B89 The Effects of Sample Preparation Optimization on Soil Mineral Analysis by Particle Correlated Raman Spectroscopy (PCRS)

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Learning Overview: The goal of this presentation is to demonstrate how optimization of sample preparation and particle dispersal parameters impact soil mineral analysis by PCRS.

Impact on the Forensic Science Community: This presentation will impact the forensic science community by providing an optimized soil mineral sample preparation and particle dispersal method as a part of the development of an analytical approach to forensic soil analysis using PCRS.

Soils consist of minerals, organic matter, microorganisms, anthropogenic, and other materials. Each of these constituents within the soil matrix can provide a significant amount of information for forensic soil analysis. Unfortunately, there has been a decrease in forensic soil examinations due to a plethora of issues, including the perceived complexity of soil analysis without accompanying statistical significance determinations, the time required for traditional microscopic analysis, budget reductions for trace examinations, and the failure of crime scene investigators to recognize the value of soil evidence. PCRS and Morphologically Directed Raman Spectroscopy (MDRS) are novel analytical techniques that have the potential to automate soil analysis methods. They provide reliable information on particle size distribution and particle morphology (circularity, elongation, etc.) and subsequently perform Raman spectroscopic analysis of isolated individual particles to accurately identify soil mineralogy.

Sample preparation is a key aspect to any microscopic investigation. Measurements are performed on individual microscopic particles, such as soil mineral grains. For meaningful results, and to maximize the evidentiary value of soil samples being analyzed, sample preparation needs to be optimized. It is important to ensure particles are reproducibly, evenly, and wholly distributed onto a substrate for analysis, and therefore the dispersion parameters must be optimized.

PCRS uses computer-controlled vacuum-type powder dispersion while MDRS employs a positive pressure dispersion technique. These dispersion systems are used to create a uniform distribution of microscopic mineral particles with good spatial separation. Poor dispersion may lead to clustering, overlapping, or fractured particles, which negatively affect the automated analysis of PCRS. Therefore, having optimized dispersion parameters allows accurate collection of data from individual particles, thus maximizing the information obtained from the soil samples and increasing the discriminating power of PCRS.

This study specifically considers the dispersion using PCRS in detail, though many of the elements are valid for both. Nine soil samples of varied geological provenance (three sand, three loam, and three silt) were collected from around the northeast United States. Each sample was air dried to a powder and subsequently coned and quartered. For each sample, one-quarter was analyzed without further processing, one-quarter was washed and dried before analysis, one-quarter was washed, dried, and sieved before analysis, and one-quarter was retained for traditional forensic soil analysis. Soil samples were then dispersed onto a glass slide or plate by computer-controlled vacuum-type powder dispersion. The variables assessed in this research included washing of the samples, sieving of the samples, and optimizing vacuum dispersion parameters (sample volume, vacuum pressure, diameter of dispersal element, and time for dispersal and settling). Dispersion was assessed microscopically via PCRS for reproducibility, uniformity, dispersion density, and the maintenance of particle morphological characteristics throughout the processing. Determination of the optimal sample preparation parameters is a critical first step in creating a reliable method for obtaining the most forensically relevant information from soil evidence using PCRS.

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