MULTI-INSTRUMENTAL CHARACTERIZATION OF TWO RED PIGMENTS IN FUNERARY ARCHAEOLOGICAL CONTEXTS FROM NORTHERN CHILE

Juan Pablo Ogalde, Bernardo Arriaza, Carolina Paipa, Patricio Leyton, Marcelo Campos-Vallette, Nelson Lara, Cristian Salas and Pedro Tapia

SUMMARY

Analysis of two archaeological red pigments from two cities in northern Chile, Calama (Calama sample) and Iquique (Iquique sample) are reported in the current work. Scanning electron microscopy and energy dispersive X-ray spectrometry (SEM-EDX), powder X-ray diffraction (XRD) and vibrational spectroscopy (IR and Raman) were used for structural studies. Hematite (α-Fe_2O_3) was the main component of the red color in both pigments. These are the first results reported for both sampled areas, helping thus to clarify the Calama funeral rites and the raw materials used for Chinchorro mummification in the Iquique region. The characterization of the raw materials provides information for future studies focused on hematite mining processes.

Introduction

Pigments from two archaeological sites, in the Calama and Iquique regions, northern Chile (Figure 1a), were chemically identified. The pigment samples were obtained from different geographic areas and the sites correspond to different cultural periods. The Calama pigment sample (Figure 1b) was found inside a *Concholepas concholepas* shell during an excavation in the Chorrillos archaeological cemetery, an early Formative site (2750-2150 B.P.), located in the Atacama Desert. The population buried in this cemetery belonged to the early agricultural period, and exhibited a 'circular-oblique' cranial deformation trend (Depósito Arqueológico de Calama – DAC– register). Previous studies in Calama (Ogalde *et al*., 2014) and a review of archaeological collections of this area have shown the presence of shells and pigments on the offering tombs. This type of offering is common in the Calama area and raises questions about the origin of the pigments. However, systematic studies concerning chemical characterization have not been done yet on this type of mineral.

The Iquique pigment sample (Figure 1c) comes from a facial mask of a Chinchorro mummy from the Sermenia site, an archaeological cemetery in the coastal desert

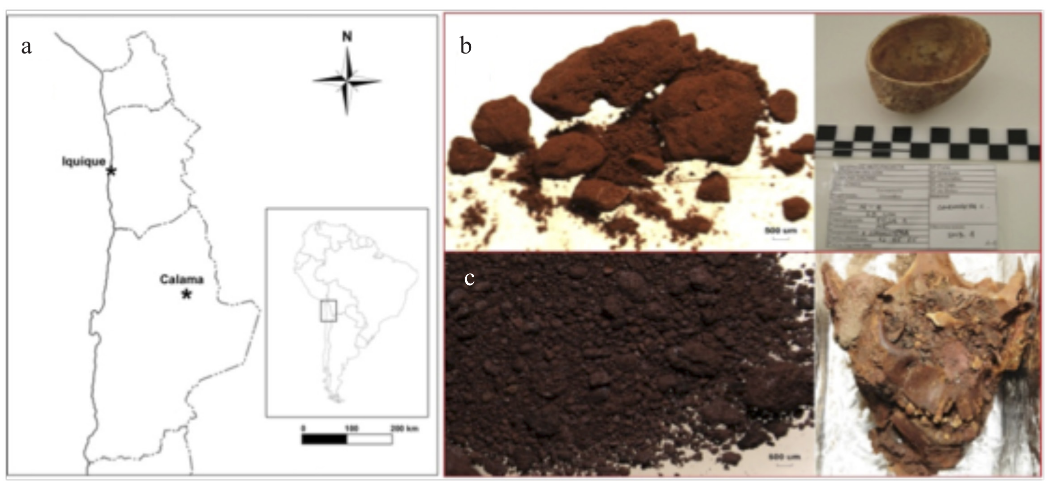

Figure 1. a) Map of northern Chile showing the cities of Calama and Iquique, where the pigment samples were found. b) Calama sample (NA-4 DAC, register) which was found in a *Concholepas concholepas* shell, and c) Iquique sample collected from the nasal area of a Chinchorro red mummy.

