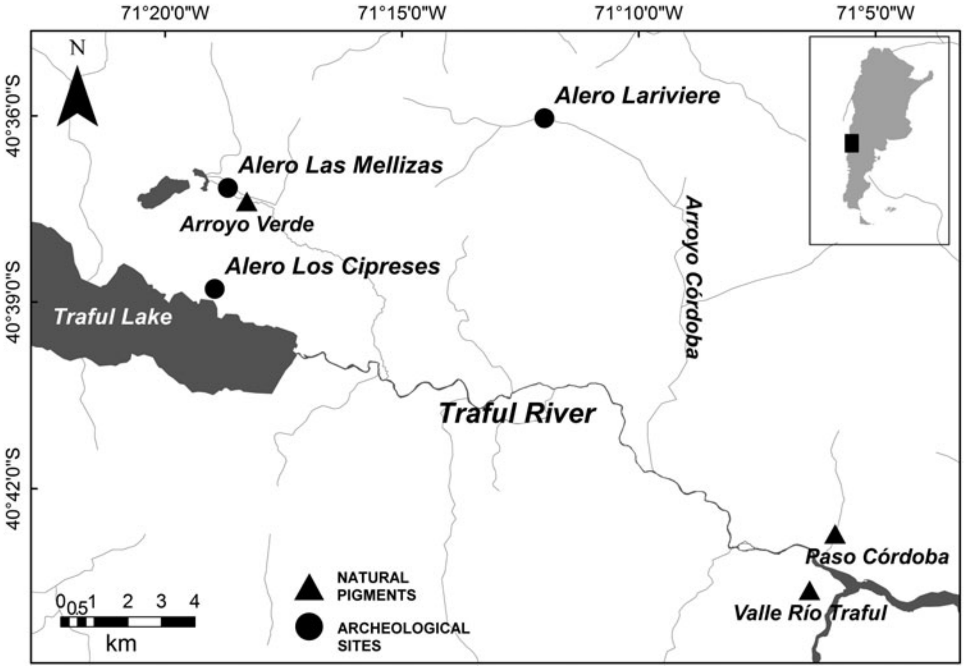

Figure 1. Location of the studied archaeological sites and places of pigments procurement.

Reports from chroniclers and travelers point out that human groups in Patagonia took advantage of mineral, plant, and animal resources for the production of pigments; and, sometimes, clay, coal, vegetable resins, or fat could be added to improve the adherence and durability. Besides, several methods were employed for the procurement of resources: exchange, purchase, and less often, direct procurement. It is worth remarking that in some cases, the supply of dyes was carried out only once a year, but large quantities of raw pigments were acquired. Moreover, the places that supplied the dyes were spread out (Prates 2009:218–219). This is apparent when we consider the references to places as far as San Juan mines (1400 km), to La Tinta and Tandil mountains (1100 km), and to some other unknown sites in the Andes (closer than 50 km) or near the Colorado River (600 km). Priegue (2007) also reports that Tehuelches people looked for green, orange, black, and red minerals towards the northern region or that they commissioned someone to do it. This variability makes it difficult to determine a potential source of procurement.

Regarding pigment processing and application, Priegue (2007:30) describes the handling of a large stone, smooth on both sides, which was used on one side to knead the paint used for pencil manufacturing; sometimes, an index finger was used as a brush. González (1965 [1798]) describes a technique of preparing pencils by rubbing the stone to obtain a very fine powder, which was then mixed with grease or similar substance to be resistant to moisture. This substance was kneaded into bars to be used as a pencil for painting blankets. He also highlights that red and black colors are of mineral origin (González 1965 [1798]:116). Musters (2005 [1869–1870]:196) and Claraz (1988[1865–1866]:130) describe the use of paintings on the body and the addition of fat or gelatin extracted from bones or water to prepare the coloring mixture.

In this work we present analysis and geochemical characterization of mineral clasts recovered from excavations. Some artifacts that may have been used in processing the pigments, such as mortars or pebbles used as strikers, were also analyzed. Although pigments were applied

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on various support surfaces such as walls of shelters and pottery, we focus on the study of natural pigments along the human occupation of the area. The results also provide information to discuss the possible uses and applications of these pigments.

Experimental

Sites and Samples

Pigment samples from three archaeological sites were analyzed: Alero Los Cipreses (ALC), Alero Larivière (AL), and Alero Las Mellizas (ALM). Evidence about the use of pigments was recorded on three different supports: grinding artifacts, pottery, and rock art. This paper is mainly focused on the analysis of natural pigments, but some grinding artifacts were also included. Time reference was obtained in all cases from vegetal charcoal collected in fire structures, by radiocarbon dating in the LATYR laboratory (*Laboratorio de Tritio y Radiocarbono*), La Plata, Argentina.

