Data Article

A dataset of the chemical composition and near-infrared spectroscopy measurements of raw cattle, poultry and pig manure

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\textbf{A R T I C L E  I N F O}

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\textbf{A B S T R A C T}

Organic waste products (OWPs) from livestock have a high fertilizer value (N, P, K), but can also lead to environmental problems when applied in excessive quantities. Because their composition varies greatly, it is important to develop fast, reliable and inexpensive methods for determining their chemical contents. Near-infrared spectroscopy (NIRS) offers the possibility of rapid analysis of samples and requires little sample preparation, and previous studies have demonstrated that NIRS could be able to determine the most important compositional parameters of solid animal manure. The recent development of low-cost miniaturized spectrometers even enables manure-spreading equipment to be equipped with sensors to measure the composition in real time, and some applications are already being commercialized for the spreading of liquid OWPs. In-situ analysis of these very heterogeneous products (roughness, humidity) is a challenge for such applications, because spectral acquisition must be performed on raw samples with no preparation. To evaluate the accuracy with which NIRS estimates dry matter content, organic matter, total and ammonium nitrogen, phosphorus, potassium, calcium and magnesium contents, we created a

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large calibration database representative of raw solid animal manures encountered in Brittany. A total of 490 samples of solid OWPs from livestock farms were collected in the early spring from 270 farms in Brittany (western France), in 2 campaigns conducted in 2018 and 2019. The sampling was designed to capture the large diversity of animal species (mainly cattle, pigs and poultry), type of farming and storage modes. Compositional parameters were analyzed according to analysis methods certified by the French standards organization (AFNOR). Samples were scanned using a Q-interline AgriQuant B8 equipped with a patented spiral sampler, which aggregates the heterogeneity of the sample. NIRS measurements were made in triplicate. Because the dataset covers a wide range of variability in the composition of solid animal manure, these data are of great interest to chemometrics experts and agronomists in search of references on the fertilizing value of products.

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**Specifications Table**

| Subject | Chemistry, Analytical chemistry: Spectroscopy |
|---------|-----------------------------------------------|
| Specific subject area | Chemical composition of solid animal manure (cattle, pig and poultry manure; poultry droppings) |
| Type of data | Table, Figure |
| How data were acquired | Chemical analyses were made for dry matter content, organic matter, total and ammonium nitrogen, phosphorus, potassium, calcium and magnesium, and followed analysis methods certified by the French standards organization (AFNOR). Samples were scanned using a Q-interline AgriQuant B8 (Tølløse, Denmark) (852–2502 nm) equipped with a patented spiral sampler, which uses a borosilicate glass tube (60 × 250 mm) (i.e. “Agritube”) as a sample container. NIRS measurements were made in triplicate. |
| Data format | Raw and analyzed |
| Parameters for data collection | The objective was to provide samples of solid animal manure that represented the compositional diversity of these organic products (e.g. animal species, type of farming, storage mode (heap vs. windrow) and potential composting). |
| Description of data collection | A total of 490 samples of solid organic products from livestock farms were collected from 270 farms in Brittany, in 2 campaigns conducted in 2018 and 2019, in early spring. Each sample was a composite of at least 18 individual samples taken from 3 distinct zones at 3 different depths of the manure heap. The composite samples were transported to the laboratory in the 24 h following collection, and then stored in a refrigerated room at 4 °C, until they were processed for chemical analysis and spectral measurements. |
| Data source location | The data come from samples of solid animal manure collected from 270 farms in Brittany, France. The dataset provides GPS coordinates of the sampling points. |
| Data accessibility | This article provides the analyzed data. Raw data are deposited in a public repository. Repository name: Data INRAE Data identification number: 10.15454/E1JI8U Direct URL to data: https://doi.org/10.15454/E1JI8U |
Value of the Data

- Near-infrared spectroscopy (NIRS) has the potential to determine the composition of animal manure in real time during spreading, but doing so requires being able to acquire spectra from raw samples. The novelty and utility of this dataset is the reliable acquisition of spectral data from raw manure samples, with no preparation before measurement.
- Because the dataset contains a wide range of variability in the composition of solid animal manure, it is therefore of great interest to chemometricians and agronomists in search of references on the fertilizing value of organic waste products (OWPs).
- These data can be used by chemometricians to test methods and parameterize predictive models. They can also be merged with other databases to increase the accuracy of chemometric analyses.