KEYWORDS / Calama / Chinchorro Mummification / Diatoms / Hematite / Iquique / Ochre /

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CARACTERIZACIÓN MULTI-INSTRUMENTAL DE DOS PIGMENTOS ROJOS DE CONTEXTOS ARQUELÓGICOS FUNERARIOS DEL NORTE DE CHILE

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RESUMEN

En este trabajo se presentan análisis realizados para pigmentos rojos arqueológicos de dos ciudades en el norte de Chile: Calama (muestra Calama) e Iquique (muestra Iquique). Para la identificación estructural se utilizó microscopía electrónica de barrido, espectrometría de energía dispersiva de rayos X (SEM-EDX), difracción de rayos X en polvo (XRD), y espectroscopia vibracional (IR infrarroja y Raman). El principal componente del color rojo en los dos pigmentos fue la hematita (α-Fe_2O_3). Estos son los primeros resultados conocidos para ambas áreas, ayudando a aclarar las costumbres funerarias de Calama y las materias primas utilizadas en la momificación Chinchorro en la zona de Iquique. La caracterización de estas materias primas provee de información para futuros estudios focalizados en los procesos mineros de hematita.

CARACTERIZAÇÃO MULTI-INSTRUMENTAL DE DOIS PIGMENTOS VERMELHOS DE CONTEXTOS ARQUELÓGICOS FUNERÁRIOS DO NORTE DO CHILE

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RESUMO

Neste trabalho são apresentadas análises realizadas para pigmentos vermelhos arqueológicos de duas cidades do norte do Chile: Calama (mostra Calama) e Iquique (mostra Iquique). Para a identificação estrutural se utilizou microscopia eletrônica de varredura, espectrometria de energia dispersiva de raios X (SEM-EDX), difração de raios X em pó (XRD), e espectroscopia vibracional (IR infravermelha e Raman). O principal componente da cor vermelha nos dois pigmentos foi hematita (α-Fe_2O_3). Estes são os primeiros resultados conhecidos para ambas as áreas, ajudando a esclarecer os costumes funerários de Calama e as matérias primas utilizadas na mumificação Chinchorro na zona de Iquique. A caracterização de estas matérias primas provê informação para futuros estudos, focados nos processos mineiros de hematita.

area. The mask was severely damaged and mainly made of red pigments. Considering the archaeological context of the Iquique sample it can be relatively dated to around 4800-3800 B.P., a period associated with the red type of Chinchorro mummies (Arriaza, 1994, 1995, 2003, 2005; Wise *et al.*, 1994; Muñoz *et al.* 1993; Arriaza *et al.* 2001, 2008a, Standen *et al.*, 2004; Arriaza and Standen, 2009). Red powder was commonly used by archaic Chinchorro people in their coastal mortuary tradition, where skeletonized individuals were reconstructed using wooden sticks and clay. In addition, the Chinchorro mummies were decorated using red and black pigments, mainly manganese (Mn) compounds (Arriaza *et al.*, 2006, 2008b, 2012; Sepulveda *et al.*, 2013, 2014), and occasionally white and green materials, not yet identified (Arriaza and Standen, 2009). As far as we know, an in-depth chemical characterization of Chinchorro mummies red pigments has not been reported thus far.

In the Andes, iron oxides were probably the raw materials mostly used as red pigments, since they are commonly found in geological deposits. The most common red chromophore compound is called hematite (α-Fe_2O_3), which provides strong color, has tinting strength, good opacity and, besides, can be easily ground and finely pulverized (Bonavia, 1959, 1985; Petersen 1970, 2010; Berthelot, 1986; Harben and Kužvart, 1996; Kroeber and Collier, 1998; Harben, 1999; Orefici and Drusini, 2003; Vaughn *et al.*, 2007, 2013; Brooks *et al.*, 2008; Vaughn *et al.*, 2005, 2007, 2013; Eerkens *et al.*, 2009, 2014; Petersen, 2010). In this paper it is shown that the hematite chromophore is associated to different uses and that it was very important in social and mortuary rituals of pre-Columbian populations of northern Chile.

Methods

Light microscopy and SEM-EDX analysis

About 1mg of the selected pigment was separated using a stereomicroscope (Olympus SZX-7) and then mounted on a stub for direct analysis in an EVO LS scanning electron microscope (SEM). SEM images were recorded at 100× and 600× with secondary and back scattered electron detectors. The samples were also analyzed on an Oxford EDX detector (8.5 WD and 450kV). The topographic qualitative information, spectrometric data and semi-quantitative results (detection sensitivity down to 0.1% by weight) were observed and interpreted using the INCA software. This analysis was carried out at the Bioarchaeology Laboratory, Instituto de Alta Investigación, Universidad de Tarapacá, Chile. A morphological and microscopic analysis of diatoms was performed at the Marine Science Laboratories, Universidad Peruana Cayetano Heredia, Lima, Perú.