Alero Los Cipreses (ALC) is a campsite that presents one aceramic and two ceramic components. The first occupation was dated at 3490 ± 80 and 2890 ± 100 BP; the earliest ceramic component was dated between 1510 ± 90 and 840 ± 90 BP (CI), and the most recent one between the sixteenth century and 1891 (CII). The rock art in this site is red and yellow colored, with simple geometric shapes (Silveira 1996). Red paint on ceramic artifacts has been observed only on six sherds from CI (Aldazabal and Micaelli 2007).

The Alero Larivière (AL) is located on the south bank of Córdoba stream, near the headwaters. It was occupied from 2760 ± 80 BP until European contact in the nineteenth century. The emergence of pottery was dated at 780 ± 50 BP (Silveira 1988–1989, 1999). The rock art of this shelter was studied in detail by Silveira (1988–1989) who distinguished three techniques: engraving, painting, and a combination of both (painted following the enclosed form of engraving). The color and tones observed were deep red, purplish red, black, and green with different frequencies in the designs: red in 36 motifs; black in 15 motifs; bicolor (black-red) in 27 motifs, and green in 2. In addition, only four pottery fragments have red paint.

The Alero Las Mellizas (ALM) is placed within a *Nothofagus* forest at 1,027 m high. The cultural remains correspond to a single occupation with the manufacture of ceramic artifacts, dated at 590 ± 90 BP. Paintings on the walls of the shelter are comprised of mostly geometric shapes and some figurative motifs (animals and humans), combining red, yellow, orange, white, black, and green pigments. Finally, five pot fragments painted with red pigment were found (Silveira et al. 2014). In the river close to this site, a green clast was recovered (ALM-022).

Pigments are defined as the natural mineral clasts whose pressure produces a colored stripe. Pigment samples were recovered in stratigraphic position during excavation throughout the archaeological sequence. Fallen fragments from the shelter wall with paint traces (Aldazabal et al. 2016) and some artifacts that could have been used for processing pigments were also analyzed and included in the discussion. The number of pigments and artifacts recovered in stratigraphy are listed in Table 1. The selection of the samples to be analyzed took into consideration that all the colors and chronologies were represented. Nevertheless, it is important to highlight that black pigment samples as raw material were not identified.

Surveys carried out in the area evidence the presence of natural pigments of different colors and dimensions on the banks of the Traful Lake and in the rivers and streams near the archaeological sites. Some of these pigments were collected and analyzed.

Instrumentation

Wavelength dispersive X-ray fluorescence spectrometry (WXRF), Raman spectroscopy (RS), and X-ray diffraction (XRD) analyses were performed on the samples. The importance of these techniques in archaeology is based, essentially, on its nondestructive nature and the possibility of analyzing samples of various shapes and sizes. Furthermore, the samples do not require prior preparation, and they are not damaged during the analysis.

WXRF analysis was performed using a Panalytical Venus 200 MiniLab, with an Sc X-ray tube

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Table 1. Mineral Pigments Recovered in Stratigraphy and Samples Analyzed.

Site	Chronology yr BP	Pigments	Artifacts with Pigment Remains
ALC[a]	S. XVI	Red (3)	Grinding tool with red pigment (2)
	840 ± 50 to 1510 ± 90	Red (10)	Pebble with black adherence (1)
		Green (1)	Painted pottery (6)
			Grinding tool with red pigment (2)
	2890 ± 100	Red (3)	Pallet with pigment red-white-black (1)
		Green (1)	Pebble with red pigment (1)
		Yellow (1)	
	3490 ± 80	Red (2)	
	ND[d]	Yellow (1)	
AL[b]	780 ± 50	Red (6)	Painted pottery (4)
		Green (2)	Fallen fragments of rock art (6)
		Yellow (1)	
		White (3)	
	2760 ± 80	Red (2)	
ALM[c]	590 ± 90	Red (10)	Painted pottery (5)
		Yellow (3)	
		White (2)	
	ND	Red (2)	

[a]ALC = Alero Los Cipreses; [b]AL = Alero Larivière, [c]ALM = Alero Las Mellizas. [d]ND: no datum.
Note: The dates are ^{14}C years BP [absolute values].

operating at fixed excitation conditions 50 kV and 5 mA. An elementary characterization (from Na to U) of the natural pigments was obtained. Light elements (Na to Ca) present higher detection limits.

