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Petrologic analysis of mineral pigments from hunter-gatherers archaeological contexts (Southeastern Pampean region, Argentina)

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ABSTRACT

Mineral colorants are frequent in the Holocene archaeological record of the Southeastern Pampean region, but they have been often unexplored. This paper presents the results of macroscopic analysis, and thin section and X-Ray diffraction techniques applied to mineral pigments from the Calera site and the Zanjón Seco and Nutria Mansa archaeological localities. The use of both analytical techniques as complementary tools is a contribution to pigment provisioning studies and the general discussion of mineral raw material exploitation in the region. In prehispanic times, pigments were obtained from mineral sources available in the Tandilia System and, in smaller proportion, from the Ventania System. Transporting these coloring raw materials to the studied archaeological contexts implied different provisioning efforts through local, middle and long distances, and their procurement was related to local and regional mobility circuits of Pampean prehispanic hunter-gatherers.

1. Introduction

Mineral pigments are frequent in the Holocene archaeological record of the Southeastern Pampean region (Mansur et al., 2004; Porto López and Mazzanti, 2005; Bonomo, 2005; Scalise and Di Prado, 2006). They also appear in different kinds of archaeological contexts (i.e., base camps, rock art sites, ritual sites), and usually in lesser proportions than other materials such as lithics, faunal remains, and ceramics. However, exceptional amounts of pigments have been recorded at some sites, such as Calera (Di Prado et al., 2007) and Amalia 2 site (Mazzanti, 2002), both located in the Tandilia System (Nágera, 1940; Fig. 1).

Despite extensive evidence of mineral pigment use among the Pampean hunter-gatherers, studies on this subject are quite recent. They have focused on provisioning strategies through provenance analysis (Mansur et al., 2004; González, 2005; Porto López and Mazzanti, 2005; Di Prado et al., 2007), production processes and use (González, 2005; Mansur et al., 2004; Pedrotta, 2005), and their relationship with ideational and symbolic aspects (Bonomo, 2005; Scalise and Di Prado, 2006), among others.

This paper presents the results of macroscopic, X-Ray diffraction (XRD) and thin section analysis conducted on pigment samples from archaeological sites located in different sectors of Buenos Aires Province, Argentina. They include the Calera site, in the Northwestern portion of the Tandilia System; the Nutria Mansa archaeological locality on the plains, close to the Atlantic coast; and the Zanjón Seco archaeological locality in the interior plains of the Interserrana area (Fig. 1). The data allowed compositional and textural characterization of the samples. The provenance of the different raw materials potentially used as pigments by hunter-gatherers of the Southeastern Pampean region during the Late Holocene is also discussed.

Methodologically, two types of techniques were applied to determine the texture and composition of larger grain-size minerals (thin sections) and smaller ones (XRD) in the tested samples. Thus, a textural and compositional database of pigments from the Southeastern Pampean region was generated. Also, provenance of these raw materials was proposed, allowing a comparative approach to the different sectors under study. The use of both analytical techniques as complementary tools is a contribution to pigment provisioning studies previously conducted in the Pampas. All the information constitutes groundwork for future analysis (i.e., Raman spectroscopy, SEM-EDAX, Spectroscopy of solid infrared) that will complement the data presented in this paper.

2. Defining the concept of pigments

A brief review of archaeological and ethnographic papers from South-central Argentina (Pampas and Patagonia regions) shows

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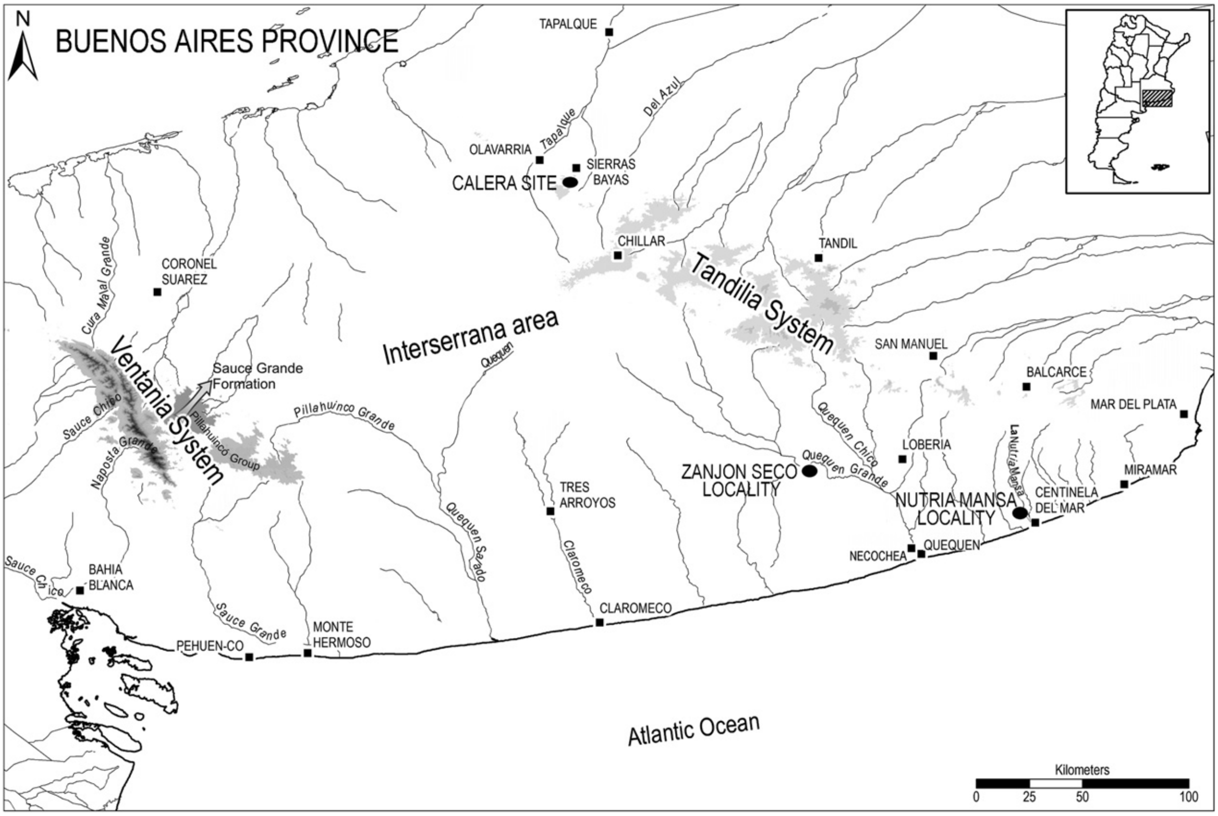

Fig. 1. Southeastern Pampean region, Buenos Aires Province. Location of the studied archaeological sites and places mentioned in the text.

that "pigment", "ocher" or "coloring substances" usually name materials of varied nature -rock fragments, non-consolidated sediments such as silts or clays, vegetables, blood, coal, and ashes- that have coloring capacity to some extent (Aguerre, 2000; Fiore, 2005; Mansur et al., 2007; Prates, 2009). In this broad meaning, pigment or coloring substances refers to colorant raw materials that could have served directly for painting, sometimes showing use-wear traces, which allow consideration as artifacts, or that were used alone or in combination with a variety of binders or additives. These latter mixtures could be manufactured artifacts, frequently referred to as *tizas* or *lápices* (chalks), *crayones* (crayons), *pastas* (cakes), and *panes* (sticks) (Boschín et al., 2002; Pedrotta, 2005; Mansur et al., 2007; Fiore et al., 2008; among others).

