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Soil Forensics - Methods and Protocols of Soil Analysis for Criminal Investigations- A Brief Study.

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ABSTRACT

Forensic soil science represents a newly-developed discipline of soil science, and has matured to the extent that well-defined questions and successful crime scene investigations can be answered in increasingly refined ways. The systematic forensic soil examination approach described in this paper uses soil morphology (e.g. color, consistency, texture and structure), mineralogy (X-ray powder diffraction) and chemistry (e.g. based primarily upon mid-infrared spectroscopy/diffuse reflectance infrared Fourier transform (DRIFT) analyses). Forensic soil characterization usually combines the descriptive and analytical steps for rapid characterization of whole soil samples for screening, and detailed characterization and quantification of composite and individual soil particles after sample selection, size fractionation and detailed mineralogical and organic matter analyses using advanced analytical methods. X-ray powder diffraction methods are arguably the most significant for both qualitative and quantitative analyses of solid materials in forensic soil science.

Keywords: Soil morphology, DRIFT, X-ray powder diffraction, Size fractionation

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INTRODUCTION

Forensic soil scientists (or forensic geologists) are more specifically concerned with soils that have been disturbed or moved (usually by human activity), sometimes comparing them to natural soils, or matching them with soil databases, to help locate the scene of crimes. Forensic soil scientists usually obtain soil samples from crime scenes and suspected control sites from which soil may have been transported by shoes, a vehicle or a shovel. Soil properties are diverse and it is this diversity which may enable forensic soil scientists to use soils with certainty as evidence in criminal and environmental investigations. Forensic soil science is a relatively new activity that is strongly 'method-orientated' because it is mostly a technique-driven activity in the multidisciplinary areas of pedology, geochemistry, mineralogy, molecular biology, geophysics, archaeology and forensic science. Consequently, it does not have a large number of past practitioners such as in the older forensic disciplines like chemistry and physics.

Identification of soil differences using various morphological soil attributes (e.g. colour, consistency, texture and structure) on whole soil samples is the first step for using soil information to help police investigators at crime scenes. The second step is to discriminate soils using the discriminating power inherent in soil materials, especially in the use of: (i) powder X-ray diffraction techniques that provide qualitative and quantitative data in the mineralogical composition of samples; and (ii) spectroscopy (e.g. Fourier transform infrared spectroscopy), which provides qualitative and semi-quantitative information about the soil organic matter (proteaceous, aliphatic, lipid, carboxyl and aromatic), mineral composition (smectite, kaolin and illite clays, quartz) and prediction of soil physicochemical properties using partial least-squares analysis (MIR-PLS) and inductively-coupled plasma spectroscopy mass spectroscopy providing elemental composition. Detailed soil characterization usually requires a joint approach that combines the descriptive and analytical steps for: (i) rapid characterization of whole soil samples for screening (Stage 1) and (ii) detailed characterization and quantification of composite and individual soil particles after sample selection, size fractionation and detailed mineralogical and organic matter analyses using advanced analytical methods (Stage 2). The same principles apply to environmental investigations of polluted sites. [1,4,6,8]

Approaches and Methods for Making Comparisons between Soil Samples

Forensic soil scientists must first determine if uncommon and unusual particles, or unusual combinations of particles, occur in the soil samples and must then compare them with similar soil in a known location. To do this properly, the soil must be systematically described and characterized using standard soil testing methods to deduce whether a soil sample can be used as evidence.

Methods for characterizing soils for a forensic comparison involve subdividing methods into two major steps, descriptive (morphological) and analytical.

Detailed soil characterization usually requires a joint approach that combines the descriptive and analytical steps in the following two stages:

Stage 1 – Rapid characterization of composite soil particles in whole soil or bulk samples for screening of samples.

Stage 2 – Detailed characterization and quantification of composite and individual soil particles following sample selection, size fractionation and detailed mineralogical and organic matter analyses using advanced analytical methods.[1,6]

Stage 1: Initial Characterization of Composite Soil Particles in Whole Samples for Screening

This stage involves morphological characterization of bulk soil samples. Soil morphological interpretation provides a visual, quick, and nondestructive approach to screen and discriminate among the various types of samples. Morphological soil descriptors are arguably the most common and simple; it is for this reason that all bulk samples are characterized first using morphological descriptors using international soil morphological methods. The eight main soil morphological descriptors of: (i) matrix color (moist and dry using Munsell Soil Color Charts), (ii) mottles (retained from geologic sources), (iii) redoximorphic features (providing an indicator of drainage or redox status because soil color relates to soil aeration of weakly reducing conditions; (iv) concentrations (nonredox; e.g., carbonates, nodules or inherited brick fragments), (v) texture

