Geociências
Geosciences

Mineralogia e química de fragmentos cerâmicos arqueológicos em sítio com Terra Preta da Amazônia Colombiana

Mineralogy and chemistry of archaeological ceramic fragments from Archaeological Dark Earth Site in Colombian Amazon

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Resumo

Vários sítios arqueológicos de Terra Preta Arqueológica (TPA) encontrados na floresta Amazônica Colombiana também contêm alto conteúdo de fragmentos cerâmicos semelhantes aqueles da Amazônia Brasileira, como mostra o sítio Quebrada Tacana. Seus fragmentos cerâmicos são amarelo a cinza, exibem matriz argilosa calcinada envolvendo fragmentos de cariapé, carvão e partículas de cinza, grãos de quartzo e micas. A matriz é composta de metacaulinita a material amorfo, quartzo, folhas de micas, clorita e sepiolita. Cariapé e cauixi são constituídos de cristobalita, da mesma forma o carvão e as cinzas. Embora não detectados pela difração de raios X, os fosfatos devem estar presentes, pois foram detectados teores de P_2O_5 de até 2,90 %, possivelmente como fosfatos de alumínio, já que o conteúdo de Ca está abaixo 0,1 %, o que elimina a possibilidade da presença de apatita. Estas características mineralógicas e químicas permitem relacionar estes fragmentos cerâmicos com aqueles encontrados nos sítios de TPA no Brasil e reforça o fósforo como um importante componente químico, originado pelo contato dos vasos cerâmicos com os alimentos do cotidiano dos povos amazônicos pré-históricos.

Palavras-chave: Fragmentos cerâmicos, Terra Preta, Quebrada Tacana.

Abstract

Several Archaeological Dark Earth (ADE) sites have been already found in the Colombian Amazon forest showing high content of archaeological ceramic fragments similarly to those in the Brazilian Amazon represented by Quebrada Tacana site. Their fragments are yellow to grey colour, display a burned clayey matrix which involves fragments of cariapé and coal and ash particles, besides grains of quartz and micas. The clay matrix is made of metakaolinite, quartz, and some mica flakes, chlorite and sepiolite. Cariapé and cauixi spicules are constituted of cristobalite, which is also the main mineral component of the coal and ashes. Although not detected by X-ray diffraction, the phosphate minerals should be present, since the contents of phosphor reach up to 2.90 Wt.% P_2O_5. Possibly it occurs as aluminium-phosphate, since Ca contents fall below 0.1 Wt.%. These mineralogical and chemical characteristics allow to correlate these ceramic fragments with those found in the ADE in Brazil and reinforce phosphor as an important chemical component, which indicates human activity by the daily use of pottery all over the Amazon region.

Keywords: *Ceramic fragments, Dark Earth, Quebrada Tacana*

1. Introduction

Several occurrences of dark earth soils of anthropic origin have been discovered in the Amazon in the last 150 years (Smith, 1879; Ranzani et al., 1962; Sombroek, 1966; Baleé, 1989; Kern & Kampf, 1989; Kern et al., 2003). They are very important since they are normally highly fertile. This property was recognized early and used by inhabitants of the region for agricultural purposes. Nowadays it is well known as Amazon Dark Earth (ADE), Archaeological Dark Earth (ADE), or Indian Dark Earth (IDE). Several researches are now considering its importance as carbon seeking; the origin of the stable organic matter and the presence of much coal; possibility of intentional soil formation by pre-historic populations; how it was possible to get highly fertile soil in the deep tropical region occupied by well known poor soils; and what is the reason for the long-term stable soil fertility (Kern et al., 2003; Glaser & Woods, 2004).