Raman microscopy analyses were performed on a LabRAM HR Raman system (HORIBA Jobin Yvon), equipped with two monochromator gratings and a charge coupled device detector (CCD). A 1600 g/mm grating and 100 μm hole resulted in a 1.5 cm^{-1} spectral resolution. A He-Ne laser line at 632.8 nm and an Ar laser line at 514.5 nm were used as excitation sources. Laser fluence was adjusted in order to avoid overheating on the sample (around 5 mW). The spectrograph was coupled to an imaging microscope with 10X, 50X, and 100X magnifications. Typically, the laser spot on the sample was about 20 and 3 μm diameter for a 10X and 50X magnification, respectively.

X-ray powder diffraction patterns were taken on a Philips a X'Pert Philips PW3020 diffractometer (Philips, The Netherlands), using graphite monochromatized Cu Kα radiation (1.54184 Å), at room temperature (1° divergence slit; 1° detector slit and 0.1 mm receiving slit). The generator was operated at 40 kV and 30 mA. X-ray measurements were performed using the step mode (0.02° per step) with a 15 s counting time per step, in the range $5° \leq 2\theta \leq 70°$. Phases were identified with the JCPDS-ICDD Powder Diffraction Database (International Centre for Diffraction Data, PA, USA). The samples were directly mounted, using an Al holder to fix them; the analysis was performed on different areas.

The applied experimental techniques provide complementary information: WXRF allows the identification of elements, whereas RS and XRD give information about compounds. Not all the compounds have a Raman signal or diffraction pattern, nor are the signals strong enough to identify them. Furthermore, the area analyzed by XRD is about 0.5 cm^2 whereas the area analyzed by Raman spectroscopy is from 7×10^{-8} to 3×10^{-6} cm^2.

Results

The results of the detected elements and the mineral compounds assigned as responsible for color are summarized in Table 2. WXRF analysis of green samples shows as major components Fe, K, Mn, and Si. In all white samples, Ca was observed, and other elements (P, Fe, and Sr) were also observed. The red samples show a

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Table 2. Elements and Compounds Reported for Analyzed Samples, Including Date and Color.

Sample	Dates yr BP	Color	WXRF	XRD	RS
ALM-022	riverside	green	Fe, Mn, Ti, Ca, Si, K	Glauconite and/ or Celadonite	
AL-E2-002	780	green	Fe, Cu, Si, P, K, Ca, Ti, Mn, Ni, Zn	Celadonite and/ or Glauconite	Celadonite
ALC-014	1590	green	Fe, Mn, K, Si	Celadonite	Celadonite Carbon
ALC-100	2890	green	Fe, Rb	Celadonite	Celadonite
ALC-103	2890	red	Fe, Mn, Mg, Ca, Ti		Hematite
ALC-107	2890	red	Fe, Zr, Cu, Mn, Zn, Ti		Hematite
ALC-109	2890	red	Fe, Ti, Mn, Cu		Hematite
ALC-110	2890	red	–		Hematite
ALC-101	1590–840	red	Fe, Zr, Mn		
ALC-259	1590–840	red	Fe, Mn, Zr, Y, Sr, Zn		Hematite Carbon
AL-D3-003	780	red	Fe, Sr, Mn, Ti	Hematite	Hematite
ALM-260	590	yellow	Fe, Zr, Sr, Ti		Hematite Magnetite Carbon
ALM-080	590	yellow	Fe, Zr, Y, Mn, Ti		Hematite
AL-002	780	white	Ca, P	Apatite	Apatite, Gypsum
AL-003	780	white	Ca, Fe, Sr	Hydroxilapatite	Apatite
ALM-254	590	white	Ca	Apatite	Apatite Carbon
ALC-369	2890	pebble with red pigment	–	Hematita Pirolusite Ilmenite	Magnetite Pirolusite
ALC-650	2890–1510	pallet with white pigment remains	–	Calcite	Calcite Carbon
ALC-034	1590–840	grinding tool with red pigment	–		Hematite
ALC-D7-037	1590–840	grinding tool with red pigment	–		Hematite Carbon

preponderance of Fe, Mn, and Ti, and in some cases other secondary elements as well (Ca, Zr, Mg); finally, the yellow ones reveal little variation with the red ones, containing mainly Fe, Ti, and Zr (Table 2). The elemental composition of similar color samples obtained by WXRF is not exactly the same, possibly representing impurities or secondary elements that could be a complementary path to infer potential sources of provenance or to explain the range of color variation observed.