1. Data Description

This article includes tables and figures that describe the chemical composition and NIR spectral data of the 490 solid animal OWPs collected in 2018 and 2019, including mean values of chemical composition parameters by type of OWP (Table 1); boxplots of 6 chemical composition parameters, representing the variability in composition by type of OWP (Fig. 1); projection of the OWPs on the plane defined by the first two components of a principal component analysis of the composition parameters (Fig. 2); a correlation matrix between each pair of composition parameters (Table 2); relations between dry matter, organic matter and total nitrogen content (Fig. 3).

The dataset is composed of a Microsoft Excel® and a csv files that contain raw data (Table 3). It includes the location of the sampling points, manure type, chemical analysis results and the NIR spectra of the 490 OWPs sampled. The dataset is available via the Data INRAE portal.

| Table 1 |
|----------------------------------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|
| Product                           | Sample size | Dry matter | OM        | AN        | Total N   | P₂O₅      | K₂O       | CaO       | MgO       |
| Cattle manure                     | 276        | 19.9       | 153.2     | 0.7       | 5.0       | 2.8       | 1.8       | 4.2       | 1.8       |
| Pig manure                        | 18         | 21.3       | 172.3     | 1.2       | 5.7       | 5.6       | 2.1       | 22.9      | 5.6       |
| Poultry manure                    | 144        | 42.2       | 310.9     | 4.8       | 16.6      | 15.1      | 5.6       | 22.9      | 5.6       |
| Poultry droppings                 | 27         | 54.6       | 350.2     | 6.1       | 21.1      | 22.6      | 7.7       | 62.0      | 7.7       |
| Compost                           | 16         | 24.6       | 183.5     | 2.2       | 7.1       | 10.0      | 5.2       | 10.5      | 5.2       |
| Other                             | 9          | 40.4       | 320.2     | 3.7       | 14.9      | 10.6      | 4.1       | 13.4      | 4.1       |

| Table 2 |
|----------------------------------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|
| Correlations between each pair of composition parameters (n = 490) (DM: dry matter content; OM: organic matter content; AN: ammonium nitrogen content; Total N: total nitrogen content; P₂O₅: phosphorus oxide content; K₂O: potassium oxide content; CaO: calcium oxide content; MgO: magnesium oxide content). |
| Parameter | DM | OM | AN | Total N | P₂O₅ | K₂O | CaO | MgO |
|-----------|----|----|----|---------|------|-----|-----|-----|
| DM        | 1.00 |     |    |         |      |     |     |     |
| OM        | 0.93 | 1.00 |    |         |      |     |     |     |
| AN        | 0.72 | 0.69 | 1.00 |         |      |     |     |     |
| Total N   | 0.88 | 0.87 | 0.86 | 1.00    |      |     |     |     |
| P₂O₅      | 0.79 | 0.69 | 0.74 | 0.79    | 1.00 |     |     |     |
| K₂O       | 0.83 | 0.80 | 0.69 | 0.83    | 0.74 | 1.00 |     |     |
| CaO       | 0.67 | 0.47 | 0.54 | 0.59    | 0.81 | 0.55 | 1.00 |     |
| MgO       | 0.80 | 0.70 | 0.72 | 0.80    | 0.90 | 0.73 | 0.67 | 1.00 |
2. Experimental Design, Materials and Methods

2.1. Sample collection and preparation

A total of 490 samples of solid OWPs from livestock farms were collected from 270 farms in Brittany, in western France, in 2 campaigns conducted in 2018 and 2019, in the early spring. Samples were collected at manure storage sites according to a protocol developed by the CAPINOV laboratory to obtain a representative sample of the manure heap. Each sample was...
Fig. 2. Projection of the 490 organic waste products on the plane defined by the first two components of a principal component analysis of the composition parameters. A concentration ellipse covers each type of organic waste product.

Fig. 3. Relation between (a) organic matter and dry matter content and (b) organic matter and total nitrogen of organic waste products (blue lines indicate linear regressions) (w.w.: wet weight).

a composite of at least 18 individual samples taken from 3 distinct zones at 3 different depths of the manure pile. The composite samples were stored in a cooler, transported to the laboratory within 24 h of collection and then stored in a refrigerated room at 4 °C, until they were processed for chemical analysis and spectral measurements.