Although recognized early in the nineteenth century as an important component of these soils, the ceramic fragments have been well studied only in their descriptive morphological aspects, which contributed to a general understanding of the ceramic technology already developed in the whole Amazon region (Hilbert, 1955; Ranzani et al., 1962; Falesi, 1974; Eden et al., 1984; Simões & Corrêa, 1987; Simões & Machado, 1987; Simões & Kalkmann, 1987; Simões & Lopes, 1987). No mineralogical and chemical research was carried out until the late 1990s (Costa et al., 1991; Coelho et al., 1996; Costa & Kern, 1999; Kern & Costa, 2001; Costa et al., 2002; Costa et al., 2004a,b; Costa et al., 2006, 2009; Kern et al., 2009). These studies concentrated on the ADE ceramic fragments along the rivers from the Oriximiná County and Caxiuanã bay in the Lower Amazon region in the state of Pará and partly in areas adjacent to Manaus and Tabatinga in the state of Amazonas (Lima, 2001), which show the importance of these researches. They identified the constant presence of phosphor in relatively high concentrations well distributed in the ceramic fragments, an element having a comparative presence in the ADE. Phosphor normally occurs as non-crystalline sub-micrometre masses in the burned clayey matrix as well as around the cariapé fragments and cauixi spicules. Chemically, the phosphor presence can be correlated to aluminium (iron) phosphates and occasionally crandallite and variscite (Costa et al., 2004a, 2006). The occurrence of phosphate in the ceramic materials of ADE can add very important insights about the exposure of the respective pot to phosphor-rich foods or even to some phosphor-bearing natural material used as fluxes which were in fact not known concerning the Amazonian archaeological potteries until the present, since they don't display any king of glass. The exposure to phosphor-rich food could be very interesting since it could contribute to understanding the diet of the pre-historic people of this very large region, which has undergone a deep environmental transformation by establishing a steadily increase of the wet and hot tropical rain forest.

In order to extend the mineralogical and chemical study of the archaeological ceramic fragments related to ADE beyond the Brazilian Amazon region, and to get information about the source of the raw material as done by Strazicich (1998), we studied the Quebrada Tacana ADE site in Colombia. ADEs in Colombia were earlier described in the 1970s in the Guyanabero and Caquetá rivers in La Pedrera and Araracuara (Von Hildebrand, 1976; Eden et al., 1984; López, 1993).

2. Location of the ADE Quebrada Tacana Site

The Quebrada Tacana is located in the Leticia County, Colombia (Figure 1) near to the Tacana creek, not far from the Brazilian border.

The ADE Quebrada Tacana occupies 2 ha of Late Holocene floodplain. The soil profile comprises a thin (10 to 15 cm) litter horizon, which overlays A, AB, and Bw1 horizons, less than 1 m thick (Figure 2). The ceramic fragments represent an Ah horizon, a typical bed (5 to 40 cm thick) formed by close sub-horizontal stacking of decimetre-large ceramic fragments (Figure 2). Coal radiocarbon dating carried out at Beta Analytic Incorporation indicates an age of 1270 to 1060 yr BP (two sigma calibrated).