The coloring materials usually preserved in the archaeological contexts are rock fragments anthropically incorporated in the sites, artifacts with possible traces of use, artifacts manufactured from pigment mixtures, or powder stains in archaeological layers. Further evidence of pigment use related to ceramic production (Aguerre, 2000; Politis et al., 2001; De la Fuente et al., 2007), rock art (Aguerre, 2000; Madrid et al., 2000; Boschín et al., 2002), and mortuary practices (Moreno, 1874; Lehmann-Nistche, 1926; Martínez et al., 2006), has been found. Pigments can appear as part of the final products of these activities (i.e., *engobe* or slip, paint, temper, rock paintings, colored human bones), or as production refuses (i.e., residues on grinding stones used for their preparation) (Adams, 2002; Di Prado et al., 2007; Logan and Fratt, 1993; Mansur et al., 2007; Politis et al., 2001; Porto López and Mazzanti, 2005).

In this paper, mineral raw materials with colorant properties, termed as "mineral pigments" or "mineral colorants", are examined. They should be considered as raw materials for paint use or production if the potential use is not proposed; in which case they could be considered artifacts with possible traces of use or artifacts manufactured from pigment mixtures.

Several eighteenth and nineteenth century chronicles mention different types of coloring raw material and the colors that Patagonian and Pampean aborigines obtained from them: ocher (this term includes rock fragments as well as clays) for making red, brown or yellow paint (Bourne, [1848] 1998; Claraz, [1865–66] 1988; Musters, [1869–70] 2005; Spegazzini, 1884); sandstones for green paint (Claraz, [1865–66] 1988); a sort of kaolin, clay or gypsum for white paint, and charchoal or coal to obtain black pigment (Claraz, [1865–66] 1988; Musters, [1869–70] 2005; Spegazzini, 1884); brown ocher (Claraz, [1865–66] 1988), among others. The uses of these colorants were also varied: hide processing and/or hide painting (Prates, 2009; Claraz, [1865–66] 1988; Musters, [1869–70] 2005); corporal painting and cosmetic use (Bourne, [1848] 1998; Claraz, [1865–66] 1988; Musters, [1869–70] 2005). This information complements the archaeological record as a useful source of hypotheses about mineral pigment uses during pre-hispanic times.

3. Regional setting

The Pampean regional mineral resources base (sensu Berón, 2006) has a restricted distribution that includes the Tandilia and Ventania systems, smaller outcrops located in the plains between these two systems, and deposits of nodules available throughout some fluvial valleys and the Atlantic coast (Bayón and Flegenheimer, 2004; Barros, 2009). The Tandilia System is located in the Southeastern portion of Buenos Aires Province, and stretches 350 km NW-SE (Fig. 1). The hills are no higher than 500 m a.s.l., and are composed of an igneous-metamorphic basement and a Precambrian and Lower Paleozoic sedimentary cover, with horizontal to subhorizontal bedding (Dalla Salda et al., 2005; Poiré and Spaletti, 2005; Poiré and Gaucher, 2009). The Ventania System is located in the Southwestern portion of Buenos Aires Province (Fig. 1). It

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stretches 180 km, with its highest ranges (more than 1000 m a.s.l.) in the central section. These mountains are composed mainly of a sedimentary Paleozoic–Mesozoic cover, which primarily includes strongly folded quartzites and subordinated outcrops of the crystalline basement of the region (Massabie et al., 2005).

On the Atlantic coast there are secondary deposits of coastal pebbles (see discussion in Bonomo and Prates, 2006) that are distributed discontinuously between Mar del Plata and Quequén Salado River (Fig. 1). Between Pehuen-Có and Monte Hermoso villages there are other secondary pebble deposits, mainly composed of quartzite nodules that were transported from Ventania outcrops by the Sauce Grande River (Bayón and Zabala, 1997; see discussion in Matarrese and Poiré, 2009).

Based on the definition outlined in section 2, a broad variety of mineral resources could be considered as pigment raw material. For this reason, petrological studies are needed before regional availability could be proposed. Mineral pigments sources are scarcely mentioned in archaeological papers, namely San Manuel (Flegenheimer, 1991; Porto López and Mazzanti, 2005; Mansur et al., 2007) and Balcarce areas (Porto López and Mazzanti, 2005; Mansur et al., 2007), both located in the Tandilia System (Fig. 1).

4. Archaeological contexts

The Calera site (CAL, 36°59′0.6″S; 60°14′1.01″W) is located in the upper section of Tapalqué stream, in the center of the Sierras Bayas hills (Western sector of the Tandilia System; Fig. 1). It consists of four pits (*cubetas*) intentionally excavated, which were filled with large amounts and varieties of archaeological materials as well as allochthonous sediments. The deposit was initially formed at the beginning of the Late Holocene, ca. 3400 BP. The burial of the materials continued, intermittently, at least until ca. 1750 BP. This suggests that the site was used for similar purposes for approximately 1650 years. The mineral pigments recorded at CAL comprise 439 pieces and many spicules smaller than 1.5 cm. This site was interpreted as a deposit of ritual origin, probably produced as the result of several ceremonies performed during a large part of the Late Holocene (Barros and Messineo, 2007; Di Prado et al., 2007; for a detailed site characterization see Politis et al., 2005).

The Nutria Mansa archaeological locality (NM, 38°24′54.2″S; 58°15′50.1″W) is comprised of two surface sites (NM1sup and NM2sup) and one buried site (NM1). They are all located on the margins of the lower Nutria Mansa stream (Bonomo, 2005; Fig. 1). Three fragments of mineral pigment were found in NM2sup. The analysis of these assemblages revealed that knapping activities were performed at the NM1sup and NM2sup sites. Various manufactured items were also discarded in these sites as the product of multiple activities (Bonomo, 2005). In NM1 13 mineral pigments were recovered (Bonomo and Matarrese, in press). NM1 represents a multiple activities site that was occupied during the Late Holocene (with AMS radiocarbon dates between ca. 3100 and 2700 BP; Bonomo, 2005).

The Zanjón Seco locality (ZS, 38°10′7″S; 59°10′8″W) is located in the middle section of Quequén Grande River, in the Interserrana plains (Fig. 1). It comprises four sites, ZS1, ZS2, ZS3 and ZS4, where abundant archaeological material was recovered during surface surveys (Politis, 1984; Martínez, 1999). ZS2 and ZS3 sites were also partially excavated. ZS2 was considered as a multiple activities site (Politis, 1984; Martínez, 1999; Politis et al., 2001), whereas ZS3 was interpreted as a refuse structure (dump) (Politis, 1984) or an activity area of a greater site where multiple activities took place (Martínez, 1999). Based on the radiocarbon data of the stratigraphic contexts (ca. 3000 BP for ZS2 and ca. 1450 BP for ZS3), ZS locality was occupied at the beginning and the end of the Late Holocene (Politis et al., 2001). As the analysis of the stratigraphic and surface materials showed a significant amount of shared traits, they were considered as part of the same component (Politis, 1984; Martínez, 1999). Spicules of ocher were observed in the archaeological levels of ZS2 (Politis et al., 2001) and 18 pigments pieces were found in superficial contexts (Politis, 1984; Martínez, 1999; Matarrese, 2007).

