Mineralogy and chemical composition of lithic artefacts for characterization of raw materials and provenance in archaeological sites of Salobo, Carajás, Pará, Brazil

Mineralogia e composição química de artefatos líticos para caracterização de material e de proveniência em sítios arqueológicos do Salobo, Carajás, Pará, Brasil

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Abstract: The Carajás Mineral Province is home to several archaeological sites, each with numerous stone artefacts elaborated in diferente materials. We studied the morphology, mineralogy, and chemistry of lithic artefacts recovered at the archaeological sites in the impacted area of the Solobo copper and gold mine in the Carajás Mineral Province. The results obtained from XRD, XRF and SEM/EDS analyses show that the starting material used was a semi-hard kaolin (semi-flint) that consisted of kaolinite, cryptocrystalline quartz, florencite, sericite, and hematite. This suggests that the same source of raw materials was involved and that this possibly reflected preference for this material. Additionally, the hardness of this raw material probably allowed the artefacts to be elaborated with ease. The mineralogical and chemical results, combined with characteristics of the artefacts' morphology and texture, indicate a relation among the archaeological sites. Similar material to that used in the production of the Solobo artefacts was found in the Alto Bonito amethyst mines that are located 40 km to the north. This site is therefore considered as the source of the raw material used for these artefacts. Finally, this indication is supported by the association of the abundant rock crystal chips and amethyst with the semi-hard kaolin present at the Alto Bonito mine.

Keywords: Beads. Pendants. Kaolin. Kaolinite. Florencite.

Resumo: A Província Mineral de Carajás abriga vários sítios arqueológicos com inúmeros artefatos líticos elaborados em diversos materiais. Estudamos a morfologia, a mineralogia e a química de artefatos líticos resgatados em sítios arqueológicos na área de impacto da mina de cobre/ouro do Salobo, na Província de Carajás. Os resultados obtidos por DRX, FRX e MEV/EDS demostram que a matéria-prima empregada na confecção dos artefatos líticos foi um caulim semiduro, constituído de caulinita, quartzo criptocristalino, florencita, sericita e hematita. Isso sugere que os artefatos tiveram uma mesma fonte de matéria-prima e que possivelmente havia preferência por esta. Adicionalmente, a dureza dessa matéria-prima provavelmente permitiu que os artefatos fossem mais facilmente elaborados. Os resultados mineralógicos e químicos, combinados com as características da morfologia dos artefatos, indicam relações intrassítios arqueológicos. Material equivalente ao utilizado na confecção dos líticos foi encontrado nas minas de ametista de Alto Bonito, a 40 km dos sítios investigados. Este local foi, então, considerado como área-fonte da matéria-prima empregada para a confecção dos artefatos. Finalmente, esta indicação é reforçada pela associação com lascas de quartzo hialino e ametista, também abundantes e associadas ao caulim semiduro presente nas minas de Alto Bonito.

Palavras-chave: Contas. Pingentes. Caulim. Calinita. Florencita.

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INTRODUCTION

Lithic artefacts are objects produced from fragments of rocks or minerals, generally of high hardness, and constitute archaeological vestiges of great durability. Consequently, these are used as important proxies in research on the identification of prehistoric human groups in order to discover production technology, provenance, and consequently human displacement (Prous, 1992). Lithic artefacts are found in many regions throughout the world, including the Amazon where they can be found in profusion. The Carajás region in southeastern Pará, an important mineral province, is one such region with numerous archaeological sites where prehistoric populations occupied both caves and the banks of rivers and streams (Caldarelli *et al*., 2005; Kipnis *et al*., 2007; Lopes *et al*., 1993; Silveira *et al*., 2008, 2009). Virtually all the predominant material culture sites contain lithic remains chipped from quartz (amethyst, citrine, and hyaline) and opal that, according to Magalhães (2006), were extracted from veins found in the nearby lowlands or plateaus which include abundant quartz. In sites located in Serra Norte, Serra Sul, and Andorinhas in Carajás, recovered lithic materials produced from rock crystal, citrine, amethyst, quartzite, and silex were used as scrapers, sharpeners, awls, chisels, arrowheads, and decorations (Magalhães, 2006; Lopes *et al*., 1993). Expressive amounts lithic materials were produced in the Amazon from quartz, one of the minerals commonly found in the Carajás region. On the other hand, lithic material collected at the archaeological sites in the area of the Salobo Project were produced mainly in semi-hard kaolin (semi-flint) material of restricted occurrence. Although there are a significant number of publications on archaeological objects found in Carajás (Lopes *et al*., 1993; Magalhães, 2006; Bueno & Pereira, 2007; Silveira *et al*., 2008, 2009; Rodet *et al*., 2014a, 2014b), mineralogical and chemical techniques have not been applied. In this case, these techniques constitute important complementary tools for the identification of the origin of lithic materials, and can thus increase our knowledge of the cultural and occupational aspects of lithic artefacts. From this perspective, this study characterizes the morphology, mineralogy, and chemical composition of lithic artefacts recovered at archaeological sites in the Salobo area of Carajás in order to identify the source of raw materials and their provenance and provide insights into the lithic industry of the investigated artefacts.

