Research Article

Micro- to Nanoscale Morphologies and Chemical Components of Soils Investigated by SEM-EDS for Forensic Science

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As a kind of microscale physical evidence, soil can provide significant assistance to forensic science. In this study, soil samples that were collected from eighteen different regions of Shandong Province, China, were examined by scanning electron microscope-energy dispersive spectrometer (SEM-EDS). The homogeneities and diameters of the samples were evaluated by SEM which has been applied to observe objects at nanoscale. The soil from Jiexiang, a city in Eastern Shandong Province, showed the maximal particle diameter and the sample from Liaocheng, another Eastern city in Shandong Province, showed the best homogeneity. The mass fraction and molar percentages of nine inorganic elements in all samples were analyzed by EDS. Oxygen and silicon showed the highest content in all of these samples. However, different samples exhibited their own characteristic elements, which can help to discriminate them from other samples. In this regard, SEM-EDS-based homogeneity and element analysis might be used as a fast and reliable technique for the soil criminological analysis in Shandong Province.

1. Introduction

Soil provides the nutrients and water required by terrestrial plants growth and exchange energy. Soil is influenced by various biotic and biological factors, such as climate, early formation of texture, microbial environment, and human activities. Therefore, the soil from various regions has tremendous differences in the components of metal, minerals, and the microbial communities [1, 2].

There is a strong connection between the soil and people's lives. Soil on the body or belongings of a person could be used as trace evidence in a large amount of criminal cases. As a kind of trace evidence, soil has played an important role in forensic science [3–6].

The physical properties of soil, including pH, color, density gradient, and inorganic components, are traditionally examined as evidences. With the development of modern technology, detection techniques for using soil as evidence are simultaneously improved [2]. Scanning electron microscopy is a type of electron microscope that produces

image of a sample by scanning it with a focused beam of electrons. The electrons interact with electrons in the sample, producing various signals containing information about the sample's surface topography and composition. Energy-dispersive X-ray spectroscopy (EDS) is an analytical technique used for element analysis or chemical characterization of samples. SEM-EDS can determine the sizes, shapes, and elemental compositions of particles, which has been adopted by many researchers to identify different particles and link them to possible sources. Cengiz et al. collected soil samples from seventeen different regions of Istanbul city, and concluded that the SEM-EDS method can discriminate the soil evidences [7]. To the best of our knowledge, morphology characteristics and elemental composition analysis about the soil particles in Shandong Province, China, have never been reported.

A biological method, T-RFLP analysis of Shandong's soil microbial community, was previously reported for forensic science [8]. As a supplement to the T-RFLP analysis, the SEM-EDS analysis technique was used as an abiotic method to

FIGURE 1: SEM images of soil from eighteen regions in Shandong province. S1 to S18, represent Zaozhuang, Yantai, Penglai (Yantai), Wulian (Rizhao), Changyi (Weifang), Rongcheng (Weihai), South mountainous area (Jinan), Liaocheng, Laiwu, Jiaozhou (Qingdao), Jiaxiang (Jining), Heze, Kenli (Dongying), Xiajin (Dezhou), Zouping (Binzhou), Qufu, Taian, and Zibo, respectively. The scale bar is 200 μm .

analyze the morphology characteristics and elemental composition of soil samples, which were collected from eighteen regions in Shandong province, China. All samples exhibited their characteristic element contents, suggesting that it is possible to identify a specific soil sample from the sample pool by SEM-EDS analysis. This SEM-EDS-based analytical method will be helpful in the criminological analysis of trace soil samples.

2. Materials and Methods

2.1. Sample Collection. Eighteen surface soil samples were collected from different regions of Shandong Province, China. The samples were numbered from Sample 1 (S1) to Sample 18 (S18), corresponding to Zaozhuang, Yantai, Penglai (Yantai), Wulian (Rizhao), Changyi (Weifang), Rongcheng (Weihai), South mountainous area (Jinan), Liaocheng, Laiwu, Jiaozhou (Qingdao), Jiaxiang (Jining), Heze, Kenli (Dongying), Xiajin (Dezhou), Zouping (Bingzhou), Qufu, Taian, and Zibo, respectively.