Infrared analysis

Infrared spectra were run on a Fourier Transform Infrared (FT-IR) Perkin Elmer Spectrum BX spectrometer equipped with a DTGS detector. The spectral resolution was 2cm^{-1}; 32 scans were performed. A pellet was prepared from 1mg of the solid sample dispersed in 200mg of KBr. The samples were processed at the Facultad de Ciencias, Pontificia Universidad Católica de Valparaíso, Chile.

Raman analysis

About 50mg of solid sample was placed on a microscope slide, and the Raman spectrum recorded using a Renishaw Raman Microscope System RM1000 equipped with a diode laser providing the 785nm line, a Leica microscope, an electrically cooled CCD detector and a notch filter to eliminate elastic scattering. The spectrum was obtained using a 50× objective. The laser power output was 2.0mW and the spectral resolution was $2cm^{-1}$. This analysis was performed at the Facultad de Ciencias, Universidad de Chile, Santiago, Chile.

Powder X-ray diffraction analysis

Powder X-ray diffraction patterns of the red pigments were recorded on a Bruker D8 Advance diffractometer using Cu Kα radiation obtained from a source tube operated at 40kV and 40mA. All patterns were recorded between 20 and 65º 2θ with a detector slit of 0.6mm and a scan speed of 0.01º/2sec. This analysis was accomplished at the Facultad de Ciencias, Pontificia Universidad Católica de Valparaíso, Chile.

Results

The Calama sample was a red pulverized material with amorphous conglomerations (Figure 1b), while the Iquique sample consisted of dark red powder with consolidated granules (Figure 1c). These optical images evidenced that homogeneity exists in the morphology and color distribution of both samples. Figures 2a and 2b are microphotographs obtained with backscattered electrons on homogenous areas from Calama and Iquique samples, respectively. SEM of the pigments allowed selecting the most representative areas for EDX analysis. Figure 2c presents the average results of the semi-quantitative EDX analysis of the total area and sub-areas of several granules. The graph shows that iron is the main component in both the Calama and Iquique samples (56 and 58% respectively), while the second highest peak corresponds to silicon concentration (20 and 15%). The high Si concentration, in addition to the presence of aluminum in both samples, suggests phases of clay and/or aluminum-silicate minerals. It is noteworthy that the lower Al concentration in the Iquique sample as compared to the Calama content indicates differences in their geological origins. Furthermore, the Iquique sample presents a high manganese content (10%), not detected in the Calama sample, which is more abundant than Al (4%). The greater concentration of calcium (5%) in the Iquique sample is also worth noting.

The IR spectrum of the Ruff standard of hematite is included in Figure 3a, where medium-strong bands at 463 and $544cm^{-1}$, assigned to hematite, are observed. In the Calama sample the bands at 470 and $560cm^{-1}$ are assigned to the hematite chromophore (Figure 3a). Bands observed at 1085 and $1037cm^{-1}$ are assigned to the (ν)ν Si-O bond stretching, while weak bands at 694 and $776cm^{-1}$ and the shoulder at about $797cm^{-1}$ are assigned to deformation modes involving the Si-O moiety and attributable to quartz (SiO_2). The band at $3434cm^{-1}$ indicates the presence of a hydroxyl group, where the largest band width at $3434cm^{-1}$ is probably due to interactions between the hydroxyl group and clay (νSi O-H: 3700-$3200cm^{-1}$) or a hydroxyl associate group (subintervals ~3300-$3100cm^{-1}$). The band at $1634cm^{-1}$ shows that a fraction of hydroxyl groups belongs to water, while a band at $898cm^{-1}$ from hydrated iron oxide compounds, such as goethite, is not observed. However, the very weak bands at 2925 and $2956cm^{-1}$ corresponding to aliphatic νC-H suggest the presence of organic materials in the Calama sample (Perez and Martin, 1967a, 1967b; Rendon and Serna, 1981; Schrader, 1995; Stuart, 2004; Toledano, 1988; Vargas-Rodriguez *et al.*, 2008; Zapatero *et al.*, 2000).