(e.g., sands, loams or clays), (vi) structure (e.g., massive or platy), (vii) effervescence class (reaction to 6N HCl, which indicates the presence of carbonates), and (viii) water repellence class are the most useful properties for visual soil characterization and assessing soil conditions. Other useful soil morphological descriptors are quartz grain shape (if sandy) and rock or other fragments (if easily observable on questioned items, such as on a shovel). [5]

Stage 2: Detailed Characterization of Composite and Individual Soil Particles

X-ray Diffraction (XRD) Methods

In many soil forensic case investigations, the amount of soil available for analyses (e.g. on the sole of a shoe) may preclude routine bulk analyses. In such situations, it is best to use an XRD fitted with a system for analysis of extremely small samples (e.g. thin coatings or single particles of the order of 2 to 10 mg) loaded into thin glass capillaries. For analysis in a Gandolfi or Debye-Scherrer powder camera, extremely small specimens (e.g. single mineral particles and paint flakes) can be mounted on the end of glass fibers. [3] Consequently, according to Kugler (2003), X-ray methods are often the only ones that will permit further differentiation of materials under laboratory conditions. According to Murray (2004), "Quantitative XRD could possibly revolutionize forensic soil examination". Methods such as XRD, XRF and DRIFT spectroscopy, whose results partially overlap, are used. These overlapping results confirm each other and give a secure result to the examination. [5]

Scanning Electron Microscopes (SEM) and Transmission Electron Microscopes (TEM)

SEM-TEM are also frequently used to examine the morphology and chemical composition (via energy dispersive spectroscopy) of particles magnified to over 100,000 times their original size making them very useful for discrimination. Soil minerals, fossils and pollen spores that occur in soils can be described and analyzed in detail by SEM and TEM and are therefore very useful indicators when studying soil samples. [3, 4]

Basic Laboratory Procedures For Initial Characterization Of Soil Specimens

Techniques of XRD and DRIFT analysis require expertise and are generally used only when detailed investigations of soil samples is required. There are certain procedures which can be easily carried out in a laboratory using basic materials to find out soil characteristics such as soil density. Here we shall highlight few of this procedures followed by us in our laboratory.

Determining Soil Density

Basic protocol is given below.

- Label six foam cups, one for each of your known and questioned soil specimens.
- Fill each cup roughly a third full with the corresponding specimen.
- Add tap water until each cup is nearly full. Add a drop of dishwashing liquid to each cup.
- For each sample, use the stirring rod to break up any clumps of soil. Stir the contents until any vegetable matter and other light material floats to the top.
- 5. Allow the contents of each cup to settle for a minute or two, and then carefully pour off most of the excess liquid. Avoid pouring off any of the solid soil.
- Repeat steps 3 through 5 for each cup. After this second wash, all or most of the light material should have been removed from the samples in all of the cups. If not, do a third wash on all of the cups.
- Pour the damp soil from each cup into an individual drying dishes, transferring as little water as possible. Place the drying dishes in the oven and heat them on low heat until they have dried completely.
- While you are waiting for the specimens to dry, fill the soda bottle with tap water and add a few drops of dishwashing liquid. Invert the bottle several times to mix the solution.
- After the samples have dried, allow them to cool to room temperature.
- Weigh out about 50 g of the dry questioned specimen and record its mass to the resolution of your balance in your lab notebook.