5. Analytical methods

5.1. Macroscopic analysis

The mineral pigments were first analyzed at a macrocospic level using a stereo microscope (Nikon SMZ800, 10 to 63× magnifications). The variables considered (Aschero, in press; Adams, 2002) were: fragmentation degree, type of rock, color (Munsell Soil Color Charts, 1994), blank type, dimensions (length, width, and thickness), weight, morphology, number of faces, and possible anthropic modifications (manufacture, striations, facets, smoothed surfaces and macroscopic residues). By the study of these attributes the mineral colorant assemblages were described. All pigments from NM ($n = 16$) and ZS ($n = 18$) were subject to macroscopic analysis. In the case of CAL pigment assemblage, only the fragments possibly modified through use ($n = 29$) and those that were analyzed by XRD and petrography ($n = 6$) were included for macroscopic analysis. Samples for XRD and thin section analysis were selected considering color and rock type diversity as selection criteria. The studies presented in this paper are an extension of previous XRD and thin section analysis (Bonomo and Matarrese, in press; Di Prado et al., 2007).

5.2. XRD analysis

XRD analysis ($n = 15$) of the whole rock and clay-size fraction were made in order to characterize the main mineral composition and identify the clay minerals and their relative abundance. Samples were disaggregated and pulverized in an agate mortar following classical methods, and clay mineral separation was later obtained from distilled water suspension and piping the $<2\ \mu$ fraction following the Stokes Law (Moore and Reynolds, 1989). Three samples were prepared through the so-called glass slide method: *i*) Natural, sample air-dried at laboratory temperature; ii) Glycoled, sample exposed to the vapours of an ethylene-glycol solution for at least 24 h; and iii) Heated, sample heated to 550 °C during 2 h. A Diffractometer (Philips PW 1011 with CuKα-anode) was used at the X-Ray Laboratory of the Centro de Investigaciones Geológicas (CIG). The scanning ranges were 2θ angles of 3°–37° for whole rock samples, 2°–30° for natural clay minerals samples, 2°–26° for ethylene-glycol samples and, 3°–15° for heated samples. The mineralogical composition of each sample was considered by means of the following intervals according to its relative abundance (Poiré, 1987): very abundant (>50%), abundant (26–50%), moderate (16–25%), scarce (6–15%), very scarce (5–1%) and traces (<1%).

5.3. Thin section analysis

The microscopic analysis of the thin sections ($n = 14$) was performed with a polarizing petrological microscope (Nikon Eclipse E200), property of the CIG. A qualitative and semi-quantitative study of the mineral components of size greater than 0.03 mm was obtained and the basic optical characteristics of minerals were identified (color, texture, etc.). Thin sections (2 × 1.5 × 1 cm, approximately) were acquired by cutting with a diamond saw and then polished to 30μ (0.03 mm) thick. Although this is a semi destructive technique, it creates permanent preparations.

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6. Characterization of pigment assemblages: results of macroscopic analysis

The mineral colorants recovered at CAL comprise 439 fragments, and many spicules which were not considered for analysis. From the preliminary study, 410 fragments of raw mineral pigment and 29 pieces possibly modified through use were identified. The modifications observed included smoothed surfaces (Fig. 2c–g) and sets of striations (parallel and oblique types, and without a specific direction). The natural blanks identified on the 29 pigments possibly modified by use were: slabs ($n = 15$; Fig. 2d), nodular concretions ($n = 10$), natural angular clasts ($n = 3$; Fig. 2b) and nodules ($n = 1$; Fig. 2h). The colors varied between white ($n = 4$), light grey ($n = 1$), very pale brown ($n = 1$), yellow ($n = 3$), reddish yellow ($n = 2$), reddish brown ($n = 3$), light red ($n = 1$), pale red ($n = 1$), weak red ($n = 4$), red ($n = 8$), and dusky red ($n = 1$). The minimum and maximum weights were 2 and 73 g, respectively. The maximum lengths were grouped between the ranges: a) $> 1.5 \leq 3$ cm ($n = 12$); b) $> 3 \leq 4.5$ cm ($n = 11$); c) $> 4.5 \leq 7$ cm ($n = 6$).

Among the 410 mineral pigments from CAL, 12 fragments were selected as representative of the variability observed (with regards to natural blanks, colors and dimensions) to be analyzed by XRD (Di Prado et al., 2007). Six of these samples were studied by polarizing petrological microscope. The samples selected included four nodular concretions, one slab and one block. The colors varied from white ($n = 1$), pinkish white ($n = 2$), reddish yellow ($n = 1$), yellow ($n = 1$) and red ($n = 1$). Their minimum weight is 20 g, whereas the maximum is 703 g. The maximum lengths were grouped in: a) > 1.5 - ≤ 3 cm ($n = 1$); b) > 3 cm – ≤ 4.5 cm ($n = 2$); c) > 4.5 cm – ≤ 6 cm ($n = 2$); d) > 6 cm – ≤ 7.5 cm ($n = 1$).

The pigments from NM locality comprise ecofacts ($n = 14$) and artifacts ($n = 2$). One of the latter is a natural clast with two smoothed faces (Fig. 2r). The second artifact was probably manufactured by mixing (pigmentary mixture; Fig. 2s). Most of the ecofacts were disintegrated material (Fig. 2q). Only four fragments showed identifable blanks: natural angular clast ($n = 3$; Fig. 2p) and block ($n = 1$). The colors registered were weak red ($n = 4$), reddish brown ($n = 3$), light brownish grey ($n = 3$), yellow ($n = 4$) and yellowish red ($n = 2$). The pigments weighed between 1 and 30 g. The maximum lengths were grouped between the ranges: a) $> 1.5 \leq 3$ cm ($n = 13$); b) $> 3 \leq 4.5$ cm ($n = 2$); c) $> 4.5 \leq 9$ cm ($n = 1$). Six of NM1 samples were analyzed by XRD (Bonomo and Matarrese, in press); three of these samples were selected to conduct thin section studies.

Macroscopically, ZS pigments ($n = 18$) includes natural angular clasts ($n = 7$), slabs ($n = 4$; Fig. 2j), disintegrated materials ($n = 2$), blocks ($n = 2$; Fig. 2n) and, oval-shaped nodules ($n = 2$; Fig. 2o). The colors observed in ZS assemblage were redish brown ($n = 7$), red ($n = 6$), weak red ($n = 4$), and dusky red ($n = 1$). The ranges of the maximum lengths of the remaining pieces included: a) $> 1.5 \leq 3$ cm ($n = 5$); b) $> 3 \leq 4.5$ cm ($n = 6$); c) $> 4.5 \leq 6$ cm ($n = 1$); d) $> 6 \leq 7.5$ cm ($n = 5$). Although the maximum and minimum weight were 2 g and 2 kg, respectively, most of the fragments do not weigh more than 80 g ($n = 16$). The heaviest artifact recorded (14 × 13 × 9 cm) was manufactured on a block pigment, and was used as a netherstone (sensu Adams, 2002) (Fig. 2i). Flat surfaces (facets) abraded by rounding and smoothing was observed in this piece, as well as parallel striations. Two other pigments also have smoothed surfaces and striations (parallel and oblique types, and without a specific direction) (Fig. 2k and l). A third fragment had a thin layer deposited over one of its faces that could correspond to macroscopic residues. Finally, another sample is probably a pigment paste (Fig. 2m). Six pigments from this archaeological locality were selected for XRD and thin section analysis.