Green samples were analyzed by X-ray diffraction (Table 2). Our results suggest the presence of iron green minerals: celadonite and/or glauconite. The approximate composition of celadonite is $K[(Al,Fe^{3+}),(Fe^{2+},Mg)](AlSi_3,Si_4)O_{10}(OH)_2$ and of glauconite is $(K,Na)(Fe^{3+},Al,Mg)_2(Si,Al)_4O_{10}(OH)_2$. Although diffraction patterns of both minerals are quite similar, complementary results from Raman spectroscopy suggest that celadonite is responsible for the green color in some of the samples. Figure 2 shows three diffractograms (green samples AL-E2-002; ALC-100; ALC-014) together with celadonite pattern (PDF 00-002-1011). A very good agreement is observed for both ALC samples; for the AL-E2-002 sample, the celadonite signal is weak and it matches to PDF 00-049-1840 (not shown in the Figure). Figure 3a shows a Raman spectrum from a green sample (ALC-014) where the typical bands of quartz (SiO_2) can be observed (peak ca 460 and 125 cm^{-1}). The remaining bands are compatible with those reported by Correia and others to different varieties of celadonite (Aliatis et al. 2009; Correia et al. 2007; Ospitali et al. 2008).

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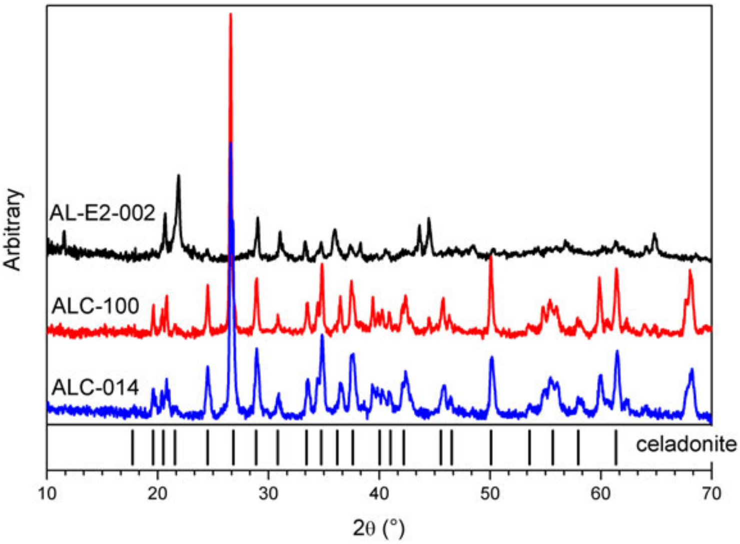

Figure 2. X-ray diffraction patterns from AL-002, ALC-100 and ALC-014 samples. The peaks of a celadonite pattern are also indicated.

Figure 3b presents two Raman spectra acquired on white samples (ALM-254; AL-002). The upper spectrum indicates the presence of apatite: the very intense peak close at 965 cm^{-1} is assigned to the stretching vibration of phosphate (PO_4^{3-}; Awonusi et al. 2007). This component is present in almost all the white samples. Only in one of them was it observed in addition to gypsum: an example is shown in the lower spectrum (Figure 3b). A peak around 1007 cm^{-1}, corresponding to gypsum ($CaSO_4$) is observed. The presence of bone is corroborated by XRD results. On the other hand, one artifact with remains of white pigment (ALC-650) shows the presence of calcite ($CaCO_3$) by RS.

Figure 3c presents examples of typical Raman spectra from red (ALC-103) and yellow (ALM-260) samples. In both cases, the color is associated with iron oxide compounds with spectra corresponding to hematite (α-Fe_2O_3; de Faria and López 2007). Results from some points (not shown) on ALM-260 (yellow sample) reveal magnetite (a band ca 670 cm^{-1}). XRD diffraction patterns of this sample are dominated by quartz and anorthite that make other assignments more difficult. Iron oxides, such as magnetite and hematite, could be present.

As mentioned above, black pigment samples as raw material were not collected. However, black dots were identified under microscope in some artifacts (ALC-650; ALC-D7-037) and in two pigment samples (ALC-014; ALC-259). The Raman bands around 1350 and 1590 cm^{-1} indicate the presence of carbon-based materials (Figure 4). Black components could be associated with minerals and organic natural materials, as graphite, humic earths, and sepia, or obtained from burnt vegetal or animal materials (Coccato et al. 2015). The determination as carbon black is consistent with vegetal charcoal.