STUDY AREA

The studied lithic artefacts are from archaeological sites located in Carajás (Figure 1) in the Tapirapé-Aquiri National Forest, Pará, Brazil. Geologically, the study area is located in the Carajás Mineral Province, known for Fe, Mn, Au, Cu, and Ni deposits. This is constituted of different lithostratigraphic units, most of which were developed and stabilized during the Archaean. DOCEGEO (1988) divided the Carajás Mineral Province into three units (Figure 1A) formed by the Xingu Complex, Itacaíunas Supergroup, and Proterozoic intrusive granites. The Salobo archaeological sites are included in the Itacaíunas Supergroup which is formed for the following groups: Salobo Pojuca, Grão Pará, Igarapé Bahia and Buritirama. The Itacaíunas supergroup is characterized by different lithotypes consisting of metavulcan-sedimentary rocks, felsic metavulcanic to mafic and iron formations. The area of the archaeological sites was geographically subdivided in three sub-basins: Igarapé Salobo, Igarapé Mirim, and Rio Cinzento, but only lithic artefacts from the first two were investigated. These are from the archaeological sites: Alex (SA), Bitoca I (SB), Bitoca II (SBII), Pau Preto (SPP), and Mirim (SM) (Figure 1B). These sites were located mainly on stream banks (Silveira *et al*., 2016).

MATERIALS AND METHODS

The lithic artefacts used were collected by *Museu Paraense Emílio Goeldi* (MPEG) researchers at archaeological sites of the Salobo Project, where copper ores are mined. For this investigation, we selected 30 pieces, including beads (10), adornments (6), pendants (2), and chips (9), among others, found in different archaeological sites, all from the collection of the Mario Ferreira Simões Technical Reserve of the MPEG (Table 1).