7. XRD and thin section analysis

Five groups of pigment samples with different provenance were proposed on the basis of the relative abundance and associations of the identified mineral species from the XRD analysis (Table 1) and the observed petrographic characteristics obtained from the thin sections. One sample (FCS.C.155) was not included in any group since source data was not established. The first group is formed by six samples from ZS, two from NM and one from CAL, with reddish to orange colors. On the basis of XRD results, Group 1 comprises samples with variable content of quartz, low potassium feldspar and plagioclases, abundant iron oxide, and abundant to little clay (Table 1; Fig. 3). Among the argilominerals, the presence of kaolinite and abundant pyrophyllite were detected. These clay species improve the adherence capacity to any surface (De Giusto, personal communication). This could have been another property sought in these mineral pigments, besides their coloring capacity.

In Group 1 eight thin sections were studied (Fig. 4). They included a ferric rock with chert clasts and cement (ZS2.P12), a ferric rock with chert clasts, clays and cement (ZS2.P9), ferric siltstones (ZS2.P7; FCS.C.259), a friable fine sandstone (NM1 4.15.23), a ferric laminated sandstone (ZS2.P2), a quartoze sandstone with quartz and chert (phthanite) clasts, and iron oxide cement (ZS2.P8), and a quartzose sandstone with rounded clasts (ZS185.21). In addition, in sample NM1 4.15.23 (Group 1) the presence of diatom frustules was also registered (Fig. 4). Among these, *Diploneis* sp. (aff. *smithii*), a typical species from continental lagoon environments (Vos and De Wolf, 1993), was identified (Gutiérrez Tellez, personal communication).

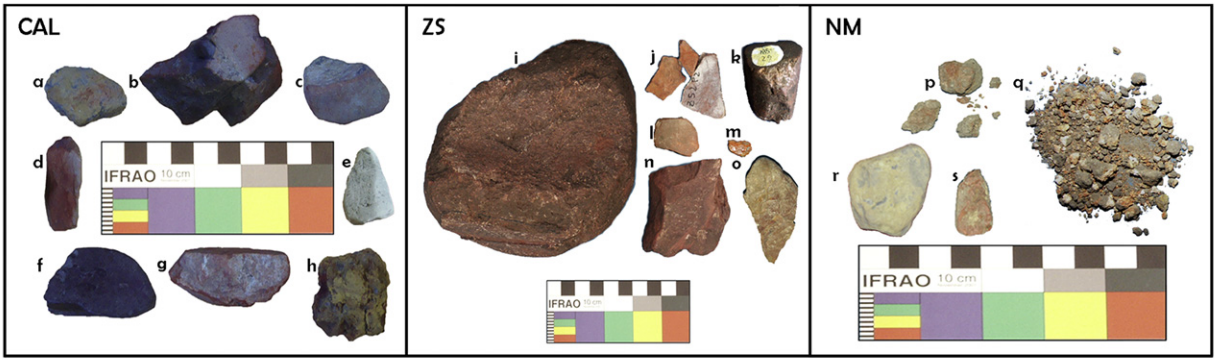

Fig. 2. Pigments from CAL, NM and ZS: *a* = nodular concretion; b, p = natural angular clasts; c–g, k-l = smoothed surfaces and striations; d, j = slabs; h, o = nodules; i = netherstone; m, s = pigmentary mixture; n = blocks; q = disintegrated material; r = natural clast with two smoothed faces. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article).

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Table 1
Results of XRD studies.

Locality	Group	Sample	Whole Rock							Clay Fraction					
			Qz	FK	Pl	Arc	Ca	OxFe	D	I-M	IS	Sm	Chl	K	Py
CAL	1	FCS.C.259	s	vs	vs	va	–	m	–	m	s	–	–	s	va
ZS	1	ZS2.P2	va	tr	vs	s	–	a	–	va	–	–	vs	s	–
ZS	1	ZS2.P7	vs	vs	vs	a	–	va	–	a	–	–	–	va	a
ZS	1	ZS2.P8	m	s	s	s	–	a	–	va	–	–	–	m	s
ZS	1	ZS2.P9	vs	vs	vs	a	vs	a	vs	va	–	–	–	s	s
ZS	1	ZS2.P12	vs	vs	s	a	vs	va	–	m	–	–	–	vs	s
ZS	1	ZS185.21	tr	tr	tr	a	–	va	–	vs	vs	tr	–	a	a
NM	1	NM1 4.15.23	s	–	–	a	–	a	–	m	–	–	m	a	a
NM	1	NM1 4.15.25 (*)	a	–	–	s	–	a	–	a	–	–	–	a	–
CAL	2	FCS.C.110	vs	–	–	vs	–	va	–	m	s	m	m	–	–
CAL	2	FCS.C.114	m	–	–	tr	–	a	–	–	–	–	–	–	–
NM	3	NM1 5.23.G1	a	m	–	s	s	s	–	a	vs	a	–	–	–
CAL	4	FCS.C.167	s	–	–	vs	va	–	–	a	m	s	s	–	–
CAL	5	FCS.C.103	m	–	–	vs	va	–	–	va	s	–	–	–	–
CAL	npd	FCS.C.155	s	–	–	tr	va	–	–	va	s	tr	tr	–	–

(*) = sample without thin section analysis; Qz = quartz; FK = potassium feldspar; Pl = plagioclase; Arc = clays; Ca = calcite; Ox Fe = iron oxide; D = dolomite; I = illite; IM = illite-mica; IS = illite-smectite interlayer; Sm = smectite; Chl = chlorite; K = kaolinite; Py = pyrophyllite; va = very abundant; a = abundant; m = moderate; s = scarce; vs = very scarce; tr = traces; npd = no provenance data available.

In relation to the provenance source for Group 1, the presence, proportion and association of minerals has been registered in the pyrophyllitic levels of Barker and San Manuel area, in the Tandilia System (Manassero, 1986; Poiré and Spaletti, 2005; Poiré and Gaucher, 2009 and references cited therein; Fig. 5). The lower section of Las Águilas Formation, as described in the Diamante stratigraphic sequence (Poiré and Spaletti, 2005) in Barker, is represented by a phthanite level (5 m thick approximately) with strongly silicified oolitic calcareous clasts. On top of the Las Águilas Formation, layers of ferric pelites (5–9 m thick) are displayed. These pelites have an iron oxide (Fe_2O_3) content of 30% on average, and iron sectors (up to 7 m thick) with up to 70% iron oxide. Their essential composition is quartz, chert, goethite, hematite, kaolinite, pyrophyllite and illite. The pyrophyllitic pelites levels of the stratigraphic sequence of the Barker hills are integrated by “a breccia of basal phthanite with pelitic matrix and purple red pyrophyllitic pelites silky and greasy to the touch, which towards the top of the sequence becomes coarser with interlayers of orthoquartzite banks” (Manassero, 1986, pp. 378).

Las Águilas Formation purple red pelites are characterized by abundant pyrophyllite and low proportions of illite and kaolinite. Feldspar and hematite are the most common impurities of the clay fraction. Quantitative average estimations of the mineralogic composition of these pelites were obtained by XRD and chemical analysis namely: kaolinite = 30% (abundant), illite = 10% (scarce), pyrophyllite = 40% (abundant), quartz = 5% (very scarce), hematite = 15% (scarce) (Iñiguez and Zalba, 1974). The general composition, the colors mainly represented (red and very dusky red), and the high concentration of iron oxides of the pelites of Las Águilas Formation coincides with Group 1 pigment samples (Table 1).

