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**MINERALOGIA E GEOQUÍMICA DE TERRA PRETA
ARQUEOLÓGICA PARA IDENTIFICAÇÃO DE PADRÃO
OCUPACIONAL PRÉ-HISTÓRICO NO VALE DO BAIXO RIO
AMAZONAS (JURUTI, PARÁ)**

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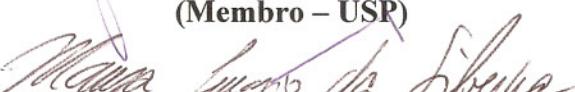
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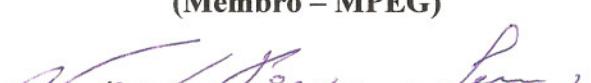
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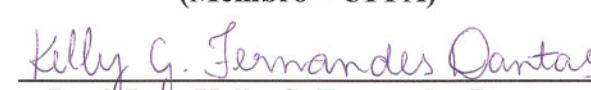
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que é pai e amigo; à minha mãe, Espírito
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RESUMO

Na Amazônia são inúmeras as ocorrências de solos modificados pela ocupação de antigos povos ceramistas. Estes solos, conhecidos como Terra Preta de Índio ou Terra Preta Arqueológica (TPA), geralmente ocupam pequenas áreas, mas também são encontrados em áreas contínuas por dezenas de hectares. Algumas dessas TPA são circundadas por solos conhecidos por Terra Mulata Antrópica (TMA). As TPA apresentam cor escura, teores elevados de Ca, Mg, P, Mn, Zn, Cu e C orgânico, fragmentos cerâmicos (FC) e carvão. Já a TMA, embora de cor escura, é desprovida de FC e os seus conteúdos de Ca, Mg, P, Mn, Zn, Cu e C orgânicos são menores em relação à TPA, porém mais elevados quando comparados aos solos adjacentes (AD). Na região do Baixo Amazonas extensas áreas de TPA são acompanhadas por faixas igualmente extensas de TMA, que foram delineadas durante as pesquisas de salvamento arqueológico para implantação da indústria de extração e beneficiamento de bauxita da ALCOA, no município de Juruti, Pará. Estes locais, conhecidos como sítios arqueológicos, ricos em fragmentos cerâmicos (FC) e matéria orgânica, foram investigados com o objetivo de conhecer as possíveis inter-relações entre os solos TPA e TMA, a real interferência da ocupação pré-histórica nos solos e delinear o padrão ocupacional, contribuindo assim para o esclarecimento da cronologia de ocupação da Amazônia. Foram realizadas amostragens de solos e de fragmentos cerâmicos (FC) em dois sítios arqueológicos com TPA e TMA selecionados para este estudo. As amostras de solo e FC foram coletadas em perfis pedológicos e em uma malha regular (60 x 120 m), representativa das TPA, TMA e AD. Em seguida foram submetidas a análises mineralógicas por DRX, MEV-EDS e microscopia óptica; e químicas (incluindo os elementos maiores e traços) determinadas por ICP-OES e ICP-MS. Além das análises químicas e mineralógicas, os FC foram ainda examinados arqueologicamente com lupa binocular e datados por termoluminescência (TL). Os fragmentos de carvão foram datados empregando-se o método radiocarbono (C^{14}) aplicando-se a técnica AMS. Os resultados obtidos mostram que os solos são constituídos essencialmente por quartzo e caulinita, e em menores proporções por illita + muscovita, goethita + hematita e anatásio. Estes minerais refletem a associação Al_2O_3 - Fe_2O_3 - TiO_2 interpretada como a assinatura dos solos derivados de crosta ferro-aluminosa de perfis lateríticos, equivalente aos solos adjacentes (AD). Embora as TPA e TMA guardem semelhanças mineralógicas e químicas entre si, diferem parcialmente nos teores de cada espécie mineral e nas concentrações dos elementos químicos. Por outro lado apatita, fosfato de Al e cristobalita são praticamente exclusivos das TPA, raramente encontrados na TMA e ausentes nos solos AD. A apatita e o fosfato de Al representam as principais fontes dos altos teores de Ca e P. Os solos

TPA e TMA se caracterizam também pelo elevado conteúdo de matéria orgânica e se deixam identificar pela associação P_2O_5 -CaO-MgO-Na₂O-K₂O-Zn-Cu-Mn-Ba-Sr-Li-Ni, interpretada como indicadora de atividade humana. Os mapas geoquímicos desses elementos e de distribuição dos FC permitiram delimitar as áreas de TPA como de ocupação permanente, delimitadas pelas concentrações mais elevadas. Foram identificadas sete manchas de solos enriquecidos e mais escuros, agrupadas em dois núcleos principais, separadas por corredores de solo equivalente à AD (associação geoquímica Al₂O₃-Fe₂O₃-TiO₂-Cr-Y-V-Zr). Tais manchas, com abundância de FC, foram interpretadas como locais de antigas aldeias. A TMA segue em extensa e contínua faixa paralela ao rio, entre a TPA e a AD, interpretada como locais de acampamento ou de atividades agrícolas. As características estilísticas e a sequência cronológica destes fragmentos indicam que a ocupação está representada principalmente por duas fases cerâmicas: Konduri, mais recente e Pocó, mais antiga. As idades obtidas por C¹⁴ e TL sugerem que estes povos ceramistas provavelmente se estabeleceram na região entre 140 AC e o século XIII da nossa era, tendo as TPA se formado subsequentemente. Os fragmentos cerâmicos constituíam vasilhas de uso cotidiano e ceremonial. Sua composição mineralógica e química se assemelha à argila utilizada pelos artesãos atuais. As diferenças se resumem aos elevados teores de fósforo (entre 1 e 4 % de P_2O_5) sob a forma de fosfatos de Al e Fe amorfos, que na argila atual se encontram em nível crustal. Isto reforça a proposta de sua origem relacionada ao uso, especialmente ao cozimento dos alimentos. Portanto, diante das similaridades mineralógicas e químicas entre os FC de culturas e cronologias distintas é possível inferir que as matérias-primas utilizadas pelos povos antigos dessa região, tiveram sempre a mesma proveniência ou ambiência geológica, iniciada há pelo menos 2.000 anos atrás, e que alcançou os povos atuais da região. Portanto, as áreas de TPA e TMA que ocupam o vale do Amazonas na região de Juruti por mais de 350 ha, indicam que elas são consequência de atividades humanas pré-históricas, extensivas e intensivas, que imprimiram significativas transformações aos solos pré-existentes. As condições de clima quente e úmido, além da densa cobertura vegetal permitiram a sua formação.

Palavras chave: Mineralogia. Terra Preta. Terra Mulata. Fragmentos Cerâmicos. Pré-história. Geoquímica. Arqueologia da Amazônia.

ABSTRACT

In the Amazon basin, the soils of a large number of sites have been modified by ancient pottery-making peoples. These soils, known as Indian Dark Earth or Archeological Dark Earth (ADE), generally cover relatively small areas, but in some cases, may extend to dozens of hectares. Some of these ADE sites are surrounded by soils known as Terra Mulata (TMA). The ADEs are dark in color, and have relatively high levels of Ca, Mg, P, Mn, Zn, Cu and organic C, ceramic fragments (CF), and charcoal. By contrast, while the TMA are relatively dark, they lack ceramic fragments, and have lower levels of Ca, Mg, P, Mn, Zn, Cu, and organic C than the ADE, even though these levels are generally higher than those found in adjacent soils or Latosol (Oxisols). Along the right bank of the lower Amazon River, at Juruti, in the Brazilian state of Pará, ample areas of ADE were found surrounded by equally extensive tracts of TMA during archeological rescue operation that preceded the construction of the ALCOA bauxite extraction and processing plant. These locations, known to be archeological sites rich in ceramic fragments and organic matter, were investigated with the aim of understanding the possible inter-relationships between the ADE and TMA soils and the specific role of the prehistoric occupation of these soils, and the determination of the pattern of occupation, and the time scale of the occupation of the Amazon basin. Samples of soil and CF were obtained from the two archeological sites with ADE and TMA selected for this study. The soil and CF samples were collected from pedological profiles within a standardized 60 m x 120 m grid, representative of the ADE, TMA and adjacent soils. The mineralogy of the samples was then analyzed using XRD, SEM-EDS and optical microscopy, and their chemistry (including major and trace elements) was determined using ICP-OES and ICP-MS. In addition to these chemical and mineralogical analyses, the CF were examined archeologically with a binocular lens, and dated using thermoluminescence. The fragments of charcoal were dated using the radiocarbon (C^{14}) method using the AMS technique. The results show that the soils are made up essentially of quartz and kaolinite, with illite + muscovite, goethite + hematite, and anatase in smaller proportions. These minerals reflect the Al_2O_3 - Fe_2O_3 - TiO_2 association that has been interpreted as the signature of soils derived from the ferrous-aluminous crust of lateritic profiles, equivalent to the adjacent soils. While ADE and TMA are relatively similar in their mineralogical and chemical composition, they are partly different in the content of each mineral and in their concentrations of the chemical elements. On the other hand, apatite, Al phosphate, and cristobalite are practically exclusive to the ADE, being found only rarely in the TMA, and entirely absent from the Latosol. The apatite

and Al phosphate represent the principal sources of the high levels of Ca and P. The ADE and TMA soils are also characterized by the high levels of organic matter, and can be identified by the P_2O_5 -CaO-MgO-Na₂O-K₂O-Zn-Cu-Mn-Ba-Sr-Li-Ni association, which can be interpreted as an indicator of human activity. The geochemical maps of these elements and the distribution of the CF permitted the delimitation of the ADE zones as representing areas of permanent human occupation, defined by the higher concentrations. Seven patches of darker, enriched soil were grouped in two principal nuclei, separated by corridors of soils equivalent to adjacent area (Al_2O_3 - Fe_2O_3 - TiO_2 -Cr-Y-V-Zr geochemical association). These patches, characterized by an abundance of ceramic fragments, were interpreted as being the sites of ancient settlements. The TMA lies in a wide, continuous belt parallel to the river, between the ADE and the adjacent soil, and is interpreted as an area of campsites or the locations of agricultural activities. The stylistic characteristics and the chronological order of these fragments indicate that the occupation of the area is represented primarily by two distinct ceramic phases, the older Pocó phase, and the more recent Konduri phase. The thermoluminescence and C¹⁴ dating indicate that these pottery-making peoples probably occupied the region between 140 B.C. and the 13th century, with the ADE being formed subsequently. The ceramic fragments represent the remains of both ceremonial vessels and everyday artifacts. Their mineralogical and chemical composition is similar to that of the clay used by the region's present-day potters. The differences are restricted to the high levels of phosphorus (between 1% and 4% P_2O_5) found in the fragments in the form of amorphous Al and Fe phosphates, which are found at crustal levels in the present-day clay. This reinforces the conclusion that their origin was related to the use of the ceramic vessels, especially for cooking food. From the mineralogical and chemical similarities between the CF of distinct cultures and ages, it is possible to infer that the raw material used by the ancient settlers of this region were invariably sourced from the same deposits or geological formations, beginning more than 2,000 years ago, and which are still used by the region's present inhabitants. Overall, then, the evidence indicates that the deposits of ADE and TMA soils in the area of Juruti, which extend for more than 350 hectares, are a consequence of extensive and intense prehistoric human activities, which imposed significant transformations on the original local soils. The hot and humid climatic conditions, and the dense forest cover were fundamental to the formation of these soils.

Key words: Mineralogy. Dark Earth. Terra Mulata. Ceramic fragments. Prehistory. Geochemistry. Amazonian archeology.

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1 INTRODUÇÃO

Na Amazônia a ocupação humana antiga ocasionou significativas alterações no ambiente. Estima-se que cerca de 60.000 Km² ou 1% dos solos amazônicos sofreram modificações antrópicas. Esses solos conhecidos como Terra Preta de Índio (TPI), Terra Preta Arqueológica ou Terra Preta Amazônica (TPA) compreendem a classe dos Arqueo-antrossolos e apresentam ampla ocorrência no Brasil e países vizinhos como Colômbia, Bolívia e Venezuela (Kern & Kämpf 1989; Kämpf & Kern 2005; Kämpf *et al.* 2010; Kern *et al.* 2010).

A formação das TPA é geralmente atribuída ao descarte de resíduos orgânicos diversos que implicaram na significativa modificação das propriedades morfológicas, físicas, químicas, mineralógicas e microbiológicas do solo (Smith 1980; Sombroek 1966; Costa & Kern 1999; Lehmann *et al.* 2003; Kern *et al.* 2003; Gleiser *et al.* 2003; Ruivo *et al.* 2009; Tsai *et al.* 2009). A cor escura, conteúdos elevados de Ca, Mg, P, Mn, Zn, Cu, C orgânico e partículas de carvão, combinados a fragmentos cerâmicos e artefatos líticos normalmente encontrados no horizonte A antrópico, faz da TPA uma variável fundamental no entendimento do cenário arqueológico amazônico diante de condições ambientais adversas.

Nas margens dos rios da região há inúmeros registros de sítios arqueológicos com terra preta (Hilbert 1955; Sombroek 1966; Smith 1980; Hilbert & Hilbert 1980; Roosevelt 1992; Gomes 2008). Por vezes, as TPA são acompanhadas por extensas faixas de solos, de cor bruno escuro, contendo pouco ou nenhum material cerâmico mas que, no entanto, apresentam teores elevados de matéria orgânica e nutrientes. Esses solos, denominados de Terra Mulata (TMA) parecem ser o resultado de atividades agrícolas praticadas pelas comunidades indígenas pré-coloniais (Sombroek 1966; Sombroek *et al.* 2002; Kämpf & Kern 2005; Denevan 2010).

Embora cerca de 80% dos sítios com TPA apresentem de 2 a 5 ha, áreas superiores a 100 ha podem ser encontradas em Santarém, Belterra, Oriximiná e Juruti, no estado do Pará; em Rio Preto da Eva, Presidente Figueiredo e Manaus, no Amazonas (Sombroek 1966; Smith 1980; Kern *et al.* 2003). Os registros arqueológicos desses locais corroboram com os registros históricos do século XVI, que descrevem aldeias e povoados grandes e numerosos, que se estendiam por 12 a 30 km, localizados em porções elevadas da paisagem (Porro 1996; Roosevelt 1992; Kampf & Kern 2005).

O estudo dos solos antrópicos representa uma ferramenta determinante para a cronologia de ocupação amazônica. No entanto, ele se encontra em estágio inicial devido às dimensões continentais, associada às poucas abordagens sistemáticas até então realizadas.

O significativo número de sítios arqueológicos e fases cerâmicas descritas sustentam os argumentos de diversidade cultural que coexistiu na região. São observados avanços significativos relativos às novas abordagens envolvendo os fragmentos cerâmicos (FC) contidos nas TPA. A abundância e resistência às condições climáticas fazem dos FC o ponto de partida das novas interpretações arqueológicas. Além de possibilitar a filiação cultural, auxiliam na identificação de proveniência de matéria-prima e permitem inferir sobre intercâmbio entre antigas comunidades (Latini *et al.* 2001; Costa *et al.* 2004a; Goffer 2007; Schaan 2007). Além da beleza estilística, as cerâmicas arqueológicas da Amazônia chamam a atenção pelos seus conteúdos elevados de fósforo (Costa *et al.* 2004a, 2004b, 2006, 2010; Rodrigues 2010; Silva 2010) quando confrontados aos conteúdos das cerâmicas européias (Duma 1972; Millani *et al.* 1998; Rathossi & Pontikes 2010) ou mesmo em relação aos solos de TPA (Kern 1996).

Grandes áreas de TPA e TMA foram delimitadas na região do Baixo Amazonas, precisamente no município de Juruti, fruto das atividades de salvamento arqueológico para instalação da completa estrutura de extração, beneficiamento e transporte ferroviário e portuário de bauxita, o minério de alumínio. Este estudo pretende, a partir da interação de dados mineralógicos, geoquímicos, arqueológicos e cronológicos obtidos a partir dos solos e fragmentos cerâmicos arqueológicos, contribuir no avanço das interpretações arqueológicas, pedológicas e áreas afins quanto às transformações causadas pela ocupação humana antiga e suas relações com a paisagem atual.

1.1 OBJETIVOS

1.1.2 Objetivo Geral

Entender os processos de formação de sítios de TPA e TMA e as inter-relações entre estes e os solos das áreas adjacentes (AD), a partir de estudos arqueológicos, mineralógicos, geoquímicos e datação por radiocarbono dos solos propriamente ditos e dos FC nele contidos. E, desta forma, determinar diferentes áreas de atividades antrópicas pretéritas, como áreas residenciais, de atividades agrícolas ou de descarte de materiais.

1.1.3 Objetivos Específicos

- Caracterização mineralógica e química dos solos TPA, TMA e AD, buscando relações pedogenéticas entre si;

- Identificação dos padrões geoquímicos multi-elementares nas TPA, TMA e solos adjacentes que permitam caracterizar cada tipo de solo e a contribuição humana pretérita;
- Determinação dos aspectos técnico-estilísticos dos fragmentos cerâmicos, na tentativa de reconhecer a filiação cultural e importância para a arqueologia da Amazônia;
- Caracterização mineralógica e química dos fragmentos cerâmicos, para auxiliar na identificação das matérias-primas de produção, bem como de sua área fonte;
- Identificação de padrões de ocupação humana antigos, com base nas associações geoquímicas, mineralógicas e na distribuição dos FC;
- Estabelecer a cronologia da ocupação humana pretérita com base em idades de radiocarbono dos solos e FC.

1.2 A ÁREA DE ESTUDO

1.2.1 Localização Geográfica

A área selecionada para desenvolvimento deste estudo compreende dois sítios arqueológicos com TPA (Terra Preta-1 e Terra Preta-2, aqui identificados como TPA-1 e TPA-2) circundados por solo de Terra Mulata (TMA-1 e TMA-2) e solos da Área Adjacente (AD). A área, predominantemente de Latossolos, está situada na margem direita do rio Amazonas, no município de Juruti, região do Baixo Amazonas, no extremo oeste do estado do Pará (Figura 1).

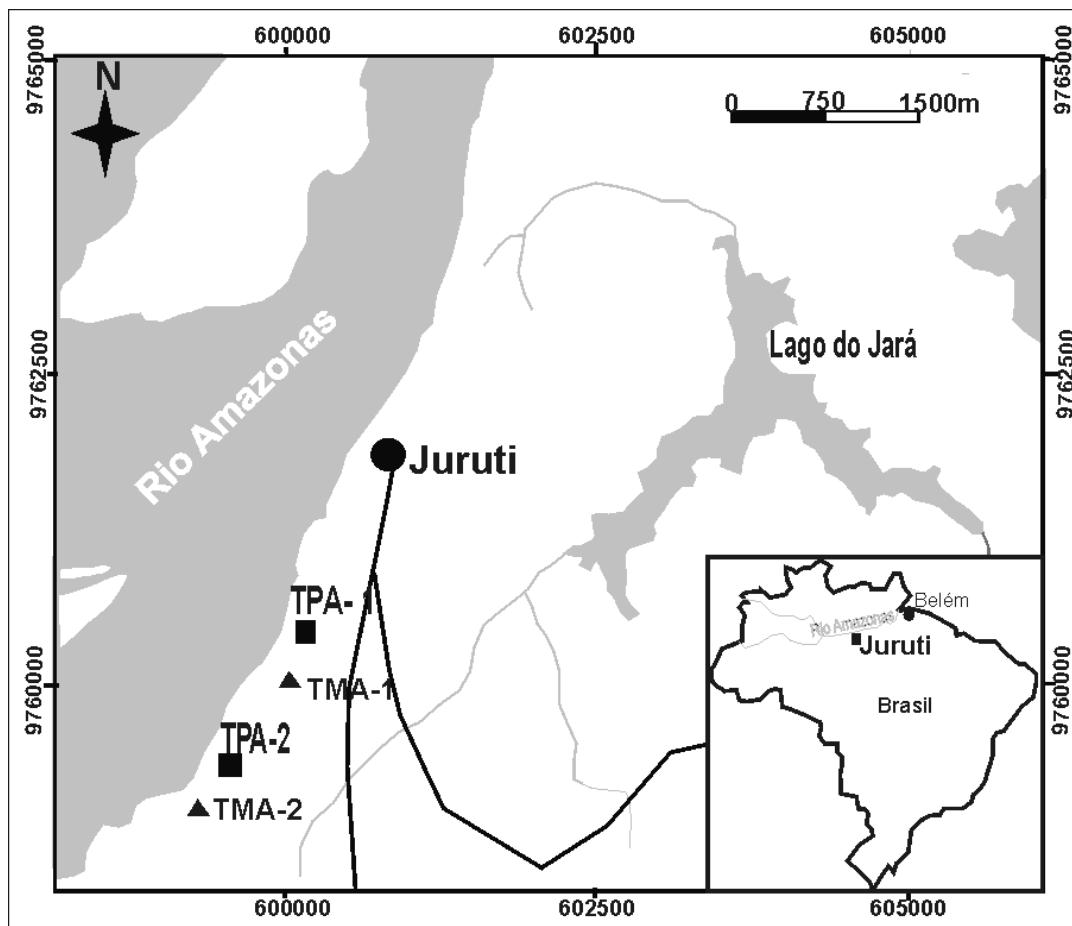


Figura 1- Mapa de localização da área de estudo, com indicação dos sítios TPA-1 (coordenada central: 21M 600159E/9760433N) e TPA-2 (coordenada central: (21M 599557E/9759353N), em torno dos quais ocorre a TMA.

1.2.2 Aspectos Climáticos

A área encontra-se em pleno domínio climático Equatorial Continental Amazônico, que combina calor e umidade elevados. A temperatura varia de 31,2°C a 23,8°C mas, dada as condições do tempo muito úmido e com nebulosidade elevada, os registros mais freqüentes ficam entre 25 e 28°C. A umidade relativa elevada varia entre 85 e 100% e a precipitação média anual é de 2100 mm (Lucas 1997; CNEC 2002).

1.2.3 Relevo e Hidrografia

Segundo o mapa de unidades de relevo do Brasil (IBGE 1993), a região estudada se situa na Depressão do Amazonas e na Planície fluvial e flúvio-lacustre do rio Amazonas. Os tipos de relevo constituinte da Depressão do Amazonas são as rampas com superfícies horizontais remanescentes da superfície sul-americana, as escarpas e as colinas pequenas, que representam os remanescentes dissecados da superfície de aplanamento Plio-Pleistocênica. As

planícies fluviais constituem o compartimento de relevo da planície fluvial e fluvio-lacustre do rio Amazonas. Os sítios arqueológicos TPA-1 e TPA-2, bem como as TMA e o solo circundantes estão sobrepostos à unidade de relevo de rampas com superfícies horizontais margeadas por planícies fluviais e flúvio-lacustres do rio Amazonas (Costa 2008). A área é drenada pela bacia hidrográfica do rio Amazonas, que tem como sub-bacia o igarapé Juruti Grande. O rio Amazonas é de água branca e se caracteriza pela instabilidade de seu leito, modificando o seu curso pela ação simultânea dos processos de erosão e de sedimentação, um dos principais responsáveis pela perda de parte dos sítios TPA-1 e TPA-2, em decorrência da erosão dos barrancos.

1.2.4 Cobertura Vegetal

A área é dominada por formações florestais que se diferenciam em Florestas Ombrófilas das Terras Baixas e Submontanas, características das vertentes e dos platôs (terra firme) respectivamente, e Florestas Aluviais, às margens do rio Amazonas. Na terra firme predomina a Floresta Ombrófila Densa, com grande riqueza em espécies. As formações vegetais de possível origem antropogênica na terra firme amazônica incluem as dominadas por palmeiras, florestas de bambu, florestas com alta densidade de castanheiras e matas de cipó são espécies indicadoras de perturbação e ocorrem associadas a carvão e fragmentos de cerâmica caracterizando vestígios de assentamentos (Balée 1989, 1993; Morán 1990). As Florestas Secundárias também ocupam as várzeas, apresentam uma riqueza bem razoável quando comparada com as de terra firme, e mostram que os estágios sucessionais entre as duas unidades de vegetação são bastante diferenciados (CNEC 2002).

1.2.5 Geologia

A região de Juruti, onde se situa a área de estudo compreende rochas sedimentares cretáceas da Formação Alter do Chão da bacia geológica do Amazonas, que afloram principalmente nos barrancos íngremes do rio Amazonas, em sua margem direita, às proximidades da cidade de mesmo nome (Costa 1991; Lucas 1997). Sobre estas rochas desenvolveram-se, entre o Paleoceno e o Mioceno, perfis lateríticos profundos, no topo dos quais ricos depósitos de bauxita (minério de alumínio), em parte lavrados pela ALCOA. Por sua vez, as bauxitas foram recobertas por material argiloso amarelo ocre, localmente espesso, conhecido como Argila de Belterra, que corresponde aos Latossolos Amarelos (Lucas 1997). Nas áreas mais arrasadas pela erosão se sucedeu a formação de espessos perfis de Latossolo

Amarelo derivados das rochas sedimentares saprolitizadas da Formação Alter do Chão e de perfis lateríticos imaturos (Costa 1991; Vasquez *et al.* 2008; Costa *et al.* 2010).

1.2.6 Solos

Na Terra Firme predominam os Latossolos Amarelos de textura média associados a Neossolos Quartzarênicos ou a Latossolos Amarelos de textura argilosa. Localmente, próximo ao rio Amazonas, destacam-se solos com horizonte A escuro, contendo fragmentos de cerâmica, objeto de estudo do presente trabalho, equivalentes à TPA e TMA, em Latossolo Amarelo. Segundo o Sistema Brasileiro de Classificação de Solos (EMBRAPA 1999), os solos TPA e TMA correspondem ao Latossolo Amarelo Tb mesotróficos, textura média, A antrópico. O solo AD compreende o Latossolo Amarelo Tb distrófico, textura média, A moderado. Na Classificação de Solos Antrópicos (Kampf *et al.* 2010), os solos TPA correspondem ao Arqueoantrossolo Hórtico-ebânico, mesotrófico, franca, ferrálico, enquanto, as TMA ao Arqueoantrossolo Ágrico-ebânico, dístico, franca, ferrálico.

Na várzea ocorrem solos hidromórficos, particularmente Gleissolos, associados, em pequena proporção, a Neossolos Flúvicos, ambos originados de sedimentos recentes aluvionares do rio Amazonas.

2 REVISÃO BIBLIOGRÁFICA

2.1 AS TERRAS PRETAS AMAZÔNICAS

Na Amazônia é considerável a ocorrência de áreas com solos de Terra Preta de Índio, Terra Preta (TP) ou Terra Preta Arqueológica (TPA) (Kern & Kämpf 1989; Kämpf *et al.* 2003, 2010; Kämpf & Kern 2005). Embora as TPA sejam descritas com maior freqüência na Amazônia Brasileira, estes solos também são reconhecidos na Bolívia, Equador, Colômbia, Guiana, Peru e Venezuela (Kern *et al.* 2003, 2010).

Os primeiros relatos a cerca das TPA remontam à segunda metade do século XIX, porém, somente a partir da última década do século XX esses solos, de fato, despertaram o interesse da comunidade científica. Desde então pesquisadores de instituições de diversos países e áreas de conhecimentos se uniram em grupos de pesquisa multi e interdisciplinares que tiveram como resultado imediato a realização de vários projetos e um significativo aumento de publicações (Myers *et al.* 2003; Kern *et al.* 2010).

O geólogo canadense Charles F. Hartt foi o primeiro a observar e registrar as Terras Pretas durante uma expedição no Amazonas entre os anos de 1870 e 1871. Em 1874, Hartt realizou pesquisas na região do Baixo Tapajós, onde descreveu as escavações realizadas no sítio Taperinha. Em 1879, Hartt e seu assistente Herbert Smith identificaram uma conexão clara entre Terra Preta e aldeias indígenas (Roosevelt 1992, Kern *et al.* 2010). No início do século XX, entre 1923-1926, o antropólogo Curt Nimuendaju produziu os primeiros mapas indicando a localização das TPA, área da região de Santarém, no estado do Pará. Em 1944, o alemão Friedrich Katzer reconheceu a fertilidade desses solos e realizou as primeiras análises químicas de solos de Terras Pretas de Santarém (Myers *et al.* 2003; Kern *et al.* 2010). Nas décadas seguintes os estudos pedológicos e arqueológicos impulsionaram as abordagens quanto à fertilidade e provável origem das TPA. Nesse período distintas hipóteses de formação lhes foram atribuídas, desde eventos geológicos, cinzas vulcânicas, fundos de lagos extintos (Barbosa de Faria 1946; Cunha Franco 1962; Falesi 1972, 1974), a áreas de antigas aldeias indígenas (Hilbert 1955; Smith 1980; Sombroek 1966; Hilbert & Hilbert 1980; Simões & Correa 1987; Kern & Kämpf 1989; Roosevelt 1992). Esta última interpretação é atualmente a mais aceita.

Os resíduos orgânicos acumulados durante a permanência do homem pré-histórico contribuíram para a formação e modificações significativas no solo. De modo que Ranzani *et al.* (1962), Sombroek (1966), Falesi (1972 e 1974), Bennema (1977), Smith (1980), Kern & Kämpf (1989), Pabst (1991), Kern (1996) e Lehmann *et al.* (2003) confirmaram a fertilidade

elevada desses solos em relação àqueles comumente encontrados na região. Em Cachoeira-Porteira, Pará, trabalhos sistemáticos efetuados em três sítios com TPA, com teores elevados de C orgânico, Ca, Mg, P, Zn e Mn indicaram áreas preferenciais para a deposição de dejetos (Kern 1988; Kern & Kämpf 1989; Kämpf & Kern 2005). Resultados semelhantes foram observados nas TPA de Caxiuanã, onde os teores desses mesmos elementos eram significativamente mais elevados que nos solos circunvizinhos (Kern 1996).

De modo geral, as TPA costumam apresentar altos teores de nutrientes e C orgânico, soma e saturação de bases moderadas ou elevadas, além de diversidade microbiológica. São também mais estáveis e melhores estruturados em relação às adjacências. São encontrados principalmente em Latossolos (Oxisols) e Argilossolos (Ultisols), que juntos recobrem aproximadamente 70% dos solos da Amazônia (Smith 1980; Rodrigues 1996; Pabst 1991; Kern *et al.* 2003; Sombroek *et al.* 2002; Lehmann *et al.* 2003).

Nas cartas de solos que abrangem a região Amazônica, apesar da freqüente ocorrência de solos com TPA, estes são catalogados como inclusões, pois ocupam em média até 5 ha. Excepcionalmente, em alguns locais, podem alcançar áreas superiores a 100 ha (Kern *et al.* 2003; Sombroek *et al.* 2002). Em Caxiuanã, no Pará, os sítios com TPA apresentaram entre 0.19 e 3.82 ha (Costa 2003). Em Belterra, próximo à cidade de Santarém foram mapeadas áreas com cerca de 200 ha de TPA (Sombroek 1966). Nas proximidades das TPA registram-se solos tipo Terra Mulata. Estes solos comparados com as TPA apresentam também coloração escura; conteúdo elevado de matéria orgânica; menores teores de P e nenhum ou muito pouco material arqueológico (fragmentos de cerâmica e de material lítico). A gênese da TMA é atribuída ao intenso uso agrícola por comunidades indígenas pré-históricas (Sombroek 1966; Sombroek *et al.* 2002; Kern *et al.* 2003).

De acordo com Kern (1996), Kern & Costa (1997), Lima *et al.* (2002), Kern *et al.* (2003) e Kämpf & Kern (2005) o horizonte A das TPA apresenta coloração escura, podendo variar de preta a bruno escuro (N2/; 2,5YR2/0; 5YR2,5/1; 7,5YR 2/0; 10YR 2/0 a 3/4), a textura varia de arenosa a argilosa, alta densidade de fragmentos de cerâmica e/ou material lítico em relação às áreas adjacentes. Kern *et al.* (2003) mostram que a espessura do horizonte antrópico ou do refugo ocupacional, em 57% dos sítios arqueológicos analisados, varia de 30 a 60 cm, podendo eventualmente chegar a 2 m, enquanto que a espessura dos solos de floresta não antrópicos, geralmente é 10 a 15 cm. As variações na profundidade podem ser resultado de processos de melanização pelo escurecimento do horizonte superficial em função do descarte irregular de matéria orgânica e processos de bioturbação (Kämpf *et al.* 2003; Kämpf & Kern 2005).

As TPA apresentam em média 38,9 g kg⁻¹ de C orgânico; 507 mg kg⁻¹ de P disponível; 9,4 cmol_c kg⁻¹ de Ca+Mg e pH 5,6, contrapondo-se às médias dos Latossolos e Argissolos da Amazônia que são de apenas 1,6 g kg⁻¹ de C orgânico; 1,7 mg kg⁻¹ de P disponível; 1,4 cmol_c kg⁻¹ de Ca+Mg e pH 4,5 (Kämpf & Kern 2005). Na região, cerca de 90% dos solos apresenta deficiência em fósforo (Rodrigues 1996) enquanto que na TPA o P disponível ou total se destaca com alto teor, e foi interpretado como um dos mais importantes indicadores de ocupação humana passada. São valores muito variáveis. Por exemplo, cerca 7.455 mg kg⁻¹ de P disponível foram encontrados nas TPA de Itaituba (Oliveira Jr *et al.* 2002), mas apenas de 30 a 448 mg kg⁻¹ em Cachoeira Porteira (Kern 1988), ambas no estado do Pará. Os teores anômalos de P e Ca são atribuídos a resíduos de origem animal, principalmente ossos e excrementos (Smith 1980; Kämpf *et al.* 2003; Kämpf & Kern 2005).

De acordo com Glaiser *et al.* (2003), as TPA são formadas por um grande depósito estável de matéria orgânica, contendo aproximadamente 30% de carbono preto, originado da queima incompleta da biomassa, sendo ele provavelmente o responsável pela grande capacidade de estoque de nutrientes no solo. Os conteúdos máximos de carbono orgânico foram encontrados nas TPA de Trairão e Belterra: chegam a alcançar valores de 243 e 210 g kg⁻¹, respectivamente (Oliveira Jr *et al.* 2002; Pabst 1991). A matéria orgânica contidas nas TPA, além de diferir em termos de quantidade, também difere em sua estruturação: é mais estável e ainda mais rica em componentes organo-metálicos que os Latossolos da Amazônia (Pabst 1991; Lehmann *et al.* 2003).

Kern (1996) e Kern & Costa (1999) apontam a associação geoquímica P₂O₅-MgO-CaO-K₂O-Ba-Cu-Cl-Mn-Sr-Zn como típica de solos de terra preta, relacionados à atividade humana pretérita, ao passo que Fe₂O₃-Na₂O-As-Cd-Co-Cr-F-Ga-Pb-V-B-Hg-Nb-Sc-Y-Zr indica assinatura dos solos adjacentes, insinuando tratar-se do substrato das TPA.

2.2 A CERÂMICA ARQUEOLÓGICA DO BAIXO AMAZONAS

As tentativas de estabelecer uma cronologia de ocupação para região Amazônica são limitadas pelas condições climáticas, especialmente temperatura e umidade elevada, responsáveis pela rápida decomposição dos vestígios de origem orgânica, como habitações, ferramentas, armas, recipientes, vestuários e ornamentos. Os vestígios inorgânicos como objetos de pedra são escassos e desta forma não permitem a obtenção de informações confiáveis sobre cronologia e proveniência. No entanto, a cerâmica é abundante, e aparentemente duradoura e costuma ser representada por inúmeros fragmentos. Esses relictos,

em sua maioria não decorados, são as principais evidências dos antigos habitantes (Meggers 1990).

O aparecimento da cerâmica, de fato, representa um impulso tecnológico que se estabeleceu como elemento necessário para o processamento e/ou o armazenamento de algum tipo de alimento, não significando que esse fato possa ser ligado mecanicamente à origem da agricultura, pois esta invenção também pode ter surgido para satisfazer a necessidade de manipulação de plantas selvagens (Scatamacchia 1991).