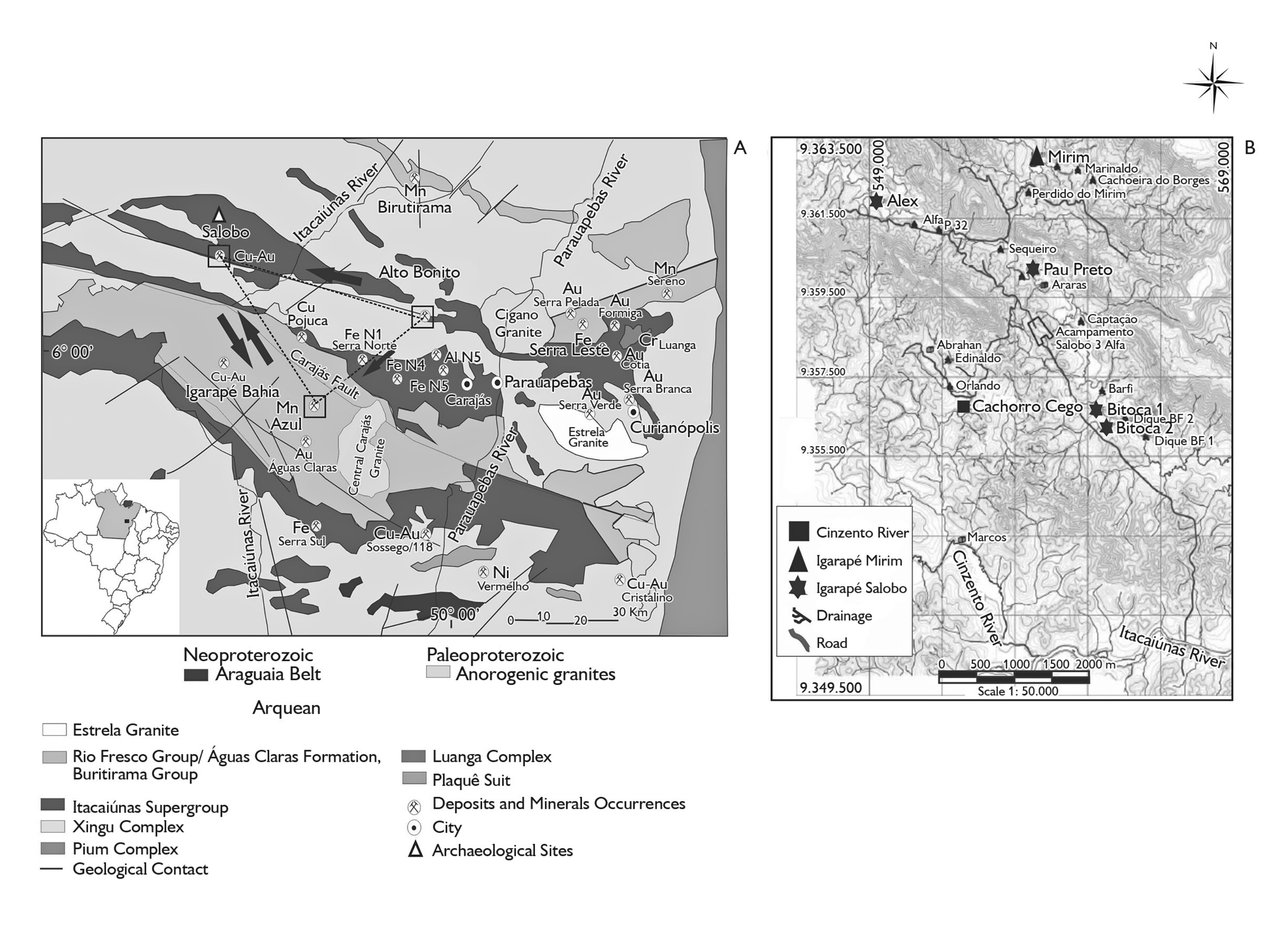

Figure 1. A) Simplified geological map of the Carajás Mineral Province, modified after Pinheiro & Holdsworth (1997); B) map showing the location of archeological sites basins (adapted from the map of Silveira *et al*., 2006).

Table 1. Frequency of anthropogenic lithic material studied, according to shape and archaeological site in the Itacaiúnas River network.

River networks	Archaeological site	Beads	Pendants	Adornments	Cores	Chips	Awls	Number of pieces
Igarapé Mirim	Mirim	1	-	-	-	-	1	2
Igarapé Salobo	Alex	1	1	1	1	-	-	4
	Pau Preto	4	-	1	-	1	-	6
	Bitoca I	4	1	3	1	6	-	15
	Bitoca II	-	-	1	-	-	-	1
Rio Cinzento	Cachorro Cego	-	-	-	-	2	-	2

The pieces are described in terms of shape, colour, size, chipping, grinding, and hole position when present. The classification of beads, pendants, and preforms was based on Rodet *et al*. (2014a, 2014b). Beads were round or cylindrical lithic units with a single central or cross hole; the various forms of pendants were mainly elongated with eccentric drilling (Barge, 1982). The preforms showed some signs of transformation (chipping, polishing, stretch marks etc.), indicating the production of this type of adornment.