In the Iquique sample (Figure 3a) the IR bands at 457 and $532cm^{-1}$ correspond to the hematite chromophore. Bands observed at 1085 and $1034cm^{-1}$ are assigned to the (ν)ν Si-O bond stretching or Si-OH bond. The weak band at 878 and $795cm^{-1}$ may come from hydrated iron oxide compounds such as goethite; the weak bands at 697, 779 and $795cm^{-1}$ are ascribed to deformation modes (Si-O) of quartz. The wide band appearing at *ca.* $3430cm^{-1}$ points to the presence of a highly associated hydroxyl group; while the band at $1634cm^{-1}$ shows that a fraction of the hydroxyl group belongs to water. However, the strong bands at 2957, 2917 and $2849cm^{-1}$ corresponding to aliphatic νC-H, suggest the presence of organic materials in the Iquique sample, which is of importance because some bands assigned to bonds of inorganic compounds could be reinterpreted. There are three bands in the zone of νC-H bond with C sp^3 hybridization; therefore, the presence of methyl ($-CH_3$) and methylene ($-CH_2-$) groups is possible, given the occurrence of a band at $1467cm^{-1}$. Indeed, the band at $1569cm^{-1}$ could correspond to the C=O bond, a signal possibly related to bending vibration at $1421cm^{-1}$ of activated methylene ($-CH_2-CO-$). In this sense , the band at $1322cm^{-1}$ could correspond to $-O-CO-CH_3$ (1380-$1365cm^{-1}$) or -CO-CH3 (1360-$1355cm^{-1}$) groups, in which case the band of CH_3 vibration (usually appears at $1380cm^{-1}$) moves to $1467cm^{-1}$ as indicated above. In order to complement the information to be discussed below, it is necessary to keep in mind that the S=O bond has several signals at 1225-$980cm^{-1}$ and a very intense band into the 1420-$1000cm^{-1}$ range (Perez and Martin, 1967a, b; Rendon and Serna, 1981; Toledano, 1988; Schrader, 1995; Coates, 2000; Zapatero *et al.*, 2000; Stuart, 2004; Vargas-Rodriguez *et al.*, 2008).

The Raman spectra (Figure 3b) of the Ruff standard hematite showed bands at 610, 496, 408, 291 and $226cm^{-1}$. These signals are also present in both Calama and Iquique samples. No other signals are visible in the Raman spectra obtained from the samples. This may be due to the intensity of the Fe-O bond signals, which mask other less intense signals, such as those of manganese oxides (Schrader, 1995; Smith and Dent, 2005).

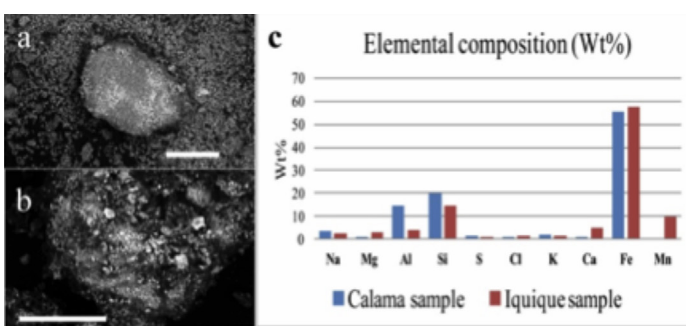

Figure 2. Micrographs obtained with backscattered electron on granules from a) Calama and b) Iquique samples. c) Average results of EDX analysis of the archaeological pigments (carbon and oxygen are not displayed in this chart).

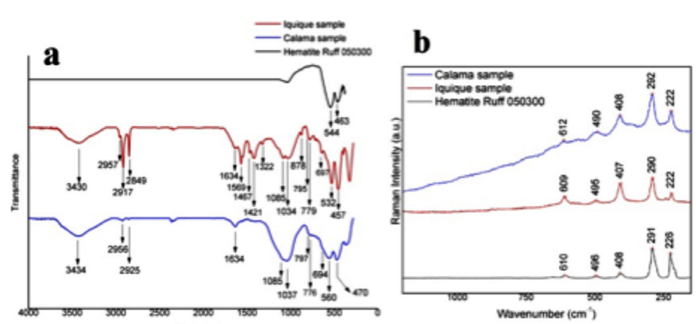

Figure 3. a) Infrared and b) Raman spectra obtained from Calama and Iquique samples along with a hematite spectral reference.