Nas Américas, a cerâmica mais antiga está relacionada ao sítio Taperinha, no Baixo Amazonas, com cerca de 7.000 anos AP (Roosevelt 1992). Esta região é reconhecida como um importante centro de produção das mais belas e refinadas cerâmicas, a exemplo dos estilos Marajoara, Tapajônico, Globular, Konduri, Pocó, Parauá, entre outras (Hilbert 1955; Hilbert & Hilbert 1980; Roosevelt 1992; Gomes 2008; Guapindaia 2008).

Segundo informações provenientes de pesquisas arqueológicas realizadas por Hilbert (1955), Simões (1984) e Roosevelt (1992) a maior concentração de grupos ceramistas pré-coloniais, especialmente dos estilos Tapajônico e Konduri, ambos pertencente à Tradição Incisa Ponteada, ocorre desde as proximidades da foz do Rio Nhamundá até o Xingu, por ambas as margens do rio Amazonas. Para Simões (1984) a falta de pesquisas com escavações estratigráficas nas áreas dos rios Tapajós e Nhamundá-Trombetas fez com que a cerâmica e outros artefatos da cultura Tapajônica e Konduri fossem tratados como complexos cerâmicos distintos. Nesta área, Curt Nimuendaju identificou sítios contendo cerâmica Tapajônica e Konduri, e concluiu que a região Trombetas-Nhamundá foi o local de origem da cultura Konduri (Hilbert 1955), definida com base em aspectos estilísticos. Por outro lado, resultados oriundos de estudos de coleções museológicas apontam alguns elementos comuns no que tange à forma de organização social das culturas Tapajônica e Konduri (Hilbert 1955; Gomes 2002).

A cerâmica Tapajônica caracteriza-se pelo uso de apliques estilizados lembrando animais (pássaros, répteis e mamíferos) e figuras humanas, além de olhos tipo grão de café, raras trípodes e bordas vazadas combinadas aos inúmeros modelados, incisões e ponteados (Hilbert 1955; Simões 1984; Prous 1992). Embora a caracterização estilística induza à homogeneidade pelas generalizações, Guapindaia (1993) ao estudar a cerâmica Tapajônica da coleção “Frederico Barata”, identificou três grupos cerâmicos distintos, denominados cerâmica tipicamente tapajó, cerâmica com influência tapajó e cerâmica de contato, demonstrando variabilidade tecnológica por provável mudança cultural.

Por sua vez, a cerâmica Konduri descrita por Hilbert (1955), Hilbert & Hilbert (1980) e Guapindaia (2008) na bacia dos rios Trombetas-Nhamundá, compreende uma cerâmica excessivamente temperada com cauixi, vasos globulares, tigelas, pratos e grelhas, bases planas, anelares, em pedestal e abundância de trípodes. Os motivos decorativos são constituídos por incisões e ponteados, com ênfase na elaboração de adornos de borda e alças, sendo traço diagnóstico a modelagem de adornos biomorfos, muito elaborados. Porém, a alta freqüência de cariapé (27,12%) e rocha triturada (10,61%), identificada por Guapindaia (2008), diverge dos parâmetros inicialmente relacionados a esse estilo.

Nessa mesma área, Hilbert & Hilbert (1980) descreveram sítios contendo cerâmica Konduri nos níveis superficiais e Pocó nos níveis mais profundos. A fase Pocó foi descrita como temperada com cauixi, cariapé ou ambos, com superfície bem alisada, parcialmente polida, cor predominantemente laranja e marrom-médio, decorada com engobo vermelho e branco, pintura vermelha e incisões espiraladas. Este estilo compõe a Tradição Incisa Modelada ou Barrancóide, que abrange principalmente as bacias do Orenoco, além do Médio e Baixo Amazonas (Lathrap 1970; Hilbert & Hilbert 1980; Lima *et al.* 2006). A análise do material cerâmico do sítio Boa Vista, da região de Trombetas apresenta características compatíveis para cada uma das ocupações, Konduri e Pocó, confirmadas por datações (Guapindaia 2008). No entanto, a clara diferenciação de datas e características tecnológicas, não se repete em termos estratigráficos, pois a mudança entre os dois estilos é imperceptível a olho nu, visto que a camada de terra preta é contínua da superfície até cerca de 60 cm de profundidade. Porém, em algumas áreas do sítio a TPA aparece como bolsões com cerâmica tipicamente Pocó.

2.3 QUÍMICA E MINERALOGIA DA CERÂMICA ARQUEOLÓGICA

Além da importância como registro cultural, investigações mineralógicas e químicas de fragmentos cerâmicos têm contribuído para o avanço das interpretações arqueológicas, trazendo novas perspectivas de abordagens. Esses dados contribuem na identificação de aspectos tecnológicos da cadeia de produção, como pigmentos, antiplásticos, temperatura de queima, fonte e procedência das matérias-primas utilizadas (Ruvalcaba-Sil *et al.* 1999; Mirti *et al.* 2004; Hein *et al.* 2004). Além destes, a presença de fosfatos nos fragmentos cerâmicos tem levantado questionamentos sobre a origem do fósforo (Duma 1972; Freestone *et al.* 1994; Costa *et al.* 2004a, 2004b, 2006, 2010).

A análise de vasos cerâmicos provenientes de sítios arqueológicos europeus, portanto sem ocorrência de solo TPA, realizada por Duma (1974) apontou que os teores de P_2O_5 (0,09

a 0,6 %) variavam conforme o uso de substâncias orgânicas. No entanto, Freestone *et al.* (1994) defendem que as elevadas concentrações de P na cerâmica arqueológica não representa um indicador de uso. Elas são principalmente reflexos do ambiente pós-deposicional e da microestrutura da cerâmica, e não necessariamente refletem adsorção de substâncias orgânicas durante a fabricação ou uso da cerâmica.

Por outro lado, Costa *et al.* (2004a, 2004b, 2006, 2010) descrevem fragmentos cerâmicos de sítios com TPA da região Amazônica com conteúdos relativamente elevados de fósforo, entre 1 e 2 % de P_2O_5 , podendo chegar a 9 % em sítios do litoral. Além disso, análise semi-quantitativa (MEV/EDS) realizada por esses pesquisadores permitiu, ainda, identificar material criptocristalino, geralmente constituído por P, Al e algumas vezes Fe, indicando fosfatos de alumínio amorfo ou criptocristalinos do tipo variscita-estrengita ($Al, Fe)(PO_4).2(H_2O)$) na cerâmica do Baixo Amazonas, enquanto que nos FC do litoral paraense a formação de crandalita-goyazita, $(Ca, Sr)Al_3(PO_4)_2(OH)_5$, é mais expressiva.

A composição mineralógica dos fragmentos cerâmicos da Amazônia é constituída basicamente de quartzo, caulinita, illita, albita, muscovita, anatásio, óxidos de ferro (maghemita ou hematita) e anfibólio (Costa *et al.* 2004a). Os fragmentos cerâmicos de Cachoeira Porteira, no Baixo Amazonas, apresentaram 12,2 % de albita e 5,3 % de fosfatos (variscita), enquanto que no sítio Manduquinha, região de Caxiuanã, as concentrações caíram para 3 % de albita e 2,9 % de P_2O_5 (Costa *et al.* 2006). Para Costa *et al.* (2004a, 2004b, 2006, 2010), Rodrigues (2010) e Silva (2010) os altos teores de fósforo encontrado nas cerâmicas, são provenientes dos alimentos durante o processo de cozimento. Além disso, defendem que a fertilidade dos solos TPA, principalmente a fonte principal de macro e micronutrientes, esteja em grande parte relacionada à gradual decomposição natural dos fragmentos durante a formação e evolução desses solos.

3 MATERIAIS E MÉTODOS

3.1 MATERIAIS

Os materiais analisados neste estudo compreendem amostras de solos e fragmentos cerâmicos (FC) coletados nas áreas dominadas por solos TPA, TMA e AD, além de amostra de argila utilizada atualmente como matéria-prima na produção de vasilhas cerâmicas. Todas as amostras são provenientes do município de Juruti, estado do Pará.

3.2. AMOSTRAGEM

3.2.1. Coleta dos solos

Após o mapeamento das TPA e TMA da área de estudo em Juruti, abriram-se 5 trincheiras assim distribuídas: duas nas terras pretas (TPA-1 e TPA-2), duas na terra mulata (TMA-1 e TMA-2) e uma na área adjacente (AD) (Figura 2). Em seguida foram descritos os perfis de solos ao longo destas trincheiras e coletadas 30 amostras seguindo os horizontes pedológicos principais. A descrição morfológica e coleta das amostras obedeceram aos procedimentos propostos por Lemos & Santos (2002) e as cores do solo foram identificadas por comparação com carta de Munsell (2000) (Figura 3). Os solos também foram coletados com auxílio de sondagens de 50 x 50 cm em uma malha regular de 60 x 120 m (Figura 2), compreendendo 43 amostras de TPA, 90 de TMA e 1 na AD, restritas ao intervalo 10 a 20 cm de profundidade, correspondente ao horizonte pedológico A₂, onde geralmente se concentra a maior quantidade de fragmentos cerâmicos (FC) nas TPA. A coleta de amostras aconteceu entre 2006 e 2007 durante o resgate arqueológico realizado pela empresa Scientia Consultoria Científica Ltda.

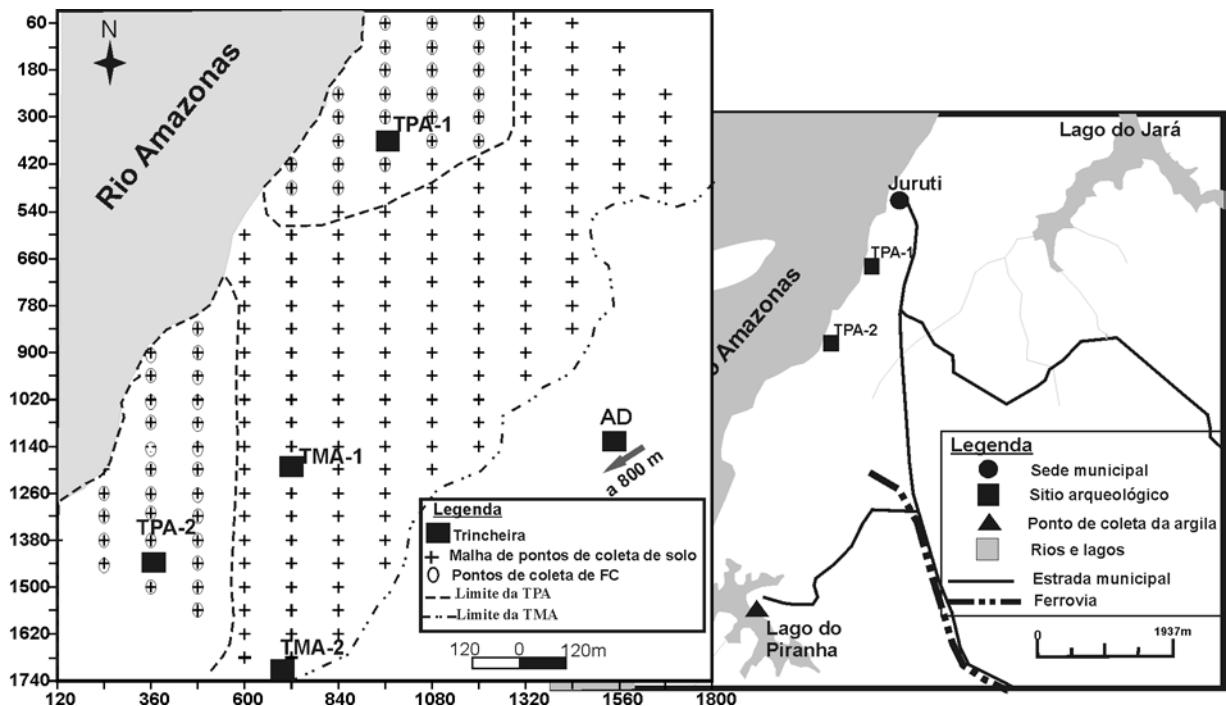


Figura 2 – Mapa da malha de coleta das amostras de solo e fragmentos cerâmicos (FC) nos sítios TPA-1 e TPA-2. Além da indicação do ponto de coleta da argila, no Lago do Piranha (coordenada central: 21M 598464E/9755450N).



Figura 3 - Descrição, coleta e determinação das cores das amostras de solo seguindo os horizontes pedológicos.

3.2.2. Coleta dos fragmentos cerâmicos

Os fragmentos cerâmicos foram coletados com auxílio de sondagens ou pequenas trincheiras medindo 50 x 50 cm, segundo uma malha regular e sistemática (60 x 120m). As coletas foram realizadas em intervalos de 10 cm, até o término da ocorrência de fragmentos cerâmicos. Em geral em cada nível de profundidade foram coletados de 1 a 22 FC, cujo tamanho variava de 2 a 15 cm (Figuras 3 a 5) (Scientia 2008).



Figura 4 - Vista da escavação em sondagem da malha regular. Foto: Scientia Consultoria.



Figura 5 - Fragmentos de cerâmica arqueológica coletados nos TPA-1 e TPA-2. Foto: Scientia Consultoria.

3.2.3 Coleta de argila

Na tentativa de identificar as matérias-primas empregadas na confecção dos artefatos cerâmicos arqueológicos, das quais derivaram os FC, coletou-se à margem do Lago do Piranha, a 15 Km da cidade Juruti, uma amostra de argila “in natura” (Figura 2). A coleta desta argila foi feita com base em levantamento etnográfico, após pesquisar as atividades ceramistas que persistem na área e que fazem uso de técnicas similares às que foram encontradas nos fragmentos arqueológicos. Segundo a história oral, a argila do referido lago corresponde à fonte de matéria-prima da atual indústria ceramista.

3.3 ANÁLISES DE LABORATÓRIO

3.3.1 Descrição macroscópica

Os fragmentos cerâmicos foram individualmente numerados e analisados sob lupa binocular ZEISS, onde foram identificados seus principais atributos técnico-estilísticos como: técnica de confecção, antiplástico, queima, acabamento, decoração, forma e volume de vasilhas, bem como a qual parte do artefato pertencia: borda, parede (corpo) ou base (Scientia 2008). No total foram analisados nesta etapa 6.300 fragmentos cerâmicos (FC). Estes procedimentos foram realizados nos laboratórios da empresa Scientia Consultoria Ltda.

Após a descrição, vinte e quatro fragmentos (doze amostras para cada sítio, coletadas entre 0 e 60 cm de profundidades) sem decoração, todos da categoria parede (corpo do pote), foram selecionados, para as determinações mineralógicas e caracterização química (Tabela 1).

Tabela 1: Relação das amostras de fragmentos cerâmicos química e mineralogicamente investigados

Sítio	TPA-1	TPA-2
Nível (cm)	Amostras	
0-10	TP1-32	TP2-7, TP2-12, TP2-18 TP2-1, TP2-5, TP2-11, TP2-
10-20	-	14, TP2-17, TP2-21
20-30	TP1-28, TP1-33 TP1-30, TP1-31, TP1- 36, TP1-37, TP1-40,	TP2-6, TP2-24, TP2-25
30-40	TP1-42, TP1-44	-
40-50	TP1-39	-
50-60	TP1-43	-

3.3.2 Difração de raios X (DRX)

Para determinação das fases mineralógicas presentes, as amostras de solo, fragmentos cerâmicos e argila, previamente pulverizados, foram submetidos à difração de raios X, segundo o método do pó. Foi utilizado o difratômetro XPERT PRO MPD equipado com goniômetro PW 3040/60 (theta-theta) PANalitical, com tubo de raio-x cerâmico de anodo de cobre ($\bullet\text{CuK}\bullet = 1,54060\text{\AA}$) e condições de operação de 40 kV e 30mA. Os dados obtidos foram interpretados com auxílio do banco de dados ICDD e o software X'pert HighScore versão 2.1b, também da PANalitical. Estes procedimentos foram realizados no laboratório de difração de raios X do grupo de Mineralogia e Geoquímica Aplicada do Instituto de Geociências da UFPA.

3.3.3 Microscopia eletrônica de varredura (MEV)

As determinações mineralógicas nas amostras de solo e FC foram complementadas por análises em microscópio eletrônico de varredura, acoplado com de dispersão de energia (MEV-EDS), o que permitiu capturar imagens e realizar determinações químicas micropontuais. As amostras foram secas a 50°C durante 4 horas, assentadas sobre plataformas de alumínio, com uma fita adesiva de carbono dupla face e metalizadas com ouro. O detector de retroespalhamento eletrônico permitiu elaborar mapas de distribuição das concentrações químicas. O equipamento utilizado foi o microscópio modelo LEO 1450 VP 500 DP e detector de SED Gresham, utilizando software IXRF. Estes procedimentos analíticos foram executados no laboratório de Microscopia Eletrônica do Museu Paraense Emílio Goeldi.

3.3.4 Microscopia óptica

Amostras de solo foram agregadas com resina Araldite da série 750 e endurecedor da série HY951 para confecção de lâminas delgadas. O estudo das lâminas foi realizado com auxilio de microscópio Zeiss Axiolab 450910, com lentes de aumento de 10, 20, 50 e 100 vezes. Estes procedimentos foram conduzidos no Laboratório de Geologia Sedimentar do Instituto de Geociências da Universidade Federal do Pará.

3.3.5 Análises químicas

As amostras de solo provenientes das trincheiras, de FC e de sedimento foram submetidas a análises químicas totais que cobriram os elementos maiores e menores (SiO_2 , Al_2O_3 , Fe_2O_3 , MgO , CaO , K_2O , Na_2O , TiO_2 , P_2O_5 , MnO), Perda ao Fogo, bem como os elementos traços (Be, Sc, V, Cr, Co, Ni, Cu, Zn, Ga As, Rb, Sr, Y, Zr, Nb, Mo, Ag, Cd, Sn, Sb, Cs, Ba, Hf, Ta, W, Hg, Pb, Bi, Th, U e os elementos terras raras). As amostras foram fundidas com tetraborato e/ou metaborato de lítio, então solubilizadas e as concentrações dos elementos mencionados determinadas por ICP-OES e ICP-MS junto a Acme Analytical Laboratories Ltd. As amostras de solo coletadas na malha regular (60 x 120 m) foram submetidas a análises químicas parciais para a determinação das concentrações de Al_2O_3 , Fe_2O_3 , MgO , CaO , K_2O , Na_2O , TiO_2 , P_2O_5 , Mn, V, Cr, Ni, Cu, Zn, Sr, Y, Zr, Li, Pb e La. As amostras foram inicialmente digeridas em meio multiácido (ácido fluorídrico, nítrico, clorídrico e gotas de perclórico) e então analisadas por ICP-MS junto aos laboratórios da Lakefield Geosol Ltda. As amostras de solo foram ainda oxidadas com dicromato de potássio em meio sulfúrico e o valor de carbono orgânico obtido por volumetria (método Walkley-

Black) foi então multiplicado pela constante 1,724 e determinado o teor de matéria orgânica junto ao laboratório de solos da Universidade Federal de Viçosa.

3.3.6 Datação radiocarbônica e por termoluminescência

As datações por radiocarbono foram realizadas em amostras de carvão coletadas em contexto arqueológico. As análises foram realizadas no Laboratório Beta Analytic Incorporation, com auxílio de acelerador de partículas acoplado a um espectrômetro de massa (AMS). As idades C-14 foram então calibradas em anos antes do presente (AP) que tem como referência o ano de 1950. Estas análises foram obtidas por Scientia Consultoria Ltda (2008). A datação por termoluminescência (TL) em grão de quartzo foi realizada em apenas dois fragmentos cerâmicos nos quais foi empregado o método de dose acumulada e dose anual junto ao laboratório Datação, Comércio & Prestação de Serviço Ltda. As idades por TL foram então calibradas em anos antes do presente, cujo ano referência é o ano de realização da análise, neste caso, 2010.

3.3.7 Tratamento estatístico

Os resultados obtidos foram tratados estatisticamente com softwares Origin 6.1 e Surf 8.0. Para as análises de correlação e agrupamento (cluster) foi utilizado o Statistica 6.0.

4 RESULTADOS E DISCUSSÃO

Os resultados são apresentados e discutidos em três artigos, submetidos a periódicos internacionais. No primeiro, são discutidos os atributos mineralógicos e químicos de perfis de solo tipo Terra Preta e Terra Mulata como forma de entender os processos de sua formação; no segundo artigo, são identificadas as principais associações geoquímicas e sua distribuição areal, mostrando como estas contribuem na identificação e interpretação de padrões de assentamentos; e, finalmente, no terceiro artigo são analisados aspectos tecnológicos, químicos e mineralógicos dos fragmentos cerâmicos provenientes de sítios com TPA, as relações entre as cerâmicas antigas e as atuais em termos de matérias-primas comuns e procedência, bem como a cronologia de ocupação.

4.1 MINERALOGICAL AND GEOCHEMICAL CONTRASTS BETWEEN THE PROFILES OF THE ARCHAEOLOGICAL DARK EARTH AND TERRA MULATA SOILS OF THE LOWER AMAZON BASIN: CONTRIBUTIONS TO THE UNDERSTANDING OF THEIR ORIGIN

(Submetido ao periódico Geoderma)

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Abstract

At Juruti, on the right bank of the lower Amazon River, Archeological Dark Earth (ADE) sites are surrounded by an extensive area of soils called Terra Mulata (TMA). The ADEs can be recognized by the high levels of Ca, Mg, P, Cu, Zn, and Mn, organic C, ceramic fragments and charcoal, which indicate the presence of ancient pottery-making settlers. The TMAs present moderate levels of these elements and organic matter, but a complete absence of ceramic fragments. The mineralogical and chemical analyses of five soil profiles representative of ADE, TMA and adjacent latosol revealed that they are composed essentially of quartz and kaolinite. Apatite, Al phosphate, and cristobalite can also be found in the ADEs, but are rare in the TMAs. The principal chemical components are SiO₂ and Al₂O₃, which are found at similar levels in ADE, TMA and latosol. However, the anthropogenic soils can be distinguished from the latosol by their relatively high levels of P, Ca and Mg, while Mn, Cu, Zn, Ba, and Sr are also typical of the geochemical signature of the ADE and TMA soils at Juruti. These mineralogical and chemical variations in relation to the adjacent soils appear to have resulted from the discarding of organic residues, in particular bones and plant remains, and successive burnings. On the other hand, the overall abundance of quartz, kaolinite, goethite + hematite, and anatase, which are responsible to the high levels of Si, Al, Fe and Ti, demonstrate that the anthropogenic soils at Juruti retain the mineralogical and chemical signature of the latosol, the original substrate for the formations of both ADEs and TMAs.

Key words: Anthropogenic soils, Terra Mulata, Dark earth, Mineralogy, Geochemistry.

1. Introduction

Anthropogenic soils are widely-distributed in the Amazonian landscape. These soils, which are known as Indian Dark Earth or Archeological Dark Earth (ADE) are generally found in relatively small patches, but may sometimes, cover an area of hundreds of hectares (Kern et al., 2003). Whatever their size, these sites present evidence of modifications caused by their ancient inhabitants, resulting from the use of the soil, the availability of resources for the subsistence of the occupants, and their cultural habits.

The ADEs are characterized by their dark coloration, relatively high levels of Ca, Mg, P, Mn, Zn, and Cu, organic C, fragments of charcoal, and large quantities of ceramic material (Kern & Kampf, 1989) when compared with the region's predominant soils. In addition, areas of ADE are often surrounded by areas of Terra Mulata (Sombroek, 1966; Woods & McCann, 1999; Sombroek et al., 2002; Kern et al., 2003). This type of soil was first described by Sombroek (1966) at Belterra, south of the city of Santarém, in central Brazilian Amazonia, where an area of approximately 1000 ha of TMA encircles 200 ha of ADE. In comparison with ADE, TMA soils are lighter in coloration – grayish or burnished – and have medium concentrations of organic C, and little or no archeological material. They typically occupy relatively extensive areas surrounding patches of ADE.

The current consensus is that, while ADEs represent ancient settlements formed by prehistoric human occupants (Gourou, 1950; Hilbert, 1955; Sombroek, 1966; Kern & Kämpf, 1989; Neves et al., 2003), the TMA soils are the product of intensive agricultural activities conducted in the adjacent areas (Sombröek, 1966; Kern et al., 2003; Sombröek et al., 2002, 2010; Denevan, 2010). Based on this interpretation, the TMA soils would be the result of the deliberate application of organic matter of plant (residues, charcoal, and ashes) and animal origin (the remains of fish and game, i.e., bones, skin, and viscera, and mollusk shells) with the objective of improving the quality of the soil (Kern et al., 2003; Sombroek et al., 2010; Denevan, 2010).

Given the controversies with regard to the origin of the TMA soils, due to the relative lack of studies, the present paper focuses on the TMAs found in the region of Juruti, in Amazonia, where TMA soils are found in association with relatively large areas of ADE. The mineralogical and chemical analyses of these soils provide insights into their origin and their relationship with the associated ADE soils.

2. Study area

The ADE archeological sites selected for the present study – denominated ADE-1 and ADE-2 – are located on the right bank of the lower Amazon River in the municipality of Juruti (Fig. 1), in the western extreme of the Brazilian state of Pará (Scientia, 2008). These ADE sites are surrounded by an extensive area of TMA soils.

The archeological sites are found on terraces with horizontal surfaces, lower than the surrounding lateritic bauxite plateaus, and bordered by the Amazon floodplain and fluvial-lacustrine plains. The areas of ADE and TMA soils are dominated by yellow Latosols derived from the underlying lateritic bauxite formations, formed over the sandy-clayey saprolitic sediments of the Alter do Chão formation (Costa, 1991; Costa, 2008).

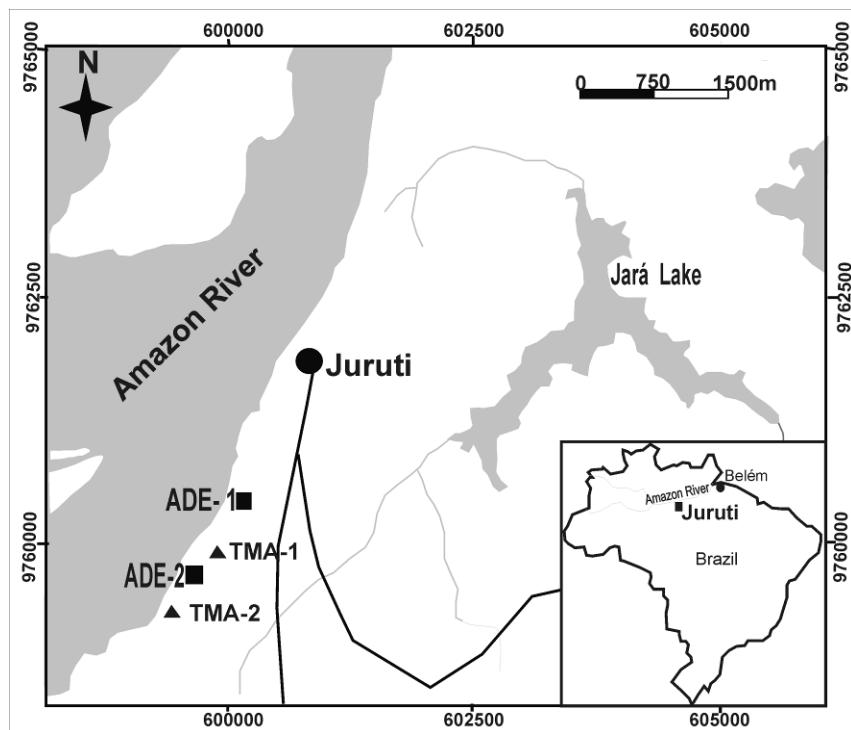


Fig. 1. Map of the study area, showing the two ADE sites and the surrounding area of TMA soil. The central coordinates of the trenches (UTM 21M) are ADE-1: 600159E/9760433N; ADE-2: 599557E/9759353N; TMA-1: 599917E/9759593N; TMA-2: 599377E/9759068N.

3. Materials and methods

Following the mapping of the ADE and TMA soils within the study area, five trenches were dug, one at each ADE site (ADE-1 and ADE-2), two within the area of TMA soil (TMA-1 and TMA-2), and one within the adjacent area, or Latosol (Fig 2). The soil profiles were described along these trenches, based on 30 samples collected from the principal pedological horizons. The morphological description was based on the scheme proposed by

Lemos & Santos (2002) and the soil colors were identified by comparison with a Munsell chart (2000).

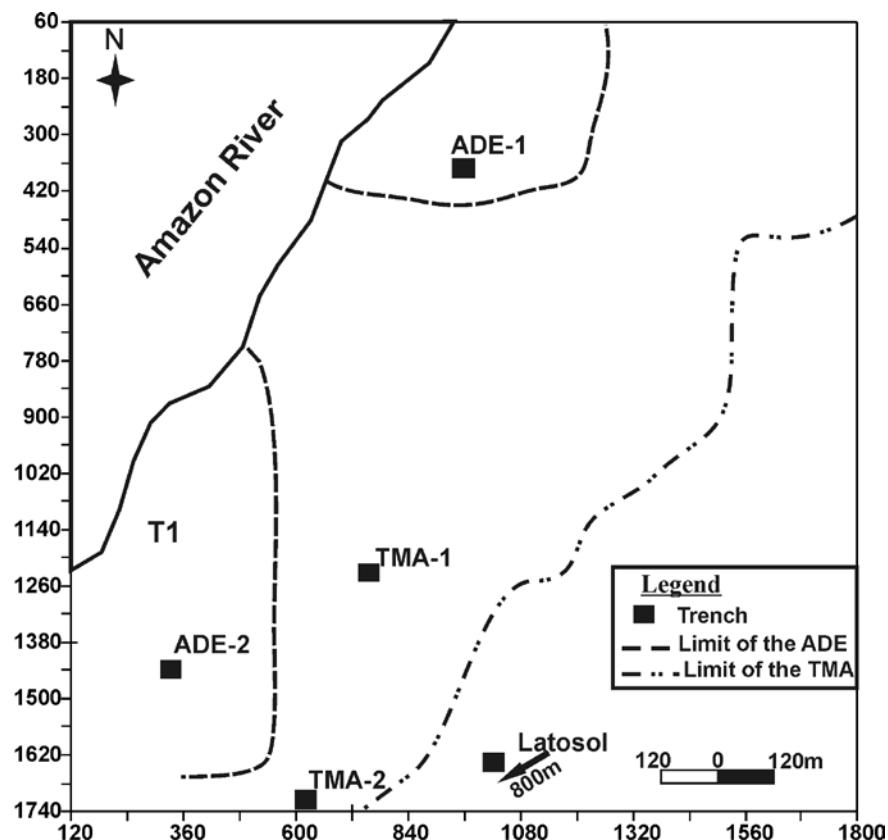


Fig. 2. Location of the soil sample collecting points in the trenches excavated within the study area (ADE-1, ADE-2, TMA-1, TMA-2, and Latosol).

3.1. Chemical and mineralogical analyses

The chemical and mineralogical analyses were conducted on samples pulverized in an agate mortar and then sieved through a <120 mesh. The mineralogical analyses were conducted in the X-ray diffraction laboratory of the UFPA Geosciences Institute, using an XPERT PRO MPD diffractometer equipped with a PW 3040/60 (theta-theta) PANalitical goniometer with a copper anode ($\bullet\text{CuK}\bullet 1 = 1.54060$) operating at 40 kV and 30mA. The data were obtained using an ICDD database and X'pert HighScore software (version 2.1b). These mineralogical measurements were complemented with analyses using scanning electron (SEM) and optical microscopes. The SEM analyses were carried out in the Goeldi Museum Electron Microscope Laboratory, using a LEO 1450 VP 500 DP microscope, while the optical analyses were conducted in the UFPA Geosciences Institute, using fine slides and Zeiss microscopes.

The chemical analyses of the total samples included the major and minor elements (SiO_2 , Al_2O_3 , Fe_2O_3 , MgO , CaO , K_2O , Na_2O , TiO_2 , P_2O_5 , and MnO) and Loss on Ignition, as well as trace (Be, Sc, V, Cr, Co, Ni, Cu, Zn, Ga As, Rb, Sr, Y, Zr, Nb, Mo, Ag, Cd, Sn, Sb, Cs, Ba, Hf, Ta, W, Hg, Pb, Bi, Th, and U) and rare earth elements (REEs), which were obtained by the tetraborate and lithium metaborate fusion method, and determined by Acme Analytical Laboratories Ltd. using ICP-OES and ICP-MS. The organic matter was estimated by multiplying by a factor of 1.724, the value of the organic carbon fraction obtained by oxidation with potassium dichromate in sulfuric medium and determined by volumetry (Walkley-Black method) in the soils laboratory of the Federal University of Viçosa.

4. Results and discussion

4.1. Characterization of the soil profiles

The ADE-1 profile presented A_1 , A_2 , A_3 , A_4 , AB, BA, and B horizons, with the same profile being found at ADE-2, except for the absence of A_4 . The A horizons reached a depth of 140 cm at ADE-1, and 40 cm at ADE-2 (Fig. 3). These A horizons are relatively sandy, with a moderate structure and diffuse transition between sub-horizons, with colors varying from black (2.5YR2.5/1) to very dark grayish brown (10YR3/2). In addition, charcoal, ashes, and an abundance of archeological ceramic fragments are common characteristics of these soils. The soil is lighter in color in the transition horizons (AB and BA), varying from dark brown (10YR3/3) to yellowish brown (10YR4/6). The texture is sandy loam, and charcoal and ceramic fragments are found sporadically.

Horizons A, AB, BA, B_1 , and B_2 were identified in the soil profile at TMA-1. The same sequence was found in the TMA-2 profile, except that it was possible to distinguish the A_1 , A_2 , and A_3 horizons (Fig. 3). The A horizons are 15 cm deep at TMA-1 and 44 cm deep at TMA-2. The texture is sandy loam, moderately structured, with diffuse transitions. Particles of charcoal are common. Horizon A is very dark gray (10YR3/2), varying to grayish brown and dark yellowish (10YR4/2 and 10YR4/4) in horizons AB and BA.

In the adjacent area, the sequence of horizons (A_1 , A_2 , AB, BA, B_1 , B_2) is similar to that of TMA-2, except for the absence of horizon A_3 (Fig. 3). The A horizons are 21 cm thick, that is, thinner than in either the ADE or TMA soils. The texture is sandy, the structure moderate, the transition between horizons diffuse, with occasional particles of charcoal, grayish brown (7.5YR3/2) in color in horizon A and dark yellowish brown (7.5YR4/2 and 7.5YR4/4) in horizons BA and AB.

Morphologically, the B horizons of the ADE, TMA, and adjacent soils are all very similar. The texture varies from sandy loam to sandy clayey, moderate structure and colors varying from yellowish brown (10YR5/6 and 7.5YR5/6) to brownish yellow (10YR6/8). Overall, then, the principal morphological differences between the anthropogenic soils, as well as those between these and the adjacent soils, are found primarily in the A horizons.

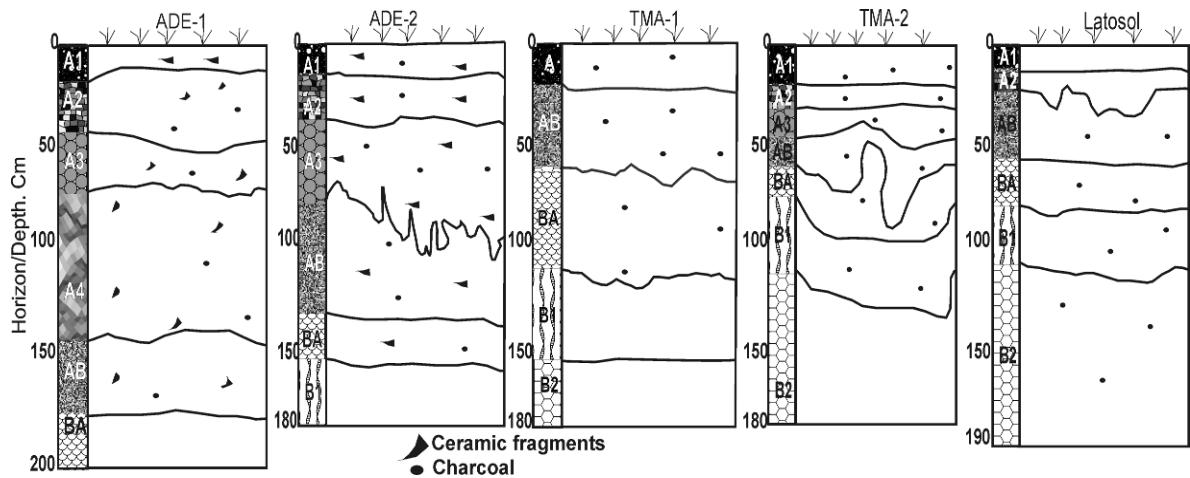


Fig. 3. Simplified profile of the horizons of the ADE, TMA, and Latosol.