Mineralogical determination was performed on all pieces. Then, 19 pieces were selected (beads, pendants, and most representative preforms) for chemical analysis. However, because these are highly relevant archaeological pieces, the material sampling was minimal, thus avoiding damage to their parts. In X-ray diffraction (XRD) they were directly held for analysis or micro-samples of approximately 10 mg of each piece were removed with the aid of a micro-electric drill with diamond crone (Dremel). The inner walls of holes and representative points of the samples received no prior treatment. Mineralogical and chemical analysis were supplemented by images and semi-quantitative chemical analysis from scanning electron microscope/energy-dispersive X-ray spectroscopy (SEM/EDS), employing LEO-1430 equipment under conditions of secondary electrons, a beam current of 90 $\mu\acute{Å}$ electrons, constant acceleration voltage of 10 KV, and a working distance of 15 mm. For this, fragments of the samples were previously metallized with gold. We used an X-ray diffractometer and X-ray fluorescence (XRF) (PANalytical X'PERT PRO MPD; PW 3040/60) with a PW 3050 goniometer (θ/θ) and a ceramic X-ray tube Cu anode ($K\alpha1$ = 1.540598 Å; model PW3373/00), in long fine focus mode at 2.200 W and 60 KV. The detector used was the X'Celerator RTMS (Real Time Multiple Scanning). Data acquisition of the records was performed with the software X'PERT HighScore version 2.1b from PANalytical. For the chemical analysis, we used a sequential wave dispersion spectrometer (WDS) Minerals PANalytical Axios, with ceramic tube X-ray anode rhodium (Rh) and a maximum power level of 2.4 KW. Given that the materials investigated are archaeological artefacts, which could not be destroyed, i.e., ground into powder, the XRF analyses were performed directly on the pieces. This procedure produces semi-quantitative results, given the lack of homogenization and specific internal standards. Therefore the Loss of Ignition was set to the next value (13.00%) of kaolinite (13.96% theoretical), the dominant mineral of the pieces. All mineralogical and chemical analyses were performed in the laboratories at the Geosciences Institute of the *Universidade Federal do Pará* (UFPA).

RESULTS

MORPHOLOGY AND MINERALOGY

All the analyzed pieces vary in size between 2 and 4 cm, the predominant colours are white and yellowish ochre, the glow is opaque, and the hardness is medium. The pieces differ visually, consisting of beads, pendants, and preforms (Figure 2). The excavated beads that

Figure 2. General morphological aspects of the investigated lithic artifacts from Salobo archaeological sites: A) shows rounded (SM 149, SB 15052 e SB 1509) and cylindric beads (SB 16182 e SB 7109); B) pendant with, free forms; C-D) preforms: kaolinite (Kln), quartz (Qtz), muscovite (Ms), florencite (Flr) and hematite (Hem).

are the objective of this study follow the morphological pattern observed in Amazonian beads (Barata, 1954; Meirelles, 2011), including the length, width, single hole, smooth, and sometimes polished surfaces. Moreover, the pendants have various shapes, including elongated and even zoomorphic forms (SBI 16315, Figure 2). They also have well-polished or ribbed surfaces, and a single or two holes can be found in various locations.

The investigated beads, pendants, and preforms are composed mainly of kaolinite, quartz, and hematite; florencite and muscovite are contained as accessory minerals (Figure 3). Kaolinite, in general, constitutes 95% of each piece (Table 2). Kaolinite presents itself in distinct morphologies and crystal sizes (Figures 4A and 4B). It occurs as booklets with pseudo-hexagonal outlines (Figure 4A) and in the form of mass aggregates with flocculated particles with indication of feldspar alteration. The XRD spectra also show high crystalline order reflections alongside undeveloped low crystalline order reflections, which could represent the booklets and pellets, respectively.

The quartz is milky and cryptocrystalline, and it is associated with kaolinite, florencite, hematite and muscovite.