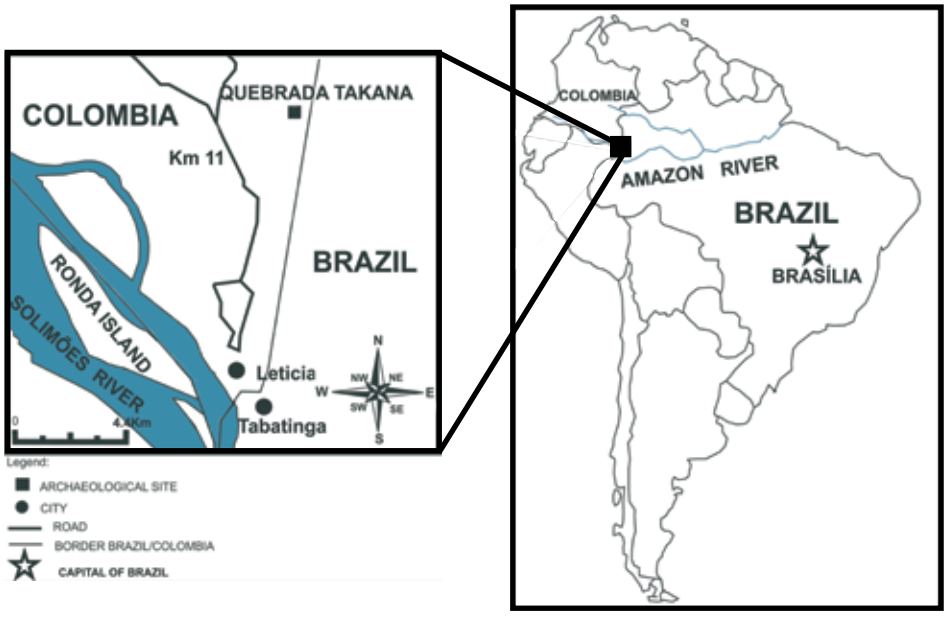

Figure 1
Location map of ADE Quebrada Tacana site. Central coordinates: 04° 07' 06.8" S and 69° 55' 16.4" W.

3. Materials and methods

Twenty-eight samples were collected from the Ah horizon (Figure 2). They are centimetre-large fragments and represent plates and semi globular basins, which are dominant, as well as pots (Figure 2). The fragments correspond to the interior wall of the ceramic vessel, displaying a dark skin made of resin. On the outer face they show simple dark brown to bluish grey traces which can be interpreted as paintings. However most of the fragments show some weathering alteration products.

All the samples have been described after their textures, colour, cohesion, painting, mineral phases and tempers, matrix, grains, or fragments, as well as weathering products and dissolution voids.

Fifteen samples have studied by optical microscope and submitted to mineral identification by the X-ray diffraction (XRD) powder method using a PANalytical diffractometer, model X`PERT PRO MPD (PW 3040/60), with a goniometer PW 3050/60 (theta to theta) equipped with a copper anode ($\lambda Cu_{K\alpha}$ = 1.7902 Å), operating conditions at 40 kV and 35mA, and detector type RTMS, X'Celerator. The data acquisition was realized with the software X´Pert Data Collector, version 2.1a, and the data treatment with X´Pert HighScore version 2.1b. The same samples were submitted to chemical analyses for major and trace element determination (Acme Chemical Laboratory procedure: dissolution by strong acid and ICP-MS determination).

4. Results and discussion

Morphology

The ceramic fragments studied present in general a grey to brown colour for the outer surface of the pot and dark grey in the inner part (Figure 3). In the recent fracture surfaces one can observe burned cariapé (wood skin, with the cellulose pattern still preserved), coal, and ashes. Soft or sandy dark brown argillaceous spots and outer films are very common and are the weathering products of ceramic fragments after they were discharged and incorporated into the ADE site.

Mineralogy and chemistry

The fragments show silt to clayey to cryptocrystalline plasmic matrix with low porosity, light coloured to brownish to reddish, which suggests a domain of clayey minerals overgrowth with cryptocrystalline iron oxy-hydroxides (Figures 4a-e) mostly XRD-amorphous. The contents of Fe_2O_3 (2.39 to 6.64 Wt. %) were high enough to be detected as crystalline iron oxy-hydroxides, if they were present. The matrix displays sub-round to angular submillimeter quartz grains, besides cariapé fragments and cauixi spicules already mentioned. The cauixi (Tubella reticulata and Parnula betesil), for example, is fresh water spongy that cumulates on trunks of trees, boats, and so on and cariapé is a tree outer skin representing a Bignomiacea, Moquilea, Licania utilis, and Turiuva (Costa et al., 2004a).

Inside the cariapé fragments occupying the cellulose vases, white cristobalite has been found (Figure 4e). Amorphous silica is commonly found in the plant tissue, and when burned can be transformed into cristobalite; in fact this has happened with the cariapé, similarly to the ceramic fragments from Cachoeira-Porteira ADE (Costa et al., 2004). The contact between cariapé and matrix is followed by a reaction aureole of the same material, cristobalite (Figure 4c), which could have taken place at the time of the ceramic burning or during use of the pot for preparing food by firing or even during both processes. As much as 1.16 to 8.36 Wt. % organic carbon has been detected, which reinforces the presence of coaled cellulose or still cellulose (Figure 4d). The cauixi spicules are also composed of silica (Costa et al., 2004) and are likely to be cristobalite, since they were burned during ceramic firing.