4.2. Mineralogy

The principal mineral components in the ADE and TMA profiles identified by XRD were quartz and kaolinite. Anatase, muscovite + illite, goethite + hematite, apatite, and cristobalite were identified as accessory minerals. Apatite, $\text{Ca}_5(\text{PO}_4)_3(\text{OH})$, was identified only at the ADE sites and TMA-1. The stoichiometric calculations also indicated the presence of aluminum phosphate ($\text{AlPO}_4 \cdot 2(\text{H}_2\text{O})$), which is amorphous to XRD, but is found frequently in soils and ceramic fragments, which are rich in Fe and Al oxy-hydroxides (Smith 1980; Valadares et al., 2003; Costa et al., 2004a, 2004b, 2006, 2010). The assemblage of minerals is repeated in the adjacent soils, with the exception of cristobalite, apatite, and aluminum phosphate, which are restricted to the anthropogenic soils (Fig. 4 and 5). The mineral content, calculated by stoichiometry, indicates that quartz is predominant in all the soils, but decreases slightly towards the base profiles, whereas kaolinite increases, in the typical pattern for tropical soils (Fig. 5).

The presence of apatite in this profile indicates the presence of bone fragments, which were in fact found during the sieving of the samples (retained in the 1 mm sieve). This suggests that the area was subject to successive deposits of bony residues. Apatite is present in horizons A₁, A₂, A₄, and AB of ADE-1 (0.97 to 4.03%), A₁, A₂, and A₃ of ADE-2 (0.7 to

1.0%) and AB of TMA-1 (1.67%). The levels of aluminum phosphate corresponded to those of apatite in the horizons of ADE-1 (0.45 to 1.92% aluminum phosphate) and TMA-1 (1.05%). The presence of cristobalite indicates the calcination of amorphous silica, that is, opaline, which is common in the make-up of plants such as cauixi, as well as in the remains of ceramic vessels which contain cauixi and cariapé, which have been transformed into cristobalite during firing (Costa & Kern, 1999; Costa et al., 2011). This mineral is found principally in horizons A₃ to AB at ADE-1, horizons A₂ to BA at ADE-2, and in horizon A at TMA-2.

The low levels of hematite and goethite (1.6 to 4.0%) in the superficial A horizons indicate possibly intense activity of organic matter, which has reduced and decomposed these minerals from the levels found in the adjacent soils, which represent the condition prior to the formation of the ADE and TMA soils, and are equivalent to the yellow latosols encountered by the study area's first human settlers. Complementary analyses of the ADE and TMA soils using optical microscopy found only fractured grains of quartz and abundant fragments of charcoal, as well as isolated residues of Fe oxy-hydroxides found throughout the clayey kaolinitic matrix (Fig. 6).

The distribution of the content of the principal minerals within the soil profiles is equivalent in the ADE and TMA soils, but is slightly different in the adjacent profile. These mineralogical differences suggest modifications provoked by the different forms in which the environment was exploited by prehistoric humans during the settlement and long-term occupation of the study area.

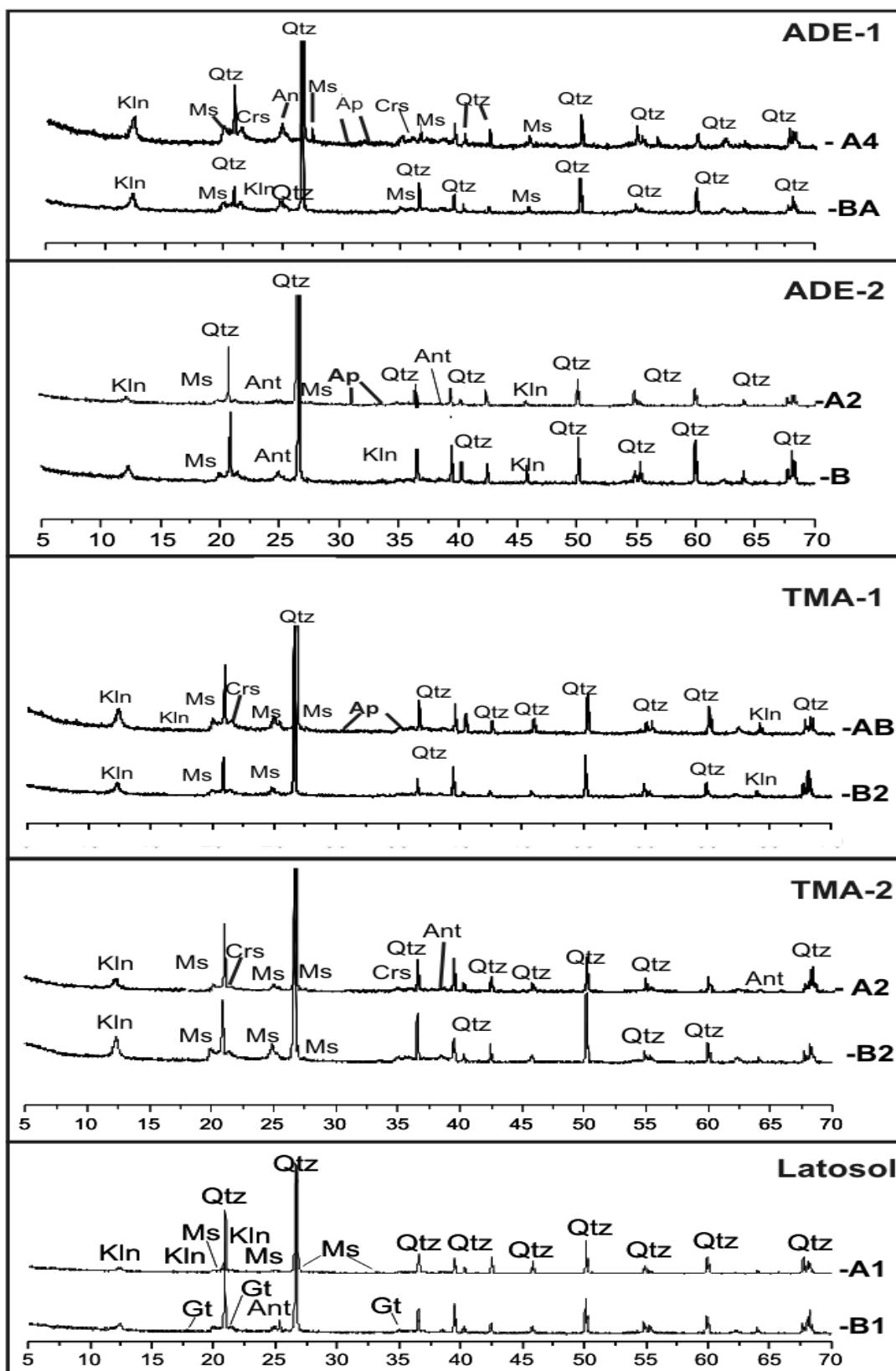


Fig. 4. X-ray diffractograms for the principal minerals identified in the soils at ADE-1, ADE-2, TMA-1, TMA-2, and latosol: quartz (Qtz), kaolinite (Kln), muscovite (Ms), anatase (Ant), apatite (Ap), and cristobalite (Crs).

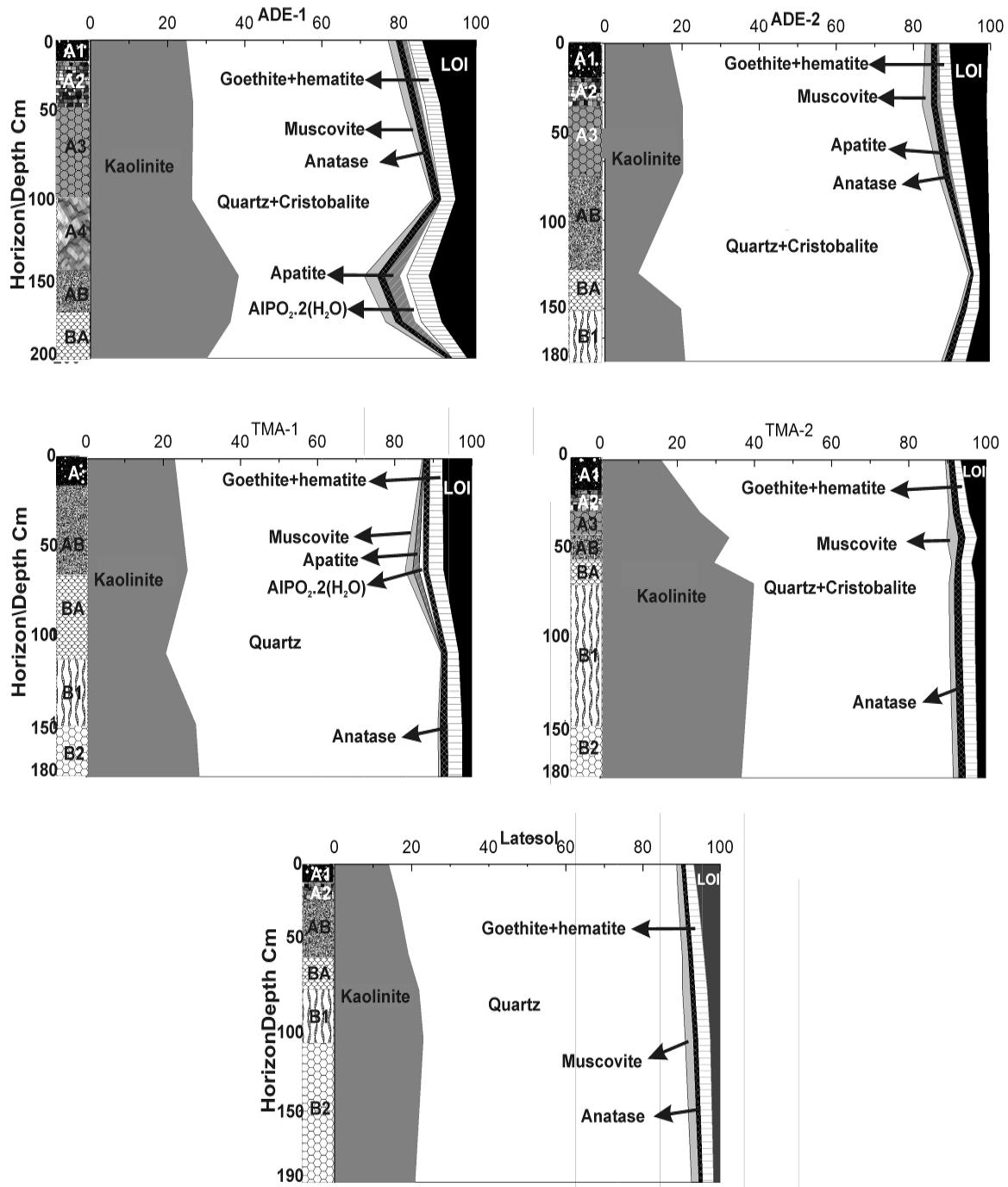


Fig. 5. Distribution of the concentrations of the principal minerals and LOI in the ADE-1, ADE-2, TMA-1, TMA-2, and latosol profiles.

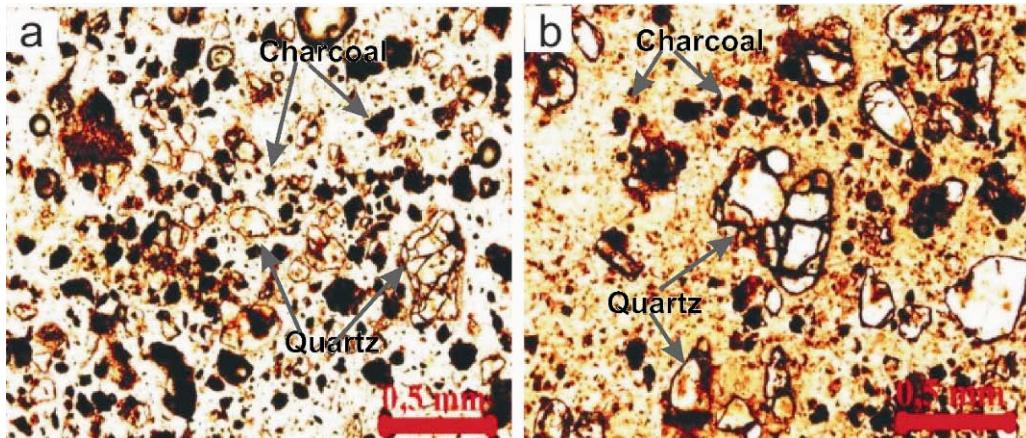


Fig. 6. Photomicrograph of the natural optical transmitted light microscopy, showing the fractured grains of quartz and the abundance of particles of charcoal in the matrix dominated by kaolinite in the soils at (a) ADE-1 and (b) TMA-1.

4.3. Chemical composition of the soil profiles

The soils are composed primarily of SiO_2 , Al_2O_3 , Fe_2O_3 , and TiO_2 which, together with LOI (represented by organic matter and H_2O) make up more than 98% of most samples, from all the different horizons, in the five profiles analyzed. The SiO_2 levels reflect the abundance of quartz, and in part, the amorphous silica resulting from the calcination of organic matter, present as cristobalite. The levels of SiO_2 vary little among the different ADE, TMA, and latosol profiles. While the levels of Al_2O_3 are variable, they represent kaolinite, with concentrations increasing towards the base of the profiles, and especially in the transition horizons (AB and BA) and horizon B at ADE-1 and TMA-1 (Table 1, Fig. 7). This is typical of the profiles of latosol-type soils, except at ADE-2, which may represent soil developed on alternating layers of sandstone and limestone, a common pattern in the Alter do Chão formation, and the basic substrate of the region's soils. The increase in the levels of Al_2O_3 in these horizons is a result of the relative concentration of kaolinite, due to the greater abundance of organic matter, which destabilizes this mineral, in the A horizon (Costa & Kern, 1999).

As for Al_2O_3 , the levels of Fe_2O_3 were lower in the A horizons (Table 1, Fig. 7). As mentioned above, this results from the partial decomposition of the Fe minerals, which are represented by iron oxy-hydroxides compounds primarily in the organic matter of the A horizon, which corresponds partly to the LOI (Fig. 7). The TiO_2 levels vary little between the different profiles and represent anatase.

In terms of their content of SiO_2 , Al_2O_3 , Fe_2O_3 and TiO_2 , then, the ADE, TMA, and latosol are chemically similar, a finding reinforced by their similarities in terms of their

composition of quartz, kaolinite, and iron oxy-hydroxides. A similar pattern was observed in the ADE soils of the region of Caxiuanã by Kern (1996) and Carmo et al. (2007). However, the levels of elements such as CaO, MgO, K₂O, P₂O₅, MnO, Cu, Zn, Ba, and Sr were divergent, suggesting that they were incorporated into the anthropogenic soils, based on an original latosol substrate.

Table 1. Chemical composition of the soil profiles.

Horiz.	SiO ₂	A ₁ BO ₃	Fe ₂ O ₃	MgO	CaO	N ₂ O	K ₂ O	TiO ₂	P ₂ O ₅	MnO	LOI	OM
	% ADE-1											
A1	64.86	10.71	3.65	0.16	0.68	0.04	0.24	1.50	0.72	0.08	17.1	6.40
A2	67.92	11.57	3.93	0.13	0.54	0.05	0.23	1.57	0.84	0.07	12.8	5.12
A3	74.77	10.54	3.86	0.05	0.01	0.02	0.05	1.81	0.06	<0.01	8.50	5.76
A4	52.04	17.03	5.71	0.23	2.25	0.07	0.39	1.77	2.57	0.07	17.60	7.68
AB	58.37	15.84	5.30	0.14	1.73	0.06	0.30	1.83	2.09	0.05	14.00	5.12
BA	75.90	12.18	3.95	0.04	0.13	0.02	0.09	1.45	0.29	0.01	5.70	0.64
ADE-2												
A1	74.83	7.21	2.86	0.13	0.32	0.05	0.18	1.40	0.29	0.06	12.40	3.84
A2	72.67	8.85	3.43	0.15	0.55	0.06	0.28	1.37	0.42	0.07	11.90	3.84
A3	76.10	8.54	3.35	0.10	0.42	0.04	0.16	1.53	0.32	0.03	9.10	3.20
AB	90.07	3.75	1.72	0.04	0.16	0.02	0.07	0.66	0.15	<0.01	3.30	0.77
BA	81.60	8.22	3.22	0.05	0.09	0.02	0.07	1.35	0.15	<0.01	5.00	0.64
B	76.52	10.15	3.87	0.06	0.07	0.02	0.09	1.74	0.16	<0.01	7.00	0.64
TMA-1												
A	75.07	9.20	3.31	0.05	<0.01	0.02	0.04	1.60	0.07	<0.01	10.3	3.52
AB	69.97	11.44	4.05	0.12	0.93	0.04	0.23	1.32	1.18	0.05	10.5	1.54
BA	81.15	8.26	2.97	0.04	<0.01	0.02	0.04	1.37	0.05	<0.01	5.90	0.90
B1	76.29	11.44	3.81	0.05	<0.01	0.02	0.07	1.89	0.06	<0.01	6.00	0.64
B2	76.01	11.83	3.77	0.05	<0.01	0.02	0.08	1.82	0.05	<0.01	6.00	0.38
TMA-2												
A1	81.46	6.54	1.67	0.07	0.18	0.02	0.10	1.40	0.12	0.02	8.20	0.52
A2	77.03	10.67	2.47	0.07	0.10	0.03	0.13	1.70	0.13	0.01	7.40	1.68
A3	72.48	13.64	3.14	0.07	0.07	0.03	0.14	1.67	0.16	<0.01	8.40	1.42
AB	75.82	12.04	2.75	0.06	0.04	0.02	0.12	1.37	0.13	<0.01	7.50	1.03
BA	69.76	16.23	3.59	0.08	0.04	0.03	0.16	1.82	0.14	<0.01	7.90	0.65
B1	70.40	15.92	3.43	0.07	0.03	0.03	0.17	1.78	0.11	<0.01	7.80	0.52
B2	72.64	15.01	3.08	0.07	0.02	0.03	0.18	1.69	0.08	<0.01	7.00	0.39
Latosol												
A1	81.82	6.00	2.26	0.06	0.01	0.03	0.15	0.92	0.07	<0.01	8.50	2.56
A2	81.04	7.07	2.72	0.06	<0.01	0.03	0.18	1.00	0.07	<0.01	7.70	0.52
AB	80.65	8.20	2.98	0.07	<0.01	0.04	0.20	1.06	0.06	<0.01	6.60	0.52
BA	79.45	9.43	3.28	0.07	<0.01	0.04	0.24	1.11	0.06	<0.01	6.10	0.26
B1	79.62	9.91	3.25	0.07	<0.01	0.04	0.26	1.05	0.05	<0.01	5.60	0.39
B2	82.26	9.01	2.84	0.06	<0.01	0.04	0.24	0.94	0.04	<0.01	4.40	0.13

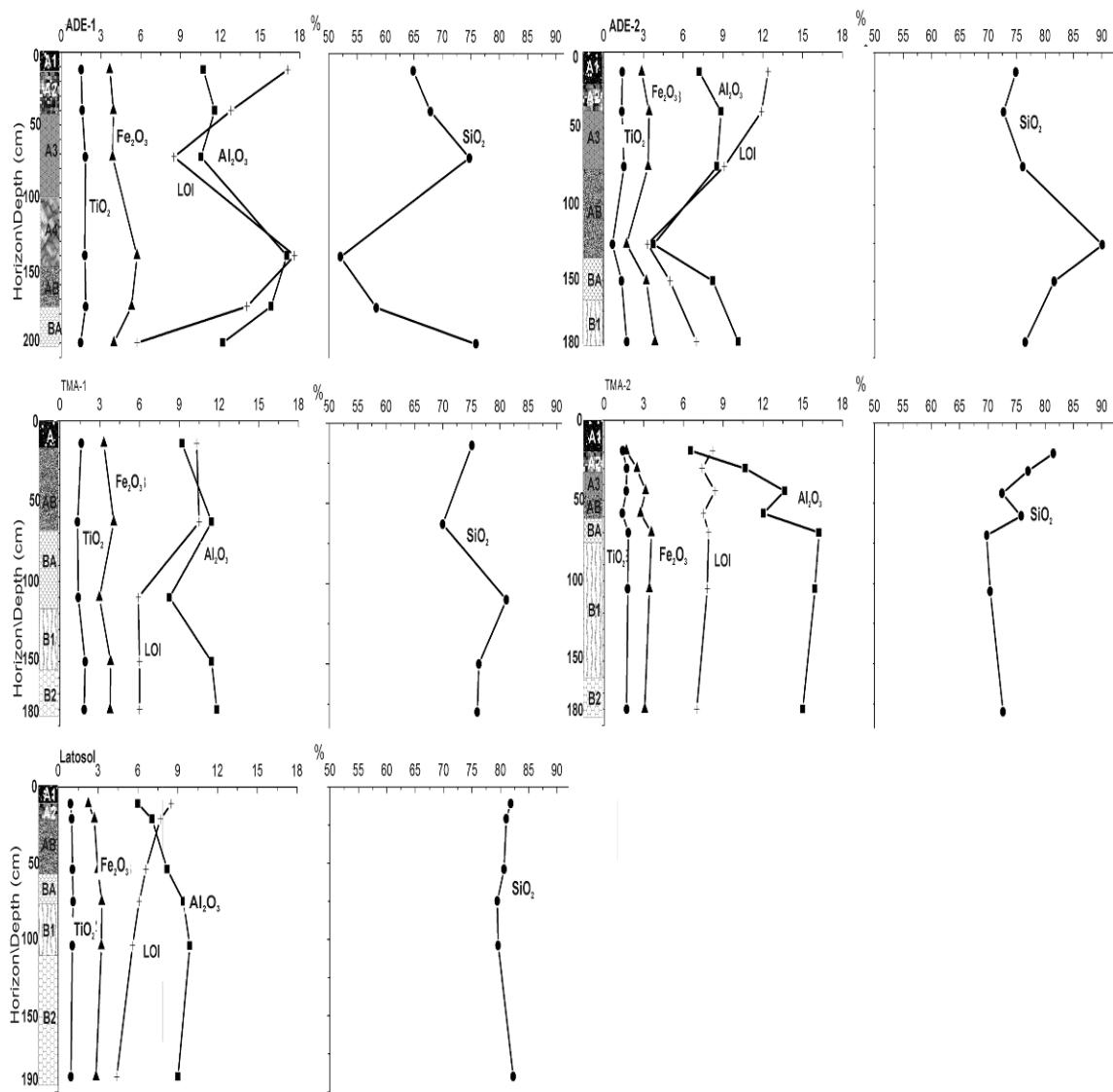


Fig. 7. Distribution of the levels of Al₂O₃, Fe₂O₃, TiO₂ and LOI in the ADE-1, ADE-2, TMA-1, TMA-2, and latosol profiles.

The highest levels of CaO and P₂O₅ are found in the A horizon, especially horizon A₄ at ADE-1 (2.25%), which is clearly different from those of all the horizons in ADE-2 and the TMA soils, with the exception of horizon AB of TMA-1 (0.93%), which is equivalent to ADE. Major contrasts in the CaO content were observed only in relation to the profile of the adjacent soil, which was at or below the detection threshold (CaO < 0.01%). In addition, P₂O₅ was strongly correlated with CaO in the ADE and TMA soils, with the highest levels being recorded in the A₄ horizon of ADE-1 (2.57%) and the AB horizon of TMA-1 (Table 1, Fig. 8). These very high values at the base of the ADE-1 profile correspond to archeological features and structures containing charcoal, ash, and fragments of bone and ceramics, which were found at varying depths of the ADE at Juruti.

These anomalous values for phosphorus and calcium are chemical indices of prehistoric refuse dumps subject to successive burn-offs. The elevated values of CaO and P₂O₅, observed in the TMA-1 profile, also reflect the presence of apatite. The significant correlation between the levels of CaO and P₂O₅ confirm the chemical contribution of apatite to the ADE and TMA soils (Fig. 9). While this is normally attributed to the presence of bone remnants, the exceptionally strong correlation observed in the ADE soils studied here indicates the historic presence of human and animal excrement (Smith, 1980; Lima et al., 2002; Kampf & Kern, 2005). The relatively high levels of phosphorus in the ADE soils at Juruti, as indicated by XRD and the stoichiometric calculations, are thus derived from two sources of phosphates – bone residues and aluminum phosphate derived from the addition of kaolinite, probably derived from the ceramic fragments, as shown by Costa et al (2004a, 2004b, 2006, 2010). In most of profiles, the levels of phosphorus are lower and those of calcium are close to the detection threshold, which means that apatite is absent.

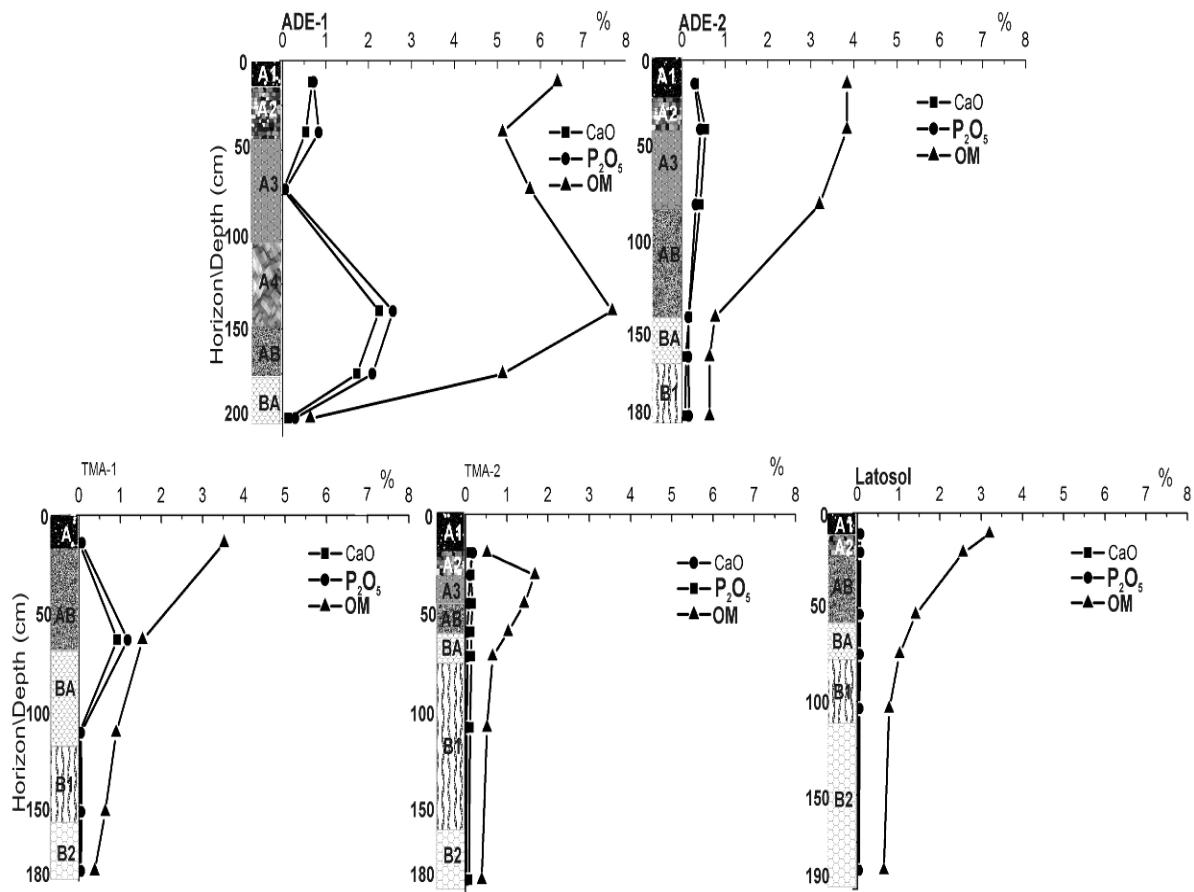


Fig. 8. Distribution of the levels of CaO, P₂O₅, and OM in the ADE-1, ADE-2, AD, TMA-1, and TMA-2 profiles. The same scale is used throughout, in order to highlight the similarities and differences in the distribution patterns between the different profiles.

The levels of MgO are equivalent to those of CaO and P₂O₅, with a limited degree of variation restricted to the A horizons of the ADE profiles (Table 1). This is supported by the strong correlation between MgO and P₂O₅ ($r = +0.94$). The MgO content of the TMA and latosol are nevertheless relatively similar (Table 1). The levels of Na₂O are also slightly higher in the ADE soils. In addition to its relatively high levels in the A horizon of the TMA, and in particular of the ADE profiles, the levels of K₂O in the B horizon of the latosol profile indicates the resilience of muscovite to weathering. Once again, these results emphasize the similarities between the ADE and TMA horizons, and to a lesser extent, with that of the AD soils.

As for phosphorus, MnO is an important indicator of human occupation (Goffer, 2007). This compound is found at high concentrations only in the A horizons of the ADE soils, and in part, in TMA-1. There is no significant variation between ADE profiles, even where the highest levels of CaO and P₂O₅ were recorded, with an overall mean of 0.06% of MnO. In the case of the TMA soils, only the AB horizon at TMA-1 was notable, whereas all the horizons of the latosol profile were below the detection threshold, i.e., Mn < 0.01% (Table 1).

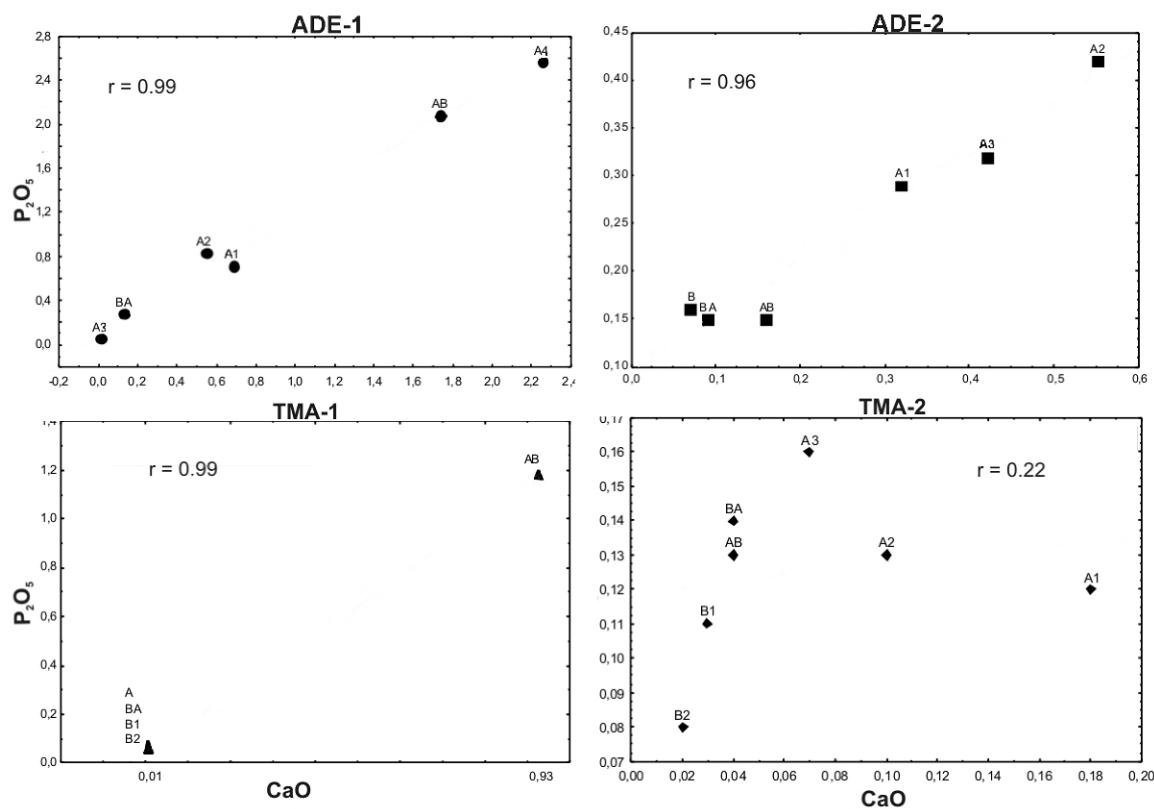


Fig. 9: Correlation between the levels of P₂O₅ and CaO in the soil horizons of ADE-1, ADE-2, TMA-1, and TMA-2.

Similarly, the concentrations of MnO, Cu, Zn, Ba, and Sr are higher in the ADE soils and TMA-1 (Table 2, Fig. 10). The strong correlations between Zn and MO, and Zn and CaO reflect the importance of the contribution of the residues deposited in the soils, especially bones (Fig. 11). The levels of Cu and Zn in the TMA-2 profile, in particular, are similar to those of the adjacent profile. Just as for Cu and Zn, the presence of Ba and Sr is related to that of organic matter, especially bone fragments, as suggested by the correlations between Ba and organic matter ($r = +0.60, +0.89$) and Ba and CaO ($r = +0.97, +0.98$) in the ADE-1 and ADE-2 profiles, respectively (Fig. 12).

Taken together, then, CaO, P₂O₅, MgO, K₂O, MnO, Cu, Zn, Ba, and Sr are the chemical elements that discriminate ADE and TMA soils in comparison with those of the adjacent area in Juruti. Kern et al. (1999) have attributed the high levels of K, Mg, Mn, Cu, and Zn in ADE soils to the presence of discarded palm leaves that had been used to thatch the occupants' cabins and had subsequently decomposed through weathering and had released these components, which were incorporated into the developing soil. These materials would have been incorporated periodically as the settlers occupied the site sequentially over long periods.