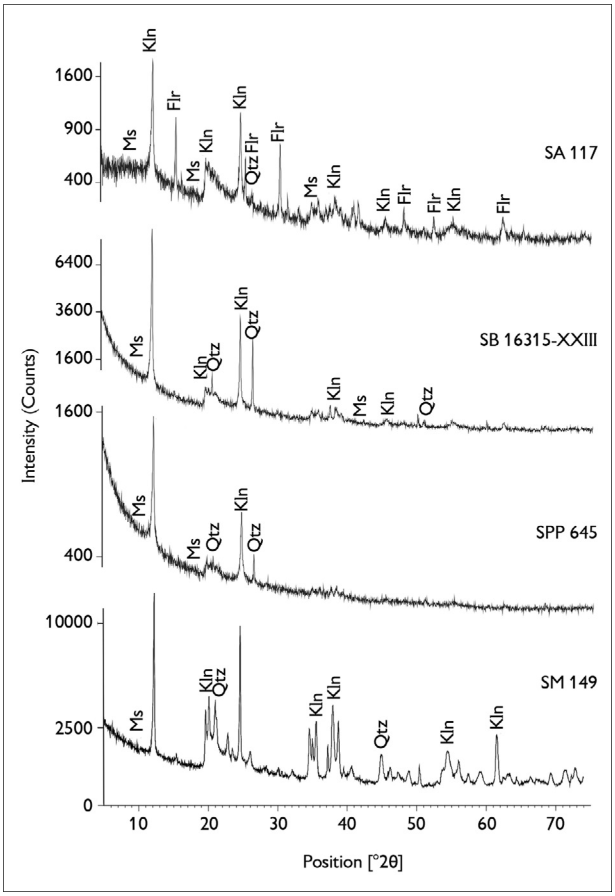

Figure 3. XRD diagrams showing the main mineral phases of some investigated lithic artefacts from the Salobo archaeological sites: kaolinite (Kln), quartz (Qtz), muscovite (Ms), and florencite (Flr).

Table 2. Mineralogical composition of the main samples investigated by XRD and XRF analyses and stoichiometric calculations. These results should be considered semi-quantitative, as previously explained in the Materials and Methods section above.

Sample identification	Kaolinite	Quartz	Muscovite	Florencite	Hematite	Total
SA 117	93.6	2.5	0.84	9.05	0.5	106.49
SB 15056	93.2	0.8	-	-	0.1	94.1
SB 15252	88.2	0.3	0.84	3.25	0.2	92.79
SB 15112	91.1	0.9	0.84	0.72	0.7	94.26
SB 16521	100.0	5.5	-	-	0.3	105.8
SB 15299	94.9	-	2.54	0.36	0.3	98.1
SB 15039 X	91.1	-	3.39	1.08	2.8	98.37
SB 15039 XX	89.1	0.6	1.69	7.23	0.2	98.82
SB 15208	17.4	-	13.55	6.87	54.5	92.32
SB 15209	94.8	0.7	1.69	1.08	0.1	98.37
SB 15052	93.9	0.6	1.69	0.72	0.5	97.41
SB 15057	94.5	0.7	2.54	0.72	0.5	98.96
SB 16315	90.3	1.5	4.23	2.89	0.3	99.22
SB 16182	87.5	-	4.23	1.44	-	93.17
SPP 607	100.0	-	0.84	-	1.1	101.94
SPP 646	87.7	1.9	5.08	0.72	0.4	95.8
SPP 607 XXXI	71.7	14.5	5.08	0.72	0.5	92.5
SPP 646 XXXXII	87.7	1.9	5.08	0.72	0.4	95.8