The XRD patterns of both red pigments were compared to reference data (JCPDS, 2004) and are shown in Figure 4. Both the Calama and Iquique samples exhibit XRD patterns similar to hematite reference data (33-0664) and quartz (46-1045). Besides, the Calama sample had peaks attributed to aluminum silicates such as albite (01-0739, $NaAlSi_3O_8$) and cordierite (09-0473, $Fe_2Al_4Si_5O_{18}$), absent in the Iquique sample. On the other hand, Iquique samples evidenced the presence of kuthnohorite (19-0234, $Ca(Ca,Mn)(CO_3)_2$) and romerite (13-0530, $Fe_3(SO_4)_4{\cdot}14H_2O$).

Finally, Figure 5 shows SEM images from two kinds of diatoms found in the red powder of th Calama sample. Diatoms are unicellular photosynthetic organisms consisting of one (unique) silica cell wall which is composed of two valves called thecae and several girdle bands. The thecae have different sizes (20-200µm diameter), and are slightly different from each other (Weiner, 2010). Based on these morphological characteristics, the largest and central diatom has been identified as a *Surirella* (cf. *S. wetzelli* Hustedt). There is a second unidentified diatom in Figure 5: the smallest oval at the lower left corner of the picture.

Discussion

The use of the presently studied red pigment in the mortuary Chinchorro tradition is important because it was associated to changes in the chromatic preparation of the body, with large social implications. The high presence of manganese in the Iquique sample (Figure 2) could be ascribed to the use of Mn-containing materials during the preparation of the Chinchorro mummies. Over the surveyed area, the use of black pigment has been reported for Archaic Acha-3 site and in the Camarones14 and 17, where the Chinchorro tradition started with the ‘black mummies’. In the middle Archaic period (7000-5000 BP), in Chinchorro 1 and Maestranza 1 sites this type of mummification was already present and Mn appears in modeled artificial mummies. During the late Archaic period (5000-3500 BP) black color was used on vegetal mats and is part of the filling of artificial mummies. On the other hand, during the late Archaic period, the use of red was fully incorporated in the Chinchorro mummification techniques, characterized as ‘red mummies’ (Arriaza, 1994, 1995, Muñoz *et al.* 1993; Standen *et al.*, 2004; Arriaza *et al.*, 2005; 2006, 2008a, b, 2012; Sepúlveda *et al.*, 2013, 2014). In the Arica area, over 400km north of Iquique, mummies with black/red dish face are found in many sites; however few of them have been thoroughly analyzed. Arriaza *et al.* (2006) study of the coating of eleven black mummies reported 36% of Mn oxide and 8.4% of Fe oxide. In six red mummies they reported 60.7 % Mn oxide and 4.3 % Fe oxide for the head (‘helmet’) and facial mask, respectively. Both types of mummies contain Fe and Mn in different percentages, which can be associated to regional and chronological variations.

The identification of hematite in the present study contributes with hard data to understand the process of chromatic change and the manipulation of the bodies. Also, in the current Iquique case, the presence of Mn could be explained by either an intentional mixture of raw materials during body preparation or natural composition of the red minerals. This mortuary practice could explain the presence of Mn in the mask of the Chinchorro mummy from Iquique. In addition, several bands observed in the IR spectrum of the Iquique sample suggest the presence of organic materials. The intense Chinchorro manipulation of the body could explain the presence of this organic compound. However, Mn is also present in large amounts in some red pigments in the Nasca region (Vaughn *et al.*, 2005; Eerkens *et al.*, 2014). Thus, the mixture of Mn and red pigments in the Iquique Chinchorro mummy sample could be the result of the geochemical origin of the hematite deposit. In this regard, the XRD patterns show quartz, kuthnohorite and romerite. The greater concentrations of Ca and Mn found by the EDX analysis of the Iquique sample are ascribed to these minerals that are associated to mummification techniques and/or geochemical origin. Romerite might be responsible for the intense signal of the νS=O mode detected in IR analysis. Similarly, the water in romerite could be related to the particular signal in the 3600-2700cm^{-1} range of the hydroxyl group from water in the IR spectrum of the Iquique sample (Figure 3a).