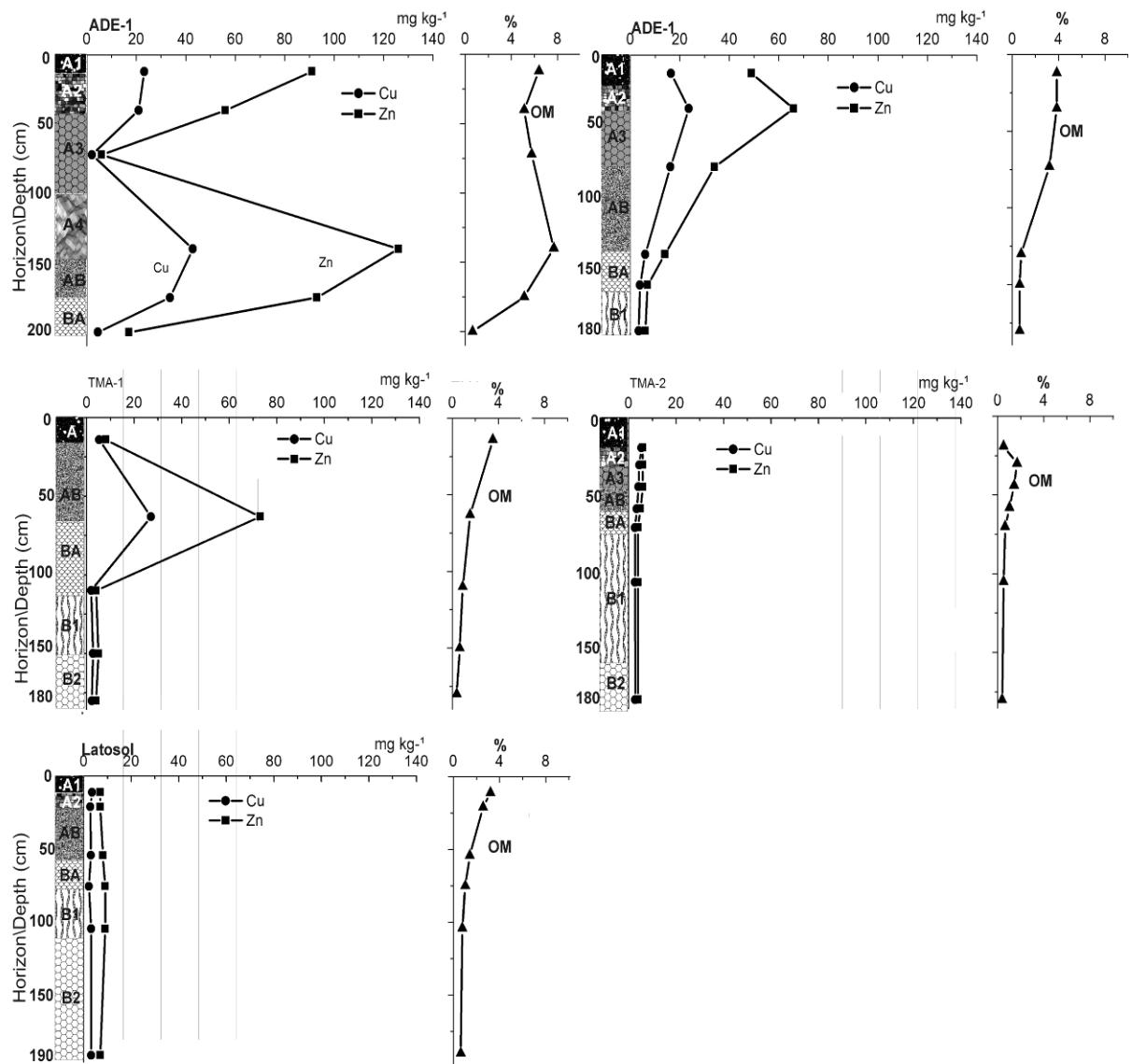


Fig. 10. Distribution of Cu, Zn, and organic matter (OM) levels in the ADE-1, ADE-2, TMA-1, TMA-2, and latosol profiles.

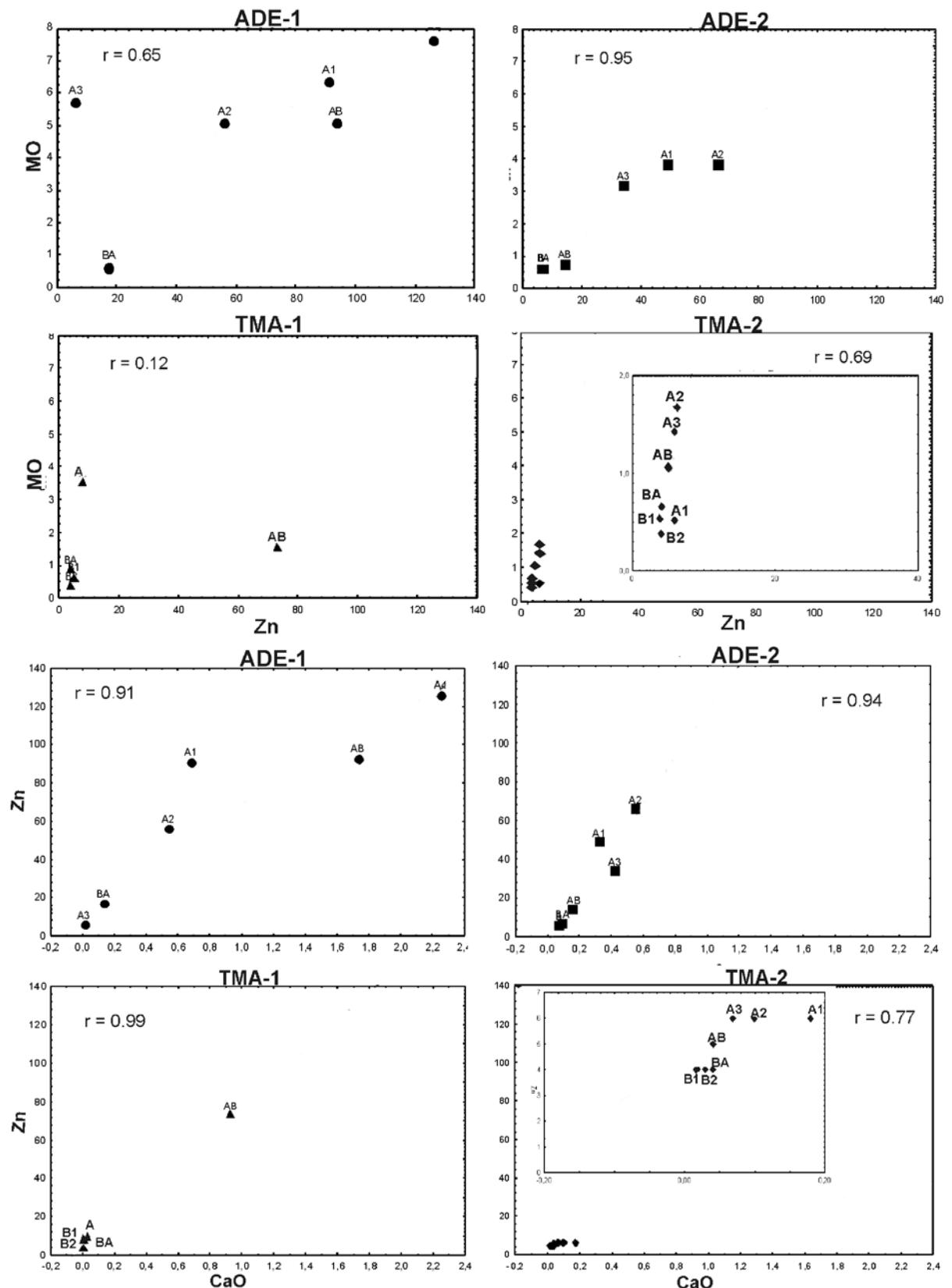


Fig. 11. Correlation between the levels of organic matter (OM) and Zn, and Zn and CaO in the ADE-1, ADE-2, TMA-1, and TMA-2 soil horizons.

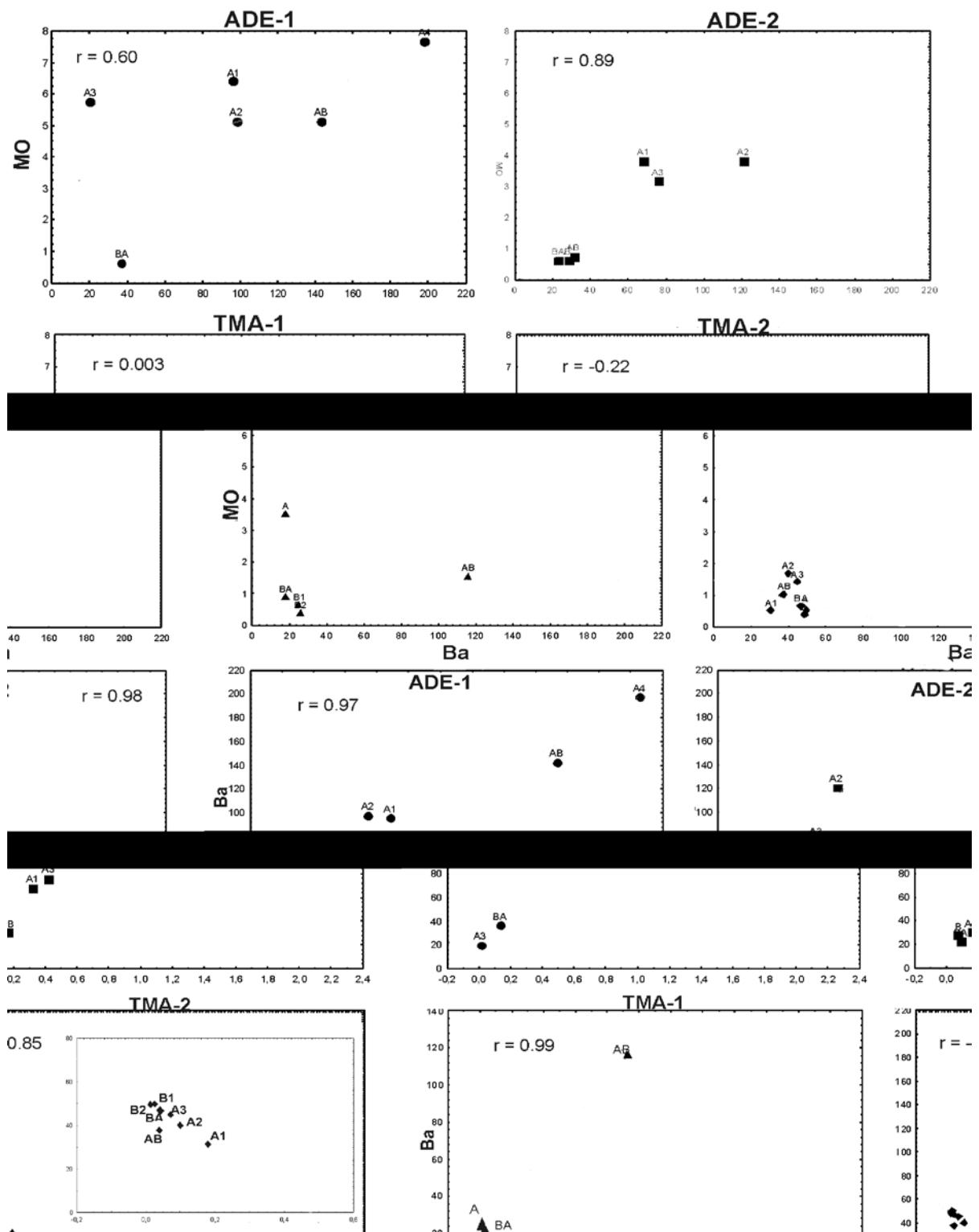


Fig. 12. Correlation between the levels of organic matter (OM) and Ba, and Ba and CaO in the ADE-1, ADE-2, TMA-1, and TMA-2 soil horizons.

While the ADE and TMA soils, and even the latosol, are highly similar in terms of their principal chemical components, i.e., SiO_2 and Al_2O_3 , then, they are clearly distinguished from adjacent soils by their relatively high concentrations of P, Ca, and Mg. At Juruti, Mn,

Cu, Zn, Ba, and Sr are also part of the geochemical signature of typical ADE and TMA soils, as described at other Amazonian sites by Kern (1996), Costa & Kern (1999), and Lima et al. (2002).

The normalization of the ADE (ADE-1 and ADE-2) and TMA (TMA-1 and TMA-2) profiles relative to the equivalent horizons of the adjacent profile, which is assumed to be the original substrate for these anthropogenic soils (both ADE and TMA), reveals that P_2O_5 , CaO, MgO, and K₂O were incorporated into these soils, especially in the A horizon of ADE (Fig. 13). As shown above, the MnO, and most of the CaO, could not be normalized, given that the corresponding horizons of the adjacent soil were below the detection threshold. The slight enrichment of A_2O_3 , Fe₂O₃, and TiO₂ in the ADE and TMA soils represents the partial signature of the latosols, the substrate from which the anthropogenic soils were formed.

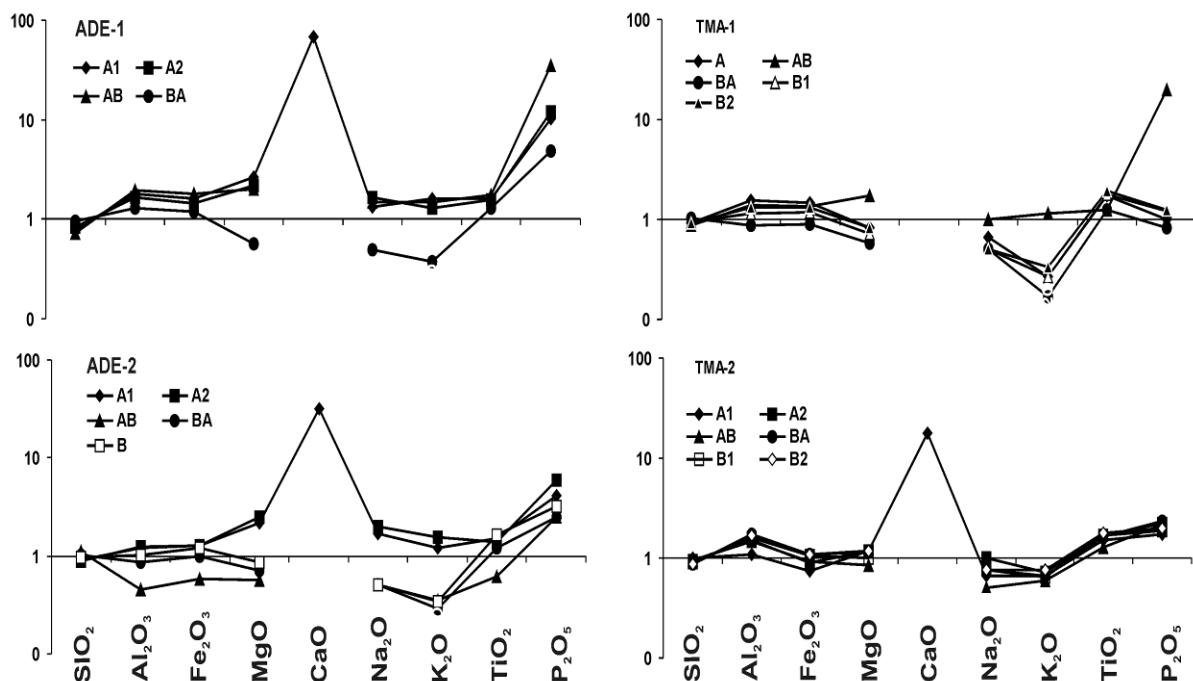


Fig. 13. Distribution of major elements in the soil profiles at ADE-1, ADE-2, TMA-1, and TMA-2 normalized to the respective horizons of the adjacent area.

By contrast, the slight enrichment of Cu, Zn, Rb, Sr, Cd, Cs, Ba, and Pb in the A, AB, and BA horizons of the ADE soils, and the AB horizon at TMA-1, or even the impoverishment observed in all the other horizons, reflects the quantity and quality of the organic matter added to the soil (Fig. 14). The levels of Cu, Zn, Sr, Ba, and Cd correlated with those of CaO, that is, they were related principally to the presence of bone fragments. Similar results were obtained by Oonk et al. (2009) and Wilson et al. (2008), who attributed the

increase in the levels of trace elements, including As and Pb, to the presence of fragments of bone and plants during the mineralization of the organic matter. The variation between horizons reflects the irregular distribution of the residues, as well as the reduced content of the rock that originated the substrate (Horbe & Costa, 1997).

By contrast, the partial enrichment of Co, Ni, and Sn reflects the presence of iron oxy-hydroxides compounds, probably in association with organometallic compounds, as indicated by the significant correlations of both Co and Ni with Fe ($r = +0.74$, $+0.59$) and the organic matter ($r = +0.93$, $+0.86$) in the ADE and TMA soils, respectively. The impoverishment of V, Cr, Ga, Y, Zr, Mo, Hf, Nb, W, Hg Ta, Th, and U in the A horizons indicates the partial decomposition of the iron oxy-hydroxides and Al compounds in the presence of organic matter, and subsequent leaching, whereas these same elements are found at similar levels in the B horizon in ADE, TMA and latosol (Fig. 14, Table 2). The geochemical similarities represent a common heritage from the adjacent soils, which formed the basis for the anthropogenic soils. These results also indicate that the yellow latosols at Juruti received different contributions from residues rich in P, Mg, Ca, Mn, Cu, Zn, Sr, and Ba, in particular bones, soft animals parts and plant residues, which were added to the basic latosols and contributed over time to the tropical pedogenetic process, to form the local ADE and TMA soils.

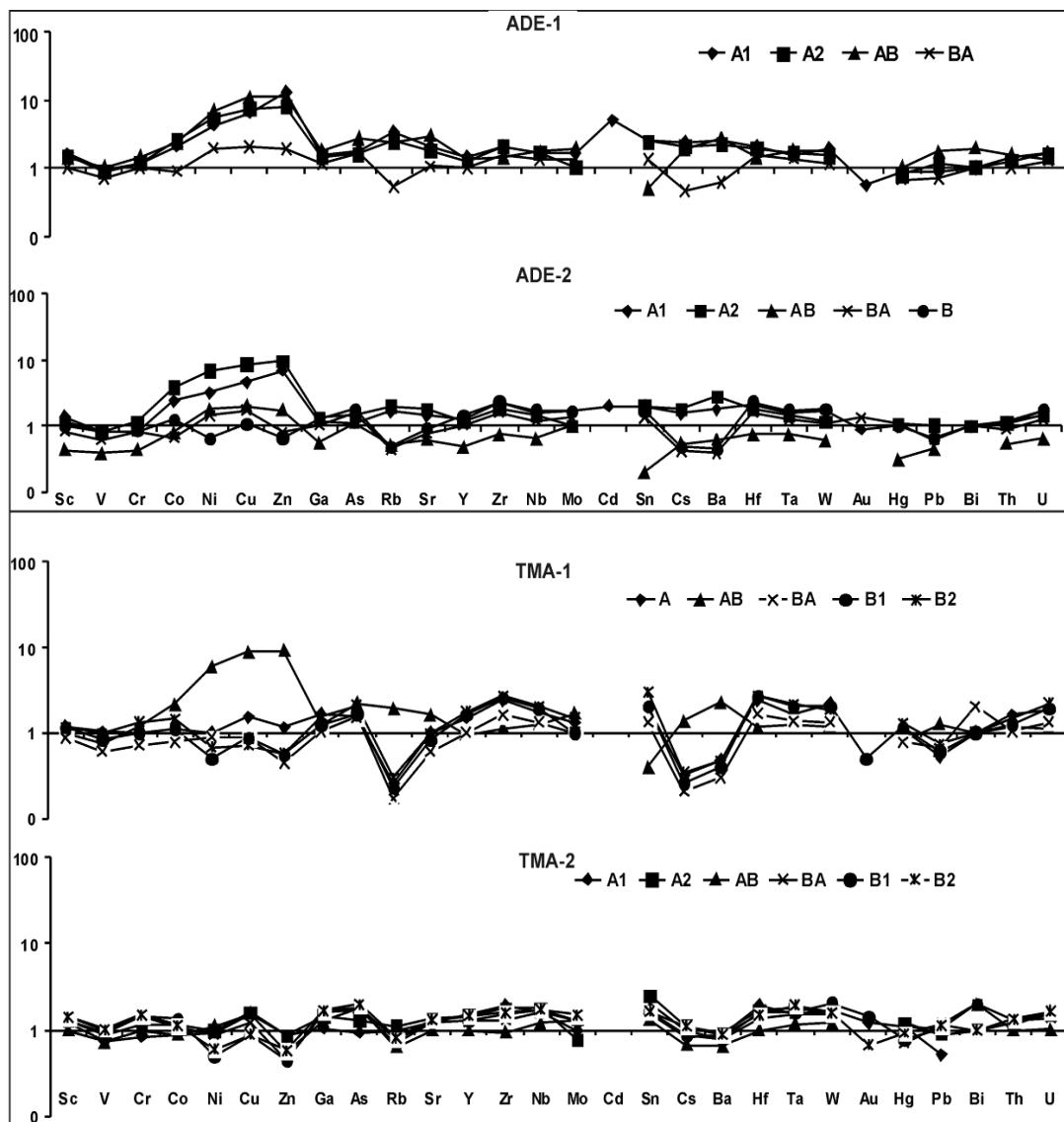


Fig. 14. Distribution of trace elements in the soil profiles of ADE-1, ADE-2, TMA-1, and TMA-2 normalized to the respective horizons of the adjacent area.

Table 2: Concentration of trace elements in the soil profiles.

Horiz.	Be	Sc	V	Cr	Co	Ni	Cu	Zn	Ga	As	Se	Rb	Sr	Y	Zr	Nb	Mo	Ag	Cd	Sr	Sb	Cs	Ba	Hf	Ta	W	Au	Hg	Tl	Pb	Bi	Th	U
mg kg ⁻¹																																	
ADE-1																																	
A1	<1.00	8.00	74.00	47.89	1.70	4.80	23.20	91.00	15.90	2.70	0.90	34.30	47.10	38.80	1812.20	29.40	0.50	<0.10	0.50	5.00	<0.10	3.90	96.00	47.50	2.20	3.00	0.50	0.08	<0.10	6.90	0.10	15.60	5.30
A2	<1.00	9.00	78.00	47.89	1.80	5.00	21.00	56.00	16.40	2.60	0.60	28.10	47.00	39.20	1955.80	31.70	0.40	<0.10	0.40	5.00	<0.10	3.70	98.00	51.50	2.40	3.10	<0.50	0.08	<0.10	6.90	0.10	17.10	5.80
A3	<1.00	8.00	95.00	54.73	0.90	1.10	2.10	6.00	20.10	2.10	<0.50	2.70	26.00	46.50	2071.30	37.00	0.40	<0.10	<0.10	5.00	<0.10	0.50	20.00	55.40	2.70	3.80	<0.50	0.12	<0.10	5.60	0.20	19.50	6.10
A4	<1.00	11.00	108.00	68.42	2.60	8.90	42.90	126.00	26.00	4.50	<0.50	37.80	103.60	40.60	1231.10	35.30	0.60	<0.10	1.30	5.00	<0.10	5.50	198.00	32.10	2.60	3.60	0.90	0.11	<0.10	10.30	0.20	22.30	5.10
AB	<1.00	10.00	101.00	68.42	2.20	6.20	33.60	93.00	24.40	4.10	<0.50	30.00	91.10	42.30	1457.60	36.20	0.60	<0.10	0.80	5.00	<0.10	4.00	143.00	40.30	2.70	3.80	0.60	0.13	<0.10	9.30	0.20	21.70	5.30
BA	<1.00	7.00	82.00	47.89	0.80	1.80	4.50	17.00	17.60	2.10	<0.50	8.30	34.00	33.50	1481.80	28.90	0.40	<0.10	<0.10	4.00	<0.10	1.10	37.00	39.50	2.20	2.60	<0.50	0.09	<0.10	4.50	0.10	16.10	4.70
ADE-2																																	
A1	<1.00	7.00	60.00	34.21	1.90	3.50	16.40	49.00	12.20	1.70	0.70	16.50	34.30	35.00	1952.90	28.00	0.50	<0.10	0.20	4.00	<0.10	2.50	68.00	51.30	2.30	2.80	0.80	0.09	<0.10	5.20	0.10	13.00	5.30
A2	<1.00	7.00	69.00	47.89	2.70	6.20	23.60	66.00	15.30	2.40	<0.50	22.70	47.10	37.30	1790.00	27.40	0.40	<0.10	0.20	4.00	<0.10	3.20	121.00	46.70	2.00	2.50	<0.50	0.11	<0.10	6.40	0.10	15.00	5.50
A3	<1.00	7.00	77.00	47.89	1.60	3.40	16.20	34.00	15.90	2.10	<0.50	15.80	41.60	40.80	2211.90	32.10	0.30	<0.10	0.20	5.00	<0.10	2.50	76.00	57.90	2.40	3.10	3.20	0.10	<0.10	4.40	0.10	16.10	5.90
AB	<1.00	3.00	37.00	20.53	0.70	1.60	6.00	14.00	7.40	1.70	<0.50	6.40	18.20	15.50	759.50	13.10	0.30	<0.10	<0.10	2.00	<0.10	1.10	31.00	20.20	1.10	1.30	0.80	0.04	<0.10	2.30	<0.10	7.40	2.40
BA	<1.00	6.00	70.00	41.05	0.60	1.30	3.90	7.00	15.40	2.10	<0.50	6.80	25.30	34.10	1608.50	26.20	0.40	<0.10	<0.10	4.00	<0.10	1.00	23.00	41.70	2.00	2.50	1.20	0.14	<0.10	3.80	0.10	13.70	4.60
B	<1.00	7.00	92.00	41.05	1.00	1.00	3.40	6.00	18.80	2.20	<0.50	8.10	30.20	44.70	2094.10	35.20	0.50	<0.10	<0.10	5.00	<0.10	1.30	28.00	55.60	2.60	3.80	<0.50	0.12	<0.10	4.00	0.10	18.70	6.20
TM A-1																																	
A	<1.00	6.00	79.00	41.05	0.90	1.10	5.30	8.00	16.60	2.30	<0.50	2.60	23.60	40.00	2057.20	32.00	0.40	<0.10	<0.10	4.00	<0.10	0.50	18.00	55.50	2.30	3.50	<0.50	0.10	<0.10	4.10	0.10	17.70	6.00
AB	<1.00	7.00	74.00	54.73	1.90	5.40	27.00	73.00	16.60	3.30	<0.50	24.10	48.50	30.30	1155.60	26.00	0.50	<0.10	0.70	4.00	<0.10	2.90	116.00	31.70	1.90	2.60	0.50	0.10	<0.10	6.60	0.10	16.60	4.30
BA	<1.00	6.00	69.00	34.21	0.70	0.80	1.90	4.00	15.30	2.00	<0.50	2.60	20.00	32.40	1634.20	28.80	0.30	<0.10	<0.10	4.00	<0.10	0.50	18.00	43.50	2.20	2.90	<0.50	0.10	<0.10	4.10	0.20	16.20	4.90
BI	<1.00	8.00	93.00	47.89	0.90	0.80	2.70	5.00	18.80	2.00	<0.50	3.80	27.30	50.90	2287.20	39.40	0.30	<0.10	<0.10	6.00	<0.10	0.70	25.00	65.50	3.00	4.20	0.70	0.14	<0.10	3.80	0.10	20.80	6.80
B2	<1.00	8.00	93.00	54.73	1.00	0.80	2.20	4.00	19.30	2.10	<0.50	4.50	28.50	45.80	2282.70	36.90	0.30	<0.10	<0.10	9.00	<0.10	0.80	26.00	61.20	2.80	3.70	<0.50	0.14	<0.10	3.90	0.10	20.70	6.60
TM A-2																																	
A1	<1.00	6.00	54.00	34.21	0.70	1.00	5.40	6.00	10.70	1.40	<0.50	9.70	28.70	40.80	1679.90	29.40	0.30	<0.10	<0.10	3.00	<0.10	1.30	31.00	45.80	2.10	2.70	1.10	0.10	<0.10	4.00	<0.10	12.90	5.30
A2	<1.00	8.00	77.00	47.89	0.80	0.90	4.50	6.00	16.60	2.10	<0.50	12.40	34.90	45.30	1706.80	33.80	0.30	<0.10	<0.10	5.00	<0.10	2.00	40.00	44.80	2.50	3.30	<0.50	0.12	<0.10	5.40	0.10	16.50	5.60
A3	<1.00	8.00	92.00	61.58	1.00	0.90	4.20	6.00	21.30	2.30	<0.50	12.60	37.10	39.00	1310.40	32.00	0.50	<0.10	<0.10	5.00	<0.10	2.30	45.00	36.20	2.40	3.10	2.10	0.13	<0.10	6.90	0.10	18.10	4.90
AB	<1.00	7.00	82.00	47.89	0.80	1.00	3.40	5.00	19.30	2.40	<0.50	9.50	32.10	31.20	934.90	26.20	0.40	<0.10	<0.10	4.00	<0.10	1.60	38.00	26.10	1.90	2.70	<0.50	0.14	<0.10	6.50	0.20	15.20	3.80
BA	<1.00	9.00	106.00	68.42	1.00	0.80	2.70	4.00	25.20	2.30	<0.50	11.80	40.80	41.60	1328.00	34.40	0.40	<0.10	<0.10	6.00	<0.10	2.40	47.00	35.80	2.50	3.40	<0.50	0.09	<0.10	7.40	0.20	20.60	5.20
BI	<1.00	10.00	106.00	68.42	1.10	0.80	2.70	4.00	24.20	2.10	<0.50	11.50	41.30	41.80	1396.80	34.80	0.40	<0.10	<0.10	5.00	<0.10	2.30	50.00	37.50	2.50	4.30	2.00	0.09	<0.10	6.60	0.20	21.00	5.40
B2	<1.00	10.00	99.00	61.58	0.80	0.70	2.80	4.00	21.40	2.00	<0.50	12.10	38.70	38.90	1314.50	32.30	0.30	<0.10	<0.10	5.00	<0.10	2.60	49.00	35.00	2.50	3.20	1.00	0.10	<0.10	6.10	0.10	19.30	4.90
Latosol																																	
A1	<1.00	5.00	76.00	41.05	0.80	1.10	3.50	7.00	10.00	1.50	<0.50	10.00	23.30	26.90	856.00	18.20	0.30	<0.10	0.10	2.00	0.10	1.60	37.00	23.30	1.40	1.60	0.90	0.09	<0.10	7.80	0.10	11.00	3.20
A2	<1.00	6.00	87.00	41.05	0.70	0.90	2.80	7.00	11.20	1.60	0.60	11.20	25.80	30.40	950.80	18.90	0.40	<0.10	<0.10	2.00	<0.10	1.80	44.00	26.10	1.40	2.10	2.40	0.10	<0.10	6.10	0.10	13.20	3.60
AB	<1.00	7.00	98.00	47.89	0.90	0.90	3.00	8.00	13.50	1.50	0.70	12.70	30.10	32.20	1030.50	20.90	0.30	<0.10	<0.10	10.00	<0.10	2.10	51.00	27.70	1.50	2.20							

As observed in the trace elements, the similarity of the distribution of rare earth elements in the horizons of the ADE and TMA soils reinforces the conclusion that they have a common geochemical source in the adjacent soils (Fig. 15). However, the heavy REEs tend to become enriched in contact with the light REEs, especially at ADE-2 and TMA-1.

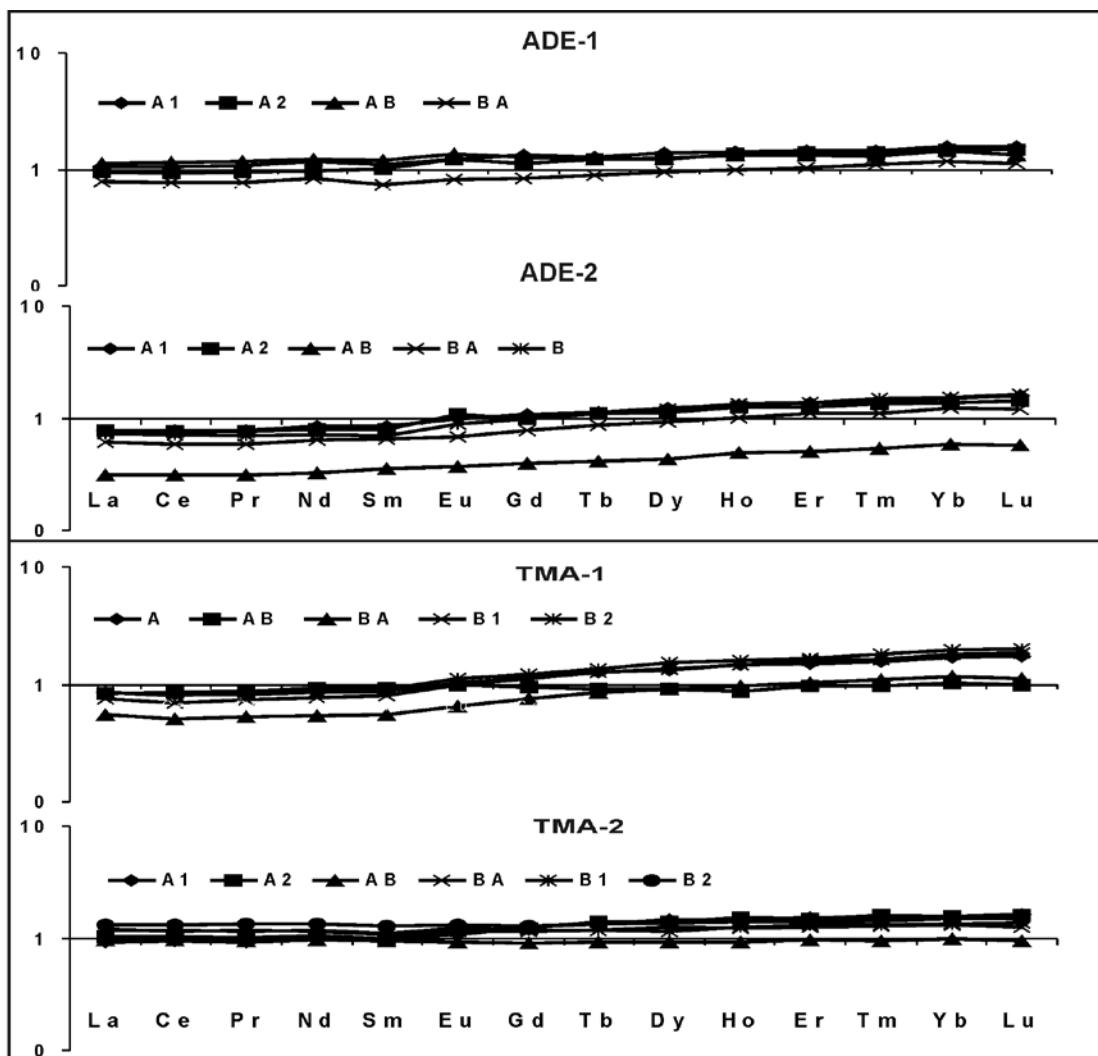


Fig. 15. Distribution of REEs in the soil profiles of ADE-1, ADE-2, TMA-1, TMA-2, normalized to the respective horizons of the adjacent area.

Conclusions

The yellow latosols that form the soils that cover the ferrous-aluminous crusts of the lateritic profiles predominate in the Amazon basin (Costa, 1991; Costa et al., 2010) appear to have provided not only the basic substrate, but also a number of components for the formation of the anthropogenic (ADE and TMA) soils at Juruti. All three types of soil are characterized by an abundance of quartz, kaolinite, goethite + hematite, and anatase, which are responsible for the high levels of Si, Al, Fe, and Ti, and testify to the mineralogical and

chemical heritage of the ADE and TMA soils at Juruti in relation to the adjacent soils. The evidence indicates that a large number of densely-populated settlements were established along the course of the lower Amazon River and its tributaries between one and three thousand years ago. These communities were inhabited by horticultural potters with complex social organization (Roosevelt, 1992; Carneiro, 2007; Heckenberger et al., 1999) who, during their occupation of the sites, accumulated large quantities of organic residues, including plant and animal remains, bones, charcoal, and an abundance of ceramic material. Thousands of years of the hot and humid weathering typical of tropical pedogenetic processes, have resulted in a thick parcel of Archeological Dark Earth type soils (Kern and Kampf, 1989; Costa and Kern, 1997; Neves et al., 2003).

The quantity and in particular the quality of the organic matter accumulated in the anthropogenic soils account for the enrichment of P, Mg, Ca, Mn, Cu, Zn, Sr, and Ba, which constitute the geochemical signature typical of the ADE soils, as at other Amazonian sites (Kern & Kampf, 1989; Kern, 1996; Costa & Kern, 1999; Lima et al., 2002). On the other hand, the Terra Mulata located in the area surrounding the ADE soils, is distinguished by the absence of ceramics or any other archeological remains. Even so, the TMA soils have clearly received a contribution from organic residues, which has resulted in the modification of the properties of its A horizon. The brownish colors and the thickness of this horizon – which is twice that of the adjacent soils – indicate the addition of organic matter through anthropogenic modifications. The occurrence of anthropogenic alterations is confirmed by the presence of apatite and cristobalite, mineral phases restricted to the ADE soils. The apatite reflects the continuous discarding of bones, which is reflected in the increased values of Ca, P, Mg, Cu, Zn, Ba, and Sr, which are also found in the TMA soils. The higher levels in the ADE soils may be related to domestic activities such as the preparation of food, the processing of game and fish, and the discarding of excrements (Smith, 1980; Kern & Kampf, 1989; Lima et al., 2002), whereas the lower concentrations of these elements in the TMA soils suggests possible agricultural practices aimed at increasing the quality of the soil, as suggested by Sombroek (1966), or simply a similar pedogenetic process based on reduced quantities of residues, in particular, bones and excrements. The levels of Ca, P, K, Zn, and Cu are as high as the ADE in some localized spots, indicates more intensively-used areas, which may represent ADE without the ceramic fragments.