Group 2 is formed by two samples from CAL. They are reddish and yellowish pigments with abundant hematite and goethite (Table 1; Fig. 3). Through the thin section study they were defined as quartzose sandstone with chert and iron cement, and ferric concretions (FCS.C.110; Fig. 4) and sandstone with chert and iron (FCS.C.114; Fig. 4). The presence of chert cements (amorphous silica) as well as the abundance of hematite and goethite supports an origin from the Las Águilas Formation, in the Barker area. Nevertheless, the presence of ferruginous cement and the absence of pyrophyllite suggest the Cerro Largo Formation, in Sierras Bayas hills, as its provenance source (Fig. 5). Sandstones with similar characteristics have also been reported in the lower section of the quartzite's levels of the Cerro Largo Formation. These levels are abundant in amorphous siliceous cement and iron oxides (Gómez Peral, 2008).

Group 3 includes a yellow pigment of NM1, where XRD analysis showed abundant quartz, moderate potassium feldspar, and a limited content of calcite, iron oxide and clays. In this last fraction, abundant illite-mica and smectite and very scarce content of interbedded illite-smectite was identified (Table 1; Fig. 3). According to thin section analysis, it is a siltstone with iron oxide in different degrees of oxidation, cementing very fine sediments (chert). Plagioclases (oligoclase and albite) were also identified through macles observation (Fig. 4). These characteristics match the descriptions of the abundant clasts of green quartzites (or plagioclasic sandstones, according to the classification in González Bonorino and Teruggi, 1952) included in the diamictites of the paraconglomerate of Sauce Grande Formation (Pillahuincó Group of Ventania System; Andreis, 1965; Fig. 1). These green quartzites can present superficial limonitic or hematitic pigmentation that gives them yellowish, brown and pink tonalities (Andreis, 1965, pp. 30). Therefore, the sample NM1 5.23.G1 could have been extrated from a green quartzite clast.

The main outcrops of the Sauce Grande Formation are located in the western flank of Pillahuincó and Tunas hills, on the right riverbank of the Sauce Grande River and on some streams banks of the area (Andreis, 1965). After the weathering of the paraconglomerate, its phenoclasts are transported by different river basins of the region. Among these, the Sauce Grande River is the most important Atlantic drainage network from the Pillahuincó hills (Furque, 1973). Coarse Quaternary conglomerates with clasts from older psephitic deposits (from Ventana and Pillahuincó hills) are observed in its valley (Furque, 1973). Thus, green quartzite clasts from Sauce Grande Formation could have also been available as secondary deposits over 220 km, from the hill sector to its mouth in the Atlantic coast.

Group 4 includes a white sample from CAL, where XRD analysis showed more than 60% of calcite, with quartz and micas (Table 1; Fig. 3). It was defined by the thin section study as a fine limestone with primary laminar structure (Fig. 4), showing the typical characteristics of the limestones from Loma Negra Formation (located in Sierras Bayas; Fig. 5). This unit is constituted by an association of carbonate facies (40 m thick) of basal reddish micritic limestones with ripples, ripples lamination, cross-lamination, and trough cross-bedding (Poiré and Spaletti, 2005; Poiré and Gaucher, 2009). The red limestone (8 m thick) is mainly brown-reddish mudstone (greenish or sometimes grey-yellowish), with a micritic grain-size and good degree of consolidation. The microscopic analysis showed a micritic to esparitic calcite occasionally crossed by veins filled with esparitic

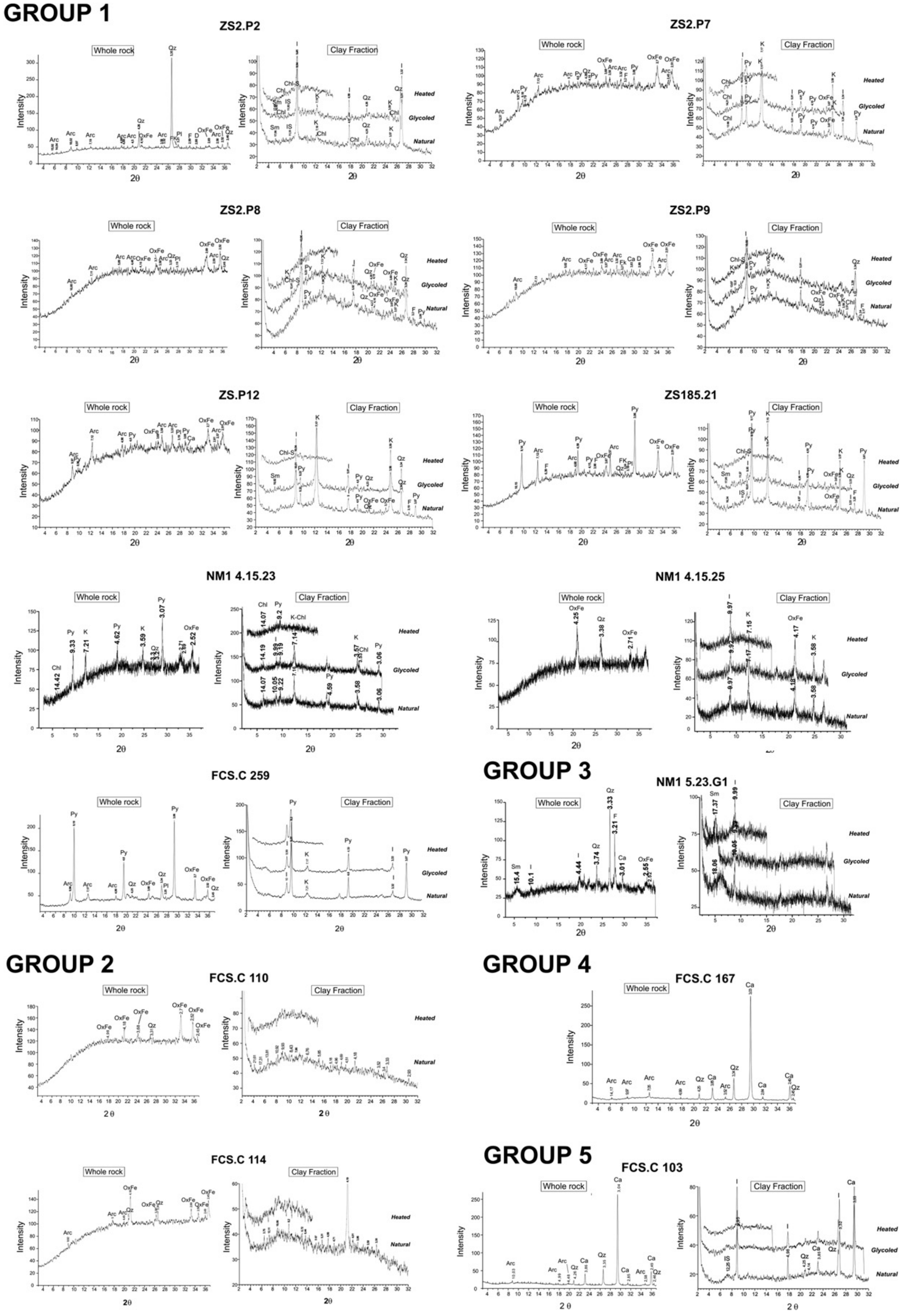

Fig. 3. Difractograms of CAL, NM and ZS pigments.