The plasmic matrix is XRD-amorphous, rich on SiO_2 and Al_2O_3 partly equivalent to metakaolinite, a possible

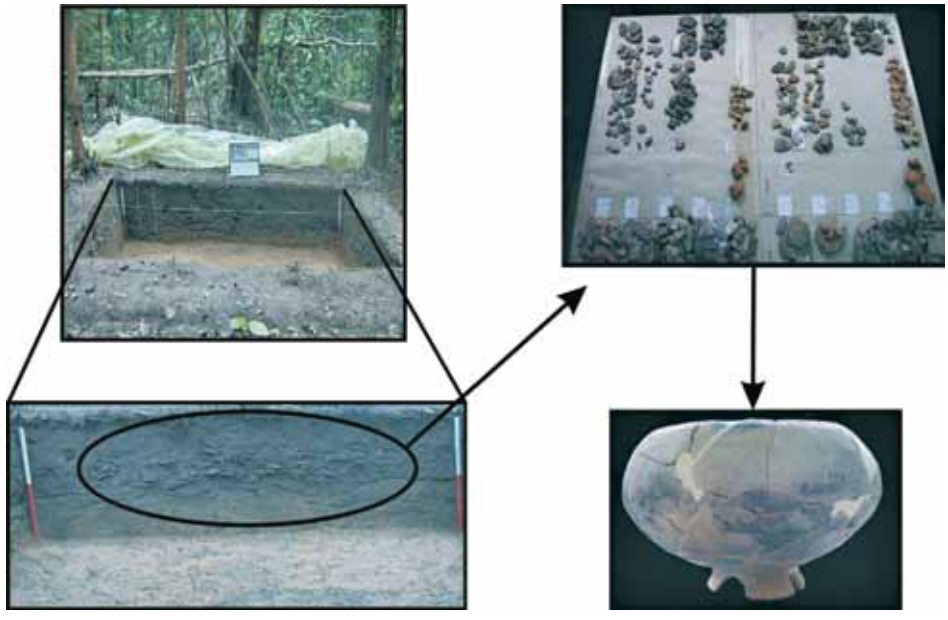

Figure 2
ADE soil profile at Quebrada Tacana site showing high accumulation of ceramic fragments and vessel reconstruction.

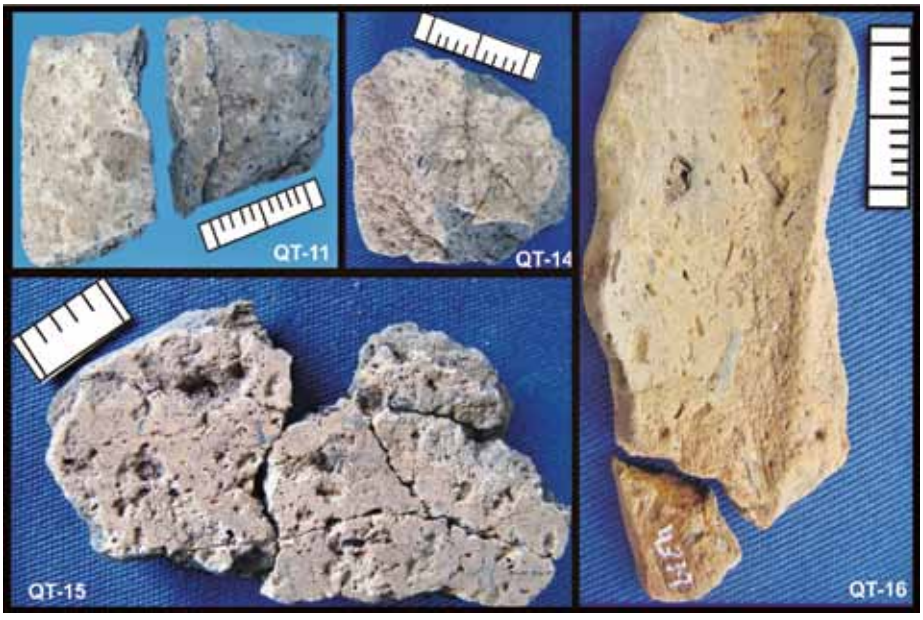

Figure 3
Mesoscopic aspects of ceramic fragments (sample QT-11, QT-14 and QT-15) showing the domain of light grey in the outer wall and grey to dark grey colour in the inner wall and interior of the ceramic mass or a typical yellow brown colour for both sides of the ceramic wall (QT-16). The scale marks show cm and mm divisions, respectively.