The identification of cristobalite in the TMA and ADE horizons represents the transformation of amorphous silica (SiO_2) into a crystalline form by firing and suggests the successive burning of organic matter, a common practice in domestic environments, related to

the clearing and preparation of the soil, for example, which is still practiced by present-day local communities that follow a traditional lifestyle (Hecht, 2003; Heckenberger et al., 1999; Denevan, 2002). Overall, the mineralogical and chemical differences found in the different horizons provide strong evidence of the modifications to the environment provoked by the presence of past human settlers, and by the pedogenetic processes that followed their occupation of the study area.

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4.2 ANALYSIS OF THE SPATIAL DISTRIBUTION OF GEOCHEMICAL SIGNATURES FOR THE IDENTIFICATION OF PREHISTORIC SETTLEMENT PATTERNS IN ADE AND TMA SITES IN THE LOWER AMAZON BASIN
 (Submetido ao periódico Journal of Archaeological of Science)

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Abstract

Extensive areas of soils of the Archeological Dark Earth (ADE) and Terra Mulata (TMA) types are found in Juruti, in the lower Amazon Basin, a region dominated by yellow Latosols. While ADE soils are dark in color and contain large quantities of ceramic fragments, the TMAs are dark, but do not contain ceramics. Multi-element chemical analyses of ADE and TMA soils from horizon A₂ (depth of 10-20 cm) revealed that the ADEs are differentiated by their relatively high concentrations of P₂O₅, CaO, K₂O, MgO, Cu, Mn, and Zn, while the TMA have medium levels of these components, and the adjacent Latosols are characterized by low concentrations. These chemical elements constitute the geochemical signature of these anthropogenic soils, while Al₂O₃, Fe₂O₃, TiO₂, Cr, Sr, La, Li, Ni, Pb, V, Y, and Zr define the geochemical signature of the Latosols, but can still be identified in the ADEs and TMA. The isoline maps of the concentrations of the elements of these two geochemical associations delimited the ADEs as areas of permanent occupation, and the TMA as temporary sites, probably linked to agricultural activities. Seven settlements were defined, grouped into two nuclei separated by corridors. The villages were established along the riverbank with the more temporary cultivated areas to the rear running almost parallel to the river. The identification of functional patterns based on the geochemical associations, and the map of the distribution of ceramic fragments and organic matter, revealed that the area occupied covered a total of more than 350 ha. The P₂O₅-CaO-MgO-Na₂O-K₂O-Zn-Cu-Mn-Ba-Sr-Li-Ni geochemical signature can be interpreted as the result of permanent human settlement on yellow Latosols.

Key words: Prehistory, anthropogenic soils, geochemical association, settlement, ceramic fragments, Amazonia

1. Introduction

In tropical regions, areas altered by paleoindigenous settlements for the construction of shelters or agriculture are increasingly well-known (Sombroek, 1966; Kern, 1996; Kern et al., 2003; Denevan, 2010). Different uses and durations of these settlements result in distinct patterns of modification of the physical, chemical, mineralogical, and microbiological properties of the local soils (Smith, 1980; Costa & Kern, 1999; Lehmann et al, 2003; Gleiser et al., 2003; Ruivo et al., 2009; Tsai et al, 2009). These modifications reflect the quantity and

quality of organic residues, specific cultural aspects of the settlers, and post-occupation (bio)-geochemical processes, such as pedogenesis.

The hot and humid environments typical of the tropics are the principal factor limiting the conservation of archeological evidence, in particular organic remains. Given this, the use of archeometric techniques has been fundamental to the interpretation of archeological contexts, such as settlement patterns (Kern & Kampf, 1989; Kern, 1996; Costa & Kern, 1999), and archeological features and structures (Wells et al., 2000; Wilson et al., 2008; OonK et al., 2009).

The anthropogenic soils known as Archeological Dark Earth (ADE) and Terra Mulata (TMA) are typical of the Amazon region, and provide the basis for the study of settlement patterns, given that they indicate the location of the functional areas of ancient settlements, due to the very distinct properties of their horizons of occupation (Kern & Kampf, 1989; Kern, 1996; Lima et al., 2002; Costa et al., 2004a; Costa et al., 2010). The A (anthropogenic) horizon is characterized by its dark color, relative thickness, presence of ceramic and stone fragments, and high levels of Ca, Mg, P, Mn, Zn, Cu, organic C, and particles of charcoal in comparison with adjacent, dominant soils (Smith, 1980; Kern & Kampf, 1989; Kern & Costa, 1997, Kampf & Kern, 2005; Sombroek, 2002).

At the Manduquinha site in Caxiuanã on the lower Amazon, elevated concentrations of Ca and P have been associated with deposits of organic matter from animal sources, whereas high concentrations of Cu, Zn, and Mn indicate areas in which plant residues have been deposited (Kern, 1996). In Piedras Negras, Guatemala, abnormal concentrations of P were associated with urban market gardens, whereas relatively high levels of Hg, Fe, Mn, and Cu indicate areas reserved for ceremonies or the production of artifacts (Wells et al., 2000). At the Tiel-Passewaij site in Holland, high concentrations of Ca and P were associated with areas that were once horse stables (Oonk et al., 2009).

Clearly, then, it is possible to link different chemical signatures with specific archeological contexts resulting from distinct patterns of political, economic, and ritualistic organization in past human groups characterized by varying levels of complexity. Based on this premise, the objective of the present study was to identify functional patterns in ADE-type soils from two prehistoric settlements of the lower Amazon Basin, based on the cross-referencing of geochemical and archeological data.

2. Study area

For the present study, two ADE archeological sites were selected – ADE-1 and ADE-2 (Figure 1). These sites are located on the right margin of the lower Amazon River in the municipality of Juruti, in the Brazilian state of Pará, and are surrounded by TMA soils.

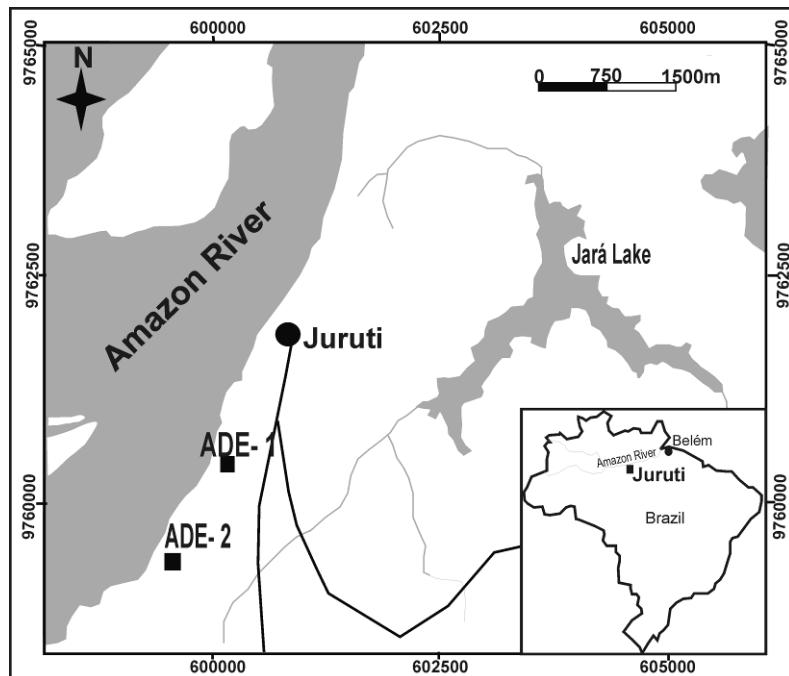


Figure 1. Location of the study area, showing sites ADE-1 (central coordinates: 21M 600159E/9760433N) and ADE-2 (central coordinates: 21M 599557E/9759353N), which are surrounded by TMA soils.

Sites ADE-1 and ADE-2, and the surrounding area of TMA soils are located on a ramped river terrace, of which the horizontal surfaces are surrounded by the fluvial and fluvial-lacustrine plains of the Amazon River. The course of the white-water Amazon is characterized by its instability, which is constantly modified by the simultaneous processes of erosion and sedimentation (Sioli, 1984). This is principal cause of the loss of part of the archeological sites originally found along the banks of the river (Costa, 2008).

The *terra firme* is dominated by medium-grained yellow Latosols, derived from the sandiest levels of the Alter do Chão Formation, which is overlaid by ADE and TMA soils in some parts of Juruti, close to the Amazon River. The vegetation at these sites is dominated by rainforest formations and secondary habitats. Balée (1989 and 1993) and Morán (1990) consider forests dominated by palms, bamboo and liana forests, and dense stands of Brazil-nut trees to be typical of habitats of anthropogenic origin in the Amazonian *terra firme*. The

presence of fruit-bearing trees such as the bacuri (*Platonia insignis* Mart.) and Spanish plum, known locally as taperebá (*Spondias lutea* L), is also indicative of disturbance and these species are often associated with soils containing charcoal and ceramic fragments, that is, the remains of agricultural settlements.

3. Materials and methods

The present study was based on the analysis of soil samples collected from the ADE and TMA areas, as well as the adjacent Latosol. Samples were collected from 50 cm x 50 cm excavations dug in a 60 m x 120 m grid, with 43 holes in ADE soils, 90 in TMA, and one in adjacent Latosol (Figure 2). Excavation was limited to a depth of 10-20 cm, which corresponds to the A₂ pedological horizon, in which the highest concentrations of ceramic fragments are found. The samples were collected in 2006 during the retrieval of archeological material by Scientia Consultoria Ltd.

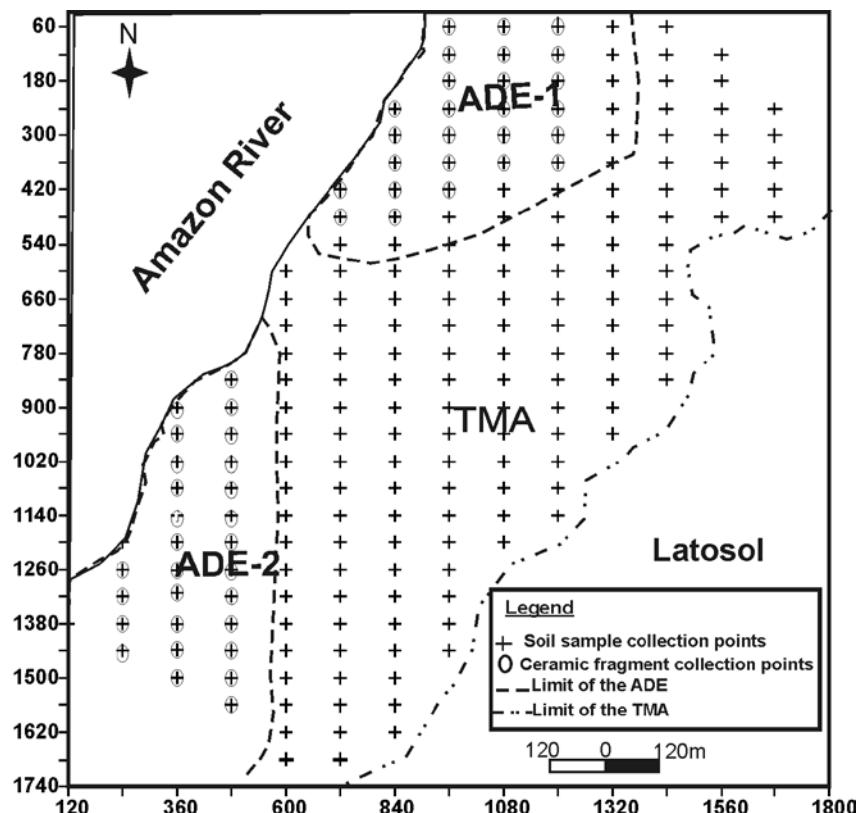


Figure 2. Map showing the distribution of ADE, TMA, and Latosol, and the sampling points for the ADE and TMA soils and the collection of ceramic fragments (CF).

3.1. Chemical analyses

The soil samples were analyzed in order to determine the concentrations of major (Al_2O_3 , Fe_2O_3 , TiO_2 , P_2O_5 , CaO , K_2O , Na_2O , and MgO) and trace elements (Mn, Cu, Zn, Cr, Sr, La, Li, Ni, Pb, V, Y, and Zr) using multi-acid digestion (hydrofluoric, nitric, and hydrochloric acids, and drops of perchloric acid) and analysis by ICP-OES, conducted by Geosol laboratories. Levels of organic matter (OM) were determined by the Walkley-Black procedure and quantified by volumetry in the soils laboratory of the Federal University of Viçosa. Statistical procedures (correlation and cluster analysis) were conducted in Statistica (version 6.0).

4. Results and discussion

The two main ADE sites (ADE-1 and ADE-2) are slightly elongated in shape, more or less parallel to the margin of the Amazon River (Figure 3). Based on Kern's (1996) reconstruction of human settlement patterns derived from the analysis of ADE soils, the sites appear to correspond to two distinct occupations. The thick layer of ADE exposed on the riverbank suggests that, in the past, these settlements extended as far as the river itself. The natural dynamic processes of the river, together with the recent deforestation of its margins, have likely contributed to increasing vertical erosion and subsidence of the banks, which in turn has provoked erosion of part of the ADE soils. The partial loss of the A horizon (occupied) is a result of the surface run-off of ADE topsoil.

Site ADE-1 covers an area of at least 29 hectares, although a considerable portion of its area (which may possibly be a distinct site) has been occupied by a recent-built suburb of the town of Juruti, known as Terra Preta (literally, "Black Earth"). The area of site ADE-2 is also at least 28 hectares. Heckenberger et al., (1999) have reported areas of ADE larger than this, such as a 30 ha site on the Xingu River and one of 50 ha in central Amazonia, which they associated with complex societies.

The two ADE sites are separated by a large area of TMA, of more than 300 hectares, although this area extends well beyond that shown on the map, which corresponds only to the area affected by the implantation of the ALCOA ore-shipping port. The area of TMA also extends as far as the riverbank (Figure 3). By analogy with the pioneering studies of Sombroek (1966) in Belterra, also in Pará, the area of TMA probably represents the settlements' plantations. At Belterra, an area of 200 hectares of ADE is surrounded by some

1000 hectares of TMA, which disperses outwards into the *terra firme*. In the present case, the two ADE sites and the TMA soils together cover an area of 350 hectares. Similar results were reported for Juruti by Smith (1999), apud Denevan (2002), who found ADE soils distributed over an area of 1 km by 3.5 km.

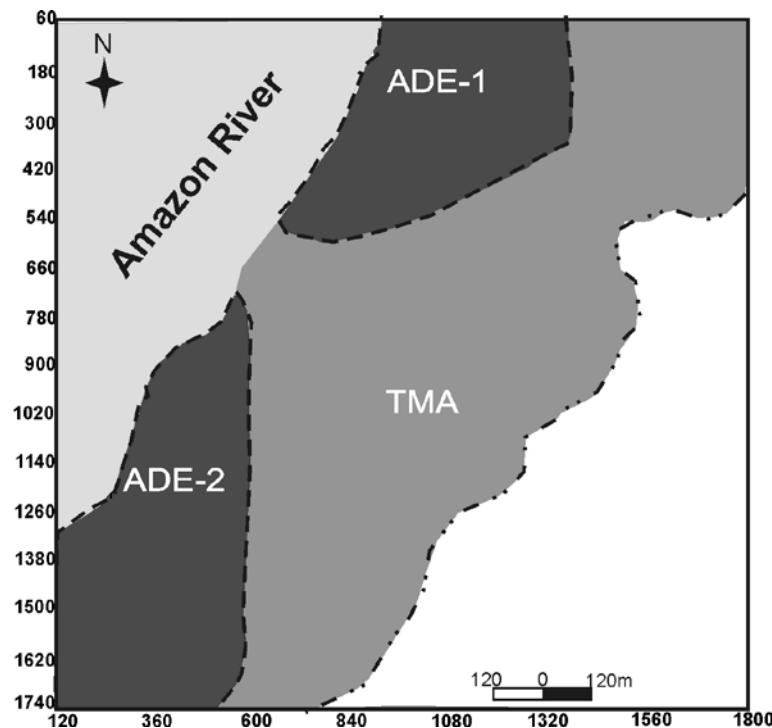


Figure 3: Distribution of the areas occupied by ADE and TMA soils, based on coloration and the presence of ceramic material.

4.1. Chemistry of the ADE soils vs. TMA

While only partial, the chemical analyses (Table 1) indicate that the soils mapped as ADEs and TMAs at Juruti cannot be distinguished on the basis of the mean concentrations of their principal components, such as Al_2O_3 , Fe_2O_3 , TiO_2 , and Na_2O . However, there is a clear distinction in the mean levels of P_2O_5 , CaO , K_2O , and MgO , the concentrations of which are much higher in the ADEs. In the case of the trace elements, Cr, Sr, La, Li, Ni, Pb, V, Y, and Zr were all present in relatively similar concentrations in ADE and TMA, although higher levels of Cu, Mn, and Zn were recorded in the ADEs. Overall, then, the set of elements P_2O_5 , CaO , K_2O , MgO , Cu, Mn, and Zn, all of which present relatively high concentrations in ADE, can be considered to be its geochemical signature on the basis of their contribution to the mineral content of the soils. Kern (1996) and Costa & Kern (1999) consider this chemical signature to be typical of ADE soils.

Table 1. Chemical composition of the ADE-1, ADE-2, TMA, and Latosol soils.

Element %	ADE-1				ADE-2				TMA				AD	
	n	x	min	max	n	x	min	max	n	x	min	max	n	
Al ₂ O ₃	24	7.5	4.50	10.00	19	6.00	5.00	6.50	90	8.00	5.00	12.50	1	5.32
Fe ₂ O ₃	24	3.5	2.10	5.00	19	3.00	2.20	4.50	90	3.00	2.00	7.40	1	1.85
TiO ₂	24	1.00	1.00	1.20	19	1.00	0.70	1.00	90	1.00	0.70	1.30	1	0.63
P ₂ O ₅	24	0.40	0.07	1.20	19	0.26	0.12	0.78	90	0.09	0.05	0.50	1	0.05
CaO	24	0.22	0.02	1.20	19	0.30	0.03	1.18	90	0.04	0.02	0.30	1	<0.01
MgO	24	0.06	0.03	0.20	19	0.08	0.03	0.21	90	0.04	0.02	0.07	1	0.06
K ₂ O	24	0.12	0.04	0.30	19	0.13	0.05	0.40	90	0.06	0.02	0.14	1	0.11
Na ₂ O	24	0.07	0.04	0.13	19	0.06	0.04	0.11	90	0.06	0.03	0.09	1	0.03
OM	1	-	-	5.12	1	-	-	3.84	2	2.60	1.68	3.52	1	2.56
mg kg ⁻¹														
Cu	24	15.00	6.00	37.00	19	16.00	8.00	36.00	90	7.00	4.00	18.00	1	6.80
Mn	24	216.00	<100.00	615.00	19	305.73	116.00	870.00	90	-	-	<100.00	1	<100.00
Zn	24	58.00	13.00	204.00	19	56.00	18.00	145.00	90	23.00	9.00	62.00	1	3.00
Cr	24	42.10	33.00	50.00	19	39.00	34.00	43.00	90	43.10	31.00	63.00	1	22.00
Ba	24	43.00	15.00	114.00	19	49.00	19.00	157.00	90	22.30	9.40	39.00	1	39.00
Sr	24	27.00	14.00	55.00	19	27.00	18.00	54.00	90	20.40	12.00	32.00	1	22.00
La	24	24.00	21.00	30.00	19	20.20	21.00	22.00	90	22.30	21.00	34.00	1	21.00
Li	24	4.50	3.40	8.00	19	5.00	3.10	11.00	90	3.50	3.10	5.00	1	4.90
Ni	24	10.00	6.50	13.00	19	8.00	5.50	13.00	90	7.50	5.00	11.00	1	3.00
Pb	24	12.00	8.70	28.00	19	10.00	8.50	17.00	90	10.00	8.10	22.00	1	14.00
V	24	82.00	57.00	108.00	19	71.00	56.50	83.00	90	90.00	53.00	135.00	1	64.00
Y	24	7.30	3.70	11.00	19	7.00	5.20	9.00	90	8.50	5.20	14.00	1	2.90
Zr	24	154.40	121.00	198.00	19	126.00	99.00	152.00	90	158.00	104.00	224.00	1	87.00

n = number of samples analyzed x = mean

The chemical differences, which are clear from a comparison of the mean values (Table 1), are further emphasized by the cluster analysis, based on Pearson's correlation coefficient. The following geochemical associations (groups) were identified within the area dominated by ADE:

The P₂O₅-CaO-MgO-Na₂O-K₂O-Zn-Cu-Mn-Ba-Sr-Li-Ni association (group 2) is formed by elements considered to be typical of ADE, in addition to the elements Na, Li, and Ni. These elements are indicative of human settlement (Kern & Kampf, 1989; Kern, 1996; Costa & Kern 1991; Wells et al., 2000; Goffer, 2007, Holliday & Garter, 2007; Wilson et al., 2008; OonK et al., 2009), and represent the ADE signature (Figure 4). These results are consistent with those obtained at other Amazonian ADE sites (Kern, 1996; Costa & Kern, 1999; Kampf et al., 2003).

The Al₂O₃-Fe₂O₃-TiO₂-La-V-Y-Zr association (group 1) can be interpreted as the signature of the soils derived from lateritic formations, which underlie the ADE sites in the present study area (Figure 4). This signature clearly represents the adjacent soils, which are of a similar composition, as identified by Kern (1996) at Caxiuanã, and by Costa (1991), Horbe & Costa (1997) and Horbe et al., (2005) at a number of other Amazonian sites. At site ADE-1, two sub-associations were recognized: Al₂O₃-TiO₂-Zr (group 1A) and Fe₂O₃-V-Cr-La

(group 1B), which represent the principal minerals – kaolinite + anatase + zircon and goethite + hematite, respectively – found in soils derived from lateritic formations (Costa, 1991; Horbe & Costa, 1997). This demonstrates that the chemical transformations induced by human settlement did not eliminate completely the geochemical signatures of the original soils, and at the same time, reinforces the assumption that the ADE, and even the TMA soils were derived from these original substrates.

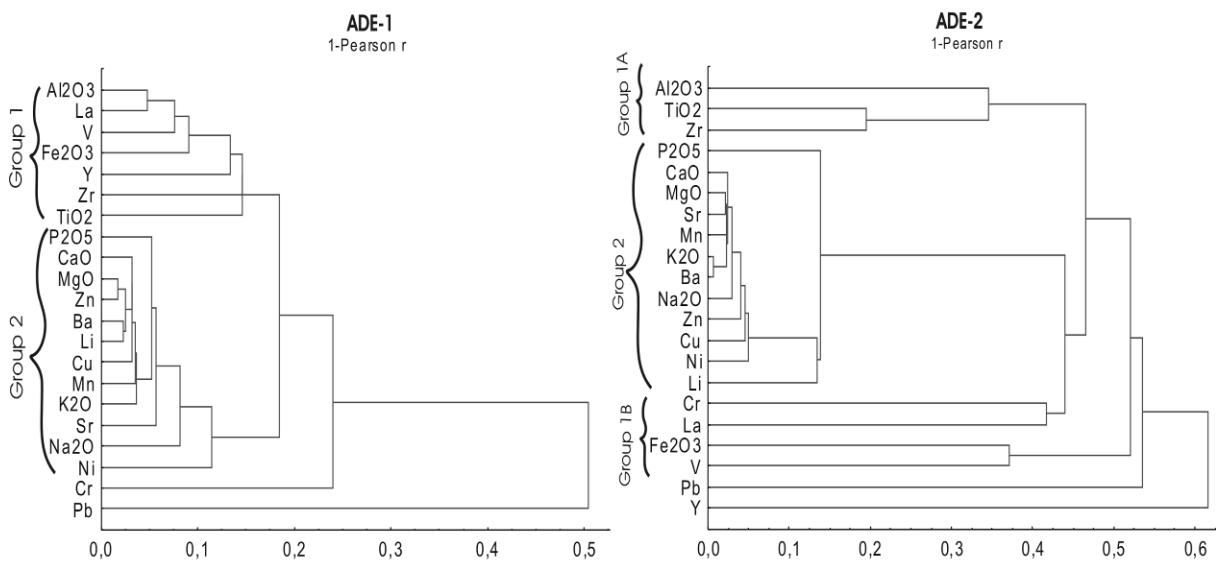


Figure 4. Geochemical associations derived from the dendrograms of the cluster analysis of the concentrations of different chemical elements in the soils from sites ADE-1 and ADE-2.

It was not possible to identify chemical groupings characteristics of the TMA soils, only the definition of a possible association by greater similarity, Al_2O_3 -Sr-Cr-V- K_2O -Ba-MgO-TiO₂ (Figure 5), a partial legacy of the signatures of both the ADE and adjacent soils. This appears to further reinforce the conclusion that the TMA soils represent the products of the physical and chemical degradation of the ADE soils, by leaching out most of the chemical information associated with the organic matter, but retaining residues of the subsurface and adjacent soils, which are hyper-stable under the prevailing tropical conditions, related to typically stable minerals, like oxy-hydroxides of iron (OHFe) such as goethite and hematite. The marked correlations between K_2O and Ba ($r = 0.96$, $n = 90$) and CaO and Zn ($r = 0.96$, $n = 90$), which are also typical of ADE soils, indicates that the TMAs maintain a partial ADE signature.

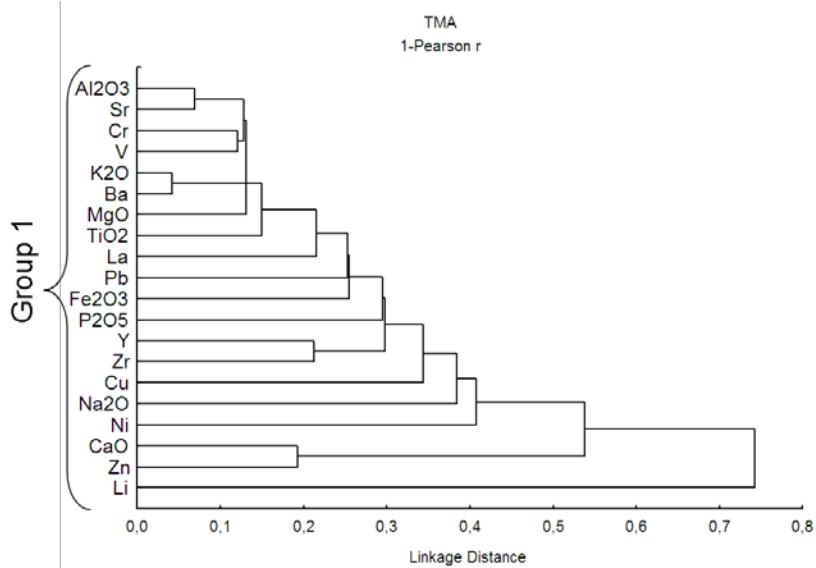


Figure 5. Dendrogram of the cluster analysis of the concentrations of chemical elements found in the TMA soils surrounding sites ADE-1 and ADE-2.

4.2. Spatial distribution of the principal chemical components in ADE and TMA soils

Given that the two principal types of soil were originally mapped based on their characteristics (color, presence of ceramic fragment, and organic content), which corresponded perfectly with their chemical composition and associations, it was possible to construct maps of isolines for the concentrations of the specific chemical elements of each association, in order to evaluate their distribution patterns and interactivity. This analysis also provided the basis for a discussion of the occupation of the region by prehistoric peoples. Isoline maps of the chemical elements of the surface soil horizon have been used as indices of functional areas in a number of archeological studies (Kern & Kampf, 1989; Kern, 1996; Wells et al., 2000; Wilson et al., 2008; Oonk et al., 2009). For example, phosphorus may be an important indicator and delimiter of human occupation (Wells et al., 2000; Goffer, 2007; Holliday & Garter, 2007).

The isoline maps of the elements of the P_2O_5 -CaO-MgO-Cu-Mn-Zn association (ceramic fragments) at Juruti discriminate the ADE and TMA domains quite clearly, with the isolines of higher values corresponding to the ADE and lower values to the TMA (Figure 6). The distribution pattern of the values for P_2O_5 , CaO and MgO reveals two ample areas with higher concentrations, running parallel to the Amazon, coinciding with the location of sites ADE-1 and ADE-2 (Figure 6). The highest values are found parallel to the riverbank, reinforcing the conclusions based on the intensity of the dark coloration and the presence of ceramic fragments. The higher concentrations recorded at ADE-1 (Table 1, Figure 6) may be

interpreted as the result of a much longer period of occupation in comparison with site ADE-2.

The distribution pattern observed at site ADE-1 supports the conclusion that the settlement originally extended further north into the urban area of the town of Juruti. The isolines for lower values delineate clearly the TMA domain, which can be interpreted as an area of agricultural activity, which has been improved by the insertion of OM (1.68-3.52%), as shown by the variable levels of CaO (0.02-0.30%), MgO (0.02-0.07%), and P₂O₅, 0.05-0.50% (Table 1). The values are consistent with those for MO (1-6%) and P₂O₅ (0.04-0.12%) recorded in ancient agricultural terraces constructed in the Peruvian Andes for growing maize, *Zea mays* (Branch et al., 2007), as well as the concentrations of Ca (0.09-0.26 %) and P (0.04-0.13 %) found in cultivated fields (Wilson et al., 2008).

The isolines delimiting the highest values for P₂O₅, CaO, and MgO overlap with the areas mapped for the two ADE sites, emphasized primarily by phosphorus and calcium, as the principal components of the phosphates, as well as apatite, the principal mineral or chemical compound of fish and mammal bones, as well as those of chelonians, and excrement (Smith, 1980; Neves et al., 2003; Goffer, 2007). These components form a clear association and together distinguish the same configuration. Microscopic bone fragments found in the ADE soils were in fact composed of apatite. This configuration is further reinforced by the similarity of the composition of the soils in the two ADE sites.

The positive correlation between P₂O₅ and CaO levels observed at ADE-1 ($r = 0.90$, $n = 24$) and ADE-2 ($r = 0.79$, $n = 19$) is quite conclusive, and contrasts clearly with the situation found in the TMA ($r = 0.17$, $n = 90$), which suggests different sources for these elements (Figure 7), considering that Ca and P normally occur at low concentrations in lateritic soils (Costa, 1991; Horbe & Costa, 1997), except in soils derived from formations rich in primary phosphates. The areas with the highest concentrations of CaO, P₂O₅, and MgO coincide with those in which ceramic fragments are most common, and probably correspond to sites occupied over longer periods, where foods were processed and prepared, and also discarded, together with parts of ceramic utensils. This conclusion is supported by the presence of fired ceramic structures, a type of oven associated with charcoal, ceramics and stone micro-chips (Figure 6). In addition, the semicircular or possibly circular (considering the intense erosion of the riverbank) configuration of the remains, together with the spatial distribution of the chemical anomalies, suggests the organization of residential cabins around a communal space (Figure 6). The whole array corresponds to an area of approximately 500-600 m across, a

pattern typical of indigenous settlements in the Amazon to this day (Heckenberger et al., 1999; Neves, 2009).

As for P₂O₅, CaO and MgO, the isolines for Cu, Zn, and Mn indicate higher values in the ADE (Figure 6). The distribution of these elements in ADE-1 is clearly related to the location of sites at which foodstuffs were manipulated and consumed, that is, where deposits of organic residues are concentrated (Wells et al., 2000; Goffer, 2007). The Cu, Mn, and Zn isolines form an elongated plot interspersed with lower levels (Figure 6). In ADE-2, two areas are delineated by isolines for higher values, separated by areas of lower concentrations, indicating less occupied areas or access paths.

The numerous spaces of approximately two hectares outlined by medium-level isolines in the TMA can be interpreted as sites which received an input of organic residues, as indicated by the higher concentrations of Cu (0.40-18.00 mg kg⁻¹) and Zn (20.00-62.00 mg kg⁻¹), as well as P₂O₅ (0.07-0.50%) and CaO (0.04-0.08%). Moderate levels of Ca (0.09-0.26%) and Zn (30.20-80.80 mg kg⁻¹) have been related to the presence of abandoned gardens and cultivated fields in the United Kingdom (Wilson et al., 2008). By contrast, the Mn content of the TMA samples is at or below the limit of detection (<100 mg kg⁻¹) (Table 1, Figure 6). This reflects the instability of the OHFe resulting from the intense leaching of deposits of organic matter.

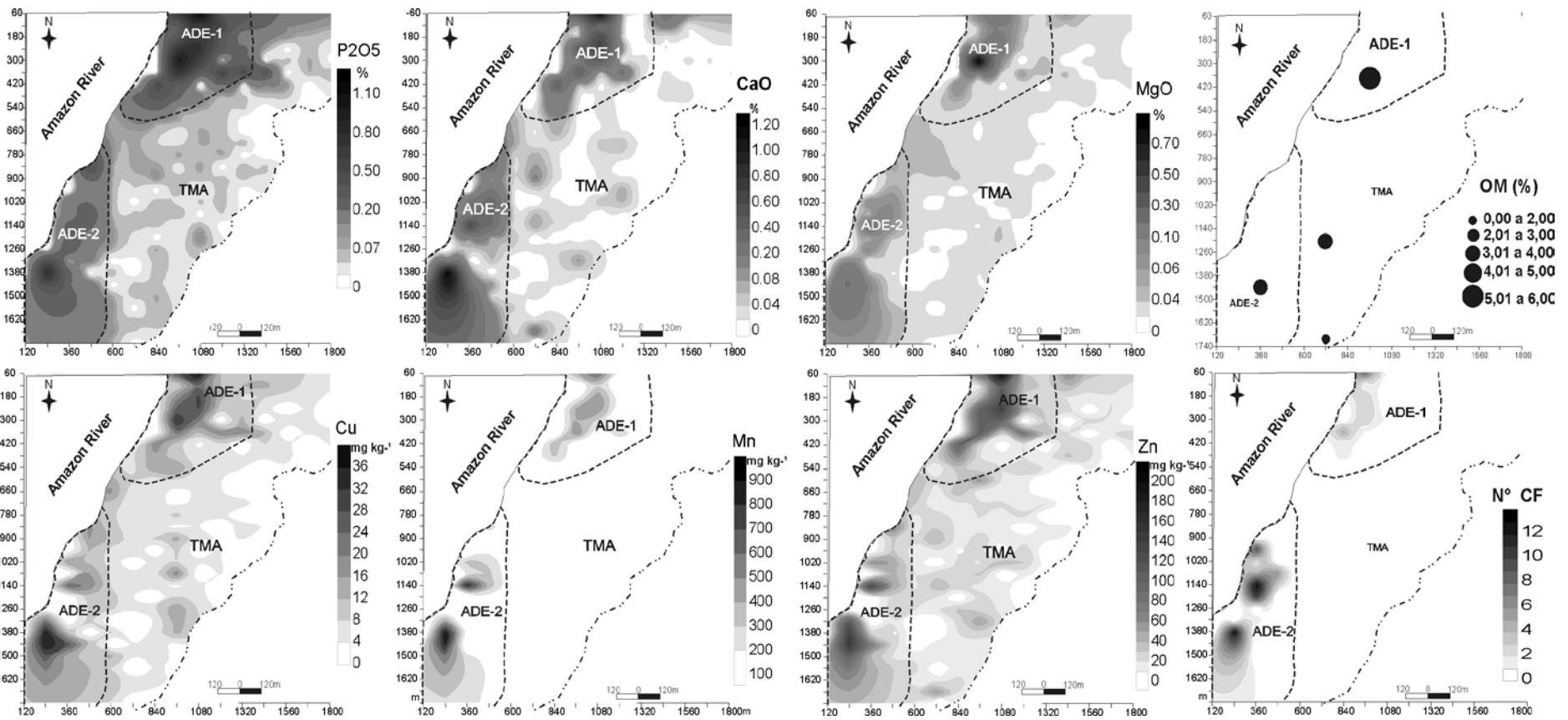


Figure 6. Spatial distribution of the concentrations of P_2O_5 , CaO , MgO , Cu , Mn , Zn , organic matter (OM), and ceramic fragments in the areas dominated by ADE and TMA soils.