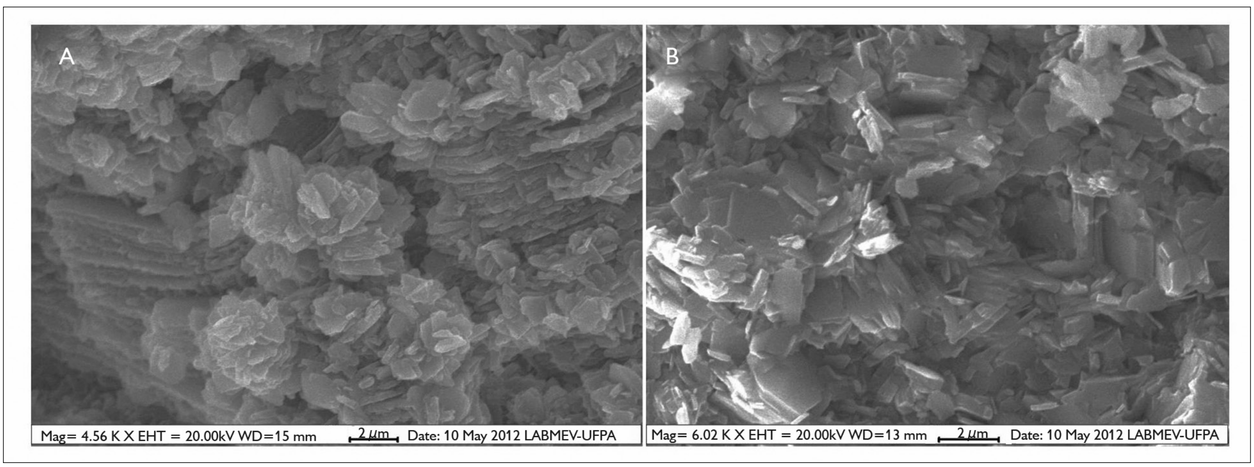

Figure 4. SEM images of well-developed booklets of kaolinite (A) and well-formed pseudo-hexagonal plate crystals of the same mineral as the main phase of some lithic artefacts (B).

The florencite, $CeAl_3(PO_4)_2\ (OH)_6$, stands out mainly in sample SA 117 and is not detected by SEM/EDS, suggesting the minimum availability of this material. In the other pieces (SB 15039, SB 15208, SB 15052 and SB 16315), it was possible to outline its main peaks in the diffraction patterns. Hematite occurs in small amounts, especially in the ochre portion of pendant SB 15140 and the pigmented lithic units (SM 149, SB 15112, SB 15039, SB 15208, SB 15299, SPP 607). Muscovite (sericite type) was identified in small amounts in almost all lithic pieces.

CHEMISTRY

In general, all the analysed pieces consist mainly of SiO_2 and Al_2O_3 (Table 3), which together account for more than 80% of the composition of each sample. The SiO_2 content ranges from 41.1 to 50.2%, and Al_2O_3 ranges from 30.5 to 42.7%, except for sample SB 15208, which is dominated by Fe_2O_3. The SiO_2 and Al_2O_3 contents, especially the average levels, are fully compatible with the kaolinite domain as indicated in Table 2. In some samples, the SiO_2/Al_2O_3 ratio approaches 1.19, typical of this mineral, and thus demonstrates that the quartz content is low. Also noteworthy are the contents of P_2O_5, up to 2.5%, CaO, up to 1.2%, Na_2O, to 3.7%, MgO < 1.3%, K_2O < 1.6, and TiO_2 < 0.2% except in two samples (SB 15052 and SB 15208) with 0.4 and 1.3%, respectively. These samples were not identified as Ti minerals; Ti probably lies in the structure of mica, as observed in sample SB 15208, which is relatively rich in K_2O. They were also not identified as Ca or Na minerals, corresponding to plagioclase feldspars, which would have altered to form kaolinite; however, these minerals were not present. Alternatively, the relatively high amounts of Na_2O reflect the chemical constitution of the sample holder used.

Table 3. Chemical composition of lithic artefacts by XRF analysis on natural samples.

Sample identification	SiO_2	Al_2O_3	Fe_2O_3	MgO	CaO	Na_2O	K_2O	TiO_2	P_2O_5	LOI	SiO_2/Al_2O_3	Total
SA 117	46.45	40.03	0.47	0.83	0.24	2.88	0.12	0.13	2.45	13.96	1.16	93.6
SB 15056	44.06	36.82	0.1	-	1.04	1.71	-	-	0.04	13.00	1.2	96.77
SB 15252	41.9	36.08	0.21	0.75	1.43	2.02	0.07	0.22	0.85	13.00	1.16	96.53
SB 15112	43.63	36.51	0.72	0.63	0.02	1.85	0.06	0.17	0.17	13.00	1.19	96.76
SB 16521	52.03	42.66	0.28	-	0.12	1.97	-	-	-	-	1.22	97.06
SB 15299	44.99	38.5	0.26	-	0.15	1.83	0.32	0.18	0.08	13.00	1.17	99.31
SB 15039 X	42.98	37.35	2.81	-	0.17	2.15	0.36	0.12	0.28	13.00	1.15	99.22
SB 15039 XX	42.57	38.04	0.16	-	0.2	1.42	0.24	0.1	1.98	13.00	1.12	97.71
SB 15208	11.83	14.11	54.45	-	0.41	-	1.57	1.32	1.86	13.00	0.84	98.55
SB 15209	45.39	38.37	0.11	-	0.18	1.59	0.23	0.09	0.25	13.00	1.18	99.21
SB 15052	44.92	37.93	0.45	-	0.18	1.5	0.23	0.42	0.18	13.00	1.18	98.81
SB 15057	45.58	38.13	0.46	-	0.11	1.56	0.28	-	0.17	13.00	1.2	99.29
SB 16315	45.44	38.14	0.25	-	0.14	1.04	0.49	-	0.79	13.00	1.19	99.29
SB 16182	41.07	36.57	-	1.26	0.26	3.65	0.49	0.19	0.39	13.00	1.12	96.88
SPP 607	45.34	39.98	1.09	-	-	-	0.11	-	-	13.48	1.13	100
SPP 646	44.98	36.8	0.39	-	0.13	1.23	0.6	-	0.16	13.00	1.22	97.29
SPP 607 XXXI	50.23	30.45	0.51	1.32	0.32	1.73	0.56	0.23	0.23	13.00	1.64	98.58
SPP 646 XXXXII	44.98	36.8	0.39	-	0.13	-	0.6	-	0.16	13.00	1.22	96.06
Theoretical kaolinite	46.54	39.5								13.96	1.18	100

