# Use of Near Infrared Spectroscopy for compost characterisation

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## Introduction and objectives

Durable management of wastes generated by human activities is a complex problem to be solved by societies. During last decades, collective awareness about the volume of waste produced and their potential nuisances has led to the development of rules to allow a clear identification of wastes natures and sources as well as to inventory past, current and future deposit sites. These steps, as well as the development of sorting and management chains, clearly participate to the wish of our societies to achieve a durable organisation and to improve its quality of life in a broad sense. On a technico-economical point of view, due to the considerable masses that must be handled, this evolution generates increasing additional costs for the society. The collection, treatment, stocking and especially valorisation pathways need to be optimised.

In the pool of wastes generate daily by human activities of all kinds, an important part consists of organic materials. So 90 %, 15-20 % and 30-35 % of wastes generated, respectively, by agricultural, industrial and familial activities are organic.

Co-products of biological processes, farm organic matters are exempt of noxious substances and recycled generally as such on the exploitation. On the other side, organic wastes issue from urban or industrial activities remain, for a too large part, evacuated towards technical burying centre. Constituted essentially of carbon, hydrogen and nitrogen, organic matters are highly fermentescible. Once buried, non controlled fermentation take place and become sources of unacceptable nuisances (combustible gases, smells, pollutant lixiviates). Technical solutions necessary to dam up these effects are then of a prohibitive cost. Now, EU legislation foresees, in this respect, the suspension of organic materials burying accounting 2010.

As far as they arise from clearly described elaboration or sorting processes, an important part of industrial and/or urban organic wastes can be considered as not harmful. This authorises to consider their valorisation through other chains than burying or cremation ones. Environmental risks bound to the burying of organic material and to the non-controlled degradation which ensue can be easily solved through controlled bio transformation processes. These can be performed under aerobic and simple conditions, through composting, or anaerobic and technically more complex conditions, through biomethanisation.

The composting of an organic product is an exothermic microbiological process requiring the presence of oxygen, water and degradable carbon and nitrogen in well-balanced proportions. At the end of the biodegradation process, the original mass is reduced and homogenised. During such process mass temperature increases till 70°C, killing pathogens, weed seeds, and deodorising the final product that can be used as a fertilizer.

During the composting, organic substratum will be the unique source of food for the micro-organisms. Dynamic balances will establish and adjust themselves according to limiting factors: quantity of elements, immediate availability, ratio between nutriments. This explains that, in their original state, all organic wastes could not be composted as such. The absence of structure; and so of free oxygenation of the mass; the absence or excess of water, the excess of carbon or contrariwise of nitrogen can be limiting factors. Plant wastes as lawn mowing, branches, straw, pulps, vegetables surpluses; liquid or semi-solid manures, wools droppings, stercoraceous materials from slaughterhouse, or complex wastes such as poultry litters, kitchen waste, etc… are as many examples.

For such materials, co-composting will consist in gathering different organic materials (agricultural, agro-industrial,…) that have not an optimal structure, dry matter content or degradable N/C and to associate them in right proportions in order to initiate an optimal bio-transformation process.

In order to follow the good evolution of composting and co-composting process, different indicators such as temperature, ratio between total carbon and nitrogen, pH or rate of dry matter evolution[1,2] are classically considered. If the optimal level that have to be reached by these parameters are relatively well known, these indicators remain, for many of them, difficult to quantify in a fast way. To solve this problem, we will explore the potentialities to use Near Infrared Spectroscopy in order to predict these classical parameters. First approaches made in this direction[3], and existing literature [4,5,6,7,8] about development of Near Infrared Spectroscopy for soil studies denote of the feasibility of an approach to be continued.

This technique would allow to optimise initial mixing carbon and nitrogen ratio, to control classical parameters evolution, to control maturity of compost and to characterise the end product in a rapid way.

## Material and methods

### Material

This project is developed in collaboration with a co-composting enterprise (Agricompost) that provided all the organic materials used in this study.

Materials used are organic mixtures of manures, vegetable wastes, fruits and brewery wastes, wool droppings, stercoraceous materials from slaughterhouse. They are initially mixed with a pile reverter MENART 3500 SP (Ménart SPRL, Montroeul-au-bois). This operation allows to homogenise raw materials and to provide oxygen which is essential for the development of aerobic micro-organism populations. Under such conditions, bio transformation can occur and the organic mixtures can follow the four steps of the composting process: mesophilic, thermophilic, cooling down and maturation phases. All these steps are characterised by different temperatures.