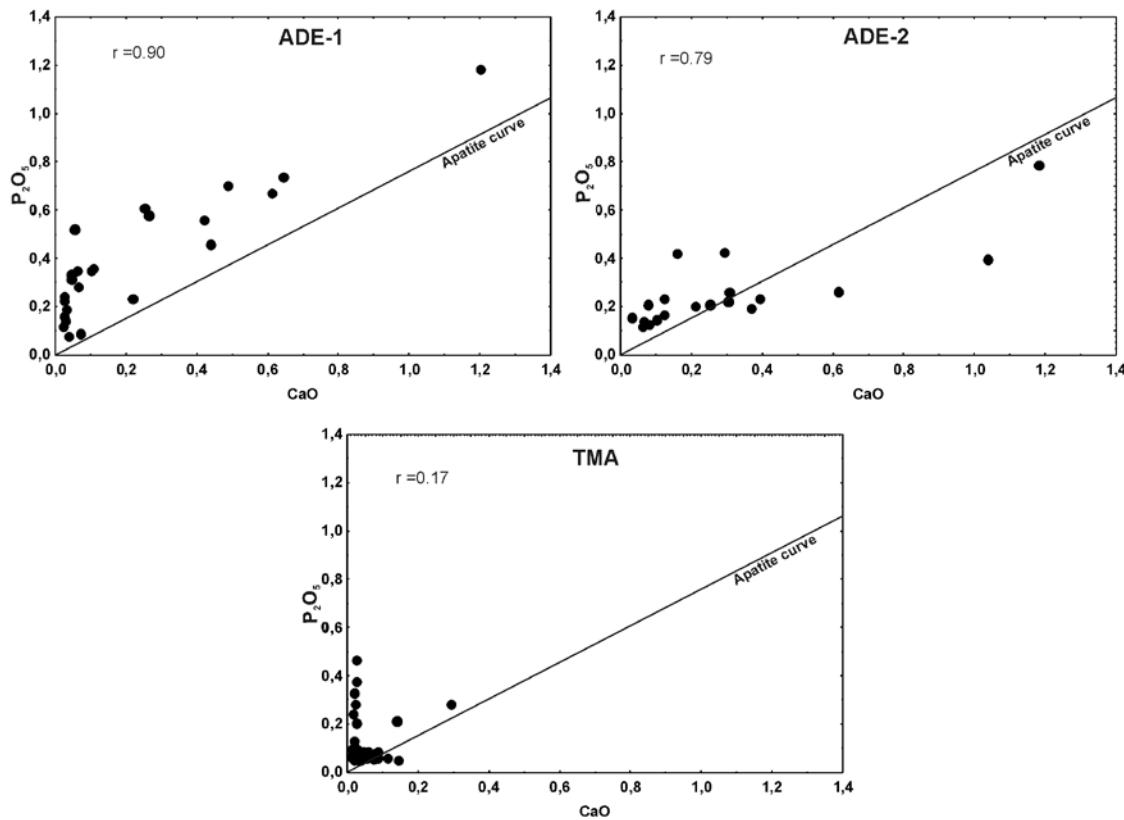


Figure 7. Correlation between the concentrations of P_2O_5 and CaO at sites ADE-1 ($n = 24$), ADE-2 ($n = 19$) and TMA ($n = 90$) in relation to the apatite curve.

On the other hand, the elements of the Al_2O_3 - Fe_2O_3 - TiO_2 - V - Y - Zr association, which is characteristic of the soils derived from the lateritic subsoil from which the ADE and TMA originated (Horbe & Costa, 1997), are strongly delineated by the isolines, and contrast with the high levels of the elements typical of human occupation found in the TMA, and in particular in the ADE. The superficial distribution of the levels of Al_2O_3 , Fe_2O_3 , TiO_2 , Zr , and Y indicates that the highest concentrations are found in the central-northern part of site ADE-1, indicating that deposits of organic matter were relatively limited in this area, or may have been eroded or leached out, leading to a greater stability in OHFe and OHAl (Figure 8). In ADE-2 and part of the TMA, by contrast, the lower levels of the elements of this association suggest the opposite pattern. The areas with lower concentrations of Al, Fe, Zr, and Y are correlated with the sites at which human settlement was probably more extensive, as indicated by the chemical signature of the ADEs and the greater frequency of ceramic fragments.

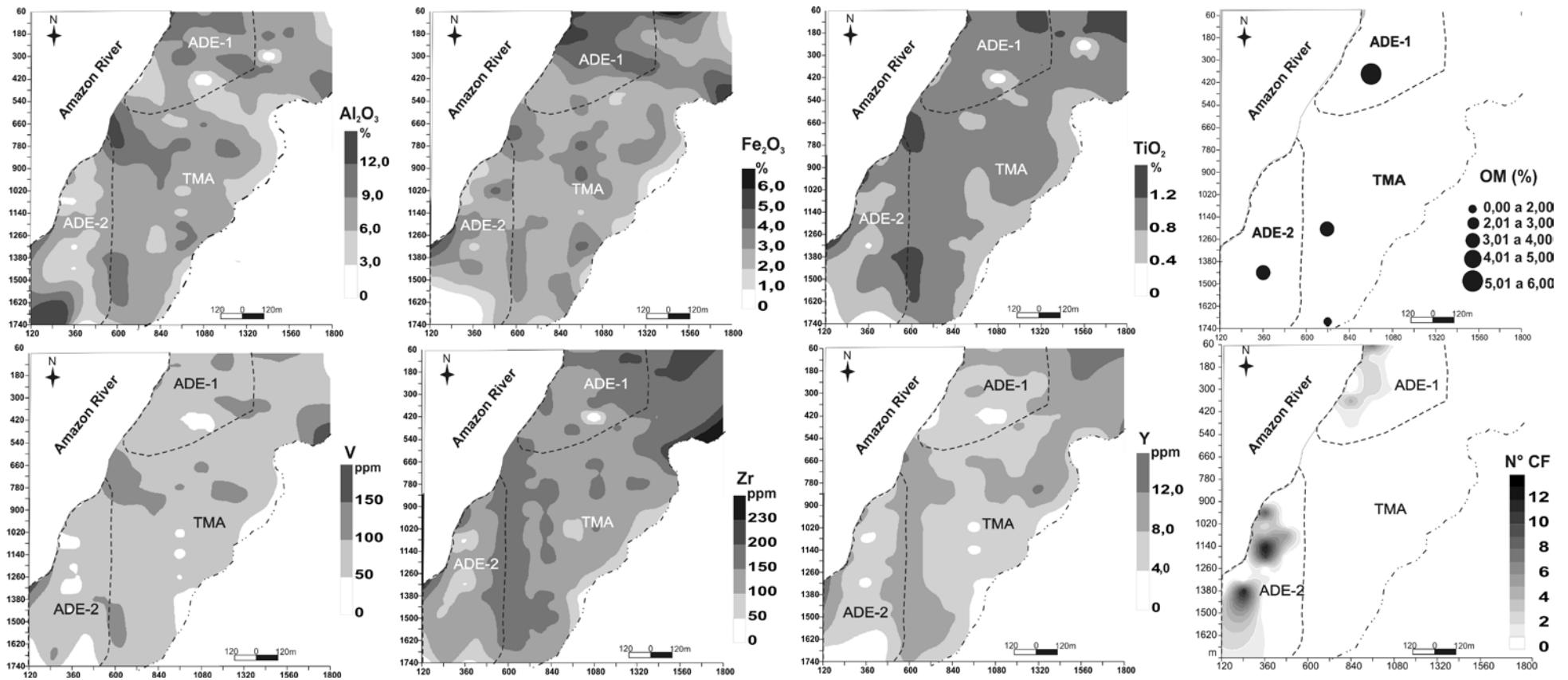


Figure 8. Spatial distribution of Al_2O_3 , Fe_2O_3 , TiO_2 , Zr, V, Y, organic matter and ceramic fragments in the areas dominated by ADE and TMA soils.

4.3. Ceramic archeology

The ceramic fragments found at sites ADE-1 and ADE-2 correspond to multi-component artifacts, with a predominance of the Konduri and Pocó ceramic styles. These styles are relatively common in the settlements of the lower Amazon basin. Konduri ceramics are characterized by an abundance of spicules of cauixi, as well as pointing and complex modeling, while the Pocó style is also defined by this complex modeling, as well as the frequent presence of large spiral incisions and elaborate painting (Hilbert, 1955; Hilbert and Hilbert, 1980; Scientia, 2008).

The subsurface distribution of the ceramic fragments runs parallel to the Amazon river (Figures 6 and 8). In these areas, the highest concentrations of ceramic fragments coincide with the highest concentrations of the elements indicative of human occupation (P_2O_5 -CaO-MgO-K₂O-Zn-Cu-Mn-Ba-Sr), which indicates that these were the sites of greatest occupancy or activity, and thus related to domestic units such as cabins, and areas at which foods were stored, prepared, and consumed. The distribution of ceramic material within site ADE-1 delineates a continuous, semicircular area of approximately 6 hectares (Figure 6). Within ADE-2, ceramic material is distributed within two distinct sites, in the north (6 ha) and south (5 ha) of the area. This distribution coincides with the typical chemical signatures of ADE soils and suggests that, during the most recent period of human occupation, the southern settlement was the most intensively occupied, which is reflected in the lower values of Al, Fe, Zr, and Y in ADE-2, as a result of the more recent, accumulated deposits of organic matter, which have diluted the concentrations of these elements.

Within the ADEs, the areas in which ceramic fragments were rare or absent corresponded with those in which the lowest concentrations of P, Ca, Mg, Mn, Cu, and Zn were found, and were thus related to the least occupied or peripheral areas of the villages. However, where there are no ceramic fragments, the chemical content (P, Ca, K, Mg, Cu, Zn, and Ba) remains high, indicating the accumulation of organic residues, so these areas were interpreted as sites at which material from game, fish or plants was discarded. No ceramic fragments or any other type of archeological structure were found on the periphery of the ADE sites, or in the TMA (Figures 6 and 8).

4.4. Hypothetical settlement pattern

The geochemical isoline maps of the chemical elements representative of the two principal associations (Figure 4) provide a baseline for the interpretation of the possible pattern of prehistoric human occupation of the study area (Figure 9), supported by the archeological data presented above. The areas dominated by anomalously high values of P, Ca, Mg, Mn, Zn, Cu, Ba, and Sr, as well as ceramic fragments and organic matter, are assumed to correspond to sites of human occupation, at which everyday activities, such as the preparation, consumption, and storage of foods, and the production of tools and artifacts were conducted, and cabins and refuse deposits were constructed. Based on these chemical signatures, the areas of archeological dark earth at Juruti appear to correspond to not two, but probably as many as seven settlements, covering 2-3 hectares at ADE-1 and up to 6 ha at ADE-2, located along the bank of the Amazon (Figure 9).

However, two of these, one located in the eastern portion of ADE-1 and the other in the north of ADE-2 were characterized by relatively high levels of P_2O_5 , CaO, Mg, Cu, Zn, Ba, and Sr, similar to those found in the settlements, but did not contain ceramic fragments (Table 2). The lack of ceramic fragments indicates that these areas may have been communal sites that included fireplaces and ovens, as well as residues of foods such as game (skins and bones) and fish, or even human or animal excrement (Smith, 1980; Oonk et al, 2009; Sullivan & Kealhofer, 2004). In other words, these sites may represent locations for the disposal of specific types of residues.

However, the dimensions of the sample grid (60 m x 120 m) were relatively ample, and may thus have been insufficient for the delimitation of specific sites such as residences, waste deposits, transit areas and communal spaces. It was nevertheless able to delimit the area of daily life in the village. The proximity of the settlements, the abundance and distribution of ceramic styles, and the dark color of the soil all contributed to the apparent homogeneity of these areas. These common features indicate that the inhabitants of these settlements shared the same habits or belonged to the same group.

The isolines that correspond to medium-range values for the typical anthropogenic geochemical association in areas with no ceramic fragments delimit an ample area to the rear of the seven villages, making up an almost homogeneous space forming the TMA (Table 2). This area can be interpreted as temporary agricultural plots on *terra firme* soils, indicated primarily by the small patches of slightly higher values (Figure 10). At least seven of these patches were also

identified. Soils of this type with no ceramic fragments or archeological structures are typical of agricultural plots (Sombroek, 1966; Sombroek et al., 2002, 2010).

The total area of TMA forms a distinct zone parallel to the margin of the Amazon, ranging inland some 700 m, where there is a transition to the adjacent soils, which have a signature typical of the region as a whole. The TMA zone reaches as far as the riverbank between the two settlements, ADE-1 and ADE-2. This zone is indicated clearly by the isolines for higher concentrations of the elements of the primary association with latosols, that is Al-Fe-Ti-V-Y-Zr (Figure 9). A north-south corridor in the transition between ADE-2 and TMA is indicated by the high values for the Latosol association (Figure 9) and the lower values for the anthropogenic geochemical association (Figure 8). This corridor appears to extend as far as the major feature that separates the two ADE sites. The corridors do not appear to have been the focus of human occupation or intense agricultural activity, but appear to form a relatively wide, long neutral area used for access to the river, possibly even to a dock. A second, similar corridor appears to have existed in the north, associated with ADE-1, with an approximately east-west orientation.

Radiocarbon dating (20-30 cm layer) indicates that the human population which (intentionally or not) left the archeological and chemical residues for the formation of the ADE and TMA soils settled here between 640 ± 50 and 690 ± 40 years ago, respectively (Scientia, 2008). This period corresponds to the 13-14th centuries, in other words, just prior to the arrival of European colonizers.

Table 2. Mean content and standard deviation (S.D) of chemical elements in the area of the villages, TMA soils and Corridor.

Element	Village		TMA		Latosol
	with CF	without CF	high values	medium values	Corridor
%	mean \pm SD	mean \pm SD	mean \pm SD	mean \pm SD	mean \pm SD
Al ₂ O ₃	6.94 \pm 1.60	7.43 \pm 1.51	8.05 \pm 1.42	7.34 \pm 1.34	9.14 \pm 1.97
Fe ₂ O ₃	3.36 \pm 0.84	3.35 \pm 1.04	3.24 \pm 0.97	2.73 \pm 0.46	3.20 \pm 0.41
TiO ₂	0.94 \pm 0.14	0.96 \pm 0.14	1.03 \pm 0.14	0.93 \pm 0.11	1.16 \pm 0.10
P ₂ O ₅	0.36 \pm 0.24	0.39 \pm 0.15	0.13 \pm 0.11	0.07 \pm 0.01	0.07 \pm 0.01
CaO	0.31 \pm 0.33	0.18 \pm 0.11	0.05 \pm 0.06	0.03 \pm 0.02	0.04 \pm 0.02
MgO	0.08 \pm 0.05	0.05 \pm 0.01	0.04 \pm 0.01	0.03 \pm 0.01	0.04 \pm 0.01
K ₂ O	0.14 \pm 0.09	0.11 \pm 0.03	0.06 \pm 0.02	0.06 \pm 0.02	0.08 \pm 0.03
NaO ₂	0.07 \pm 0.02	0.07 \pm 0.01	0.06 \pm 0.01	0.06 \pm 0.01	0.06 \pm 0.01
mg kg ⁻¹					
Mn	292.13 \pm 228.18	182.50 \pm 87.14	< 100.00	< 100.00	< 100.00
Zn	64.48 \pm 49.38	55.50 \pm 21.05	27.18 \pm 10.68	20.27 \pm 7.83	22.76 \pm 7.79
Cu	16.80 \pm 8.49	15.45 \pm 3.97	7.39 \pm 3.46	5.26 \pm 1.10	9.78 \pm 2.65
Ba	50.45 \pm 34.63	41.75 \pm 12.58	23.09 \pm 5.85	20.03 \pm 5.25	25.53 \pm 7.72
Cr	41.06 \pm 3.87	42.50 \pm 2.89	43.45 \pm 6.31	40.11 \pm 5.71	49.35 \pm 6.86
La	22.39 \pm 3.25	22.50 \pm 2.89	23.03 \pm 3.45	20.54 \pm 3.41	24.94 \pm 4.85
Li	4.88 \pm 1.80	4.25 \pm 0.70	3.56 \pm 0.39	3.62 \pm 0.48	2.96 \pm 1.56
Ni	9.12 \pm 2.44	9.25 \pm 2.90	8.16 \pm 1.33	7.16 \pm 1.20	7.54 \pm 1.00
Pb	10.50 \pm 2.48	13.62 \pm 2.70	11.17 \pm 3.78	8.56 \pm 1.08	9.80 \pm 2.69
Sr	28.90 \pm 10.27	26.00 \pm 6.16	20.97 \pm 3.53	18.43 \pm 3.20	24.49 \pm 4.40
V	77.70 \pm 13.60	82.55 \pm 15.29	90.89 \pm 13.89	84.55 \pm 14.12	101.14 \pm 13.20
Y	7.30 \pm 1.38	6.83 \pm 1.45	8.38 \pm 2.27	7.77 \pm 1.86	10.21 \pm 1.07
Zr	142.49 \pm 27.78	146.50 \pm 20.57	160.12 \pm 27.80	147.77 \pm 21.19	176.59 \pm 13.53

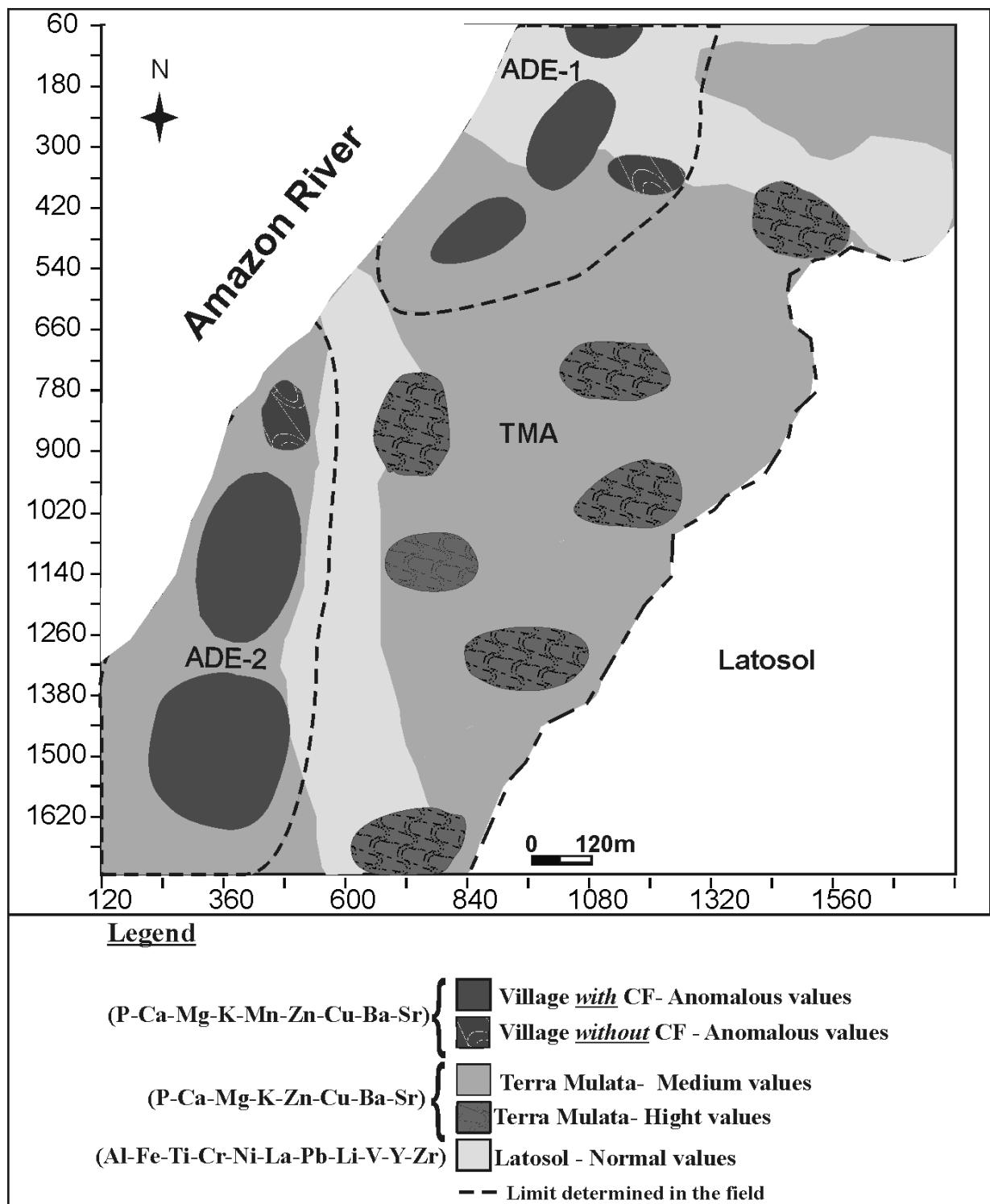


Figure 9. Hypothetical settlement pattern for the study area.

5. Conclusions

The ADE (Archeological Dark Earth) and TMA (Terra Mulata) soils found at Juruti are very similar to those found at other Amazonian sites located within an area of yellow latosols. The ADEs are found in areas adjacent to the Amazon River, while the TMAs are peripheral, with the whole area being surrounded by latosols. The ceramic fragments are typical of ADEs, but are much rarer in the TMA. The chemical composition of the ADEs and TMAs indicates their derivation from adjacent soils, in particular with regard to the levels of Al_2O_3 , Fe_2O_3 , TiO_2 , V, Y, and Zr, although their concentrations of P_2O_5 , CaO, MgO, Mn, Zn, Cu, Ba, and Sr are divergent, with distinctly higher levels in the ADE soils and medium levels in the TMA in comparison with the adjacent soils. The concentrations of P_2O_5 and CaO in the ADE reflect the presence of microfragments of bone, in the form of apatite as well as amorphous phosphates, probably of aluminum.

Two geochemical associations were identified – $\text{P}_2\text{O}_5\text{-CaO-MgO-Mn-Zn-Cu-Ba-Sr}$, which distinguished the ADEs categorically, and the TMAs partially, and $\text{Al}_2\text{O}_3\text{-Fe}_2\text{O}_3\text{-TiO}_2\text{-Y-V-Zr}$, which is typical of the adjacent latosols, and can be identified in both ADEs and TMAs, indicating their derivation from these adjacent substrates. The geochemical maps of the elements associated with anthropogenic soils delimit perfectly the area within which ceramic fragments were found, as well as two smaller areas, which are interpreted as possible communal sites, and were not occupied by waste deposits during shifts in occupation. The TMA zone is demarcated by medium values for the anthropogenic association, and the absence of ceramic fragments. Slightly higher values were identified as possible campsites. Corridors between the two ADE sites and between these sites and the area of TMA were identified through the higher values of the association with Al_2O_3 .

The large areas of both ADE and TMA indicate that human activity was both extensive and intense. Overall, the geochemical signature typical of ADE soils, i.e. $\text{P}_2\text{O}_5\text{-CaO-MgO-K}_2\text{O-Na}_2\text{O-Zn-Cu-Mn-Ba-Sr-Li-Ni}$ (Kern and Kampf, 1998; Kern, 1996; Lima et al., 2002), covered an area of 350 ha, probably with distinct patterns of spatial organization.

The same ceramic styles were found in the two ADE sites, suggesting that the human groups that occupied these settlements between the 13th and 14th centuries probably shared the same customs or practices, or belonged to the same group. The greater abundance of ceramic

fragments in the more superficial levels of the soil, which coincide with lower values of the Al₂O₃ association, indicates that the recent occupation of ADE-2 was more intense.

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4.3 TECHNOLOGY, MINERALOGY, AND CHEMISTRY OF ARCHEOLOGICAL CERAMICS FROM TWO DARK EARTH SITES IN THE LOWER AMAZON BASIN
 (Submetido ao periódico Archaeometry)

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ABSTRACT

In the context of Amazonian archeology, Juruti, in the lower Amazon basin, is one of the most culturally diverse and complex, given its location at the center of the region that produced the most refined and elegant ceramic styles during this period. At this site, large quantities of ceramic fragments in the Konduri and Pocó styles can be found throughout a 60-hectare area of Dark Earth (ADE). The archeological analyses indicate that the ADE areas were occupied by pottery-making peoples, since approximately 140 B.C. and up until the 14th century. The archeological analyses, mineralogy and multi-element chemical of samples of ancient ceramic fragments and the clay used by present-day potters for the production of ceramic vessels, which has thus been used locally for the production of ceramics for more than two thousand years. The ceramic fragments are rich in phosphorus (1 to 4% P_2O_5) in the form of Al and Fe phosphates. The phosphorus is nevertheless found at crustal levels in the local clay, which indicates that it originated from other sources, such as the use of the vessels for cooking food, a pattern recorded previously at other Amazonian sites.

Key words: Ceramic fragments, Raw material, Mineralogy, Archeology, Amazonia.

INTRODUCTION

The prehistoric cultures of the lower Amazon Basin are characterized by their complexity and diversity, emphasized by a rich ceramic tradition, which is the oldest of the New World, with the earliest finds coming from Taperinha, south of the city of Santarém, dating to 7000 BP (Roosevelt 1992). These ceramics are among the most refined and elegant known, and include the Tapajônica, Marajoara, Konduri, Globular, Pocó, and Parauá styles (Hilbert 1955; Hilbert & Hilbert 1980; Roosevelt 1992; Gomes 2008; Guapindaia 2008). The Parauá style has been discovered only recently (Gomes 2008).

The Tapajônico and Koduri ceramic styles dominated the Trombetas-Nhamundá river basins as far as the confluence of the Tapajós River with the Amazon, and share many stylistic

and chronological similarities. The ceramic artifacts are rarely found intact, but more typically as fragments inserted in soils of the Archeological Dark Earth (ADE) type. These soils correspond to recent prehistoric settlements in the region (Hilbert 1955; Kern & Kampf 1989, 2005; Roosevelt 1992). The two styles are contemporary and represent the incision and stippled tradition, which date from between the tenth and the sixteenth centuries (Simões 1984; Gomes 2002). These styles are characterized by extensive modeling, stippling and deep, dense incisions. Artifacts include relative fine and carefully elaborated vessels with narrow necks and caryatids, which are typical of the Tapajônico style, while the conical (tripod) bases are typical of the Konduri style (Hilbert 1955; Roosevelt 1992). The Konduri style was more widely distributed, and has been considered a derivative of the Tapajônico style (Hilbert 1955; Simões 1984; Gomes 2008).

While it is sometimes found together with Konduri pieces, the Pocó style is normally found in the upper archeological strata (Hilbert & Hilbert 1980; Guapindaia 2008). The principal characteristics of this ceramic style are the modeling, large and spiraled incisions related to the Barrancoid or modeled incision tradition, from the first millennium, found primarily in the Orinoco basin, and the middle and lower Amazon (Lathrap 1970; Hilbert & Hilbert 1980; Lima *et al.* 2006). The marked chronological and technological differentiation of the Konduri and Pocó styles is not reflected in stratigraphic patterns, given that the continuous formation of Dark earth deposits obscures the shifts between the different stylistic complexes (Guapindaia 2008).

In addition to the importance of this cultural record, these ceramic artifacts and their fragments are noted for their resilience to the intense weathering provoked by the region's hot and humid equatorial climate, which sustains its dense rainforests, but also decays most material deposited in the soil. The ceramic fragments are practically the only archeological remains to survive, but these remains permit the identification of technological aspects of the production process, such as painting, appliqués, tempers, firing temperatures, dimensions, forms, and designs, as well as the sources of the raw material, all indicators of cultural complexity. All these features can contribute to the understanding of cultural exchange and shifts, and the distribution of commercial routes between ancient settlements (Latini *et al.* 2001; Costa *et al.* 2004a; Goffer 2007; Schaan 2007).

Mineralogical, chemical, and geochemical studies have provided important insights for the development of archeological research, based on new approaches to the analysis of

specimens. For example, the presence of phosphates in ceramic fragments has raised a number of questions with regard to the origin of the phosphorus. Costa et al. (2004a, 2004b, 2006, 2011) and Rodrigues (2010) demonstrated that the phosphorus is derived from the function of the artifacts and the diet of their users. In addition, Costa *et al.* (2004, 2006, 2011), Rodrigues (2010), and Silva (2010) argue that the fertility of ADE soils, in particular as a major source of macro- and micronutrients, is related primarily to the gradual decomposition of the ceramic fragments they contain. Knowledge of the distribution of the phosphorus within the structure of the vessels may help reconstruct them from the ceramic fragments (Duma 1972). The high levels of phosphorus found in Amazonian ceramics, generally of the order of 1-3% P₂O₅ (Costa *et al.* 2004a, 2006, 2011), are much higher than those found in equivalent residues at European sites (Millani *et al.* 1998; Rathossi & Pontikes 2010).

Recent archeological studies in the lower Amazon Basin, in particular those in the municipality of Juruti, where archeological sites have been salvaged prior to the installation of a bauxite processing plant, have mapped large areas of ADE soils rich in ceramic fragments, which are still partially preserved from weathering. The composition of these remains was investigated using the mineralogical and geochemical techniques discussed above, and the results of the analyses are presented and discussed in the present study.

STUDY AREA

The area surveyed in the present study is located on the right bank of the lower Amazon River, in the municipality of Juruti, in the Brazilian state of Pará (Fig.1). The area is characterized by ample areas of ADE, surrounded by “Terra Mulata” (slightly dark earth), from which two archeological sites, denominated ADE-1 and ADE-2, were selected for the present study. The two sites cover a total of approximately 60 hectares of ADE soils. These soils were established over the yellow latosols which dominate the area and are found adjacent to the main survey area. The archeological horizon of the ADE soils is found at depths of between 60 and 150 cm, and is rich in ceramic fragments.

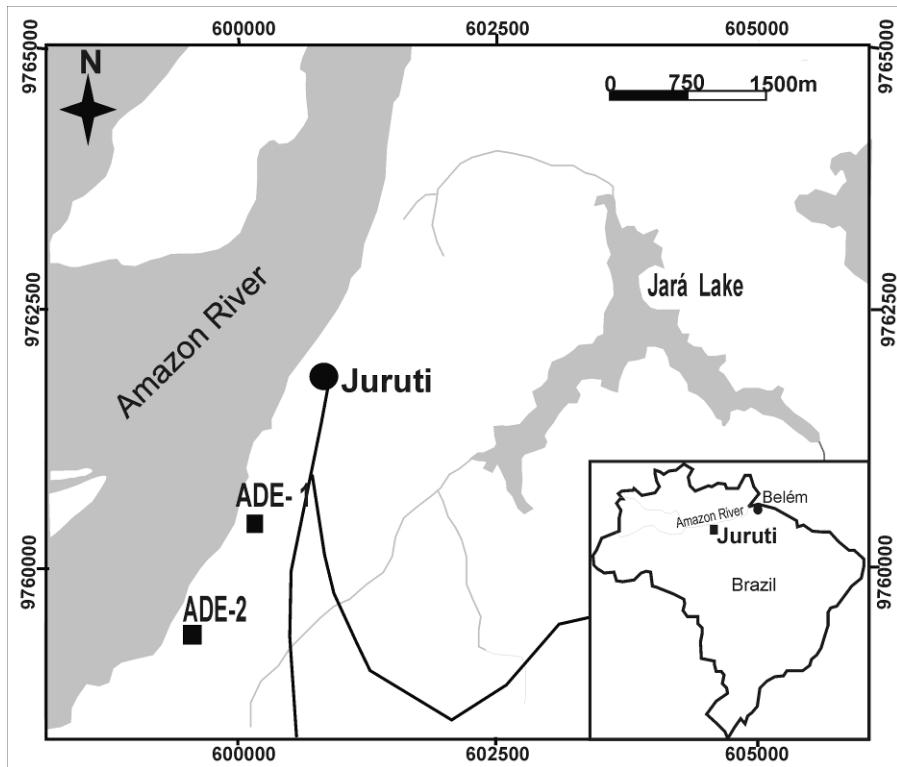


Figure 1. Map showing the locations of sites ADE-1 (central coordinates: 21M 600159E/9760433N) and ADE-2 (central coordinates: 21M 599557E/9759353N)

As for the rest of the Amazon basin, the archeological context of the Juruti site does not present a clear chronological sequence of occupation. Hilbert (1955) recorded ceramics with characteristics of the Konduri style in the dark earths located on the margins of the Juruti-Velho and Juruti-Mirim lakes. The limited archeological evidence available indicates that the principal concentrations of pre-colonial groups were located along both margins of the Amazon River between the mouths of the Nhamundá and Xingu rivers. In general, the settlements in these areas are associated with dark earth soils (Hilbert 1955; PRONAPA 1968; Hilbert & Hilbert 1980; Roosevelt 1992; Costa & Kern 1999). While inconclusive, the ethno-historic data from the European conquest of the Amazon basin, between the mid-sixteenth century and the nineteenth century, describe continuous and densely-populated settlements along the margins of the region's principal rivers. These settlements appear to have been based on socially and politically stratified societies, such as that of the Tapajó people on the Tapajós River, and their neighbors, the Canuri or Konduri, on the Trombeta-Nhamundá. According to historical reports, these peoples produced fine pottery, which was appreciated not only by neighboring peoples, but also the European

colonizers (Roosevelt 1992; Porro 1996; Hilbert 1955). Other groups living in the same area included the Bobui, Jamundá, Parucoató, Uabói, and the belligerent Mundurucu (Barbosa de Farias 1946; Hilbert 1958).

MATERIALS AND METHODS

The ceramic fragments were collected from a series of small excavations of 50 cm x 50 cm in area and varying depths, which were dug in a 60 m x 120 m grid within both ADE sites (Fig. 2). The trenches were excavated in 10 cm intervals down as far as the level at which ceramic fragments were absent. Between one and 22 fragments were observed in each interval, with sizes varying from 2 cm to 15 cm. Once collected, the fragments were washed to eliminate excess soil particles and then observed under a binocular hand lens for the identification of technical and stylistic attributes, such as manufacturing technique, tempers, finishing, and decoration, as well as the part of the artifact represented, i.e. rim, wall or base (Scientia 2008). A total of 6300 ceramic fragments were analyzed in this phase of the study.

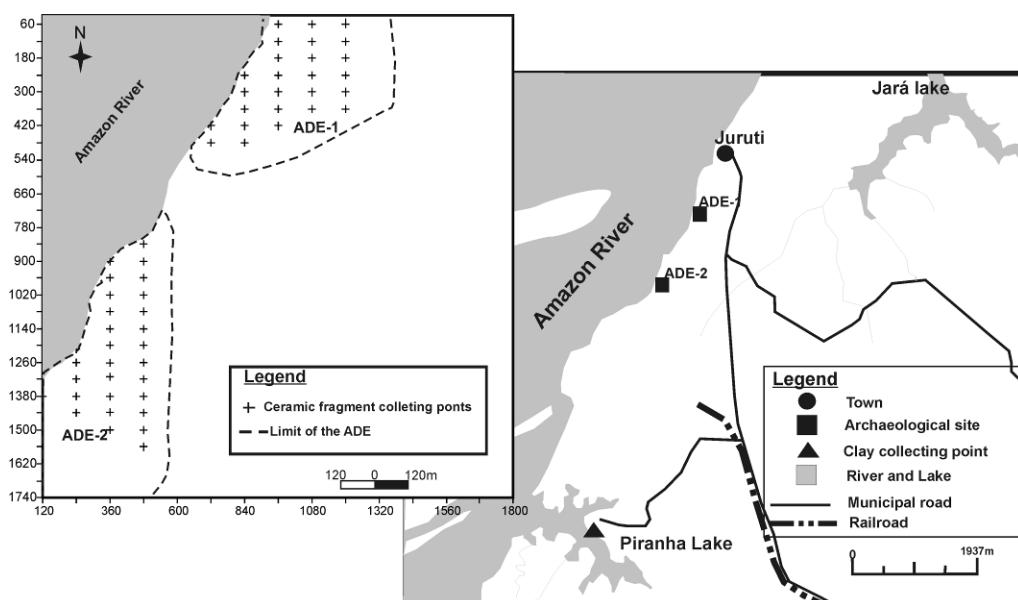


Figure 2. Maps showing the excavation grid used for the collection of ceramic fragments at sites ADE-1 and ADE-2, and Piranha Lake (central coordinates: 21M 598464E/9755450N), from which samples of clay were collected.