obvious product of the calcinations of the kaolinite around 550°C. Kaolinite is the most frequent clay mineral of the ceramic raw material. Quartz is abundant and described as floating grains in the matrix. Clinochlore occurs formed during the burning of pottery or even during the firing cooking. Mica flakes occur and is being represented by the relatively high contents of K_2O, besides MgO (Table 1). Illite, smectite, and even kaolinite are present in small quantities and possibly formed lately during discharging of the potteries and their subsequent weathering, similarly goethite from haematite. Microcline was sometimes detected. Anatase is XRD-identified in constant low concentration throughout the ceramic fragments. The presence of anatase is confirmed by chemical analyses, which show 0.95 to 1.15 Wt. % of TiO_2. Garnet was found rarely.

The phosphates in the fragment ceramics could not be normally detected either by XRD or by optical microscope. However the chemical analyses show 0.50% to 2.90 Wt.% of P_2O_5 (Table 1), which could correspond to ca. 1.5 to 8.7 Wt.% of aluminium phosphates, the possible phosphate minerals, found in the ceramic fragments (Costa et al., 2004a, 2006, 2009). SEM images and EDS chemical analyses confirm the presence of phosphor in the clayey matrix. In the ADE site at Bragança, located at north eastern of state Para, well formed crandallite crystals have been identified (Costa et al., 2006). Variscite and variscite-strengite have been identified at Cachoeira-Porteira, in the Lower Amazon region (Costa et al., 2004a). The phosphor contents of Quebrada Tacana cannot be related either to apatite, rarely found as bone fragments