The objective of the study was to provide samples of solid animal manure that represented the compositional diversity of these OWPs by considering the following criteria: (i) animal species (i.e. cattle, pigs and poultry (layers, broilers and turkeys) bred mainly in Brittany), (ii)
Table 3
Contents of the dataset (w.w.: wet weight).

| File name       | Variable name | Content                                      |
|-----------------|---------------|----------------------------------------------|
| chemical_analysis.xls | Id_sample | Sample identification number |
| LAT_WGS84       | Latitude of the sampling point                  |
| LONG_WGS84      | Longitude of the sampling point                  |
| Manure_type     | Manure type                                         |
| DM              | Dry matter content (%)                             |
| OM              | Organic matter content (g kg⁻¹ w.w.)              |
| AN              | Ammonium nitrogen content (g N kg⁻¹ w.w.)         |
| Total_N         | Total nitrogen content (g N kg⁻¹ w.w.)            |
| P2O5            | Total phosphorus oxide content (g P₂O₅ kg⁻¹ w.w.) |
| K2O             | Total potassium oxide content (g K₂O kg⁻¹ w.w.)   |
| CaO             | Calcium oxide content (g CaO kg⁻¹ w.w.)           |
| MgO             | Magnesium oxide content (g MgO kg⁻¹ w.w.)         |
| spectra.csv     | The first column contains the sample identification number, and the other columns contain the absorbance data recorded from 802–2502 nm |

The type of farming for poultry manure (e.g. poultry production on litter, which produces poultry manure vs. egg production in buildings without litter, which produces poultry droppings), (iii) type of farming for cattle manure (i.e. deep litter, strawed slope, stalls) and (iv) storage mode (i.e. heap vs. windrow) and potential composting.

2.2. Chemical analyses

The CAPINOV laboratory performed the chemical analyses. Dry matter content was measured according to the NF EN 13,040 standard by drying a subsample at a temperature of 103 ± 2 °C. Organic matter content was determined by calcination at 450 ± 25 °C (standard NF EN 13039 [1]). Total N and ammonium N were analyzed in raw subsamples to avoid gaseous N losses during drying; subsamples were obtained by homogenizing and fragmenting samples into particles smaller than 5 mm. Total N was measured by the Kjeldahl method according to NF EN 13654-1 [2], and ammonium N was measured by steam distillation, using a method (similar to the Kjeldahl method) that volatilizes ammonium by adding a strong base to the distillation mash. Ammonia volatilized during distillation is trapped in a known quantity of boric acid, and the ammonium content is measured by titration with a 0.1 N HCl solution. Total N analyses were performed in duplicate.

Contents of total phosphorus, potassium, calcium and magnesium were determined in a subsample dried at 75 ± 5 °C, according to sample preparation standard NF EN 13040 [3]. The analyses were performed using inductively coupled plasma (Element XR Thermo Scientific) after extraction with aqua regia, according to NF EN 13650 [4].

2.3. Spectroscopic analysis

Samples were scanned using a Q-interline AgriQuant B8 (Tølløse, Denmark) equipped with a patented spiral sampler, which uses a borosilicate glass tube (60 × 250 mm) (i.e. “Agritube”) as a sample container. The QJC1021 Reference tube is a similar glass tube filled with reference material. During analysis, the tube turned at a linear speed that was influenced by the weight of the tube. The maximum linear speed of the tube recorded was 3 mm·s⁻¹. In this study, with a sample weight of 500–1000 g, the mean linear speed was ca. 2 mm·s⁻¹, which created a scanned area equivalent to 300 cm². The acquisition device considers the heterogeneity of the sample. Three tubes of each sample were scanned. Each replicated measurement was the mean of 180 scans, and absorbance (Abs) was recorded from 1990 to 15,506 cm⁻¹, with
a step of 8 cm$^{-1}$, using the following equation: $\text{Abs} = \log(1/\text{reflectance})$. The spectral regions from 11,737 to 15,528 cm$^{-1}$ and 1990–4000 cm$^{-1}$ were excluded since they were noisy. The final spectrum was the mean of the three replicates after the inter- replicate variability was verified. The wavenumber (4000–11,737 cm$^{-1}$) was converted to wavelength (852–2502 nm).

2.4. Spectral pre-treatment

To reduce the influence of particle size and optical path difference on reflectance spectra [5], five pre-treatments were tested: first- and second-derivative transformation over a 21 nm gap with second-order Savitzky-Golay smoothing (SG1 and SG2, respectively), standard normal variate, multiplicative scatter correction and detrending. For each chemical component, the pre-treatment that yielded the lowest root mean square error of cross-validation was used in subsequent calculations.

Ethics Statement

Not applicable

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that have, or could be perceived to have, influenced the work reported in this article.

CRediT Author Statement

Thierry Morvan: Conceptualization, Methodology, Writing – original draft, Writing – review & editing, Visualization, Supervision, Data curation, Project administration; Fabien Gogé: Conceptualization, Methodology, Validation, Formal analysis, Investigation, Data curation, Writing – original draft, Visualization; Thierry Oboyet: Methodology, Validation, Formal analysis; Odile Carel: Methodology, Validation, Formal analysis; Youssef Fouad: Conceptualization, Methodology, Writing – original draft, Supervision, Data curation, Project administration.

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