2.2. SEM-EDS Analysis. All of the soil samples were dried for 72 hours in an oven at 110–120°C in order to remove moisture. Before they were subjected to the SEM-EDS analysis, samples were coated with a layer of gold using a gold-plated machine for 120 s. Sample analysis was performed in a Hitachi S-2500 Scanning Electron Microscope. The images were quantitatively analyzed with the software Image J [9]. SEM was equipped with an Oxford 3294X energy dispersive spectroscopic detector and was automated by Oxford Instruments INCA Energy and Feature Software. The results were detected and recorded with the Genesis 60E software.

2.3. Results and Discussions

2.3.1. SEM-EDS Analysis of the Samples. SEM is capable of imaging at a much higher resolution than light microscopes, owing to the small de Broglie wavelength of electrons. This property enables samples to be examined in fine details, as small as a single column of atoms, which is tens of thousands of times smaller than the smallest resolvable object under a light microscope. Objects can be observed in the limit of 0.1 nm (10^{-10} m). SEM images of the eighteen soil samples from different regions of Shandong Province were shown in Figure 1. All the soils are composed of micrometer-scale grains which consist of smaller particles with nanoscale structure characterization. The particle sizes of S1, S8, S10, and S13 were more homogeneous than those of others. Due to the presence of larger particles, S2, S5, and S6 were inhomogeneous, while S8 was the most homogeneous in all of the tested samples.

The particle area of these soil samples is shown in Figure 2. Samples from different regions showed varied diameters. For all of the samples, the area of soil particles was generally in the range of 100 to 500 μm^2 . The increasing order of particle sizes was S11, S7, S18, S13, S12, S9, S16, S17, S15, S14, S4, S3, S2, S10, S1, S5, S6, and S8.

There are various elements in soil, such as oxygen, silicon, aluminum, iron, calcium, sodium, potassium, magnesium,

FIGURE 2: The particle area of soil samples from eighteen regions in Shandong province. Each bar represented the mean $\pm$ SD of three replicates.

and titanium. Soil samples from different environments contained varied elements, which were characterized by element types and their content ratios.

The mass fraction and the atomic percentage of nine elements from the eighteen samples were measured by EDS. As shown in Figure 3, the contents of oxygen and silicon were the highest in all of the samples, because they are the composition elements of SiO_2 and silicate, two of the most important components of soil. The contents of sodium and aluminium were relatively stable in all of these samples and ranged from 6.53% to 10.61% and from 0.46% to 1.54%, respectively. Contents of other elements varied obviously among different samples. Contents of calcium and magnesium in S11, S12, and S13 were higher than other samples, because many lands in Kenli, Xiajin, and Zouping are saline and alkaline. The content of the element titanium was the highest in S3 in comparison with all of the other samples, and the contents of the elements iron and potassium in S3 were the lowest, because this sample was collected near a coating plant. S9, S12, and S18 contained the lowest amount of magnesium, 0.98%, 0.80%, and 0.87%, respectively. The contents of the element calcium in the soil samples S1, S6, and S7 were higher than those in other samples, likely due to the native limestone constituents.

The atomic percentages of oxygen, silicon, sodium, and aluminium were relatively stable in all of these samples. Contents of calcium and magnesium were higher in S11, S12, and S13 than other samples. The atomic percentage of nine elements (Figure 4) showed the same trend as the mass fraction in Figure 3.

3. Conclusion

SEM-EDS-based analytical method could be used for the automatic examination and measurement of the geometric and morphological parameters, including the homogeneities and particle sizes of soil samples, which are important for the identification of the soil. The difference in soil elemental

FIGURE 3: Element mass fraction of soil samples from eighteen regions in Shandong province.

FIGURE 4: Element atomic percentage of soil from eighteen regions in Shandong Province.

components represents the variety of the soil texture from different regions, which is important when narrowing the tracking range of investigation. As a soil database in China is still in its infancy, this research will be helpful in performing the convenient batch detections of a large number of soil samples to establish a national and global SEM-EDS database for forensic science.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

Authors' Contribution

Yingzi Liu and Yumei Li contributed equally to this work.

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