K_2O and MgO contents are compatible with the accessory presence of muscovite sericite (Table 3). Unfortunately, it was not possible to determine the rare-earth element (REE) contents, i.e., La and Ce, to confirm the presence of florencite. P_2O_5 content above the crustal average (> 0.10%) occurs in most of the artefacts analysed, and in three samples (SA 117, SB 15039 XX and SB 15208) the levels are between 1.9 and 2.5%, which in terms of florencite corresponds to 9% of this mineral. Sample SBI 15208 is unique for its intense red colour, clearly indicated by the Fe_2O_3 domain in the form of hematite; it also contains some kaolinite and quartz, and it has the highest content of muscovite and even florencite.

DISCUSSION

PROVENANCE OF RAW MATERIALS

The mineralogical and chemical similarity among the vast majority of the investigated lithic pieces suggest they derive from a single raw material source, the same mine source area, and provenance (Figure 1). The raw material corresponds to a semi-hard kaolin, comprises kaolinite with small quantities of cryptocrystalline quartz, florencite, and sericite (muscovite). This microcrystalline quartz probably indicates the nature of the flint kaolin. The constant presence of sericite and florencite, a rare mineral, shows the peculiarity of this mineral occurrence. Florencite is a member of the crandallite group, in which the most common solid solution is crandallite-goyazite, but it often contains florencite in small proportions, which are difficult to distinguish by XRD. As it was not possible to determine the REE, florencite will be considered an individual mineral, phosphate member and/or phosphate-sulfate alunite supergroup member, which includes crandallite and woodhouseite (Toledo, 1999; Dill, 2001, 2003).

Kaolinite and florencite (or any mineral of the alunite supergroup) associated with 'semi-hard' kaolin are found in hydrothermal veins in subvolcanic venules and pyroclastic rocks with high sulfidation (Dill *et al*., 1995a, 1995b; Dill, 2003). This association was also observed in altered sedimentary rocks, alkaline-carbonatitic complexes, rocks of low grade metamorphism (Dill, 2001, 2003), and in lateritic profiles (Costa, 1991; Toledo, 1999; Lottermoser, 1990a, 1990b; Dill, 2001; Pöllmann *et al*., 2002) in laterite. They are usually soft.

In the Carajás Mineral Province, in the region not far from the area of the investigated archaeological sites, and close to the Salobo copper-gold mine, similar material in terms of textural aspects and mineralogical composition was found in the underground amethyst mines in Alto Bonito (Figure 5). Medium-hard hydrothermal kaolin without identification of phosphates was also reported in the manganese mine in Azul (Costa *et al*., 2005) and the exhausted gold mine of Igarapé Bahia. Here, florencite was found locally. The occurrence of Alto Bonito is closest to the raw material of the investigated lithic remains and is only 40 km north of the investigated sites. The kaolinite-phosphate (florencite)-quartz-sericite material formed veins and cemented breccia-like material (Oliveira, 1999), which cross cut the hyaline quartz veins and amethyst hosted by Proterozoic metarenites (low grade metamorphism) or inside of shear zone (Oliveira, 1999; Lima & Villas, 2002; Costa *et al*., 2008). The fault zone environment and consequent formation of veins and breccias favoured the development of semi-flint kaolin, equivalent to that of the investigated lithic material in terms of mineralogical composition and similar textural; in addition, given its relative proximity to the archaeological sites, it may have been the raw material for these archaeological sites. This assumption is strengthened by the occurrence of hyaline quartz chips found in archaeological sites in the same region (Silveira *et al*., 2008, 2009), whose main source area would also have been located on the outskirts of the current amethyst and quartz hyaline mines (Oliveira, 1999; Costa *et al*., 2008).