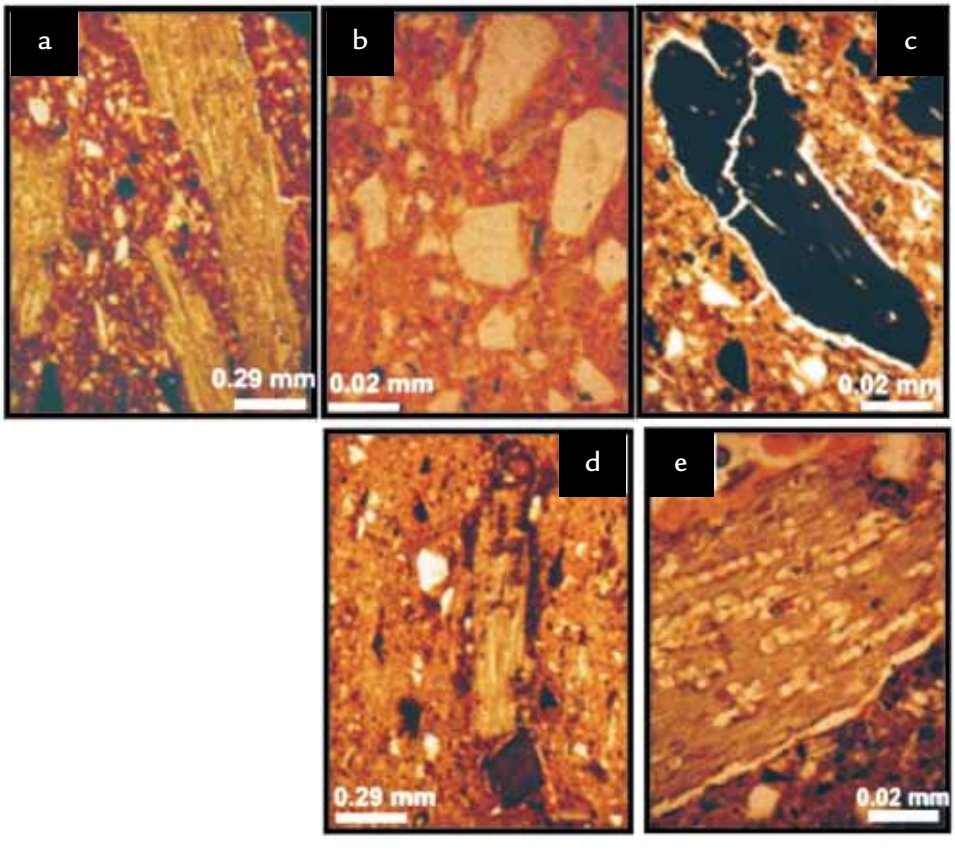

Figure 4
Photomicrograph images obtained by optical microscope of the ceramic fragments showing:
a) general aspects of matrix and fragments of cariapé, cauixi spicules, coal, and quartz.
b) Details of quartz grains, coal fragments, and/or ash particles inside a clayey matrix.
c) Detail of a stark burned cariapé (which looks like coal) with a reaction aureole and veinlet of cristobalite.
d) Fragment of cariapé slightly burned, close to ash particles.
e) Details of the cellulose vase of cariapé composed of cristobalite.

inside of the fragments, for example at Cachoeira-Porteira (Costa et al., 2004a), nor to crandallite or crandallite-goyazite, since the contents of CaO (Table 1) and SrO (not indicated at Table 1) are very low (< 0.10 Wt.%).

The chemical composition of the ceramic fragments is not homogeneous in terms of phosphor and iron (Figure 5) and suggests a raw material for pot production based on quartz and kaolinite as the main mineral phases, besides iron oxy-hydroxide minerals in variable proportions. This iron-rich clay raw material was significantly modified by the addition of silica-rich cariapé, ashes, coal, and cauixi, but did not receive any contribution of feldspars (except for a small proportion of K-feldspar), differing from the other ceramics of the Amazon. Phosphor is an important component of archaeological ceramic fragments and has been found in Quebrada Tacana. It is not linked either to special raw materials with phosphate minerals or to organic tempers. It may be introduced later during the daily use of the ceramic artefact for food preparation and storage, a phosphor contamination, as proposed by Costa et al. (2004).

SAMPLES	SiO_2	Al_2O_3	Fe_2O_3	MgO	CaO	Na_2O	K_2O	TiO_2	P_2O_5	MnO	T/C	T/S	LOI
QT-02	59.28	18.09	2.98	0.40	0.05	0.05	1.12	0.98	0.59	<0,01	1.99	0.04	16.4
QT-03	55.74	17.94	5.51	0.66	0.03	0.11	1.57	1.00	0.71	0.02	3.24	0.02	16.6
QT-05	53.89	20.07	3.30	0.57	0.05	0.07	1.44	1.15	1.32	0.02	2.76	0.04	18.0
QT-06	60.79	17.33	2.96	0.42	0.05	0.06	1.08	1.00	1.15	<0,01	1.71	0.04	15.0
QT-10	67.35	13.62	3.02	0.30	0.05	0.07	0.93	0.96	1.08	0.01	1.72	0.03	12.5
QT-12	63.63	15.79	3.04	0.52	0.03	0.09	1.38	0.96	1.09	0.01	1.16	0.05	13.3
QT-13	54.88	19.18	3.62	0.30	0.05	0.05	1.42	1.00	1.70	0.01	1.99	0.06	17.6
QT-14	49.45	16.37	2.96	0.40	0.05	0.04	1.24	0.95	1.86	0.01	8.36	0.07	26.5
QT-15	52.75	17.54	2.50	0.25	0.03	0.04	0.98	1.12	0.87	0.01	6.28	0.03	23.8
QT-16	54.39	17.64	6.64	0.62	0.04	0.05	1.78	0.97	1.02	<0,01	0.97	0.07	16.7
QT-18	57.49	16.07	2.47	0.36	0.06	0.05	1.22	0.96	2.39	<0,01	3.82	0.06	18.8
QT-19	57.88	17.28	2.71	0.49	0.08	0.09	1.46	0.98	2.90	0.01	2.39	0.04	16.0
QT-22	53.10	16.62	2.58	0.29	0.02	0.05	1.04	1.03	1.25	0.02	6.15	0.03	23.8
QT-25	57.50	17.42	3.94	0.33	0.04	0.05	1.29	1.01	1.18	0.01	1.86	0.04	17.1
QT-27	49.83	19.48	3.24	0.35	0.04	0.05	1.12	1.07	0.99	0.02	4.80	0.03	23.9
AVERAGE	56.33	17.36	3.43	0.42	0.04	0.06	1.27	1.01	1.34	0.01	3.28	0.043	18.4
C.-PORTEIRA(1)	65.55	16.37	5.79	0.63	0.43	0.69	0.90	0.86	2.37	na	na	na	na
CAXIUNÃ(2)	71.35	8.60	4.54	0.26	0.19	0.98	0.59	0.40	1.31	na	na	na	na
MANAUS(3)	36.81	20.82	6.13	1.35	1.02	nd	7.29	0.57	3.62	0.088	na	na	na

