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Soil forensics: How far can soil clay analysis distinguish between soil vestiges?

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ABSTRACT

Soil traces are useful as forensic evidences because they frequently adhere to individuals and objects associated with crimes and can place or discard a suspect at/from a crime scene. Soil is a mixture of organic and inorganic components and among them soil clay contains signatures that make it reliable as forensic evidence. In this study, we hypothesized that soils can be forensically distinguished through the analysis of their clay fraction alone, and that samples of the same soil type can be consistently distinguished according to the distance they were collected from each other. To test these hypotheses 16 Oxisol samples were collected at distances of between 2 m and 1.000 m, and 16 Inceptisol samples were collected at distances of between 2 m and 300 m from each other. Clay fractions were extracted from soil samples and analyzed for hyperspectral color reflectance (HSI), X-ray diffraction crystallographic (XRD), and for contents of iron oxides, kaolinite and gibbsite. The dataset was submitted to multivariate analysis and results were from 65% to 100% effective to distinguish between samples from the two soil types. Both soil types could be consistently distinguished for forensic purposes according to the distance that samples were collected from each other: 1000 m for Oxisol and 10 m for Inceptisol. Clay color and XRD analysis were the most effective techniques to distinguish clay samples, and Inceptisol samples were more easily distinguished than Oxisol samples. Soil forensics seems a promising field for soil scientists as soil clay can be useful as forensic evidence by using routine analytical techniques from soil science.

1. Introduction

Although underestimated in forensic investigation, soils can provide crucial information to solve crimes [1]. Lack of interest in soils as forensic evidences may be due to misconceptions regarding soil homogeneity [2], and difficulties in processing soil traces and interpreting the results. However, many crimes take place under circumstances where soil traces are transferred from their original site to tools, weapons, tires, shoe soles, clothes, skin, hair, and other surfaces, leaving evidence that may link an individual, animal or object to a crime scene [3–7]. Reports on the successful use of soil as forensic evidence dates from 1856 in Germany [8], but over a century later soil analysis has yet to become a common practice in forensic investigations [4,5].

Examination of soils in a forensic context can contribute to the investigation of alleged criminal events by providing a link between a particular scene or a suspect, victim, or an object, or by indicating that such a link does not exist [5,7]. The inferences for forensic intelligence and researches on soil science are different [9], but analytical methods

from soil science have been transferred to forensic analysis without major problem [3–6,10–18].

Soil is a complex mixture of silicate and oxide minerals, organic matter, microorganisms and other compounds that create an extremely large variety of soil types, which allows for their differentiation, even within a pedogenically uniform area [1,5,18]. Forensic studies have achieved varied success when they have focused on the characteristics of bulk soil samples such as color, pH, soil particle distribution, shape and texture, mass loss upon ignition, elemental analysis, carbon and nitrogen isotope ratios, magnetic susceptibility, and others [1–6,10–18]. The analysis of soil clay separately from the other soil components is particularly promising for crime investigations [4], because pedogenic processes act on parent materials and create long-lasting signatures in soil clays that fit the desired criteria for forensic evidence of singularity and stability [3]. Soil clay fractions contain minerals with distinct physical structures and chemical composition, which may place a particular clay vestige at a specific site. Ephemeral soil characteristics, such as concentration of available plant nutrients

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should be avoided in forensic analysis due to significant variability and instability caused by soil moisture, weather conditions, fertilizer application, land use, and other transitory conditions [3,4].

Screening soil evidences according to their particle size distribution is simple and effective [13], but soil evidence may change particle size distribution along the way when transferred from the crime scene to elsewhere [7]. In this sense, the smallest soil particle fractions (clay) would preferentially persist adhered to a person or an object as a vestige rather than the larger soil fractions (sand) [7].

Another distinguishable soil characteristic for forensic investigation is color [3,19], which results from a mixture of minerals and organic components [1,20]. The bulk soil color is primarily attributed to clay, especially to iron oxides that coat the surfaces of mineral grains. Therefore, color comparison of soil clay fractions is expected to provide more reproducible measurements and less variation bias than intact soil samples [11].

Soil forensics has greatly developed in recent years although it is a poorly explored field among soil scientists and forensic experts [5–7,19]. Aiming to evaluate the discriminatory power of soil clay for forensic use, we used routine soil analysis methods and multivariate statistics to test 1) the hypothesis that clay fractions gather singular characteristics for each soil type in a way that fits with forensic analysis, and 2) the principle that soil samples collected at shorter distances are more similar than samples collected at longer distances one from each other [21]. Clay color analysis (hyperspectral imaging), clay chemical fractioning, crystallographic analysis of clay minerals (X-ray diffraction), and determination of kaolinite and gibbsite contents in clays (differential scanning calorimetry and thermogravimetry analysis) were the techniques used for discriminating the collected soil samples.

2. Materials and methods

2.1. Study area

Soil samples for this study were collected in the Brazilian Federal District - BFD, which extends for 5814 km² on the Brazilian Central Plateau (Fig. 1). Oxisols cover 55% of BFD territory and inceptisols cover another 31% of it. The remaining 14% of the BFD territory is covered by eight different soil types (9%) and water reservoirs (5%) [22]. Regional topography varies from flat to gently sloped, with average altitude of 1100 m. Climate is Tropical Savanna (Aw - Köppen-Geiger) with well-defined wet and dry seasons. Annual mean temperature ranges from 21 °C to 24 °C and annual rainfall ranges from 1200 to 1600 mm, with 84% of precipitation occurring in summer.