Twelve samples of ceramic fragments representing the Konduri and Pocó cultures were collected from each site at depths of up to 60 cm (Table 1) for the determination of their mineralogical composition and chemical characteristics. All the samples were chosen from pieces representing the main body (wall) of vessels with no decoration.

Table 1- Samples of ceramic fragments analyzed in the present study by site, depth, and probable culture.

Depth (cm)	Samples collected at site:		Probable culture
	ADE-1	ADE-2	
0-10	DE1-32	DE2-7, DE2-12, DE2-18	Konduri
10-20	-	DE2-1, DE2-5, DE2-11, DE2-14, DE2-17, DE2-21	Konduri
20-30	DE1-28, DE1-33	DE2-6, DE2-24, DE2-25	Konduri
30-40	DE1-30, DE1-31, DE1-36, DE1-37, DE1-40, DE1-42, DE1-44	-	Konduri and Pocó
40-50	DE1-39	-	Pocó
50-60	DE1-43	-	Pocó

The identification of the probable raw material used for the production of the ceramic artifacts, and their possible origin, was based on the analysis of samples of clay obtained from the margin of Piranha Lake (21M 598464E, 9755450N), located some 15 km south of the town of Juruti (Fig. 2). This site was selected based on an ethnographic survey of the region's present-day potters who have adopted techniques similar to those used by the original settlers. These artisans obtain their raw material from the margins of the lake.

The samples were pulverized in an agate mortar until their grain size had reached a <125 mesh. The mineralogical analyses were carried out in the X-ray diffraction laboratory of the UFPA Geosciences Institute, using an XPERT PRO MPD diffractometer equipped with a PW 3040/60 (theta-theta) PANalitical goniometer, copper anode ($\bullet\text{CuK}\bullet 1 = 1.54060$) run at 40 kV and 30mA. The data were obtained using an ICDD database and X'pert HighScore software (version 2.1b). The mineralogical analysis were complemented with data from scanning electron microscopy (SEM), using a microscope coupled to an energy-dispersed spectrometer (EDS),

which provided both images and chemical microanalysis. The backscatter electron detector provided data for the production of maps of the distribution of chemical concentrations. All procedures were conducted in the electron microscopy laboratory of the Goeldi Museum in Belém, using a LEO 1450 VP 500 DP microscope.

The chemical analyses of the total samples included major and minor elements (SiO_2 , Al_2O_3 , Fe_2O_3 , MgO , CaO , K_2O , Na_2O , TiO_2 , P_2O_5 , MnO), Loss on ignition (LOI), as well as trace (Be, Sc, V, Cr, Co, Ni, Cu, Zn, Ga As, Rb, Sr, Y, Zr, Nb, Mo, Ag, Cd, Sn, Sb, Cs, Ba, Hf, Ta, W, Hg, Pb, Bi, Th, U) and rare earth elements (REEs), which were obtained by fusion with lithium tetraborate, and determined by ICP-OES and ICP-MS by the Acme Analytical Laboratories Ltd.

Two ceramic fragments were used for quartz-grain thermoluminescence dating by Datação, Comércio & Prestação de Serviço Ltda, while samples of charcoal were used for radiocarbon 14 dating. This analysis was obtained by Scientia Consultoria Ltda (2008) from the Beta Analytic Incorporation Laboratory. The general data on the frequency of the 6300 ceramic fragments, and the results of the chemical analyses of the 24 samples were processed statistically using the program Statistica (version 6.0), while the distribution map was produced using Surfer (version 8.0).

RESULTS AND DISCUSSION

Frequency and spatial distribution

The ceramic fragments are found between the surface and depths of up to 180 cm, although most are concentrated into the 0-40 cm stratum (Figure 3). Fragments are abundant at the surface (depths of 0-10 cm) only at site ADE-2, whereas the greatest abundance was recorded in the immediately lower levels at both sites, at depths of between 10 cm and 40 cm. The frequency of fragments falls away rapidly at ADE-2 below the 40-50 cm stratum, and they disappear completely below 80 cm, whereas at ADE-1 they remain relatively abundant at lower levels, but within a reduced area (Fig. 3).

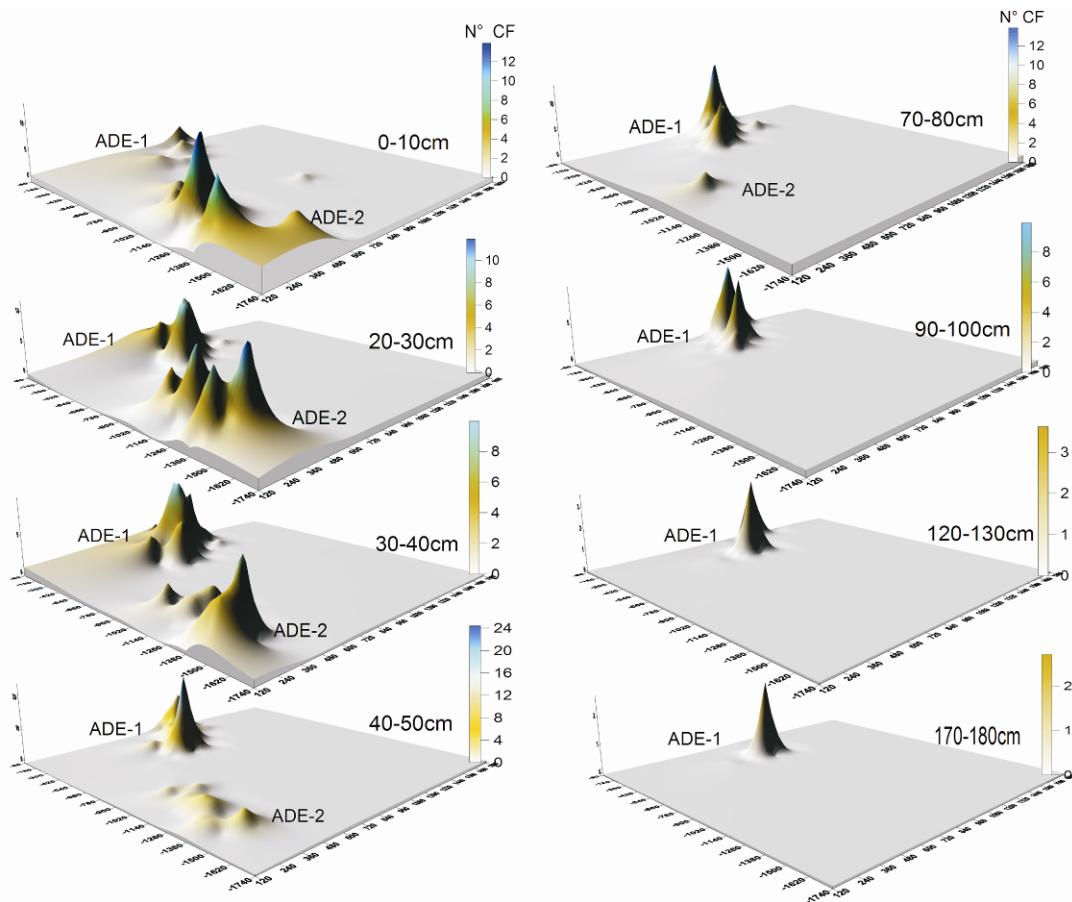


Figure 3. Map of the frequency of ceramic fragments (CF) by archeological horizon at sites ADE-1 and ADE-2 in Juruti, Pará.

Ceramic technology and corresponding culture

The morphological analysis of the 6300 ceramic fragments indicated that only 24% of the remains at ADE-1 represent utensils or undecorated vessels, whereas the proportion at site ADE-2 was 60%. In general, the fragments have a porous surface, often worn or weakened, varying in color from gray to ochre, with a fine microporous matrix containing grains of quartz and micro-particles calcined of organic matter such as cariapé (*Licania utilis* or *Licania turiuva*) and cauixi (*Tubella reticulata* or *Parnula betesil*). Cauixi is the principal temper used in these ceramics, being found in more than 90% of the fragments at both sites. This material is widely-used as one of the principal components of the production of ceramic throughout the lower Amazon basin (Hilbert 1955; Simões 1984; Roosevelt 1992; Gomes 2008).

The reconstruction of the vessels' shapes permitted the identification of distinct functional classes, although artifacts with an open or slightly closed morphology and a flat or rounded base

predominate. These vessels were clearly cooking pots, plates, and rounded bowls with volumes varying from 2.5 l to 24.0 l. The vessels with a more closed structure and medium volume (3.5 to 4.5 l) had thicker walls (1.2 to 2.0 cm) than the larger vessels (5.5 to 8.4 l), with walls of between 0.5 and 1.1 cm. Rye (1981) and Viana (1996) found that vessels with thinner walls were more appropriate for cooking, given their better heat conduction, and thus faster cooking times, as well as greater resistance to thermal shock. Closed forms are also more appropriate for cooking food.

It was also possible to confirm that the principal pottery technique was coiling method. Modeling was less common, and mainly involved attachments, adornments, and other decorative motifs. The ceramic fragments of the upper strata (0-40 cm) are characterized by grooving, stippling, brushing, and fine incisions with details in red paint. The appliqués are zoomorphic and anthropomorphic, typical of the Konduri style, related to the stippled incision tradition (Fig. 4). Clearly, then, the Konduri was the last prehistoric culture to occupy the ADE sites at Juruti.

By contrast, the ceramic fragments from the deeper strata (> 40 cm) are characterized by ample spiral incisions painted red, located on both the walls and the internal and extroverted rims of the vessels, which were sometime painted in red and orange over white (Fig. 4). Multi-colored vessels were almost certainly used in religious ceremonies or rituals. This style is typical of the Pocó phase, representing the modeled incision or Barrancoid tradition, which is found throughout the Orinoco basin, and the Trombetas-Nhamundá and Negro basins in central Amazonia (Lathrap 1970; Hilbert 1980; Roosevelt 1992; Lima *et al.* 2006; Neves 2010). In Juruti, the Barrancoid style is found exclusively at site ADE-1 (Fig. 3). The occupation of the area by pottery-making human populations probably began at site ADE-1, with the Pocó culture, an interpretation compatible with the chronological sequence determined by the radiocarbon and thermoluminescence dating (Table 2), which indicated that the oldest inhabitants arrived around 140 B.C., and remained until 1300 A.D., that is, a continuous occupation of some 1400 years. The level immediately above, consisting of 90 cm of discarded material, now representing soil with ceramic fragments, indicates either a prolonged occupation or dense population, which began at site ADE-2. The Konduri style predominates in these deposits (Table 2, Fig. 3).

The more limited dating results from site ADE-2 indicate a much shorter sequence of occupation, given that the oldest date is 690 A.D., and the most recent is 1300, indicating that the area was occupied for some 600 years. While ADE-1 was settled for longer overall, the data indicate that the two sites were occupied simultaneously for a long period up until the 14th

century, given the presence of ceramic fragments at ADE-2 in the 70-80 cm stratum. The greater concentration of ceramic fragments in the 20-40 cm strata indicate that the peak of the occupation of site ADE-2 occurred between the 3rd and 9th centuries. The gradual decrease in the abundance of ceramic material in the upper levels of site ADE-1 indicates that it was the first to be abandoned, while the increasing abundance of ceramics at ADE-2 suggests a possible increase in the population of this site during the same period. It thus seems likely that the population of ADE-1 migrated to ADE-2 (Figure 3). The dating that corresponds to the 4th century at ADE-1 and the 10th century at ADE-2 indicate stratigraphic disturbances.

Table 2. Chronological sequence of the occupation of sites ADE-1 and ADE-2 based on radiocarbon and thermoluminescence dating.

Site	Depth (cm)	Age	Year/Century
ADE-1	20-30	640 ± 50 BP (a)	XIII A.D.
ADE-1	40-60	1760 ± 40 BP (a)	II A.D.
ADE-1	70-80	1960 ± 40 BP (a)	10 B.C.
ADE-1	80-90	2040 ± 40 BP (a)	90 B.C.
ADE-1	90-100	2090 ± 50 BP (a)	140 B.C.
ADE-1	120-130	1710 ± 50 BP(a)	III B.C.
ADE-2	20-30	690 ± 40 BP (a)	XIII A.D.
ADE-2	20-30	1400±160 BP (b)	VII A.D.
ADE-2	40-50	1170±180 BP (b)	IX A.D.

(a) by C¹⁴ dating (Scientia 2008), (b) by thermoluminescence dating.

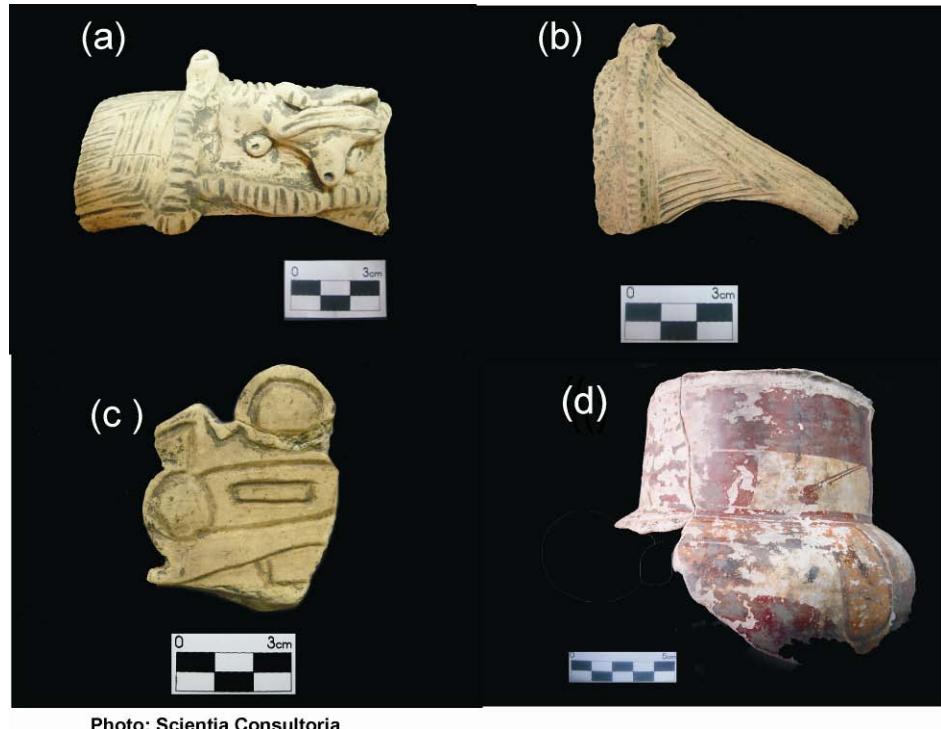


Photo: Scientia Consultoria

Figure 4. Stylistic features of the artifacts and ceramic fragments of the (a, b) Konduri and (c, d) Pocó cultures found at sites ADE-1 and ADE-2.

Mineralogical composition

The ceramic fragments from sites ADE-1 and ADE-2 are constituted (in decreasing order of abundance) of quartz, metakaolinite, illite+muscovite, microcline, albite, maghemite, hematite, and anatase. Metakaolinite is amorphous, and is normally derived from the dehydration of kaolinite – the principal mineral in ceramic clays – during the firing process. These same minerals make up the natural clay used by potters in the present day. However, in its raw form, this clay lacks metakaolinite, and contains only kaolinite (as well as smectite), which confirms the derivation of metakaolinite from the kaolinite in the clay. The maximum temperature for the thermal dehydration of kaolinite is 550°C, which also ruptures the structure of smectite. As the vitrification of ceramics occurs at temperatures above 600°C, this evidence indicates that the vessels were fired at between 550°C and 600°C (Goffer 2007; Costa *et al.* 2011).

The mineralogical composition of the ceramic fragments from Juruti is equivalent to that of fragments from a second site in the lower Amazon basin, Cachoeira Porteira (Costa *et al.* 2004a). In fact, the chemical and mineralogical composition was identical, indicating that the raw

material was derived either from the same source or from different sources with the same lithology and geological environment.

In addition to the minerals identified by XRD, which includes the amorphous forms, such as metakaolinite, the total chemical analyses identified significant levels of P₂O₅, varying from 0.94% to 4.35% (Table 3) in different samples, although the mineral phase responsible for this pattern was not found. Chemical analyses using SEM-EDS also identified phosphorus levels of 1 to 7 wt%, in particular in the matrix dominated by metakaolinite. The values for P₂O₅ suggest the presence of phosphates in the Juruti ceramics in a pattern similar to that found at other Amazonian sites (Costa *et al.* 2003; 2004a, 2006, 2010; Rodrigues 2010; Silva 2010). These phosphates appear to be of aluminum, aluminum-iron, and sometimes calcium/strontium-aluminum, amorphous to microcrystalline, represented by variscite, variscite-strengite, and crandallite-goyazite (Costa *et al.* 2004b, 2006, 2010; Rodrigues 2010). The SEM-EDS analysis of the matrix of sample DE2-24, which contains cariapé as temper, found that the microspherulites (Fig. 5) are composed of phosphorus, aluminum, and iron, which suggests the variscite-strengite series (Al, Fe)(PO₄).2(H₂O) identified by Costa *et al.* (2004a, 2010) in ceramic fragments from other ADE sites.

The SEM-EDS microchemical mapping confirms that these phosphates are distributed throughout the matrix, given the homogeneous distribution of Si, Al, Fe, P, and K (Fig. 6). However, phosphates were not identified as minerals in the clay presumed to be the raw material for the ceramics. Within this same metakaolinite matrix, the chemical analysis identified occasional concentrations of P and Ca consistent with apatite. This mineral is a component of rare, minuscule bone particles (Fig. 6). Bone fragments in the form of apatite were described by Lima *et al.* (2002) and Costa *et al.* (2004a) from ceramic fragments collected from ADE soils at other sites in the Amazon basin, and were likely added accidentally to the clay matrix during the production of the artifacts, given that they are not normally used as temper. This same chemical mapping demonstrated that the high values of Si highlight the spicules of cauixi and its fragments in the metakaolinite matrix, which are originally constituted of amorphous silica (Costa *et al.* 2004a). These chemical maps show that the areas occupied by cauixi have no Al, P, Fe, Ca or K whatsoever, and are composed exclusively of SiO₂.

The data presented here indicate that the ceramic fragments analyzed from the superficial layers (down to 30 cm) of ADE-2, in which the Konduri style predominates, or even the lower

levels (20-60 cm at ADE-1), where Konduri and Pocó may occur simultaneously, are mineralogically homogeneous. This supports the deduction that the peoples that produced the vessels from which the fragments were derived, whatever their depth or age, were almost certainly produced from the same raw materials obtained from the same sources in the vicinity of the settlements, and possibly also used the same firing technology.

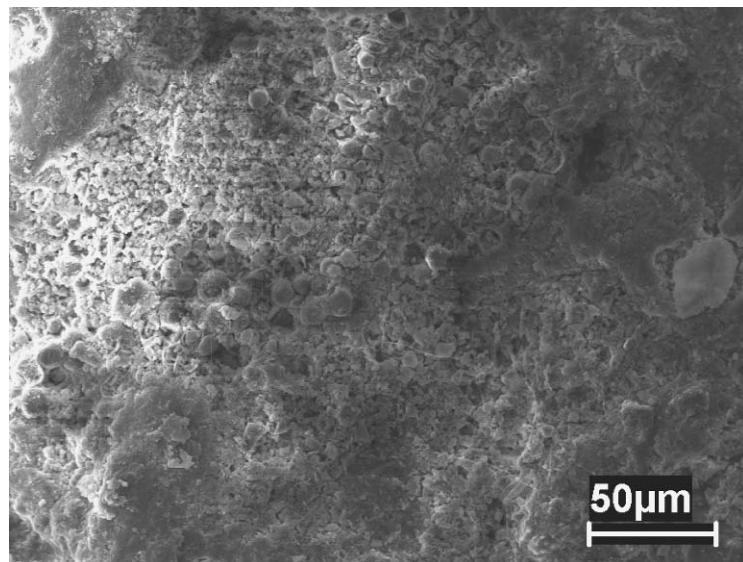


Figure 5. Scanning electron microscope (SEM) image showing the spherulitic features of the matrix of a ceramic fragment. The overall pattern suggests the substitution of the cellulose tissue of the cariapé, as observed by Costa *et al.* (2004) in ceramic fragments from Cachoeira-Porteira.

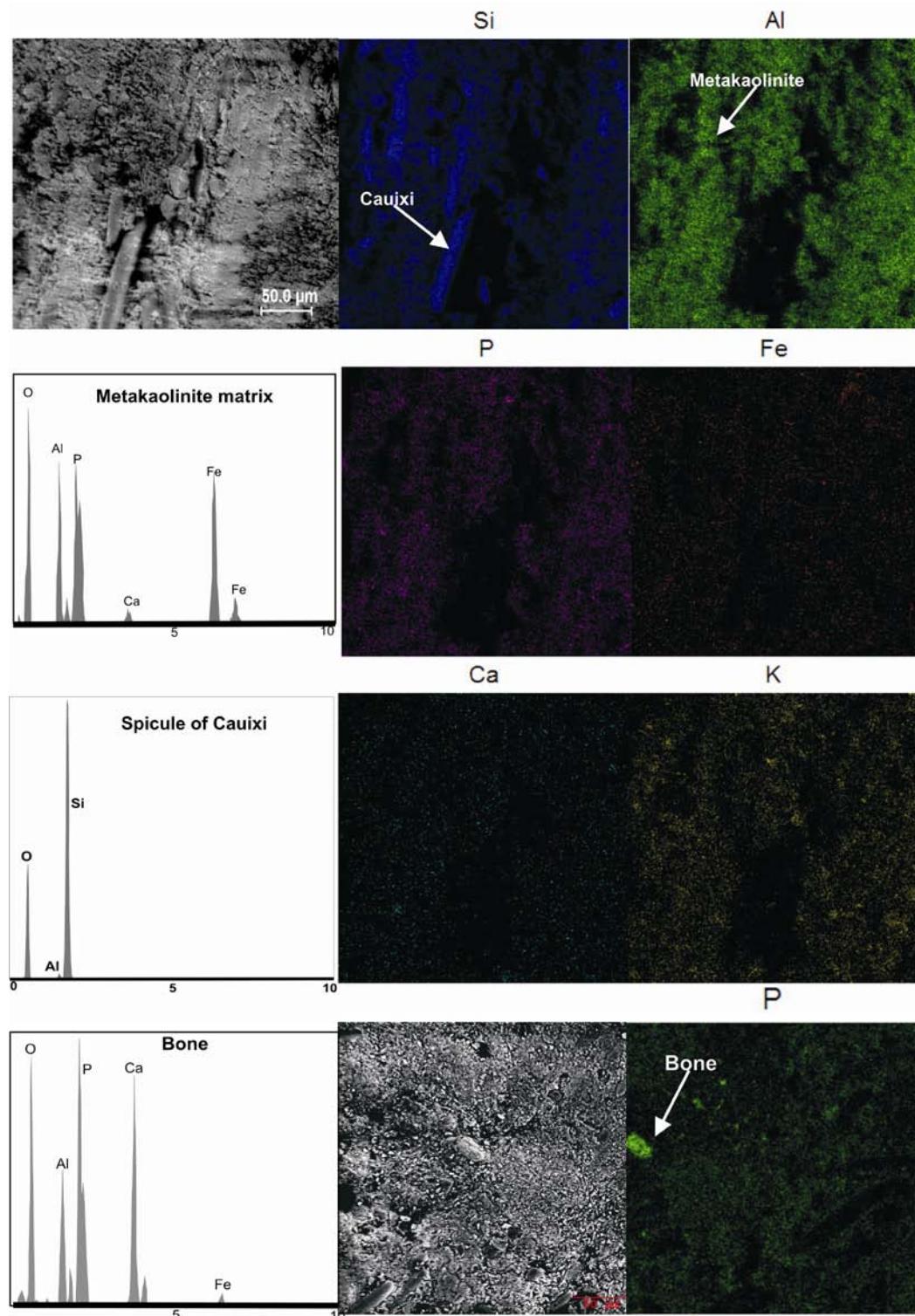


Figure 6. SEM image of the respective chemical maps for the elements Si, Al, P, Fe, Ca, and K. The EDS spectra of the metakaolinite matrix, indicating the presence of Al, P, Fe, and to a lesser extent, Ca, which correspond to phosphates of Al and Fe. Spectrum of a spicule of cauixi composed of Si, as well as microfragments of bone, indicating the dominance of Ca and P, in addition to Al and Fe, interpreted as apatite + phosphates of Al and Fe.

Total chemical composition

The chemical composition of the ceramic fragments is dominated by SiO_2 , with a mean of 65.69% of the fragments from ADE-1 and 62.43% of those from ADE-2, followed by Al_2O_3 , with means of 16.46% (ADE-1) and 16.41% (ADE-2), and Fe_2O_3 , with mean values of 4.41% and 4.01%, respectively (Table 3). The levels of SiO_2 and Al_2O_3 constitute basically the principal minerals metakaolinite and quartz, as well as the predominant temper, cauixi, or occasionally, cariapé, both of which are composed principally of SiO_2 . Remains of diatoms found in the metakaolinite matrix of the fragments (Fig. 7) also contribute to the SiO_2 levels, and were probably present in the raw material, which permits the classification of this material as a sediment or sedimentary rock. The Fe_2O_3 concentrations, which were higher at ADE-1, represent hematite (+maghemite), and occasionally goethite. The levels of these three chemical compounds (SiO_2 , Al_2O_3 , and Fe_2O_3) vary very little overall (Table 3). The levels of TiO_2 vary even less and the mean concentrations at the two sites were statistically equal, at 75%, which represents anatase. Similarly, while the mean values for K_2O – 1.82% (ADE-1) and 1.74% (ADE-2) – and, to a lesser extent, MgO – 0.67% (ADE-1) and 0.66% (ADE-2) – were equal, individual values were relatively more variable. While these values of K_2O reflect the presence of illite/muscovite + microcline, $\text{K}_2\text{O} + \text{MgO}$ are the principal components of illite/muscovite. The reduced correlation between these two chemical components (Fig. 8) confirms the presence of the two mineralogical phases, illite/muscovite and microcline.

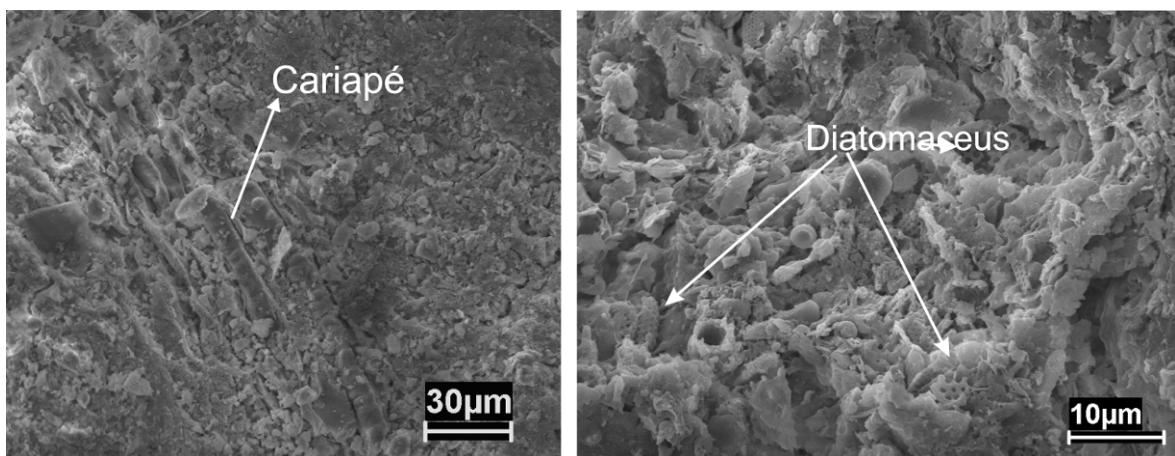


Figure 7. SEM images of ceramic fragments with cariapé, which was used as coarse temper, and diatomic skeletons, which were probably primary components of the original clayey raw material, both of which are composed of amorphous silica.

On the other hand, while the concentrations of Na₂O were well below 1.0 % (mean values of 0.28% and 0.23% at ADE-1 and ADE-2, respectively), they vary considerably among different fragments, and correspond to small concentrations of albite. A similar pattern was recorded for CaO, with mean values of 0.28% and 0.26 % at sites ADE-1 and ADE-2, respectively (Table 3). By contrast, the levels of P₂O₅ were expected, given that it appears to be a common component of the ceramic fragments found at Amazonian ADE sites (Costa *et al.* 2004b, 2006, 2010; Rodrigues 2010; Silva 2010). The mean levels of this compound – 2.92 % at ADE-1 and 2.47 % at ADE-2 – are of the same order of magnitude as those found in ceramic fragments at Cachoeira Porteira (Table 3). However, the levels of P₂O₅, like those of CaO and Na₂O, vary considerably between samples, ranging from 0.94 % to 4.35 %.

As in the fragments studied at other Amazonian sites, these high levels of P₂O₅ are not expressed in the form of a phosphatic mineral, which can be identified by XRD or optical microscopy. As discussed previously, these are in fact amorphous aluminum phosphates, as demonstrated by Costa *et al.* (2010) in ceramic fragments from ADE soils in Cachoeira Porteira, Caxiuanã, and to some extent in Jabuti. These are not Ca-Al phosphates, given that the levels of CaO, as shown above, are very low, in addition to which, they do not correlate with P₂O₅ (Fig. 8). Even so, it seems reasonable to assume that they represent a less abundant phase, such as crandallite, which is supported by the strong correlations between CaO and Ba ($r = 0.68$) and CaO and Sr ($r = 0.84$), a pattern also found by Costa *et al.* (2010) and Rodrigues (2010) at Jabuti. These extremely low values for CaO indicate clearly that apatite is not present as the principal phosphate. It is important to note that the levels of P₂O₅ in the clay identified as the raw material for the ceramics were crustal, i.e. < 0.10 %. As the phosphorus was not found at equivalent levels in this clay, it was almost certainly incorporated subsequently. These results confirm the originating process defined by Costa *et al.* (2003, 2004b, 2006, 2009, 2010, 2011), in which the high levels of phosphorus found in the ceramics were incorporated after the production of the vessels, and may have occurred during the cooking of foods rich in phosphorus. During this phase, the formation of aluminum phosphates is favored, given the establishment of a hydrothermal reaction between the vessel wall, dominated by metakaolinite, and the liquid, an environment which is propitious for the formation of phosphates, whether in nature or the laboratory.

One unexpected finding is the fact that, with the exception of P₂O₅, the mean levels of the other main components of the clayey raw material – SiO₂, Al₂O₃, Fe₂O₃, K₂O, MgO, CaO, Na₂O (slightly higher) and TiO₂ – were similar to those found in the ceramic fragments from both sites. The H₂O lost from the fragments during calcination was replaced through their exposure to the weathering process, partly through the accumulation of P₂O₅, such that it was not necessary to apply mathematical corrections to recalculate the concentrations for comparison (Table 3). Overall, then, the chemical elements and compounds, such as SiO₂, Al₂O₃, TiO₂, K₂O, MgO, and even Fe₂O₃ (Fig. 9), varied little between sites or archeological strata (and thus, cultures), and are chemically, and also mineralogically homogeneous.

Table 3. Chemical composition (%) of the ceramic fragments from sites ADE-1 and ADE-2, compared with that of local raw clay and archeological ceramics from Cachoeira Porteira.

Samples	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MgO	CaO	N ₂ O	K ₂ O	TiO ₂	P ₂ O ₅	MnO	LOI
%											
ADE-1											
DE1-28	65.73	14.23	4.50	0.75	0.13	0.22	1.95	0.67	2.29	0.02	9.40
DE1-30	63.86	15.07	3.60	0.51	0.35	0.35	2.04	0.72	4.24	0.03	9.10
DE1-31	64.82	14.74	3.99	0.56	0.20	0.41	1.99	0.78	3.04	0.04	9.30
DE1-32	54.17	20.81	5.40	0.63	0.33	0.09	1.49	0.90	2.22	0.02	13.80
DE1-33	63.85	15.46	4.22	0.83	0.17	0.22	1.67	0.74	1.37	0.06	11.30
DE1-36	61.18	16.88	3.96	0.48	0.26	0.26	1.54	0.83	4.10	0.05	10.30
DE1-37	63.02	16.41	4.25	0.78	0.35	0.42	2.12	0.72	3.10	0.04	8.60
DE1-39	58.11	19.32	5.46	0.84	0.46	0.39	2.02	0.83	4.02	0.03	8.30
DE1-40	62.87	15.00	5.01	0.61	0.21	0.17	1.78	0.62	2.83	0.03	10.70
DE1-42	61.47	15.74	4.71	0.78	0.41	0.36	2.06	0.78	4.16	0.05	9.30
DE1-43	54.23	21.18	3.44	0.20	0.03	0.04	1.55	0.77	2.27	0.01	16.10
DE1-44	66.92	12.72	4.41	0.90	0.22	0.37	1.67	0.84	1.38	0.03	10.40
Mean	61.69	16.46	4.41	0.66	0.26	0.28	1.82	0.77	2.92	0.03	10.55
ADE-2											
DE2-1	59.59	18.33	2.05	0.87	0.22	0.05	1.10	0.83	2.56	0.02	14.20
DE2-5	60.14	15.76	5.78	1.21	0.24	0.28	2.08	0.71	0.94	0.10	12.70
DE2-6	64.00	15.01	3.37	0.43	0.20	0.32	1.67	0.80	2.62	0.03	11.50
DE2-7	61.64	16.75	4.30	0.67	0.45	0.32	2.21	0.79	4.27	0.04	8.40
DE2-11	63.90	16.35	4.18	0.63	0.13	0.24	1.87	0.76	1.85	0.03	10.00
DE2-12	62.17	16.26	4.40	0.96	0.24	0.45	2.03	0.83	0.76	0.02	11.70
DE2-14	68.79	16.18	4.68	0.88	0.72	0.44	1.94	0.75	0.96	0.09	4.40
DE2-17	59.98	18.42	2.68	0.27	0.14	0.04	1.23	0.65	3.91	0.03	12.50
DE2-18	58.91	16.41	5.12	0.35	0.15	0.28	1.62	0.73	1.40	0.04	14.90
DE2-21	61.32	18.24	2.51	0.33	0.29	0.09	1.95	0.70	4.35	0.02	10.00
DE2-24	65.11	13.74	4.74	0.60	0.29	0.02	1.00	0.71	2.71	<0.01	11.00
DE2-25	63.57	15.50	4.30	0.79	0.28	0.27	2.21	0.68	3.34	0.04	8.90
Mean	62.43	16.41	4.01	0.67	0.28	0.23	1.74	0.75	2.47	0.04	10.85
Clay	51.71	24.60	3.60	1.00	0.07	0.17	2.21	0.79	0.07	0.01	15.60
C. Porteira ⁴	65.55	16.37	5.79	0.63	0.43	0.69	0.90	0.86	2.37	0.01	na

na = not analyzed

⁴=Costa et al., 2004a

The chemical similarities of the ceramic fragments from the two sites are reinforced by the multivariate statistical analysis (Fig. 10), in which only three samples are outliers, but group with the local clay. Relatively high levels of Sr, Ba, and Zn are characteristic of these samples. This further reinforces the conclusion that the ceramic vessels of the upper levels, which represent the Konduri culture, were produced from the same raw material, probably obtained from the same source site or local geological unit as the ceramics of the Pocó culture, the fragments of which were found in the lower levels. The chemical and mineralogical composition of these clays is consistent with that of the raw material used for the production of the artifacts (Table 3). This conclusion based on the major chemical elements is further reinforced by the concentrations of trace elements (Table 4), which link the fragments and the clay unequivocally, and are related intimately with the Fe and Al oxy-hydroxides compounds. Of the elements that have some affinity with Fe_2O_3 , only Co presents a strong correlation ($r = 0.75$), whereas Cr, Ga, Sc, Th, U, and the REEs correlate more closely with Al_2O_3 ($r > 0.6$), which is probably due to the greater reactivity of metakaolinite to reduced concentrations of Fe, while V and Y are correlated more weakly with Al and Fe ($r < 0.5$).