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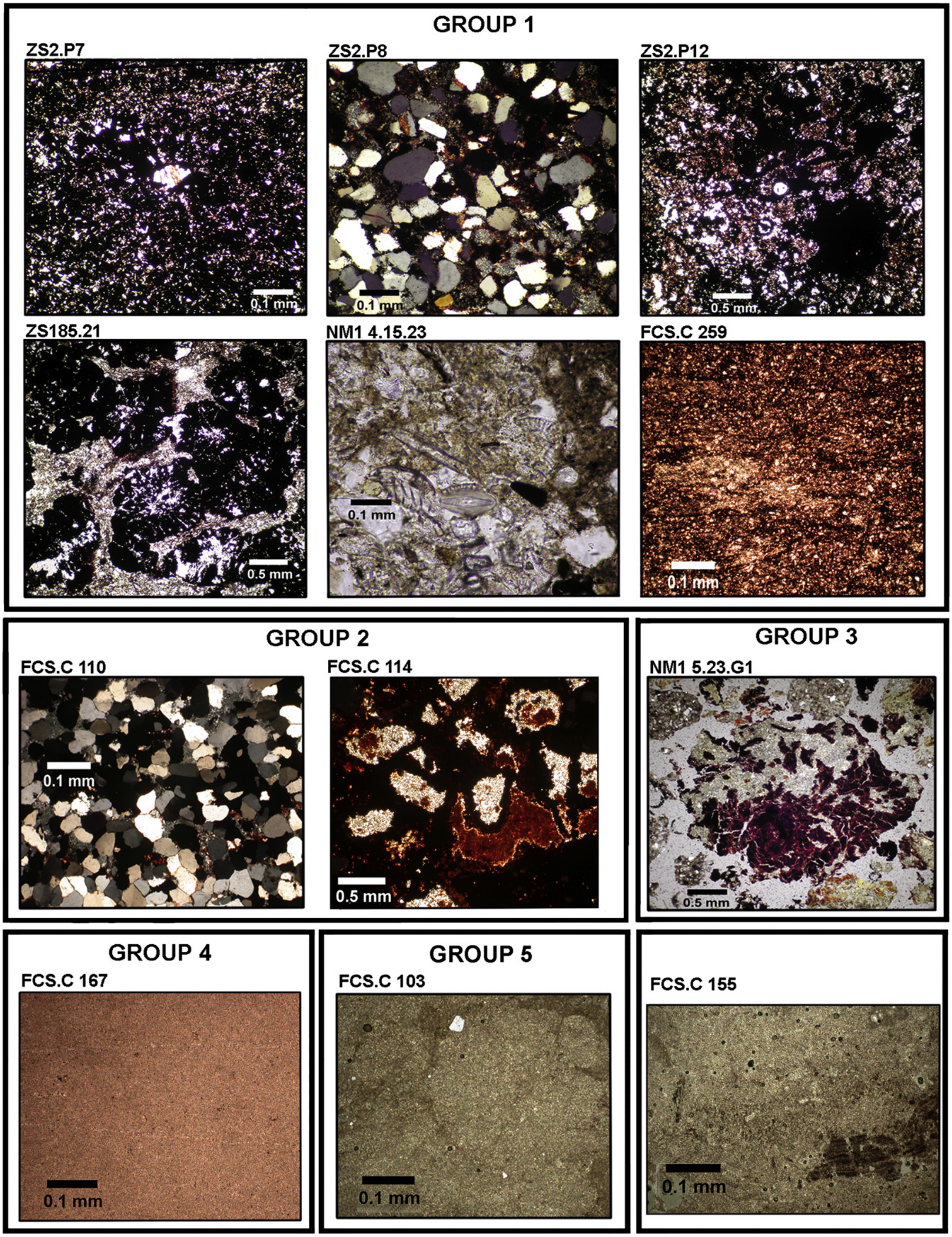

Fig. 4. Thin section photographs of CAL, NM and ZS pigments.

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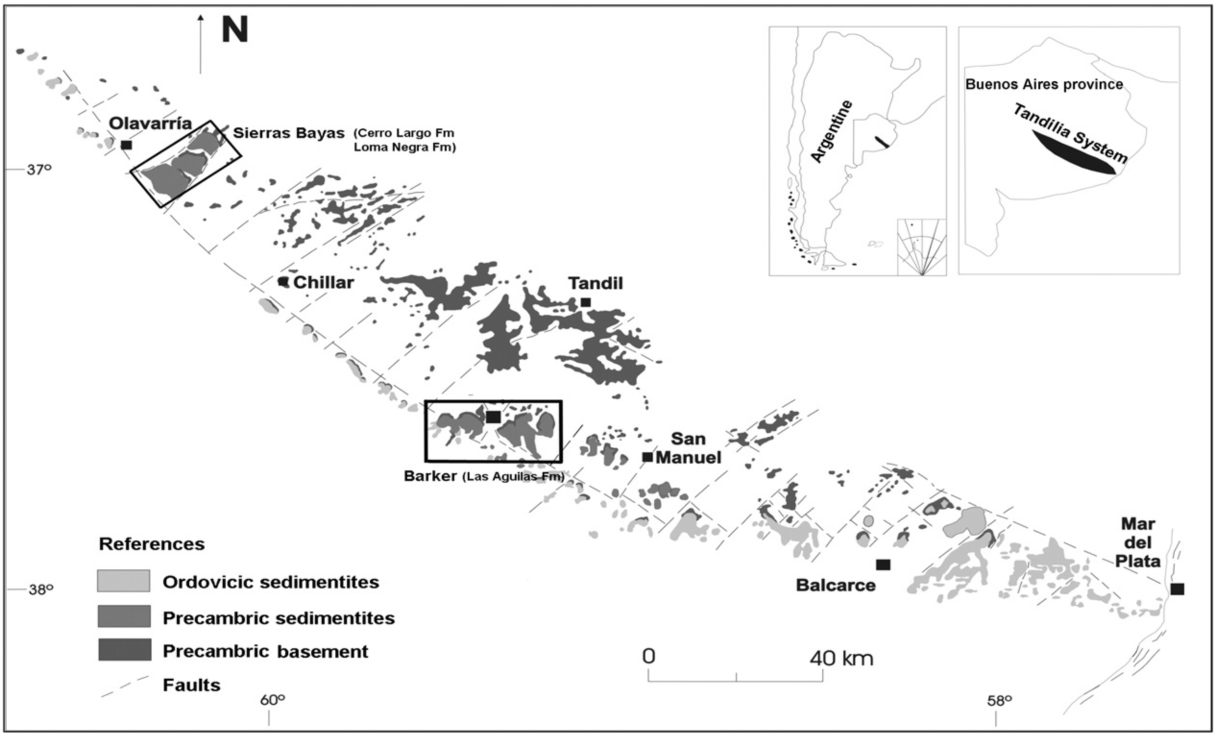

Fig. 5. Geologic map of Tandilia System.

calcite. The interstitial argillaceous material is essentially illitic, with a very scarce presence of amorphous silica. A typical reddish limestone shows an average of 74–76% $CaCO_3$, accompanied by 3–3.5% Al_2O_3 and 0.5–3.5% Fe_2O_3. The value of carbonates varies between 72 and 85%, whereas the argillaceous material is 12–19% and phthanite on the order of 3% (Poiré et al., 2005).