Besides the minerals associated with colored pigments, several compounds were detected by both Raman and XRD techniques. Quartz (SiO_2) was present in most of the samples. Titanium dioxides (TiO_2) was identify mainly as anatase, but rutile and brookite were also determined to be present. Aluminum silicates were frequently detected. The XRD results also suggest the presence of ulvoespinel, biotite, attakolite, and gypsum, among other minerals. The numbers of collected pigments samples of each color from the three sites are shown in Figure 5, to analyze frequency of presence.

Discussion

The results of the three techniques match and complement each other, showing the variability

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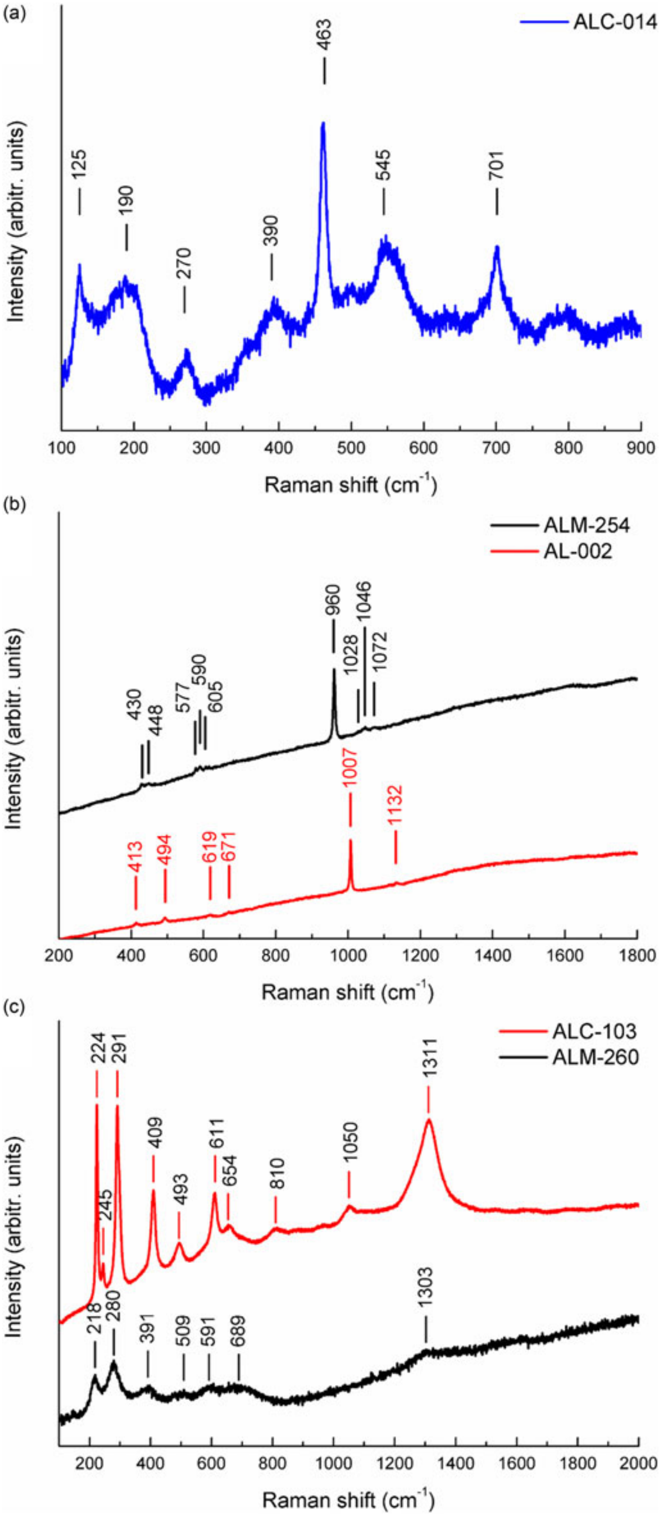

Figure 3. Raman spectra on samples: (a) ALC-014 (green); (b) ALM-254 and AL-002 (white); (c) ALC-103 (red), and ALM-260 (yellow).

