The effect that storage conditions, drying method, and length of time that a soil sample is stored on FTIR results.

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Abstract

This study compares soil samples stored in a freezer at -20° C versus samples stored at room temperature and samples dried at room temperature versus dried in an oven at 100°C. Analysis of the Fourier transform Infrared Spectra is performed to determine if differences between length of storage time and storage conditions can be distinguished, and at what point differences might be detected. After the completion of the research it was found that there was little to no difference between the different methods when using FTIR. Thus whatever techniques are available to a laboratory can be used for comparison.

Introduction

When researching soil analysis in various articles such as Cox et al and Calderon et al, there is little mention on how samples are stored or dried. With this research we aimed to see if the storage conditions and drying methods can effect the results of soil samples when using an FTIR instrument to identify difference in the soil samples spectra. This research compared the ashed, un-ashed, and the organic content to see if difference could be seen between the sites and differences between length of storage time and storage conditions. The organic spectra were obtained by subtracting the ashed from the un-ashed spectra.

Materials and Methods

Collected soil samples from three locations: Marshall University Forensic Science Center at 1401 Forensic Science Drive, Huntington, WV 25701 and two from the Crime Scene House at 1524 5th Avenue Huntington, WV 25703.

Sample Storage from 0-5 weeks:

- Room temperature
- Freezer at -20°C

Drying Methods:

- Air dried till water weight evaporated
- Oven at 100°C till water weight evaporated

Sieving Method:

- Samples were lightly grounded with mortar and pestle
- 2mm size sieve was used to remove large debris
- Samples were lightly grounded with mortar and pestle a second time
- A 75 µm sieve was used to obtain the wanted fraction

Ashing Method:

- Samples were heated at 650°C till organic content was ashed

Instrumentation:

- Nicolet Nexus 670 FTIR with a diamond ATR "smart accessory."
- IR parameters were set to: 128 scans, 4cm^{-1} resolution, 4000-400 cm^{-1} range
- Bands were then labeled using the Ominc software Nicolet.

Results

Spectra were collected for the overall sample and the inorganic fraction of the samples. The inorganic spectra were subtracted from the overall spectra to reveal the organic fraction. The following spectra compared the different variables of the study.

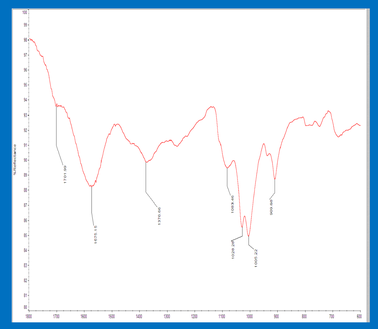

Figure. 1. Depicts a sample of a humic acid standard. This sample was run on a newer computer system which is why it looks different than the other figures

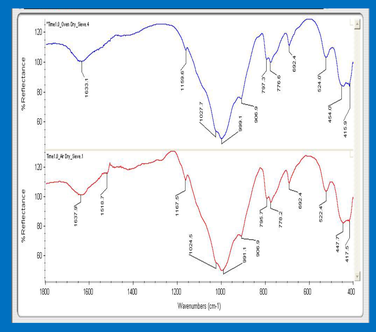

Figure 2. Compares an Oven Dried sample to a Air Dried sample.

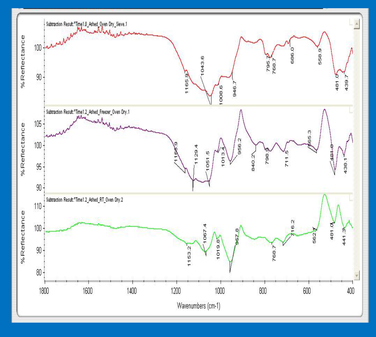

Figure 3. Compares Freezer stored samples to Room Temperature stored samples.

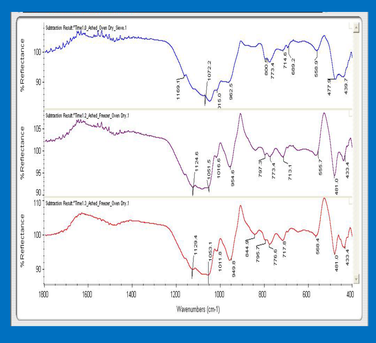

Figure 4. Compares the length of time in storage of the sample.

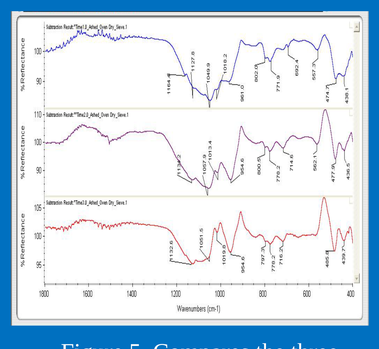

Figure 5. Compares the three different site locations

Conclusion

The main organic component of soil is humic acid. A humic acid standard was used for comparison with the study samples. The humic acid's spectrum should have peaks that match with the organic content of the soil samples that were collected from the three sites. However, that was not completely the case for the obtained results. The other peaks found in the other spectra could have come from the other living organisms that are made up by the soil, and not the humic acid.

It is always better to analyze evidence as soon as it is collected, and the same is found with soil samples. The superior results are always achieved when a sample is dried and analyzed immediately being collected. However, immediate analysis is not always an option. When comparing the storage conditions, both samples stored at room temperature and in the freezer exhibited similar results. Samples stored in the freezer exhibited results closer to the samples analyzed soon after collection; however, for the length of this study, differences between room temperature and freezer storage conditions were negligible. When comparing the drying techniques, both the oven dried and air dried samples gave the same results. So there is no reason to pick one drying method over the other, when looking only at the FTIR results.

References

Stevenson FJ, Goh KM. Infrared Spectra of Humic Acids: Elimination of Interference due to hygroscopic moisture and Structural changed accompanying Heating with KBr. Soil Science 1972; 117(1): 34-41.

Calderón FJ, Reeves III JB, Collins HP, Paul EA. Chemical Differences in Soil Organic Matter Fractions by Diffuse-Reflectance Mid-Infrared Spectroscopy. Soil Sci. Soc. Am. J 2009; 75(2):568-79.

Bornemann L, Welp G, Amelung W. Particulate Organic Matter at the Field Scale: Rapid Acquisition Using Mid-Infrared Spectroscopy. Soil Sci Soc Am J 2009;74(4):1147-56.

Cox RJ, Peterson HL, Young J, Cusik C, Espinoza EO. The forensic analysis of soil organic by FTIR. Forensic Science International 2000; 108:107-16.

Acknowledgments

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