- Fill the 100 mL graduated cylinder to 50.0 mL with water from the soda bottle, using a disposable pipette to add water dropwise until the cylinder contains as close as possible to 50.0 mL. Record this initial volume as accurately as possible in your lab notebook.
- Withdraw a few mL of the water from the graduated cylinder with each of two pipettes. Set them aside, inverted to make sure none of the water leaks from the pipettes.
- Using a folded sheet of paper, carefully transfer the weighed questioned soil specimen to the graduated cylinder. Make sure as little as possible of the soil specimen adheres to the walls of the cylinder above the liquid level.
- Your goal is to make sure all of the soil is immersed in the liquid. If you get air bubbles under the surface of the liquid, tap the cylinder or use the stirring rod to eliminate them.
- Use the liquid stored in the disposable pipettes to rinse down any soil that adheres to the inside surface of the graduated cylinder above the liquid line. Make sure to expel all of the liquid from both of the disposable pipettes, restoring the exact amount of liquid to the cylinder that was present at the initial measurement.
- Determine the new liquid volume as accurately as possible and record it in your lab notebook.
- Subtract the initial volume from the final volume to determine the volume of liquid displaced by the specimen. For example, if the graduated cylinder initially contained 50.4 mL and the final volume was 84.2 mL, calculate the displaced volume as $84.2 - 50.4 = 33.8 \text{ mL}$.
- Record the displacement volume in your lab notebook.
- Divide the mass of the specimen by the volume displaced to determine the density of the specimen in grams per milliliter. For example, if your specimen mass was 50.39 g and the displacement volume was 33.8 mL, calculate the density of the specimen as $50.39 \text{ g} / 33.8 \text{ mL} = 1.49 \text{ g/mL}$. Record this value in your lab notebook.
- Repeat steps 10 through 17 for each of the known specimens. Compare the density value you obtained for the questioned specimen with those you obtained for the known specimens to determine if one or more of the known specimens is similar in density.

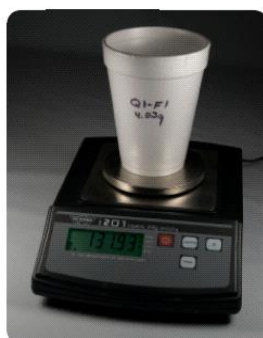
Now let us discuss about calculation of **soil settling time**:

- Label six test tubes Q1 and K1 through K5.
- Transfer the questioned soil specimen to tube Q1 until the tube is about one quarter full. Tap the tube gently to settle the soil specimen.
- Fill tube Q1 with tap water (with detergent added) to about 1 cm from the rim.
- Agitate the contents of the tube to suspend the soil in the liquid, and immediately note the start time in your lab notebook. Replace the tube in the rack and observe it as the soil settles.
- When the soil appears to have settled completely, record the finish time in your lab notebook.
- Subtract the start time from the end time to determine the elapsed time needed for the specimen to settle completely. Record that elapsed time in your lab notebook.
- Repeat steps 2 through 6 for specimens K1 through K5. Unless your specimens settle very quickly, you'll have time to start some or all of the remaining tubes before settling completes in the first tube.
- Compare the settling times of the questioned specimen and the known specimens to determine if the questioned specimen is consistent with one or more of the known specimens.

Determine soil particle size distribution

- If you have not already done so, put on your splash goggles, gloves, and protective clothing.
- Assuming that you have six soil specimens, label six foam drink cups Q1, K1, K2, K3, K4, and K5.
- Assuming that you will separate each soil specimen into four fractions, label four foam drink cups "Q1-F1" through "Q1-F4," four more cups "K1-F1" through "K1-F4," and so on until you have 24 labeled fraction cups, four for each of the six soil specimens.
- Weigh each fraction cup and record its mass to the maximum resolution of your balance. Write the mass of each cup on the cup itself.
- Weigh about 200 g of the questioned specimen to the maximum resolution of your balance and record that mass in your lab notebook. (If the capacity of your balance is too small, simply weigh the 200 g of soil in multiple portions.)
- Transfer the specimen to cup Q1 and add sufficient water to the cup to form a soupy mix.