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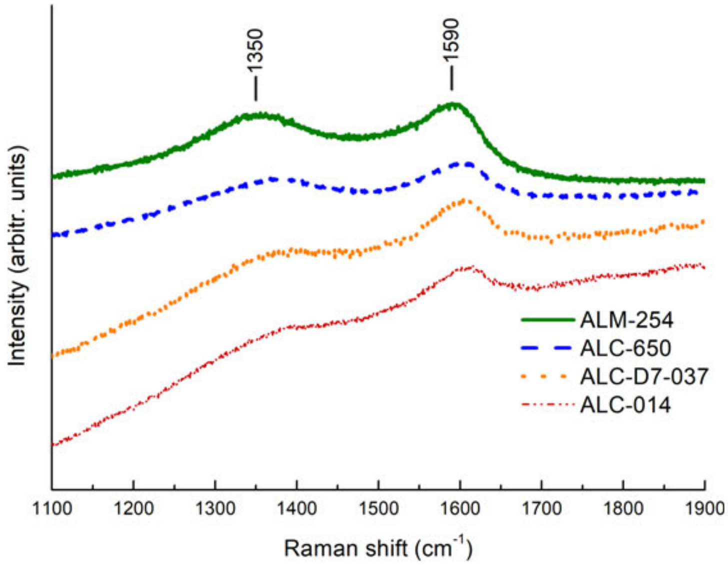

Figure 4. Raman bands associated with carbon signal from spectra on black dots from several samples.

of colorant raw materials used. The analysis reveals the presence of iron-based mineral in green, red, and yellow colors. Green color is associated with the presence of celadonite or glauconite; red and yellow colors with iron oxides, mainly hematite; and white color with bone.

Celadonite is a phyllosilicate, from the montmorillonite-vermiculite group. It appears as filling cracks in basalts and andesites with the crystal structure of mica. Because of the color properties and similar occurrence, it is often confused with glauconite (Odin et al. 1988). Although the chemical composition of the two is similar, because of geological conditions they are found in widely different environments. Archaeologically, celadonite has been identified

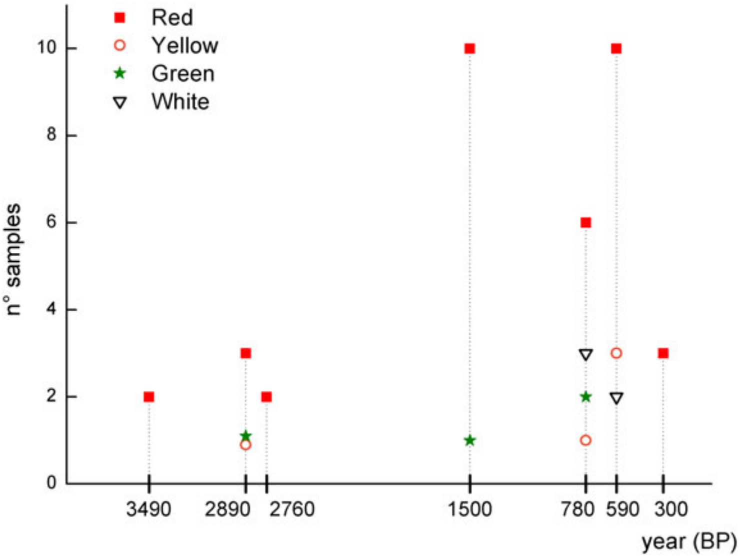

Figure 5. Total number of collected pigments samples of each color, by strata.

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in the rock art paintings of the Comallo I Cave (50 km SE of the study area), but the surveys carried out in the nearby area of the cave showed neither celadonite nor glauconite. Several authors propose that these pigments could have been brought by trade (Boschín 2009; Boschín et al. 2003; Masaferro et al. 2012). In Patagonia, the presence of celadonite is reported in basaltic rocks of Sañico in the Aluminé Lake (Neuquén), Los Menucos (Río Negro), and Paso de los Indios (Chubut; Hayase and Manera 1973). It is worth highlighting that in our case, green pigments were recovered from ALC and AL sites, even if in the last one there are no pictograms in this color. At ALM, many pictograms were made in green, but no pigment was recovered. However, green clasts collected from Arroyo Verde, near ALM, and from the Arroyo Córdoba, near AL, were identified as celadonite. The novelty of finding green clasts in two of the three archaeological sites and their presence in the streams makes it possible to argue for, unlike Comallo I cave, a local provisioning.

White pigment samples analyzed in the present work are mainly bones. These charred bones formed a small set in stratigraphic position; they were interpreted as pigment because they are potentially usable for painting as the process is described in historical documents. However, this interpretation is subject to the identification of this color on the rock art paintings or other kind of support. One flat lithic artifact with white pigment remains was found at ALC. However, the analysis only revealed the presence of calcite. This could suggest that bone was not the only source of white pigment. Archaeological research allowed the authors to determine various ways of obtaining this color. In Comallo I cave, the authors described a mixture of quartz, plagioclase, and mica (Boschín 2009; Boschín et al. 2003, 2011).