Group 5 (Figs. 3 and 4) includes a pinkish white pigment from CAL that has 60% calcite and abundant quartz sand grains. It also has lumpy structures and mica. These characteristics are observed in the calcitic deposits (commonly called *toscas*) from B-calcitic Horizons of the Neogene or Quaternary Pampean paleosoils, wich are widespread in southeast Buenos Aires Province (Giai and Visconti, 2002) and are especially common in the Sierras Bayas hills. The thin section studies conducted on *toscas* located in the Balcarce area (Fig. 1) show calcitic nodules with clay coverings, feldspar microclasts and quartz (Buscchiazzo, 1988). Some mineral pigments from CAL with possible use-wear traits are macroscopically similar to sample FCS.C.103 (i.e., Fig. 2e). This, and the presence of a sherd with white paint, support its inclusion within the mineral colorant repertoire of CAL. Nevertheless, the abundance of *toscas* in the Pampean region soils indicates that some of these rock fragments could have been incorporated to the site by natural agents, instead of anthropic ones.

FCS.C.155 is a pinkish white pigment without primary natural rock texture (Table 1; Fig. 4) and its source could not be established definitively. It is composed of lithoclasts, where the spacing between pores is stained with iron oxide. Angular quartz and calcite are also present. This last component is typically observed in the Pampean loess that can be included in the Quaternary *toscas*. Therefore, it is proposed that it is a mixture manufactured with mineral species available in the Sierras Bayas hills.

8. Discussion

The compositional analysis presented in this paper shows the variablility of colorant raw materials utilized in Pampean archaeological sites (CAL, ZS and NM). The geological provenance was identified for most of the samples. The studied pigments were rock fragments for which colorant properties were inferred, due in most cases to the presence of iron oxides and hydroxides, which are the result of rock alteration primarily during diagenesis and weathering (Tucker, 2001). The oxides and hydroxides are unstable minerals, changing between mineral types due to the variable oxide-reduction conditions. Goethite, hematite and limonite are the most common coloring agents, as noted on most of the studied archaeological pigments. However, detecting the presence and types of these oxides in the samples was not enough to propose potential provisioning sources, but required more complete petrologic information.

In two cases, the colorant property was given by the presence of minerals such as calcite, widening the range of mineral resources that could be potentially used as white pigment. In sample FCS.C.167 (Group 4) this carbonate mineral comes from the marine limestones of the Precambrian sea of the region. The origin of the calcite in sample FCS.C.103 (Group 5) is the B-calcitic Horizons of the Neogene or Quaternary Pampean paleosoils (Buscchiazzo, 1988; Giai and Visconti, 2002). In this case, the presence of iron oxide in a very low proportion gave a pinkish coloration.

The description of macroscopic features has relevance since this is the observation level applied by pre-hispanic groups for selecting, provisioning and use of these coloring raw materials. At the macroscopic level, diverse rock supports (blanks) were observed. They included mainly slabs, nodular concretions, natural angular clasts and, to a lesser extent, blocks. A few nodules were also found, possibly obtained from secondary deposits. Disintegrated material was frequent in the studied assemblages. This is in part because of the low hardness observed in many of the samples. It could be due to the high content of a soft mineral such as calcite or it could be the product of matrix alteration and/or weakening of grain cementation. The hardness of the coloring raw material might have been an important factor regarding how usable or extractable the pigment fraction was. Although related to the final application of the mineral pigments, some of these mineral resources could have allowed more variants than others. For example, some might have required scraping the section(s) where the coloring oxides or minerals are concentrated, as could be the case for slabs and nodules. Most of the remaining cases could have been ground or used as chalks (the striations and facets observed in some pieces could have been part of this kind of usage). Finally, the netherstone from ZS locality could have been used as a ground stone to process pigments and also be a part of the colorant raw material processed.

Some of the combinations of pigment production described in chronicles and ethnographic papers were mineral pigment rock and water (Musters, [1869–70] 2005, pp. 196); mineral pigment rock, water and crushed roots (Aguerre, 2000, pp. 99–100); powdered mineral colorant and fat or melted bone marrow (Claraz, [1865–66] 1988, pp. 86, 130; Musters, [1869–70] 2005, pp. 91, 196); mineral colorant, fat and vegetal rubber (Spegazzini, 1884, pp. 234); and mineral colorant, fat and blood (Bourne, [1848] 1998, pp. 44). The presence of diatoms in the sample from NM1 could be related to similar procedures. The diatom group identified matches the ones determined on the site soil samples (Bonomo, 2005). One possibility is that this introduction was the product of the preparation of a pigmentary mixture, which involved, among other ingredients, sediment and water. In the same way, it is also possible that the modification of the natural texture (presence of lithoclasts) on one CAL sample (FCS.C.155) could have been part of intentional aggregates for making pigmentary mixtures. The fatty acid presence determined by infrared spectroscopy and gas chromatography-mass spectrometric analysis on another two CAL's samples (Di Prado et al., 2007), could be also due to similar manufacturing procedures.

From the data presented in this paper, it can be proposed that pigment raw materials are present in a variety of supports and frequencies within the regional mineral resources base:

Tandilia System: i) Quartzitic/illitic red ocher from the lower section of the psamopelites of the Cerro Largo Formation, and from the upper dolomite levels of the Villa Mónica Formation (see Poiré and Iñiguez, 1984), both in the Sierras Bayas area; ii) Pyrophyllitic red ocher (with the pyrophyllite-kaolinite-illite argillomineral association) from the lower section of the Las Águilas Formation in the Barker area; and iii) Limestone from the Loma Negra Formation in the Sierras Bayas and Barker areas;

Ventania System: iv) Yellow limonite from the green plagioclase sandstones of the diamictites of Sauce Grande Formation (Pillahuncó Group).

Finally, despite the abundance of *toscas*, they do not have an enclosed distribution in the Pampean Region; they crop out in the Sierras Bayas area. Regarding the potential sources of the pigment raw materials, analysis showed that 13 of the 15 pigment samples originally came from different sectors of the Tandilia System. It is proposed that nine (Group 1) were obtained from the pyrophyllitic levels of the Las Águilas Formation in the Barker area (Fig. 5), and one from the Loma Negra Formation in Sierras Bayas hills (Group 4; Fig. 5). Group 2 samples have two possible places of provenance: the Las Aguilas Formation in Barker or the Cerro Largo Formation in Sierras Bayas hills (Fig. 5). Finally, one sample (Group 5) could be a *tosca* fragment from B-calcitic horizons of the Neogene or Quaternary Pampean paleosoils.

In the Southeastern Pampean region, other researchers (Madrid et al., 2000; Porto López and Mazzanti, 2005) have also emphasized the Tandilia System as recurrently visited by Pampean pre-hispanic groups for pigment provisioning, among other lithic raw materials. There are ethnohistorical references that mention Tandilia (also called *Sierras de la Tinta* [Sierras of the Ink]) as a place for pigment procurement (Claraz, [1865–66] 1988; Prates, 2009). Similarly, by the end of the nineteenth century Hautal reports the *Mina de la Pintura* (Paint Mine) hill as one of the first red ocher quarry exploited (Madrid et al., 2000). This outcrop was later mentioned by Nágera (1940) and it would have been located in an area close to the current limestone quarry of Loma Negra (near Olavarría city).