Number of intervention of the pile reverter depends of parameters evolution. In order to have different maturity stages, samples are collected at different time during the process, so they are characterised by a more or less important decomposition.

### Calibrations on fresh material

Fresh matter is analysed by references analytical methods. For pH and conductivity, determination is made on a mixture of 100g of sample and 1000ml of demineralised water after 1 hour agitation, 10 hour of rest and a filtration. Measurement are realised with a pH meter WTW pH538 (VWR International SA, Leuven) and a conduct meter HANNA instruments HI9033 (Hanna Instruments SA, Tense).

Ammoniacal nitrogen is determined by a titration ($H_2SO_4$) of the ammoniac realised after an alcalinisation of 25ml of the mixture used for pH and conductivity measurment with NaOH. This measurement is performed with a KJELTEC AUTO 1030 analyser TECATOR (Foss Benelux SA, Hansbeke).

On the other hand, fresh sample are analysed in rectangular cup with a FOSS-NIRSystems 6500 scanning monochromator (Foss Benelux SA, Hansbeke). NIR calibrations were derived for dry matter (DM), total carbon, pH, conductivity, ammoniacal nitrogen and total nitrogen. Total nitrogen corresponds to total organic nitrogen (Kjeldahl) on dry matter.

### Calibrations on dry material

Fresh matter is dried at 65°C during 48 hours in a drying oven. Samples are grinded with a sieve of 1mm. Residual moisture is determined at 103°C during 3 hours.

Ashes are determined after a treatment at 550°C. Total carbon and total nitrogen were determined by a Perkin-Elmer 2400 CHN Elemental Analyser (PerkinElmer Analytical Instruments SA).

Samples are finally scanned with a NIRSystems 5000 (Foss Benelux SA, Hansbeke) in small ring cup. Studied parameters in NIR are residual moisture, organic nitrogen, ashes, total nitrogen and carbon.

## Results and discussion

### Calibrations on fresh material

Results present interesting determination coefficient for dry matter (0.95) and total carbon (0.87). Population standard error/standard error of prediction ratios (SD/SEC) are of 5.3 and 3.4, respectively for dry matter (DM) and total carbon. This ratio allows a comparison of the developed equations which is independent of the units and has already been used by other authors. SD/SEC values of 2.5-3.0 are considered adequate fo screening the samples for quality but values of at least 3.0-5.0 are required for quality assurance[9].

Determination coefficient for total nitrogen, ammoniacal nitrogen, pH and conductivity are lower with values included between 0.51 and 0.72. Standard error population/standard error of prediction ratios are under three for all these parameters: 1.6 for pH, 1.9 for conductivity and 1.6 for ammoniacal nitrogen (Table 1 and Figure 1).

These results are probably due to matrix complexity. Indeed, as underlined previously, initial organic mixtures are made with a wide range of raw materials. This diversity especially influence results for samples took at the beginning of the process. At this time, bio transformation start and most of materials have maintained their initial structure. For some parameters, especially for conductivity, the low population size could also partly explains these results.