Hematite is the mineral form of ferric oxide. It appears as aggregate of small red crystals or as large dark ones and is difficult to crush (Clottes et al. 1990). In its natural state, it contains 70% iron and traces associated with titanium, aluminum, manganese, and water. Its colors can include yellowish ochre, reddish brown, red to silver-gray, and black. It usually occurs in sedimentary deposits but is also found in metamorphic rocks due to contact metamorphism, and occasionally as a sublimate in extrusive igneous rocks. It is also integrated as a colorant in red soils (Brodtkorb et al. 2007; Leal et al. 2011). Archaeological studies on rock paintings in the region show that the red color is the result of using two kinds of minerals: natural hematite and thermally processed goethite or maghemite (Boschín 2009; Boschín et al. 2003; Massaferro et al. 2012; Podestá and Albornoz 2007; Rousaki et al. 2015; Wainwright et al. 2000). Nevertheless, the red samples presented in this work only evidence hematite as the principal chromophore. This could suggest that no thermal processing was necessary because natural hematite was available to be directly used. Hematite red pigments were registered from the earliest levels in the three archaeological sites with a tendency to increase in presence in later times (Figure 5). This is consistent with the established dates for the beginning of the registered rock art painting style and could suggest a previous use requiring less material over nonlasting supports. Guanaco skin fragments painted with geometric designs were found in Puerto Tranquilo I, Nahuel Huapi Lake, in a ceramic occupation context dated at 640 ± 60 BP (Hajduk et al. 2007). Evidence of painting activity on leather was recovered in Campo Moncada 2, Province of Chubut, in archaeological contexts dated between 5000 BP and 800 BP (Marchione and Belleli 2009). In addition, lithic fragments with remnants of ochre pigment were recovered at Traful I cave, in the initial occupation dated to 9430 ± 230 BP; flakes and faunal bone fragments with ochre were also collected at the level dated 6240 ± 60 BP (Crivelli Montero et al. 1993). In the area under study, in addition to the use of pigments in rock art, pigments were registered in a few ceramic fragments from recent layers, where they were applied as slip (Table 1). Taking into account all the available information, we could suggest a use for corporal or animal skin painting as well as a likely secondary treatment of the dead human bones. The use of red pigments is also evident through the remains found on different artifacts. Some artifacts with traces of red pigment were recovered at the ALC archaeological site at levels dated 2890 ± 100 BP. Their presence supports

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the hypothesis of an early local processing of pigments. Moreover, pebbles with red pigment adhered, dated at 840 ± 90 BP from ALC and at AL dated 780 BP, show the continuity of these practices over time. Processing artifacts does not appear in ALM, which could suggest, issues of preservation aside, that the pigments were used without previous preparation or were brought already processed.

Few yellow samples were collected; they revealed the presence of iron oxides. Iron-based yellow pigments are commonly associated with limonite, lepidocrocite, and goethite among others (Shepard 1985). Raman signal of these minerals is weak in comparison to others also present, such as hematite. X-ray diffractograms, on the other hand, are dominated by feldspars and quartz that make the detection of iron-based minerals difficult.

The determination of carbon black is consistent with vegetal charcoal. The evidence, as dots on samples of natural pigments and on some processing artifacts, may be interpreted as a result of matrix contamination. In addition to the chromophores determined, some complementary compounds were identified as quartz and anatase. Both are present in many mineral pigments and also in sediments. They appear in fractures of certain metamorphic rocks such as schist or gneiss. They can also be formed as a secondary mineral in igneous rock crevices, such as granite or pegmatite, and finally, they can be found in alluvial sediments carried by streams from high mountains and redeposited. They are commonly associated with brookite, rutile, titanite, ilmenite, titaniferous magnetite, hematite, and quartz (Brodtkorb et al. 2007).