- Swirling the cup to keep the soil suspended, pour the suspension through your largest mesh, capturing the liquid and solids that pass through the mesh in another container. Add more water to the cup as necessary to make sure that all of the soil in the cup is rinsed into the mesh, but try to use as little water as possible while still transferring all of the soil.
- Transfer the soil particles captured by the first mesh into cup Q1-F1. If necessary, rinse the particles off the mesh, but try to use as little water as possible.
- The large particles captured by the first mesh should settle very quickly. Once those particles have settled, use a pipette to remove and discard as much water as possible from cup Q1-F1 to speed drying. Make sure not to remove any of the soil particles. Set cup Q1-F1 aside to dry.
- Set up your second sieve, and pour the soil/water suspension that passed the first sieve through the second sieve, again capturing the liquid and solids that pass the mesh in another container. Make sure that all of the soil is transferred from the cup into the second sieve, using as little water as possible to rinse the soil into the sieve.
- Transfer the soil particles captured by the second mesh into cup Q1-F2, again using as little water as possible to do a complete transfer. Once the particles have settled, again use a pipette to remove as much water as possible from the cup without removing any soil particles. Set cup Q1-F2 aside to dry.
- Repeat the preceding steps with each of your sieves until you have isolated each fraction into its own fraction cup and set it aside to dry.
- Repeat the preceding steps with soil specimens K1 through K5.
- At this point, you have a large array of cups, all of which contain damp (or wet) soil specimen fractions. You can allow these specimens to dry naturally, which may require several days. Alternatively, you can dry them in an oven set to its lowest temperature (typically about 120°F or 50°C). Before you do that, perform a test with an empty cup to make sure it won't melt.
- Once all of your specimens are dry, weigh each cup to the maximum resolution of your balance. Subtract the empty mass of the cup from the mass of the cup with the specimen fraction to determine the mass of the specimen fraction, and record that value in your lab notebook.
- For each soil specimen fraction, divide the mass of that fraction by the total mass of all fractions, multiply that result by 100 to determine the fraction mass percentage, and enter that value in your lab notebook. Note that we are calculating the fraction mass percentages based on the total mass of all fractions isolated rather than on the initial mass of the specimen. That's because some of the material in the original specimen may have been soluble and so dissolved in the water we used to separate the fractions.
- We have the data necessary to calculate one more possibly useful value, the insoluble mass percentage of each of our specimens. We know the original mass of each specimen, and we know the total mass of all of the fractions we isolated for each specimen. Divide the total mass of the isolated fractions of each specimen, divide that value by the original mass of that specimen, and multiply by 100 to determine the percentage of the original specimen that was insoluble in water. Record that value in your lab notebook.



In our session, the mass of the foam cup was 4.03 g. With the dried Q1-F1 soil specimen fraction added, the mass was 131.93 g. Subtracting the mass of the empty cup gives us the mass of the Q1-F1 soil fraction, $(131.93 - 4.03) = 127.90$ g.

Figure 1: Weighing of specimen on electronic balance

Examining microscopic characteristics of soil samples

- If you have not already done so, put on your splash goggles, gloves, and protective clothing. (In this lab session, the purpose of these safety items is less to protect you from the specimens than to protect the specimens from you. Forensic technicians and scientists always wear protective gear to avoid contaminating specimens.)
- Label six well slides Q1 and K1 through K5, and transfer small amounts of the corresponding soil specimens to each slide. You needn't fill the well completely. Ideally, you want just enough soil in each well to provide a single layer of particles.
- Examine each specimen with the magnifier or at low magnification under a stereo microscope. Sometimes, similarities and differences between specimens are more clearly visible at lower magnification. At higher magnification, you may not be able to see the forest for the trees.
- With the compound microscope set to 40X magnification, observe the questioned soil specimen. Record detailed observations for that specimen in your lab notebook. Here are some questions to keep in mind as you observe the specimen
- When you finish examining the specimen at low magnification, switch to medium and then high magnification, which may reveal additional details.
- Repeat steps 4 and 5 for each of the known specimens.
- After you complete step 6, you should have a good idea of which of the known specimens, if any, is consistent with the questioned specimen. If you find a consistent known specimen, compare it directly with the questioned specimen, switching the slides in and out of the microscope stage. Record your observations during the direct comparison in your lab notebook. [4,5,6]