Since crimes occur more frequently in urban areas [23], and 86% of BFD is covered by only two soil types, sixteen reddish Oxisol samples (Ox₁ to Ox₁₆) and sixteen Inceptisol [24] samples (Ic₁ to Ic₁₆) were collected in the BFD urban area from 10 cm of topsoil along two transects beside roads and sidewalks. Each sample weighed approximately 100 g and transects were located on sites of same geology and geomorphology for each soil type to reduce sources of variability among soil samples (Fig. 1). The sampled Oxisol developed from slate at the Brasília pediplain (950–1200 m altitude), and the sampled Inceptisol developed from sandy meta-rhythmites at the Contagem Rodeador pediplain (1200–1400 m altitude). The Oxisol transect was established along slopes from 0% to 2% and Inceptisol transect along 9%–11% of slope. Despite the homogeneity of the environment beneath each soil type, it is expected that soil characteristics become less similar as distances between soil samples increase along transects [21]. Thus, to check the minimum distance necessary to detect consistent variations between soil samples of the same soil type, we collected four Oxisol samples every 2 m (Ox₁–Ox₄), every 10 m (Ox₅–Ox₈), every 100 m (Ox₉–Ox₁₂), and every 1000 m (Ox₁₃–Ox₁₆). This totaled a transect of 4446 m that started at the following GPS coordinate: 15° 46' 36.2" S and 47° 56' 14.7" W. Likewise, we collected four Inceptisol samples every 2 m (Ic₁–Ic₄), every 10 m (Ic₅–Ic₈), every 100 m (Ic₉–Ic₁₂), and

every 300 m (Ic₁₃–Ic₁₆), along a transect of 1646 m that started at the following GPS coordinate: 15° 52' 47.7" S and 48° 0' 0.3" W (Fig. 1). The shorter transect was due to the absence of a larger area of similar geology and geomorphology covered by Inceptisol.

2.2. Soil sample preparation

Soil samples were air-dried, passed through a 2 mm sieve and manually homogenized before analysis. Approximately 50 g of each soil sample were treated with 30% (v/v) solution of H₂O₂ in 70 °C water bath until all organic matter was removed. Then, soil samples were mixed with 200 mL of 0.2 mol L⁻¹ NaOH solution and agitated for 90 min in a circular shaker. Subsequently, samples were macerated with a rubber baton and washed with deionized water through a 0.053 mm sieve to separate the sand fraction. Suspensions containing silt and clay particles were collected in porcelain capsules. This process was repeated until the water following maceration was limpid. Silt and clay fractions were separated by particle sedimentation, based on Stokes' Law [25]. The silt fraction was discarded, and the clay fraction was oven-dried at 45 °C and grounded into a powder for color analysis (hyperspectral imaging), mineralogical identification (X-ray diffraction), determination of iron oxides concentrations after chemical fractioning, and contents of kaolinite and gibbsite (differential scanning calorimetry and thermogravimetry analysis).

2.3. Hyperspectral imaging (HSI) of clay samples

Powdered clay samples were submitted to reflectance spectral analysis in a ChemImage HSI Examiner™ 200 QD equipment. Samples were placed on Petri dishes and continuously imaged at 5 nm pace from 400 nm to 1100 nm wavelengths with a tungsten halogen light source under a 10 × magnification. Each sample was imaged ten times and reflectance mean values were calculated at each 5 nm reading pace. Clay samples displayed reflectance peaks at 730, 760, 780, 790, 825, 860, 925, 945, 995 and 1085 nm. Values of reflectance on these peaks were selected for the comparison of the clay samples (Table S1).

2.4. Chemical fractioning of clay samples

2.4.1. Extraction of poorly ordered iron (Fe) and aluminium (Al) oxides

Powdered clay samples of 0.4 g were placed in 200 mL tubes and extracted under dark condition with 10 mL of 0.2 mol L⁻¹ ammonium oxalate (AO) solution adjusted to pH 3.0. Tubes were mechanically agitated for 2 h and subsequently centrifuged at 3000 rpm for 10 min.

Suspensions were analyzed for extracted Fe in a Shimadzu 1240 UV/VIS spectrophotometer at 510 nm and recorded as Fe_{AO}. Clay mass reduction due to AO-extraction refers to the content of poorly ordered Fe and Al oxides removed from samples, which were recorded as AO_{Clay} (Table S2).

2.4.2. Extraction of crystalline iron oxides (Fe₂O₃)

Powdered clay samples of 2.5 g were placed in 100 mL tubes and extracted four times with a solution of citrate-bicarbonate-dithionite (CBD): 40 mL of 0.3 mol L⁻¹ sodium citrate + 5 mL of 1.0 mol L⁻¹ sodium bicarbonate + 1.0 g of powdered sodium dithionite. Samples in solution were manually agitated while heated at 70 °C for 30 min in a water bath apparatus. After cooling, solutions were centrifuged at 3000 rpm for 10 min. Suspensions were analyzed for extracted iron (Fe) in a Shimadzu 1240 UV/VIS spectrophotometer at 510 nm and recorded as Fe_{CBD} (Table S2). Deferrified solid phases left in tubes were washed with 80 mL of 0.5 mol L⁻¹ (NH₄)₂CO₃ solution, subsequently with 50 mL of deionized water and finally oven-dried at 45 °C. Oven-dried deferrified solid phases were analyzed for kaolinite (Kt) and gibbsite (Gb) in an X-ray diffraction (XRD) crystallographic study, differential scanning calorimetry (DSC), and thermogravimetry (TG) analysis. Clay mass reduction due to CBD-extraction refers to the

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