Within Tandilia, other places could have been used for pigment exploitation. In this sense, Porto López and Mazzanti (2005) have studied the composition of pigment samples extracted at different modern quarries near San Manuel (Southwestern Tandilia; Fig. 1), as well as archaeological samples from sites located within a 60 km radius from the quarries. By XRD and fluorescent X-ray analysis they were able to propose San Manuel as the provisioning source of a yellow pigment from the El Abra site, and the El Volcán quarry as the origin of a white sample from the same site. The whole rock fraction X-Ray data presented by these authors is not coincident with any of the samples studied here. In the archaeological quarry site of La Liebre (Reconquista hill, San Manuel area; Fig. 1), limonites were found among other remains (Flegenheimer, 1991). The plagioclases identified through macles observation in the NM limonite sample are absent in those from San Manuel, and therefore it is discarded as a provisioning source.

The supply of lithic raw materials from Ventania System has been proposed for different archaeological contexts of the Interserrana area in relation to the production of lithic knapped artifacts and, recently, for grinding tools (Bayón et al., 2006; Matarrese and Poiré, 2009). Using the data generated in this paper, the procurement of mineral pigments (at least from the eastern orographic group of this system) could be added to this framework.

From the above information, distances from which pigments were transported to ZS, NM and CAL are now discussed. Different approaches have been proposed regarding potential provisioning circuits on hunter-gatherer groups (see Bayón and Flegenheimer, 2004 and references cited therein). Meltzer's proporsal, as adapted by Bayón and Flegenheimer (2004) for the analysis of the production of lithic materials in Pampean archaeological contexts, is here explored. These authors consider raw materials as *immediately available* when their provenance source is located within a radius of 10 km from the sites. Beyond this distance the provisioning will be considered *local* when the exploited resources were transported less than 60 km. *Medium range* and *long distance* raw materials will be those available at 60–100 km, and more than 100 km from the sites, respectively. Three mobility circuits can also be delimited in which the lithic resources are involved: 1) an immediate circuit that would not exceed 5 km around the localities or places; 2) a regional circuit where the search for resources required a combination of planned or logistic incursions, and; 3) an extraregional circuit including diverse ecological zones and involving different social, ethnic and/or kin groups (Berón, 2006).

In the studied archaeological contexts, the presence of mineral colorant from Barker would indicate a medium range supplying to ZS (located approximately 68 km from Barker), and a long distance provisioning to CAL and NM (at 112 km and 139 km from Barker area, respectively). On the other hand, due the location of CAL in the Sierras Bayas hills, supplying carbonate rocks would have involved the exploitation of an immediately available resource as close as 5 km. The presence in NM of mineral pigments from the Sauce Grande Formation implies long distance supply, whether it occurred in the outcrop area or at the secondary deposits in the Sauce Grande River valley. This is because the nearest outcrops are about 315 km from the site. Distances from different sections of the Sauce Grande River and NM site varied between 259 and 297 km. These provisioning distances suggest that the ZS and NM and part of CAL assemblages of pigment raw materials were probably obtained from prehispanic Pampean groups regional mobility circuits. CAL's carbonate rocks are an exception, as supply might have taken place through immediate procurement circuits.

9. Final remarks

From a methodological point of view, the level of specificity achieved in the present study in relation to the provenance of mineral colorant raw materials of the studied Pampean archaeological sites could have only been obtained through the complementary information from XRD and thin section analysis. Components of these lithics resources that confer coloring capacity (oxides and calcite) are not sufficient to identify possible procurement places. This is why it is necessary to consider the different minerals associated in a sample, and their relative proportions and textural characteristics. Future sampling on the geologic units proposed as potential pigment source will allow a better adjustment of the provenance data here obtained. Furthermore, chemical studies (identification of chemical elements and their proportions) could be a complementary approach, especially for the cases in which the mineralogic composition was ambiguous.

On the basis of the recovered information, conclusions are outlined. CAL's pigments stand out within the analyzed archaeological assemblages, with a much greater amount of pigments in comparison with NM and ZS (see site functionality below). The size, weight and morphology of some of the pieces make them difficult to manipulate; these mineral pigments do not show any macroscopic alteration of their surfaces or extraction signs. Other mineral colorants from CAL are small, easier to manipulate and could possibly have macroscopic use-wear traits. Independent evidences of pigment processing and use in CAL are the macroscopic red residues observed on the surface of a fragmented grinding artifact, as well as pottery painted red, yellow, and white.

Three of the six samples from CAL can be considered as raw materials immediately available, possibly collected through an immediate procurement circuit. One sample was obtained from a long distance and, therefore, within a regional provisioning circuit of mineral resources that would have probably required planned or logistic incursions, or a combination of both. In addition, the results of this study are coincident with those obtained by Barros and Messineo (2007) at CAL regarding raw materials for lithic artifacts. A quartzite with good knapping quality was identified. This rock does not crop out in the Sierras Bayas hills, but in the Barker area (Barros and Messineo, 2007). This makes the Barker area a prime source for the exploitation of mineral resources.

CAL was interpreted as a ritual site, formed by the intentional burial of various cultural materials by Pampean hunter-gatherers during diachronic ceremonial events over 1700 years. During these events, trading of goods, people, prestige objects, information, consumption and the redistribution of resources could have taken place. The materials could have been buried as offertory aims and/or could have been trash generated in ceremonies (Politis et al., 2005). NM and ZS archaeological contexts represent Pampean group occupations where several activities occurred related to subsistence, and the production and use of lithic tools, pottery, and pigments. The NM pigment set includes mainly brittle and small fragments. Greater size diversity was observed in ZS, where different natural supports were identified. Thus, the mineral colorant recorded in these Pampean localities could be potential pigment raw material or the remains of pigment processing and/or paint activities. In ZS, the larger and heavier pieces could have been strategically stored for future returns to the locality. Similar strategies have been proposed in relation to large quartzite cores used under its potential, and the presence of large grinding assemblages discarded at ZS and NM (Martínez, 1999; Matarrese, 2007; Matarrese and Poiré, 2009). In addition, it is possible that different ways of processing and use of pigments were developed at this locality (for example, the use of the netherstone ZS.185.21). Regarding procurement transport of pigment raw materials, in ZS and NM they were obtained during regional circuits of provisioning.

The studied archaeological contexts correspond chronologically to the Late Holocene. For this period, it has been proposed that the Pampean hunter-gatherers had diversified their exchange networks and extended their territorial limits (Martínez, 1999; Politis and Madrid, 2001). Significant technological innovations include the bow and arrow, pottery, and rock art in rock shelters. The use of coloring raw materials is placed within a regional context. Exchange routes or logistic trips may have been employed to obtaining varied resources where the mineral pigments were procured. In the case of CAL, given the proposed functionality of the site, part of the transported materials through long distances could have come from people gathered at this place, and not necessarily from procurement trips.

The procurement of mineral pigment raw material was part of the provisioning strategies of the Pampean hunter-gatherers, along with other mineral resources involved in the production of lithic technologies (i.e., knapped and ground stone tools). In some of the Southern Pampean sites, evidence of paint production and use were found, such as colored artifacts, and tools used to process pigment and/or for making paint. These studies are a contribution to describing compositional variability of pigment raw materials, and the understanding of their exploitation by pre-hispanic Pampean groups.

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