Figure 2: Sand grains under the microscope

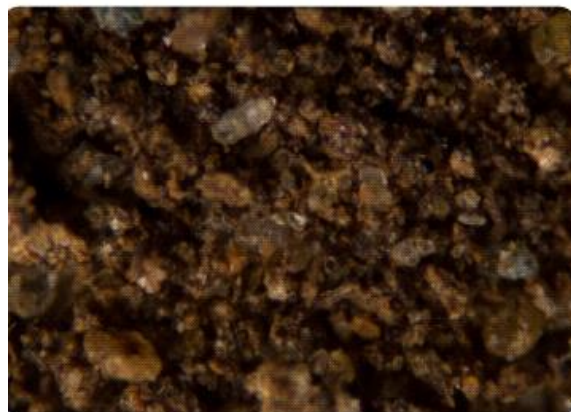


Figure 3: Sand grains as observed under 40x

CONCLUSIONS

Soil materials are routinely encountered as evidence by police (physical evidence branch) for crime scene investigators and forensic staff. However, most forensic and physical evidence laboratories either do not accept or are unable to adequately characterize soil materials. The main reason for this is that morphological, mineralogical and spectroscopic analytical knowledge required to examine and interpret such soil evidence needs a large amount of training and expertise, but initial analysis of soil characteristics such as determining soil density is relatively easy and can be performed easily in a laboratory. This initial analysis of soil may play a major role in determining whether detailed analysis of soil is required or not. The crime scenario example illustrate the use of combined pedological , mineralogical and spectroscopic methods in the forensic comparison of transported soil samples to forensic evidence items (e.g. shoes) with control soil samples from either the scene of the crime or a site traversed by the suspect in association with the crime. Forensic soil examination can be complex because of the diversity and in-homogeneity of soil samples. However, such diversity and complexity enables forensic examiners to distinguish between soils, which may appear to be similar.

Future Prospects

Soil Forensics is relatively a new branch in forensic science. Techniques such as DNA fingerprinting, Blood spatter analysis etc had been developed years before investigators realized that soil also could be used as integral evidence in criminal investigations.

Though the importance of soil forensics was realized much later, in recent years there has been significant development in this field. Techniques such as XRD, DRIFT analysis have proved to be efficient in soil analysis for criminal investigations.

As a whole Soil Forensic is yet to develop into more advanced science, so if special emphasis is laid on importance of soil as evidence then the investigators and scientists could develop more advanced techniques for soil analysis in future. For research and practical application in this area to grow appreciably, it will need to be considered and taught as an integral part of both soil science and forensic science courses. Finally, an attempt should be made to develop and refine methodologies and approaches to develop a practical 'Soil forensic manual with soil kit for sampling, describing and interpreting soils. Development of extensive soil databases similar to fingerprint databases will go a long way in helping the investigators to match their soil analysis results to the database thus speeding up the process of criminal investigation. Also efficient training of Soil scientists and geologists will help them to serve as expert witness and thus will enable soil analysis results as admissible proof of evidence in court of law.

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REFERENCES

- [1] Blackledge RD and Jones EL , All that glitters is gold! In: Forensic Analysis on the Cutting Edge: New Methods for Trace Evidence Analysis, (Ed. RD Blackledge),Wiley,New York,2007, pp. 1–32.
- [2] Chisum, W.J., & Turvey, B. "Evidence Dynamics: Locard's Exchange Principle & Crime Reconstruction," *Journal of Behavioral Profiling*, January, 2000, Vol. 1, No. 1
- [3] Dawson LA, Campbell CD, Hillier S and Brewer MJ , Methods of characterizing and fingerprinting soil for forensic application. In: Soil Analysis in Forensic Taphonomy: Chemical and Biological Effects of Buried Human Remains,(Eds. M Tibbett and DO Carter), CRC, Boca Raton, FL. 2008, pp. 271–315.
- [4] Fitzpatrick RW , Nature, distribution and origin of soil materials in the forensic comparison of soils. In: Soil Analysis in Forensic Taphonomy: Chemical and Biological Effects of Buried Human Remains (Eds. M Tibbett and DO Carter), CRC, Boca Raton, FL. 2008, pp. 1–28.



- [5] Fitzpatrick RW, McKenzie NJ and Maschmedt D , Soil morphological indicators and their importance to soil fertility. In: Soil Analysis: An Interpretation Manual (Eds. K Peverell, LA Sparrow and DJ Reuter), CSIRO Publishing, Melbourne, Australia, 1999, pp. 55–69.
- [6] Fitzpatrick RW, Powell B, McKenzie NJ, Maschmedt DJ, Schoknecht N and Jacquier DW ,Demands on soil classification in Australia. In: Soil Classification: A Global Desk Reference(Eds. H Eswaran, T Rice, R Ahrens and BA Stewart), CRC, Boca Raton, FL.2003 pp. 77–100.
- [7] Fitzpatrick RW, Raven MD and Forrester ST, Investigation to determine if shoes seized by South Australia Police contain soil materials that compare with a control soil sample from the bank of the Torrens River, Adelaide. CSIRO Land and Water Client Report CAFSS_027. January 2007.
- [8] Fitzpatrick RW, Raven M and McLaughlin MJ (2006). Forensic soil science: an overview with reference to case investigations and challenges. In: Proceedings of the First International Workshop on Criminal and Environmental Forensics (Ed. RW Fitzpatrick). Centre for Australian Forensic Soil Science, Perth, Australia. <http://www.clw.csiro.au/cafss/>