How to minimise loss in spectral quality when going from laboratory to field measurements using a portable FTIR instrument?

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Use of FTIR in the MIR range for characterisation of Soil

- Overall chemical profile with both organic matter and full mineralogy represented
- Qualitative analysis - interpretation and visualisations allows insight into the nature of a soil sample
- Quantitative analysis – gives predictions of soil properties
- Generally still a Lab based method
Instrumentation for FTIR analysis in the field

- Development of suitable handheld FTIR instrumentation is relatively recent
- This study used both an Agilent 4100 and the newer Agilent 4300 FTIR spectrometer
- Spectral quality is as good as for Lab based instruments
- Both spectrometers had DATR and diffuse reflectance sampling options

- a – Agilent 4300 in lab using diffuse reflectance
- b – Agilent 4100 and 4300 in lab using DATR with clip
- c – Agilent 4300 in-field using DATR
Datasets Studied

- A subset (15 soils) from the National Inventory of Scotland (NSIS) dataset
- Variation in OM from 1-47%
- Fresh, fresh-dried, dried sieved soils of the NSIS subset were used
- “Best practice” FTIR spectra of the dry milled soils were already available for the subset

- 80 Swedish soils from 4 fields
- 2%C to ~25%C
- Lab and field measurements

Scottish Soils

Swedish Soils
Aims of the study

- Comparison of different sampling methods to analyse field condition soil
- Determine the effects of particle size, heterogeneity and soil moisture on the quality, reproducibility and representativeness of soil spectra
- Comparison of different versions of the FTIR spectrometer
- Aim of achieving the best possible spectra
Sampling Accessories – 1) Schematics

DATR

Diffuse Reflectance

The IR beam interacting with a sample in a diffuse reflectance experiment

Diamond/KRS-5 crystal

Sample

Thermo Fisher
Sampling Accessories – 2) Perceived Advantages and Disadvantages

**DATR:**
- small sampling area (1 mm x 2 µm) – disadvantage for heterogeneity/representativeness
- Close contact required between sample and DATR window
- Some bands in the spectrum are weak

**Diffuse reflectance:**
- larger sampling area/depth of penetration
- No contact required with sample
- Spectra of neat soil has inversion of strong bands
Results – 1) Heterogeneity

- The diffuse reflectance resulted in inversions of the strongest bands in the spectra.

- Measurements on the dried non-milled soil was still quite reproducible using diffuse reflectance for some samples.

- Organic soils are not well differentiated by diffuse reflectance but give the most reproducible spectra for the DATR.

- Replication for the DATR method was far better than anticipated, especially when using field condition soil.

- For some soil types, especially with coarse texture, averaging of multiple scans may be needed to overcome the heterogeneous nature of the soil.
Results – 2) Particle Size

- For particle size, an awareness of differences compared with milled samples is needed
- Minerals (other than clay minerals) are under represented in the un-milled spectra
- Mineral bands may be poorly resolved
- Relative proportions of SOM and clay minerals are enhanced
- Sloping baselines can also occur

Laboratory measured MIR spectra from two Scottish soils; a) organic soil, and b) mineral soil. I) are fresh samples measured using diffuse reflectance. II to IV are measured using DATR on II) fresh, III) dried, IV) dried and milled soil samples.
Results – 3) Water content

- Water presented a major problem for diffuse reflectance with difficulty in getting sufficient signal for fresh soil or measurements in the field.
- Moisture proved a mixed blessing with the DATR spectra - good contact and more representative spectra could be achieved but water bands were variable in strength and did obscure other bands in the spectra.

Allowing the fresh soil to dry on the DATR window produced spectra showing the same features as the fresh dried samples, but often less noisy, and with better replication.
Conclusions

- In general, the DATR method proved easier to use and appeared to perform better than the diffuse reflectance
- There seemed to be a major problem getting enough signal to do diffuse measurements on field condition soil
- Smearing soil on the DATR window, and then allowing to dry, proved the most successful approach for field condition soil
- Producing reasonable quality, representative, spectra was possible for field condition soil
- Spectra recorded using the Agilent 4300 appeared slightly less noisy than those from the Agilent 4100
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