Table 1
Chemical composition (Wt. %) of the ceramic fragments from ADE Quebrada Tacana, Colombia.

(1) C.-PORTEIRA: Cachoeira-Porteira; costa et al., 2004. (2) Coelho et al., 1996. (3) Lima, 2001.
T/C: Total Carbon
T/S: Total Sulphur
LOI: Lost Of Ignition
na: not analyzed

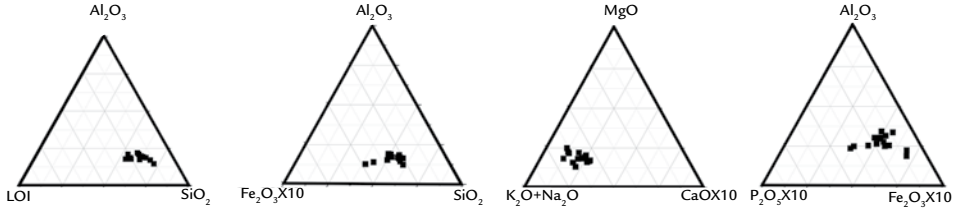

Figure 5
The chemical composition of the ceramic fragments from ADE site Quebrada Tacana after the diagrams SiO_2 - Al_2O_3 - L.O.I.; SiO_2 - Al_2O_3- Fe_2O_3; CaO - MgO - (K_2O + Na_2O); Fe_2O_3 -Al_2O_3 - P_2O_5 from left to right.

5. Conclusions

The morphological data of the ceramic fragments studied at ADE Quebrada Tacana are parts of ceramic plates and pots daily used for food preparation, cooking and storage. By correlation they are as old as the ADE site, around 1170 cal yr BP. The grey colour may be related to low temperature of burning as well as to the first stage of chemical weathering. The most common tempers are of organic origin and silica-rich. Besides quartz and kaolinite-derived amorphous phase (metakaolinite), the matrix is made of chlorite, sepiolite and microcline, which reinforce the thermal treatment (burning) and the hydrothermal environment originated by cooking. The mineral composition of the ceramic fragments from Quebrada Tacana is similar to most archaeological ceramics of ADE studied in the Amazon, except for its higher content of organic-silica. It means that the pre-historic peoples used much more organic silica to improve the raw material. Illite, montmorillonite, goethite, and kaolinite may be weathering products after discharge of the ceramics. No phosphate minerals were XRD detected, but their presence is clearly indicated by chemical analysis. They may be XRD - amorphous aluminium phosphates, as found in other Amazon ADE. Apatite or any other Ca-phosphate is not possible, since the ceramic materials are calcium-poor. The chemical composition in terms of SiO_2, Al_2O_3, CaO, MgO, K_2O and Na_2O and the mineralogy suggest a common and slightly homogeneous raw material, possibly a saprolitic and/or sedimentary material, derived from quartz + aluminium silicate rocks with some Mg and Fe as ceramic raw material. To improve the raw material the prehistoric peoples of the Quebrada Tacana introduced an expressive quantity of calcined silica rich organic tempers. Phosphor probably came later during the use of ceramic vessels for food preparation, normally rich on phosphor, similarly to other ADE ceramics as proposed by Costa et al (2004b).

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