**Table 1 : NIRS calibration characteristics for the prediction of co-compost parameters on fresh material**

| | N | Mean | SEC | RSQ | SECV | 1-VR | SD | SD/SEC |
|---|---|---|---|---|---|---|---|---|
| FRESH MATTER | | | | | | | | |
| DM | 147 | 25,17 | 2,08 | 0,96 | 2,44 | 0,95 | 10,99 | 5,28 |
| Total Nitrogen | 127 | 2,42 | 0,23 | 0,82 | 0,29 | 0,72 | 0,55 | 2,39 |
| Total Carbon | 129 | 34,93 | 2,16 | 0,92 | 2,68 | 0,87 | 7,44 | 3,44 |
| pH | 149 | 8,48 | 0,35 | 0,63 | 0,40 | 0,51 | 0,57 | 1,63 |
| Conductivity | 74 | 1478 | 307 | 0,72 | 377 | 0,59 | 585 | 1,90 |
| $N_{NH3}$ | 147 | 0,18 | 0,09 | 0,63 | 0,10 | 0,52 | 0,14 | 1,56 |
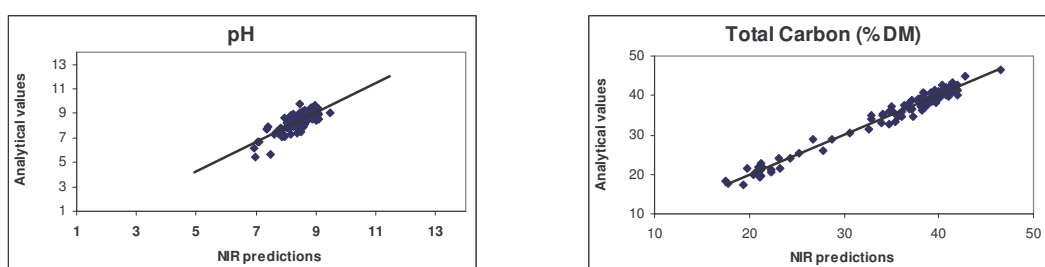


**Figure 1: Correlation between labs and NIR measurement, on fresh material, for pH and total carbon (%DM)**

Calibrations on dry material

Determination coefficients for dry matter are always above 0.77. The best results are for ashes (0.96) and total carbon (0.92). Standard error population/standard error of prediction ratios (SD/SEC) is above 3 for organic nitrogen (3.7), total carbon (4.1) and ashes (6.0). Calibrations can then be used for analytical determination of these parameters.

Standard error population/standard error of prediction ratios are less good for residual moisture (2.5) and for total nitrogen (2.4) but are promising (Table 2 and Figure 2).

The results obtained on dry material are better than the ones obtained on fresh material due to a better samples homogeneity. Indeed, sample drying and grinding will destroy raw materials structure, especially for the samples took at the beginning of the process.

**Table 2 : NIRS calibration characteristics for the prediction of co-compost parameters on dry material**

| DRY MATTER | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | N | Mean | SEC | RSQ | SECV | 1-VR | SD | SD/SEC |
| DM | 252 | 94,9 | 0,78 | 0,84 | 0,96 | 0,77 | 1,98 | 2,54 |
| Organic Nitrogen | 154 | 2,08 | 0,15 | 0,93 | 0,18 | 0,89 | 0,56 | 3,74 |
| Ashes | 225 | 39,39 | 2,81 | 0,97 | 3,54 | 0,96 | 16,71 | 5,95 |
| Total Nitrogen | 324 | 2,28 | 0,23 | 0,83 | 0,25 | 0,80 | 0,55 | 2,39 |
| Total Carbon | 324 | 35,36 | 1,83 | 0,94 | 2,14 | 0,92 | 7,41 | 4,05 |
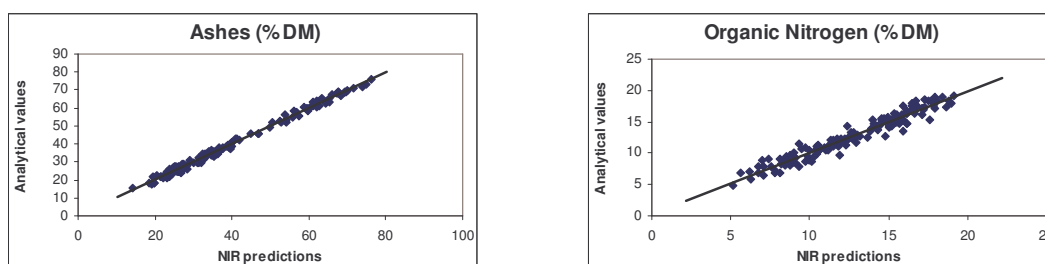


**Figure 2: Correlation between labs and NIR measurement, on dry material, for ashes and organic nitrogen (%DM)**

## Conclusions

NIR seems to be an appropriate technique for a rapid, low cost and "*in situ*" compost analysis. Its rapidity will allow to take suitable decision according the association of organic matter to perform in co-compost mixtures.

Due probably to a larger number and a better homogeneity, performances on dried and grinded materials are higher than results on fresh matter. Structure of raw material interferes with the fresh matter measurements, so a larger number of samples will be needed to improve fresh matter calibrations.

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