The NIRS principle

NIRS is an analytical technique that characterizes materials according to their reflectance in the wavelengths ranging between 800 and 2500 nm. A light source emits a radiation that is received by the sample. This light react with the sample and is then (i) transmitted through the sample, (ii) absorbed by the sample and (iii) reflected by the sample. The way the light is divided into these 3 parts depends on the molecules that constitute the sample. The reflected light is caught by a light receptor. The reflectance (R) or absorbance (log 1/R) is put in graph as a function of wavelengths. This graph is the spectral signature of the sample (Figure 2).

What you need to use the NIRS method routinely in a lab

1. An analytical protocol and a spectrometer
   The spectral signature is influenced, among others, by soil humidity, particles size and obviously by the laboratory or the analyst. Different tests have been performed to find the better analytical protocol, easy to proceed and repeatable (Genot et al., 2008).
   We work with 2 mm sieved, dried samples (following ISO 11464) placed in a quarter cup. Each sample is scanned in duplicate. The spectra are compared calling the RMS (Root Mean Square): RMS ≤ 10,000: the measurement is accepted and the spectra are averaged; RMS > 10,000: the measurement needs to be done again.

2. An initial spectral library
   As many other analytical methods, NIRS doesn’t allow to directly obtain the wanted value (clay content of the soil); the spectral signature needs to be interpreted.
   To build the interpretation model, a spectral library is needed. It contains clay content determined with the reference method (ISO 11277) and the associated spectrum (couple REF value – NIR spectrum). The main characteristics of the library are (for the studied territory – Wallonia):
   • Representativeness of the clay content;
   • Representativeness of the land uses (cultivars, grasslands, forests, etc.);
   • Representativeness of the spectral diversity.

3. Model to predict the value from the spectrum
   To build the interpretation model, 3 steps can be distinguished: pre-treatment, building of the predictive model and validation of the model. For each step, different methods exist:
   - Pre-treatment: 1) baseline correction; 2) derivative; The pre-treatment allows to get a better view on the spectrum, get rid of the background noise, etc.
   - Predictive model: local PLS. The PLS method consists of replacing p correlated variables by k non-correlated new variables (the PLS factors). The PLS factors are then combined to write a predictive equation. The “local” approach means that, for each unknown sample to be predicted, the closest samples (from spectroscopic point of view) are selected in the library, used to define the PLS factors and write a predictive equation. As the closest samples are different for each unknown sample, the predictive equation is also different.

4. Grow the spectral library
   Growing the initial spectral library and its representativeness’s (clay content, land uses and spectral), leads to strengthen the predictive model, a better accuracy of the predicted values and a higher amount of correctly predicted samples.
   The contributors are:
   • the analytical laboratories of REQUASUD network; spread on the Wallonia region, they are the most able to feed the library on an efficient way;
   • the European LUCAS spectral library, for croplands and grasslands uses (survey implemented in 2009).
   These contributors work with the same reference method (ISO 11277) and the same equipment. That way, the so built spectral library is homogeneous and usable by every lab of the network, using the same predictive model.
   Nowadays, the spectral library is so big that it has been divided into 3 parts according to land use: croplands, grasslands et vegetable gardens.
   Here are the current basic statistics of the REQUASUD spectral library for clay content:

<table>
<thead>
<tr>
<th>Amount of samples</th>
<th>Claybands</th>
<th>Grassbands</th>
<th>Vegetable gardens</th>
</tr>
</thead>
<tbody>
<tr>
<td>9,603</td>
<td>277</td>
<td>77</td>
<td></td>
</tr>
</tbody>
</table>

   N.B. 1) A same laboratory may be represented by a different number over time. These graphs don’t show the evolution of a performance of the contributor over time.
   N.B. 2) A REF value means that GH = 3 and/or NH ≤ 1.

Control performances of the laboratories

PT’s (Performance Testing’s) are organized within REQUASUD network (2 per year), and NIRS has been implemented recently. Five samples are sent to the participants, each sample in duplicate so the repeatability can be assessed. Some samples are incorporated into several following PT’s, allowing the participants to better follow their performances over time.

Maximum difference between the predicted values of all the participants on identical samples, over time

<table>
<thead>
<tr>
<th>Sample</th>
<th>N/1</th>
<th>N/2</th>
<th>N/3</th>
<th>N/4</th>
<th>N/5</th>
<th>N/6</th>
<th>N/7</th>
</tr>
</thead>
<tbody>
<tr>
<td>NIRS2015-1</td>
<td>1.75</td>
<td>7</td>
<td>3.00</td>
<td>6</td>
<td>1.67</td>
<td>6</td>
<td>3.68</td>
</tr>
<tr>
<td>NIRS2015-2</td>
<td>2.38</td>
<td>6</td>
<td>2.38</td>
<td>6</td>
<td>2.38</td>
<td>6</td>
<td></td>
</tr>
</tbody>
</table>

The amount of correctly predicted samples is high (more than 80%). RPD values show a performant model for croplands and a medium model for grasslands. The performances of the model can be improved for vegetable gardens. This can happen by adding couples REF value – NIR spectrum in the spectral library.

Performances

Here are the current values for the statistical parameters indicating the performances of a predictive model, for the REQUASUD spectral library and clay content:

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References

Genot V et al., 2008. Study of the near infrared reflectance spectroscopy performances for the determination of soil parameters useful for the fertility diagnosis, European Geosciences Union, Vienna, Austria, 15 – 18 April 2009.

LUCAS database: https://ec.europa.eu/eurostat/fr/web/lucas/overview