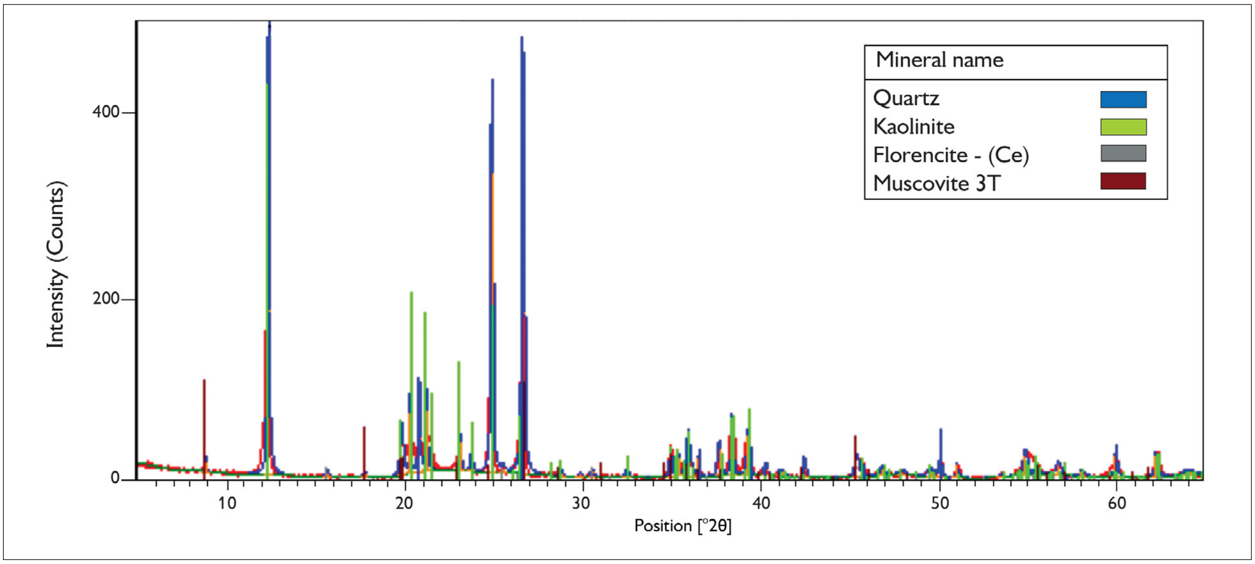

Figure 5. X-ray diffraction spectrum of a contact rock in the amethyst vein of high ordered kaolinite, quartz, florencite, and muscovite (sericite). Modified after analysis from Oliveira (1999).

CONCLUSIONS

The beads, pendants, and preforms found in the archaeological sites in the Salobo region were derived from semi-flint kaolin, white in colour, sometimes with a slight brown tint. This kaolin mainly consists of kaolinite, with some cryptocrystalline quartz, muscovite and florencite. The kaolin variations are mainly due to different phosphate concentrations since, often, no phosphate is present. It is likely that the cryptocrystalline quartz, phosphate, and sericite enhanced the nature of the semi-hard kaolin. The mineralogical and chemical results, combined with characteristics of artefacts morphology and texture, indicate relations among the archaeological sites. The beads, pendants, and preforms are morphologically different, but the raw material is similar, suggesting that they came from the same source and were preferred as the materials for manufacture of lithic artefacts. Similar material in terms of textural and mineralogical properties was found north of Salobo, in the Alto Bonito mines in the amethyst veins contained in metarenites. Knowledge of mineralogy and chemistry was crucial for the interpretation of the source area of the raw materials. Therefore, Alto Bonito may have been the source of raw materials of the lithic artefacts studied in Salobo.

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