The availability of the aforementioned minerals in the region is widespread. Based on the geological information, similar lithologies and minerals have been identified in the nearby area and could have constituted sources of supply. As mentioned above, green clasts were found in the streams near the archeological sites. Leal and colleagues (2011) describe hematite at Paso Córdoba, just 10 km away from the sites. The Collon Cura and Angostura Colorada formations exhibit outcrops of mineral rich in iron oxide and are located closer than 100 km from the sites (Danieli 1995; Ferrer et al. 1999; Manassero and Maggi 1995; Zappettini and Dalponte 2009). At La Ventana formation, just 50 km away, even if reddish tuffs predominate, greenish gray and beige colors could also be found. At Cajon Grande and Cajon del Medio (NW Neuquén province, 200 km away), biotite and primary amphiboles (edenite, ferro-edenite and magnesiotaramite) were identified, and in secondary igneous rocks (andesite, diorite and microgabbro), associated with skarn, the presence of plagioclase, quartz, magnetite and ilmenite, apatite, and titanite have been reported (Ecosteguy et al. 2013; Leanza et al. 2011). A first approach to the supply areas of mineral resources found throughout the occupation sequence of the area was summarized by Silveira and others (2013). In addition, specific surveys carried out near the archaeological sites, to search for potential sources of pigment raw materials, were positive, registering pigment clasts of all colors on the surface and in the streambeds (Aldazabal et al. 2016). These results led us to propose secondary sources for pigment supply. Moreover, comparisons with previous studies point out that the found raw materials were widely used in the region (Albornoz et al. 2008; Aldazabal et al. 2015, 2016; Hajduk et al. 2007; Rousaki et al. 2015; Sepúlveda 2011; Vázquez et al. 2008; Wainwright et al. 2000).

Finally, even though the absence of rock art paintings at early times could be a consequence of conservation problems, studies about rock art in this cordillera region postulate a chronology no older than 2800 years BP (Crivelli Montero 2006). On the other hand, the initial chronology for the style to which the art of these sites is assigned, the Lacustrine Forested Modality, is estimated around 1300 years BP (Podestá et al. 2008).

Ethnographic information and chronicles referring to the Patagonian region reveal that paintings were used by the human groups inhabiting the Patagonia region, and Argentina in general, for different purposes. These purposes include decoration of clothes or tents, as well as body and facial ornamentation (Claraz 1988 [1865–1866]; Fiore 2005; Moreno 2007 [1877]; Musters 2005 [1869–1870]; Onelli 1904). These uses not only had an aesthetic

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value but also a practical aim (protection) and a symbolic meaning. The color palette was mainly reduced to red, black, white, and yellow. A detailed compilation of ethnographic information referring to the different motives and symbolic significance of body painting can be seen in Bolcatto (2015).

Conclusion

The natural pigments studied were collected at stratigraphic position, in association with cultural remains from the beginning of the occupation of the three sites located at the Traful Lake area. Additionally, artifacts associated with the processing of pigments were recovered at the Los Cipreses site, supporting the hypothesis of an early use of color on other supports prior to art or pottery painting. The information provided by the first chronicles allows us to consider the use of pigments from the initial times of the human settlement of these sites, possibly related to treatment of the body, or in materials that are not preserved such as leather, textiles, or basketry. It is notable that red pigments are always predominant and increase their relative abundance in recent levels, but yellow and green are also recovered. In relation to the processing techniques and preparation of the pigments, we recorded grinding artifacts with pigment powder remains, but did not find "pencils" or any evidence that supports the hypothesis of the mixing with other products such as grease. The presence of hematite as unprocessed natural pigment suggests that there was no need for heating other related minerals to obtain a red color, as noted in some of the archaeological papers cited, in which the use of goethite, hematite, and limonite with thermal processing was described. We have interpreted the calcined bones as potential white pigments. These bones could also be a result of processing food resources; however, this kind of thermal alteration was not observed on the faunal collection. Therefore, until new evidence is obtained, we support the hypothesis that these bones are associated with the pigments. Black color, as pigment raw material, could not be discriminated among the archaeological remains because it is generally produced with charcoal, and there are many combustion structures related to many other purposes (warming, cooking, etc.). In relation to pigment supply, the presence of natural clasts in the streams of the area with similar geochemical composition to pigments recovered in excavation would support the hypothesis of their availability when the area was occupied and make it unnecessary to search for fixed remote potential sources. We suggest that the geological characteristics of the area enable direct procurement of these natural pigments as clasts resources in secondary sources. Future surveys and additional ochre sources studies in the region would estimate the geographical extent of ochre procurement.

Finally, the chosen analytical techniques have improved a specific sensitivity and provided precise information. The results from complementary analytical techniques allow more accuracy in the determination of minerals and help to avoid misinterpretations. The information on natural pigments presented in this paper constitutes the first step toward studying the importance of color throughout the human occupation of the area and its use on different supports. There are numerous archaeological works that refer to paintings on supports such as pottery, rock art, and to a lesser extent on other artifacts. This study focused on raw materials recovered in archaeological contexts. In order to continue with the aim of study, pigments on pottery and rock art samples from the area will be analyzed.

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