Another important fact about red and black coastal pigments is that during the middle Archaic period (7000-5000 BP) they are found deposited on sea shells as funeral offerings (Sepulveda *et al.*, 2014). This is precisely the ‘funeral situation’ of the hematite identified in the Calama sample of Chorrillos archaeological cemetery, located more than 279 km south of Iquique. In the Calama sample a diatom (*Surirella*, cf. *S. wetzelli* Hustedt) was discovered, which is normally found in waters with a high salt content. Also, the XRD patterns show quartz and peaks attributed to albite and

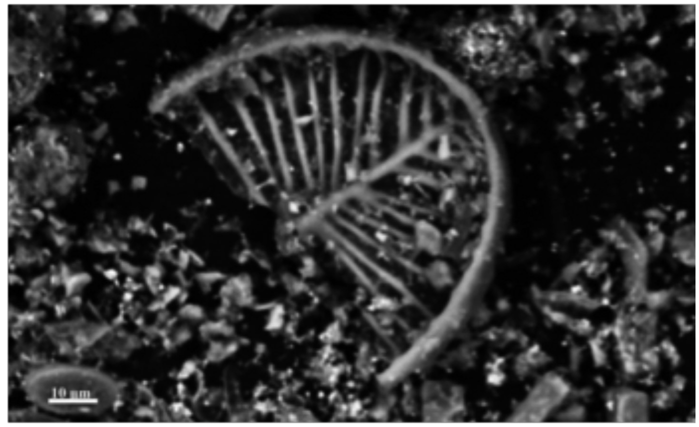

Figure 5. Diatomes in Calama sample (1.97KX).

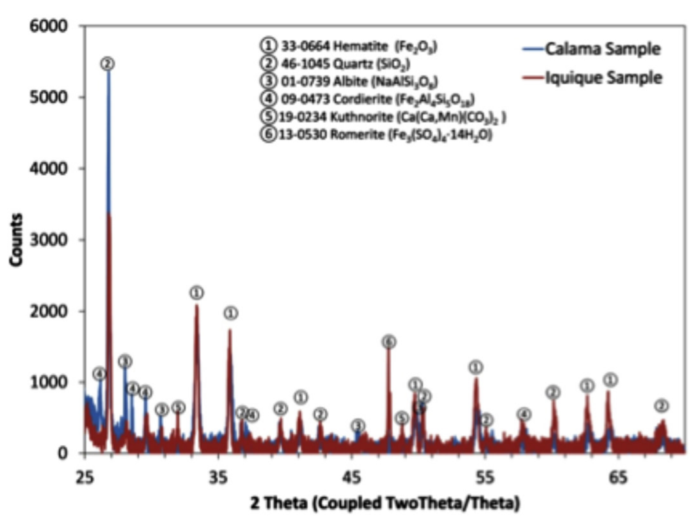

Figure 4. XRD powder diffraction patterns from a) Calama and b) Iquique samples.

cordierite, coherent with EDX spectra. These minerals could be related to the geochemical origin of the hematite deposits in the Calama area, which have silicates rich in Al and water, with high salt content. Previous studies in Calama (Ogalde *et al.*, 2014) have shown the presence in grave goods of a yellow chromophore called orpiment (As_2S_3), which is potentially toxic. Subsequently, red, black and yellow pigments were deposited in valves of *C. concholepas* as funerary offerings at the archaic and formative cemeteries of the studied area. In ancient times people handled different types of minerals with various degree of toxicity. For instance, in Huancavelica, Lima, Peru, cinnabar (HgS) was used as red pigment to decorate pottery, for textile dyes, etc. Consequently, the handling of toxic substances could have been the consequence of trial and error during pre-Columbian era. This type of research could help understand the mining processes, as well as the mineral sources and distribution routes (Salazar *et al.*, 2010a, 2010b, 2013; Salazar and Vilches 2014). This is of interest considering that the extraction of hematite is the first evidence of mining in South America (Vaughn *et al.*, 2007, 2013; Eerkens *et al.*, 2009).

Final Comments

The Iquique sample studied herein is associated to the Archaic period and the Calama one to the Formative period of northern Chile. In summary, the results allow to conclude that in this case, hematite was used in the Chinchorro mummification tradition in the Iquique region. In the Calama area, ancient people also used the same red pigment as part of their funerary offerings for the care of the dead in the afterlife. In both cases, these red pigments are specifically related to mortuary practices and in both cases the red chromophore was hematite. This cultural relationship between pigments and afterlife has a long history in northern Chile and the Andes. The funerary character, toxicity and antiquity of the pigments are important, since they allow investigating the acquisition, processing and ritual aspect of these materials. Future studies could focus on the processes necessary for mining these pigments and the social implications.

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