Of the other elements analyzed, Be, Cd, Mo, Hg, Ag, and Sb all presented concentrations close to or below the limit of detection, and only Zn, Ga, Cr, and V are above crustal levels (Table 4). The trace elements in the fragments also vary little, with the most marked fluctuations being observed in Sr, Ba, Zn, Rb, and the REEs (Table 4, Fig. 9). These subtle chemical variations reflect the mineralogical similarities between the raw materials used for the production of the vessels and the chemical modifications resulting from the daily use of these artifacts for the preparation of foods by the populations that produced them.

When normalized to chondrite (meteorite) levels, the distribution of the REEs in the fragment samples was highly similar to that of the clay presumed to be the raw material. The curves show the enrichment of light REEs, with a strong positive anomaly for cerium and subtle negative anomalies for Eu and Ho (Fig. 11). The geochemical signature of these rare earth elements may contribute to the identification of the raw material used for the production of the ceramic vessels.

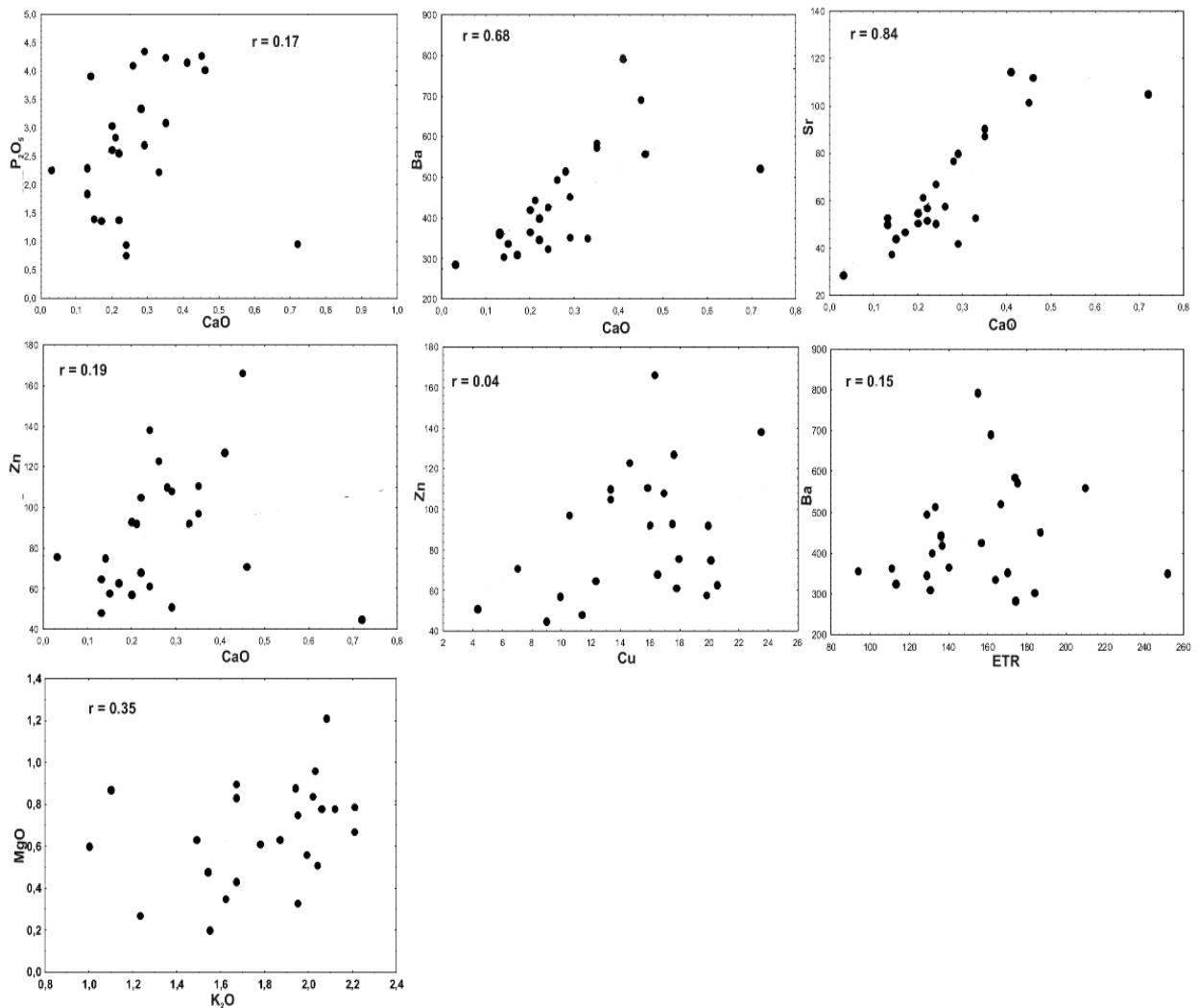


Figure 8. Correlation between the levels of CaO and P₂O₅, CaO and Ba, CaO and Sr, CaO and Zn, Cu and Zn , ETR and Ba, MgO and K₂O in the samples of ceramic fragments (n= 24) obtained from sites ADE-1 and ADE-2.

Table 4. Concentration of trace elements (mg kg^{-1}) in the ceramic fragments of the sites ADE-1 and ADE-2, compared with those of the raw clay and upper terrestrial crust.

Samples	Be	Sc	V	Cr	Co	Ni	Cu	Zn	Ga	As	Rb	Sr	Y	Zr	Nb	Mo	Ag	Cd	Sn	Sb	Cs	Ba	Hf	Ta	W	Hg	Pb	Bi	Tl	U	ETR
mg kg^{-1}																															
ADE-1																															
DE1-28	<1.00	15.00	93.00	61.58	6.10	15.80	12.30	65.00	19.10	3.90	100.40	52.90	19.80	122.00	12.70	0.20	<0.10	0.30	3.00	<0.10	11.10	365.00	3.50	1.00	1.50	0.10	19.70	0.40	14.60	2.70	139.90
DE1-30	2.00	14.00	82.00	61.58	6.20	16.60	10.50	97.00	18.50	1.90	119.60	87.30	24.60	147.20	14.50	0.30	<0.10	0.20	3.00	<0.10	7.50	585.00	4.20	1.10	1.60	0.02	17.00	0.20	13.70	3.00	173.53
DE1-31	2.00	13.00	88.00	54.73	4.00	15.80	17.50	93.00	18.50	3.10	104.60	54.80	22.10	179.00	15.00	0.60	<0.10	0.40	2.00	<0.10	8.80	420.00	4.90	1.10	1.60	0.02	16.70	0.30	12.30	3.00	136.67
DE1-32	2.00	18.00	136.00	75.26	10.40	13.80	16.00	92.00	24.20	4.10	161.60	52.70	36.40	187.10	17.10	0.20	<0.10	<0.10	3.00	<0.10	9.60	351.00	5.60	1.20	1.90	0.01	23.10	0.30	16.00	3.90	251.70
DE1-33	<1.00	15.00	103.00	61.58	6.20	15.10	20.50	63.00	20.70	9.10	112.80	46.80	18.80	151.70	13.40	0.20	<0.10	<0.10	3.00	<0.10	9.70	310.00	4.20	1.10	1.70	0.02	21.00	0.40	14.30	3.00	130.49
DE1-36	<1.00	15.00	94.00	61.58	7.10	15.10	14.60	123.00	21.80	3.30	92.70	57.60	21.60	152.90	16.20	0.10	<0.10	<0.10	3.00	<0.10	8.30	495.00	4.70	1.20	1.50	0.01	20.00	0.30	12.80	3.30	128.56
DE1-37	2.00	15.00	91.00	68.42	7.60	17.40	15.80	111.00	20.40	2.50	139.90	90.50	23.60	150.40	13.80	0.20	<0.10	0.20	3.00	<0.10	13.60	573.00	4.50	1.10	1.80	0.01	17.90	0.30	13.30	2.80	173.17
DE1-39	2.00	17.00	99.00	68.42	9.70	13.80	7.00	71.00	23.50	2.10	89.20	112.10	28.50	169.20	15.70	0.30	<0.10	<0.10	3.00	0.20	5.60	559.00	4.60	1.20	1.70	0.01	15.80	0.20	15.40	3.50	209.45
DE1-40	3.00	14.00	89.00	61.58	7.40	17.70	19.90	92.00	18.90	6.70	118.60	61.40	18.50	110.00	11.70	0.30	<0.10	0.20	2.00	<0.10	10.50	444.00	3.00	0.90	1.50	0.02	21.80	0.30	12.30	2.70	135.91
DE1-42	2.00	15.00	87.00	61.58	8.10	19.60	17.60	127.00	20.20	4.20	117.10	114.50	21.90	141.00	14.30	0.70	<0.10	0.20	3.00	<0.10	9.10	792.00	4.10	1.10	1.80	0.09	18.30	0.20	12.40	2.80	154.94
DE1-43	2.00	22.00	122.00	82.10	3.40	15.10	17.90	76.00	27.90	3.80	81.30	28.90	19.40	148.20	14.80	0.60	<0.10	<0.10	4.00	<0.10	8.10	285.00	4.20	1.20	1.90	0.05	30.10	0.40	28.40	5.30	174.08
DE1-44	<1.00	14.00	93.00	54.73	6.50	15.30	16.50	68.00	17.60	4.50	95.00	57.00	22.40	218.40	15.80	0.20	<0.10	0.20	3.00	<0.10	9.10	347.00	5.50	1.10	1.80	0.03	13.20	0.20	11.80	3.40	128.66
Mean	2.13	15.58	98.08	64.43	6.89	15.93	15.50	89.80	20.94	4.10	111.07	68.00	23.13	156.43	14.58	0.33	<0.10	0.24	3.00	0.20	9.25	460.50	4.42	1.11	1.69	0.03	19.55	0.29	14.78	3.28	161.58
ADE-2																															
DE2-1	<1.00	12.00	79.00	88.94	3.50	8.80	13.30	105.00	20.80	2.60	108.30	51.90	14.50	150.80	12.70	0.20	<0.10	0.20	3.00	<0.10	8.30	400.00	4.40	1.00	1.00	0.02	25.40	0.30	15.90	3.30	131.70
DE2-5	<1.00	15.00	120.00	61.58	11.50	28.30	23.50	138.00	20.50	11.60	125.40	50.20	17.00	113.70	13.40	0.20	<0.10	0.20	3.00	<0.10	8.80	324.00	3.60	1.10	1.20	0.06	19.20	0.40	15.20	2.60	112.92
DE2-6	<1.00	13.00	100.00	61.58	4.40	12.40	9.90	57.00	21.40	5.00	89.90	50.70	20.50	163.50	15.60	0.10	<0.10	<0.10	3.00	<0.10	6.60	364.00	4.70	1.10	1.60	0.07	12.80	0.30	13.30	2.80	110.92
DE2-7	2.00	15.00	85.00	68.42	7.80	24.80	16.30	166.00	22.40	2.40	116.20	101.60	25.00	164.30	15.70	0.40	<0.10	0.20	3.00	<0.10	6.90	691.00	4.70	1.10	1.70	0.01	16.50	0.20	14.20	3.20	161.19
DE2-11	<1.00	15.00	116.00	68.42	5.60	11.60	11.40	48.00	21.90	3.70	122.50	49.90	19.20	140.20	15.10	0.20	<0.10	<0.10	3.00	<0.10	9.30	358.00	4.30	1.20	1.70	0.01	18.00	0.30	14.90	2.80	93.65
DE2-12	2.00	15.00	119.00	68.42	7.70	19.40	17.80	61.00	21.60	2.90	125.60	67.00	23.70	173.80	16.50	0.10	<0.10	<0.10	3.00	<0.10	10.60	426.00	4.50	1.30	1.70	0.05	18.00	0.40	15.10	3.20	156.52
DE2-14	2.00	15.00	101.00	61.58	9.90	7.20	9.00	45.00	19.10	2.70	100.50	105.20	24.90	126.00	14.40	0.10	<0.10	<0.10	3.00	<0.10	7.20	521.00	4.00	1.00	1.50	0.01	8.10	0.10	12.60	3.10	166.43
DE2-17	2.00	16.00	80.00	61.58	3.10	12.90	20.10	75.00	23.70	2.70	67.20	37.40	25.20	160.40	13.20	0.40	<0.10	<0.10	3.00	0.20	5.10	303.00	4.60	1.00	1.60	0.02	29.70	0.50	19.70	4.70	183.70
DE2-18	2.00	15.00	119.00	68.42	6.20	13.50	19.80	58.00	21.90	5.40	79.00	44.10	23.40	144.40	13.80	0.40	<0.10	<0.10	4.00	<0.10	5.90	336.00	4.10	1.00	1.70	0.03	19.30	0.30	14.70	3.30	163.87
DE2-21	2.00	15.00	89.00	68.42	2.70	11.90	16.90	108.00	22.00	3.60	125.40	79.90	22.50	200.60	14.00	0.20	<0.10	<0.10	3.00	<0.10	113.0	453.00	5.60	1.10	1.50	0.01	27.50	0.40	18.50	3.70	186.69
DE2-24	2.00	13.00	83.00	68.42	1.90	6.70	4.30	51.00	18.20	4.00	54.20	41.90	26.70	211.80	13.60	0.60	<0.10	<0.10	3.00	<0.10	3.80	353.00	6.30	0.90	1.30	0.02	31.10	0.30	13.80	3.80	170.13
DE2-25	2.00	14.00	90.00	54.73	6.10	17.10	13.30	110.00	19.90	4.30	112.80	76.80	19.90	115.60	13.00	0.30	<0.10	<0.10	3.00	<0.10	8.10	515.00	3.60	1.00	1.50	0.06	17.40	0.30	12.80	2.80	133.16
Mean	2.00	14.42	98.42	66.71	5.87	14.55	14.63	85.17	21.12	4.24	102.25	63.05	21.88	155.43	14.25	0.27	<0.10	0.20	3.08	0.20	7.66	420.33	4.53	1.07	1.50	0.30	20.25	0.32	15.06	3.28	147.50
Clay	2.00	19.00	173.00	68.42	6.00	20.00	20.40	27.00	29.10	1.50	114.90	84.40	31.30	137.70	15.70	4.00	0.40	<0.10	4.00	<0.10	12.80	413.00	4.20	1.30	2.60	0.08	32.90	0.50	18.70	4.00	288.70
Earth's Crust ¹	3.00	7.00	53.00	35.00	11.60	19.00	14.30	52.00	14.00	2.00	110.00	316.00	20.70	237.00	26.00	1.40	0.55	0.10	3.00	0.03	5.80	668.00	3.42	1.50	1.40	0.05	17.00	0.12	10.30	2.50	146.82

¹Wedepohl, 1995

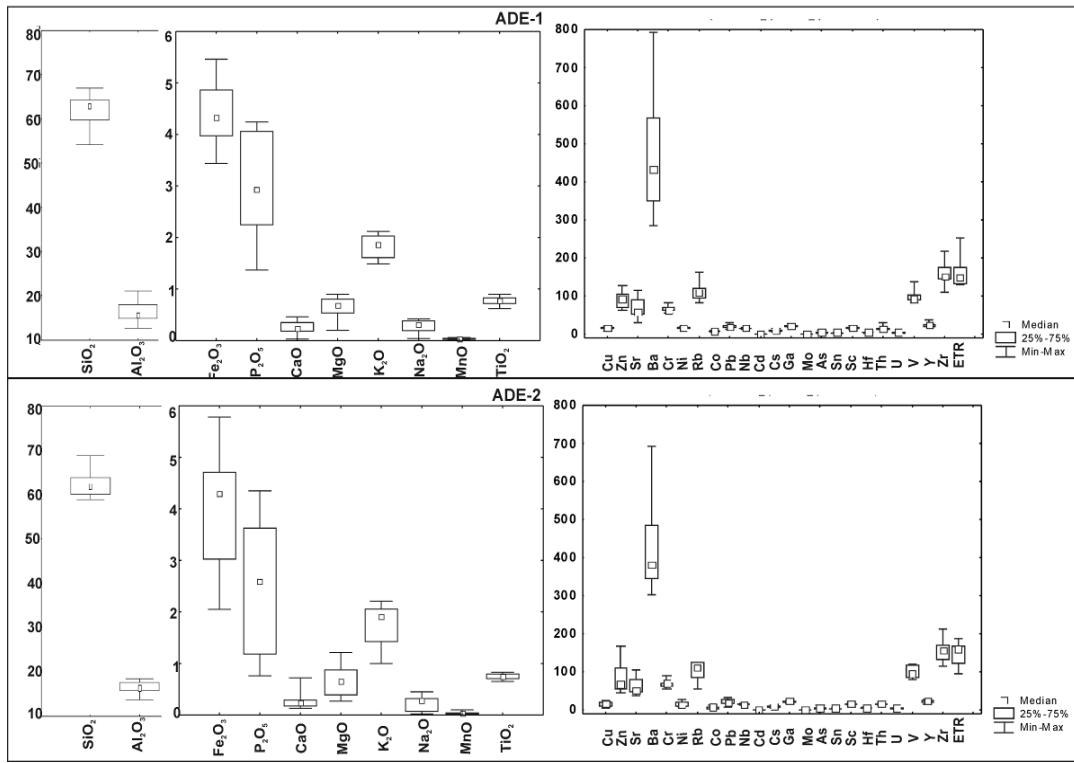


Figure 9. Diagram showing the dispersion of mean, minimum, and maximum chemical concentrations of in the ceramic fragments from sites ADE-1 and ADE-2.

The cluster analysis (Fig. 10) indicated that the ceramic fragments from both sites were relatively homogeneous, further reinforcing the conclusion that they were produced from the same raw material using the same technology. The only discrepancy in the composition of the fragments and the clay is the relatively high concentrations of P₂O₅ found in the former.

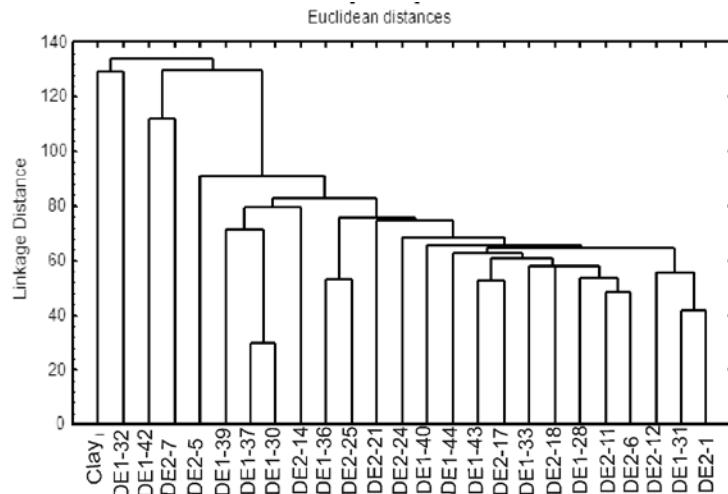


Figure 10. Dendrogram of the similarity of the chemical composition of the ceramic fragments from sites ADE-1 and ADE-2, and local clay.

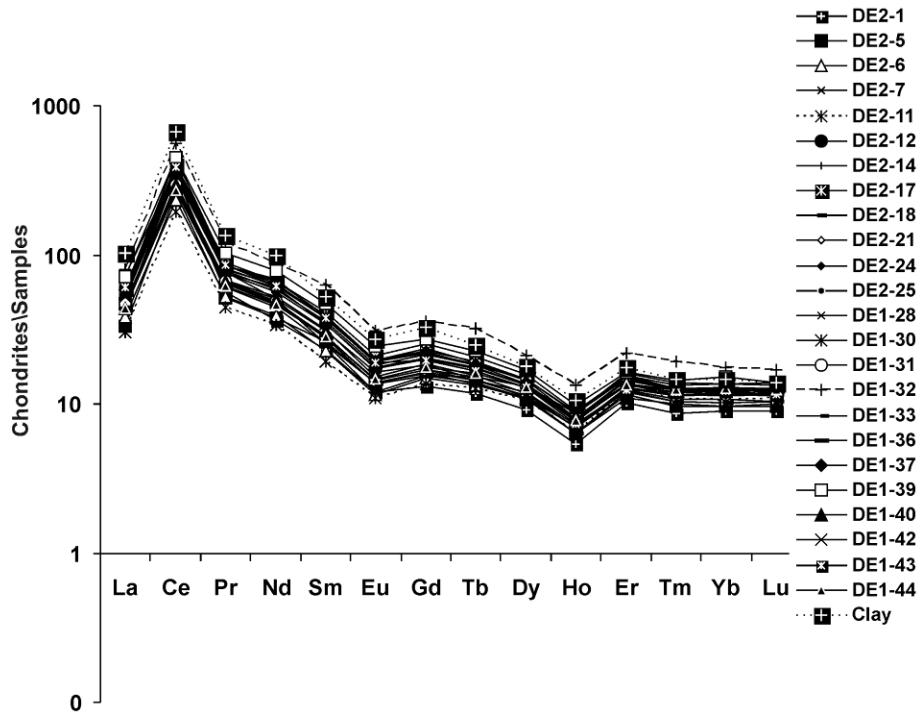


Figure 11. Normalization to chondrites of the concentrations of rare earth elements in the ceramic fragments and local clay (Evensen et al., 1978).

The spatial distribution of P_2O_5 , CaO, MgO, and Sr concentrations in the ceramic fragments varied considerably both within and between sites. The highest levels are relatively more frequent at ADE-1. In addition to the conclusion that most of the ceramic fragments at ADE-1 were derived from vessels used for cooking, the evidence indicates that the fragments from ADE-2 suffered more accentuated weathering, related to the fact that they were collected from more superficial levels, and were thus more exposed to micro-organisms (Fig. 12). Overall, then, the analysis of the chemical components indicates that the ceramic fragments were derived from vessels produced from the same raw material, which were all used for the preparation of food.

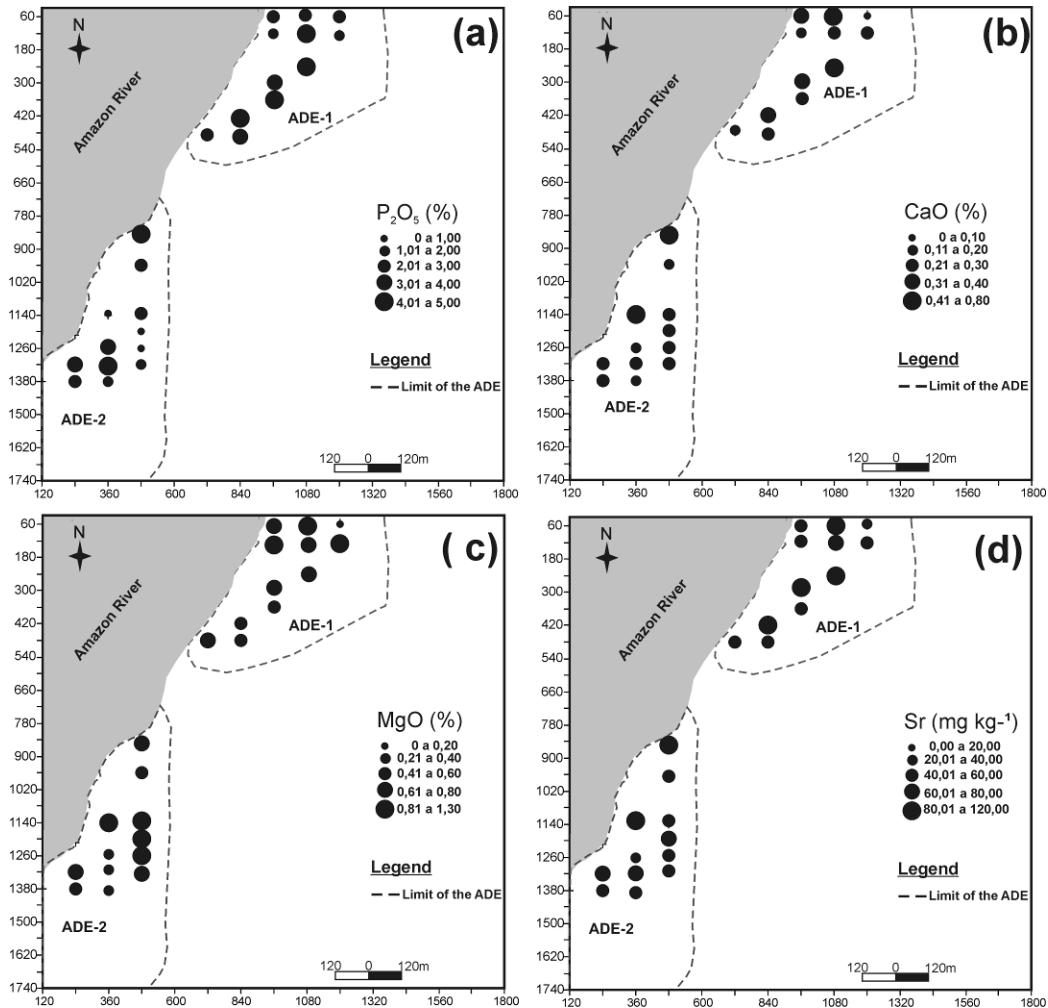


Figure 12. Isoline maps for the concentrations of (a) P_2O_5 , (b) CaO , (c) MgO , and (d) Sr in the ceramic fragments found at sites ADE-1 and ADE-2.

CONCLUSION

The occupation of the ADE areas at Juruti by pottery-making human populations probably began at site ADE-1 with the Pocó culture, which is compatible with the dating of the arrival of the first inhabitants to approximately 140 B.C., with continuous occupation until the 14th century. The more superficial levels are occupied by the Konduri style, which predominates at site ADE-2. The ceramic material from these sites is made up of fragments of vessels made for daily and ceremonial use, which were tempered predominantly with cauixi, and produced using techniques common in the Amazon Basin and neighboring areas, that can be recognized based on their decorative motifs, composed of incisions and stippling, which is characteristic of the Konduri style. The incisions and modeling, as well as the painting in bands, are typical of the Pocó or Barrancoid styles.

The principal minerals in the ceramic fragments are quartz and metakaolinite, supported by OHFe, illite+muscovite, microcline, albite, and anatase. Kaolinite and smectite were found only in local clays. The Al and Fe phosphates, variscite-strengite, which are amorphous to the XRD, are restricted to the ceramic fragments, and originated from a phase subsequent to the production of the vessels, given that they are absent from the clay, while calcium phosphate, found in rare bone fragments, was probably introduced accidentally during the production of the artifacts. The Al phosphates are abundant and amply distributed throughout the metakaolinite matrix. The high concentrations of phosphates (1.37 to 4.30% P₂O₅) are related to the use of the vessels for cooking foods rich in phosphorus, as shown by Costa *et al.* (2004ab). The slightly lower values (0.7 to 0.9% of P₂O₅) recorded for some of the fragments indicate that their primary function was not cooking, but rather the storage or processing of solids and liquids.

The chemical and mineralogical similarities of the fragments from different archeological strata and cultures, including those more than two thousand years old, indicate that the raw materials were extracted from the same sources or geological unit as those used by local potters in the present day.

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5 CONCLUSÕES

Parcelas dos terrenos de terra firme situados à margem direita do rio Amazonas em seu trecho conhecido como Baixo Amazonas, em Juruti, são ocupadas por extensas e contínuas áreas de solos tipo TPA e TMA. Os estudos arqueológicos sugerem que entre o primeiro milênio antes e o primeiro depois de Cristo houve densas ocupações de horticultores ceramistas que alcançaram níveis complexos de organização social. A grande quantidade de matéria orgânica, representada por húmus, carvão, bem como fragmentos de ossos, sugere que estas áreas de TPA e, em parte, TMA, representem acumulações deixadas por grupos humanos durante sua permanência nestes locais, conclusão que é reforçada pela grande quantidade de material cerâmico contido nos solos de TPA. Os resultados arqueológicos, mineralógicos e químicos relativos aos solos e seus fragmentos cerâmicos permitiram identificar processos pedológicos decorrentes do clima tropical quente e úmido, com contribuição de antigas ocupações humanas.

Os solos das TPA, TMA e AD são Latossolos Amarelos que recobrem a crosta ferro-aluminosa dos perfis lateríticos dominantes na área. Os minerais predominantes são quartzo e caulinita, responsáveis pelos teores elevados de SiO_2 e Al_2O_3 , seguidos de goethita + hematita e anatásio, como acessórios. Os minerais apatita e cristobalita estão restritos aos solos TPA e TMA. A apatita constitui os fragmentos de ossos, sendo assim uma das principais fontes de Ca e P das TPA e TMA. A cristobalita identificada nos horizontes de TPA e TMA está como constituinte de carvão e cinzas resultantes da queima sucessiva de matéria orgânica, prática comum entre comunidades tradicionais no preparo do solo para o cultivo. Nas TPA certamente ainda há a contribuição dos relictos submilimétricos de FC, que contêm cariapé e cauxi que, ao serem calcinados, se transformam parcialmente em cristobalita.

A associação $\text{SiO}_2\text{-Al}_2\text{O}_3\text{-Fe}_2\text{O}_3\text{-TiO}_2\text{-Cr-Y-V-Zr}$ representa a assinatura geoquímica dos Latossolos, portanto encontrada nas TPA, TMA e AD. Enquanto que associação geoquímica $\text{P}_2\text{O}_5\text{-CaO-MgO-Mn-Zn-Cu-Ba-Sr}$ é comum nas TPA e TMA, demonstrando que esses solos sofreram influência antrópica. Porém, nas TMA a associação geoquímica é menos clara, sugerindo um uso diferente da área.

A distribuição areal tanto dos elementos $\text{P}_2\text{O}_5\text{-CaO-MgO-Mn-Zn-Cu-Ba-Sr}$ quanto da freqüência de fragmentos cerâmicos permitiram identificar um conjunto de sete manchas, que variam de 2 a 6 ha, distribuídas em dois núcleos paralelos ao rio Amazonas. Supõe-se que nelas as atividades humanas cotidianas permitiram acumular resíduos orgânicos vegetais e animais,

além de fragmentos cerâmicos. Nestes locais as pessoas moravam; preparavam, consumiam e guardavam alimentos; confeccionavam ferramentas e artesanato, principalmente utensílios cerâmicos; realizavam rituais e cerimônias religiosas. Enfim, ao que tudo indica, toda a gama de atividades cotidianas de aldeias.

Por sua vez, o domínio das TMA, entre as TPA e a AD, delineado por valores medianos da mesma associação, permite delimitar possíveis locais de acampamento ou de atividades agrícolas. De modo que é possível inferir que a ocupação humana que levou à formação das TPA e TMA foi intensa e se estendeu por mais de 350 ha por pelo menos 1.400 anos. A julgar pela abundância de fragmentos cerâmicos, uma grande quantidade de artefatos cerâmicos foi confeccionada, a maioria destinada ao uso diário. Muitos desses artefatos que obedeceram a regras rígidas de elaboração, reconhecidas nos motivos decorativos relativos aos estilos Pocó e Konduri. De ocorrência restrita às TPA, os aspectos técnicos e estilísticos dos fragmentos cerâmicos sugerem que os povos que se estabeleceram na região compartilhavam costumes ou hábitos semelhantes ou pertenciam aos grupos Pocó e Konduri.

A cronologia de ocupação horticultora ceramista em Juruti provavelmente se iniciou ao norte, pelo sítio TPA-1 com a cultura Pocó, por volta de 140 AC, posteriormente sobreposta pela cultura Konduri, que ocupou simultaneamente os dois sítios com TPA e ali permaneceu até a chegada do colonizador europeu.

Os fragmentos cerâmicos pertencentes tanto a vasilhas de uso diário como às de uso ceremonial eram temperados principalmente com cauixi e confeccionados por acordelamento, técnica ainda hoje utilizada na região. São fragmentos constituídos por quartzo, metacaulinita e, em menor proporção, oxi-hidróxidos de Fe (goethita e hematita), illita + muscovita, microclínio, albita e anatásio, além de fosfatos de Al e Fe, variscita-estrengita, abundantes e amplamente distribuídos na matriz de metacaulinita. Provavelmente os fosfatos se originaram do uso das vasilhas para preparação de alimentos, portanto se constituem em mais uma informação sobre a contribuição antrópica para o desenvolvimento dos solos de TPA.

A similaridade mineralógica e química entre os fragmentos cerâmicos e a argila local, utilizada ainda hoje para produção de utensílios cerâmicos do cotidiano, permite inferir que as fontes de matéria-prima utilizadas pelas populações que ocuparam a região há mais de 2.000 anos têm a mesma proveniência ou ambiência geológica das fontes empregadas pela população atual. Portanto, as TPA e TMA que margeiam o vale do Amazonas por centenas de hectares indicam

que elas estão relacionadas diretamente às intensas atividades de grupos pré-históricos ceramistas horticultores nos Latossolos Amarelos. Os resíduos vegetais e animais, sólidos e líquidos, bem como aqueles do descarte das vasilhas cerâmicas, os FC, em contato e/ou incorporados aos Latossolos, submetidos a quase 2.000 anos de transformações pedogenéticas (geológicas e biogeoquímicas) pós-ocupacionais, sob intensa ação do clima tropical quente e úmido amazônico, deram origem às expressivas coberturas de solos tipo TPA e TMA, que preservaram parcialmente, direta ou indiretamente os registros antrópicos do seu passado.

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UNIVERSIDADE FEDERAL DO PARÁ
INSTITUTO DE GEOCIÊNCIAS
PROGRAMA DE PÓS-GRADUAÇÃO EM GEOLOGIA E GEOQUÍMICA

PARECER

Sobre a Defesa Pública da Tese de Doutorado de JUCILENE AMORIM COSTA

A banca examinadora da tese de doutorado de **JUCILENE AMORIM COSTA** intitulada “**MINERALOGIA E GEOQUÍMICA DE TERRA PRETA ARQUEOLÓGICA PARA IDENTIFICAÇÃO DE PADRÃO OCUPACIONAL PRÉ-HISTÓRICO NO VALE DO BAIXO RIO AMAZONAS (JURITI, PARÁ)**” composta pelos Professores Doutores Marcondes Lima da Costa (Orientador-UFPA), Renato Kipnis (USP), Kelly das Graças F. Dantas (UFPA), Maura Imazio da Silveira (MPEG) e Vanda P. Lemos (UFPA) após a apresentação oral e arguição da candidata, emite o seguinte parecer.

A candidata apresentou contribuição relevante ao conhecimento sobre os processos de formação da Terra Preta Arqueológica e da Terra Mulata, identificação e interpretação de áreas de atividades, e para o estudo das cerâmicas arqueológicas em sítios de terra preta. A apresentação oral foi clara, bem estruturada e de conteúdo relevante, demonstrando conhecimento da literatura e discussão dos dados apresentados. Na arguição a candidata defendeu sua tese e a hipótese principal nela formulada, bem como respondeu às várias questões gerais e específicas apresentadas. O documento está bem redigido e bem estruturado. Contribuições foram feitas pela banca visando melhoria do trabalho para de artigos científicos submetidos para publicação.

Com base no exposto, a banca examinadora decidiu por unanimidade aprovar a tese de doutorado.

Belém, 16 de dezembro de 2011

Prof. Dr. Marcondes Lima da Costa (Orientador-UFPA)

Prof. Dr. Renato Kipnis (USP)

Prof.ª Dr.ª Kelly das Graças F. Dantas (UFPA)

Prof.ª Dr.ª Maura Imazio da Silveira (MPEG)

Prof.ª Dr.ª Vanda Pôrpolo Lemos (UFPA)