

# Prediction of organic compositions in manure by near infrared reflectance spectroscopy

**Masahiro Amari, Yasuyuki Fukumoto and Ryozo Takada**

*National Institute of Livestock and Grassland Research Science, National Agricultural Research Organisation, Tsukuba Norindanchi, POB 5, Ibaraki 305-0901, Japan*

## Introduction

Many organic materials are included in the excreta of livestock. These materials are important resources for the production of organic manure and for improving soil quality which are still not being used effectively. Therefore, it is suggested that one standard factor for quality evaluation of manure made from excreta of livestock should be established. The subject of this study is to develop a rapid and accurate analytical method to analyse the organic composition of manure made from excreta of livestock and to establish the quality evaluation method based on some compositions measured by near infrared (NIR) reflectance spectroscopy.

## Materials and methods

### Samples

Samples were collected from each batch of manure every week from 1<sup>st</sup> to 7<sup>th</sup> week of the processing period. A total of 15 samples were used, including the source of the samples. The manure was made from 560 kg of swine feces and 60 kg of sawdust, with a moisture content of 65% RH. The manure was made in the two ways; one by using transparent cover sheets to expose it to sunlight and the other by using black cover sheets to shield it from the light.

### Chemical analysis

The 15 samples of manure were analysed for moisture, nitrogen and organic matter (OM) content by an approximate analysis method.<sup>1</sup> Fibrous fractions were also measured using the enzymatic method<sup>2,3</sup> and the detergent method.<sup>4,5</sup> The enzymatic method was used for determining organic cell wall (OCW) and fibres with low digestibility (Organic b fraction: Ob) content, while the detergent method was used for the determination of the neutral detergent fibre (NDF), acid detergent fibre (ADF) and acid detergent lignin (ADL) content. Biological oxygenation demand (BOD) was analysed by the core-metre method.<sup>6</sup>

### NIR reflectance analysis

Spectra of samples from 400 nm to 2500 nm were measured with an NIR instrument, Model 6500 (NIRSystems). Calibration equations were developed using eight manure samples and the rest of the samples were used to test the developed calibration equations.

**Table 1. Minimum, maximum and ranges of chemical compositions of samples.**

	Min.–Max.	Ranges
NDF (DM%)	44.3–55.2	10.9
ADF (DM%)	32.7–41.5	8.8
ADL (DM%)	12.9–21.2	8.3
OCW (DM%)	54.5–65.7	11.2
Ob (DM%)	53.2–62.9	9.7
BOD ( $\times 10^4$ mg kg <sup>-1</sup> )	0.0–19.9	19.0

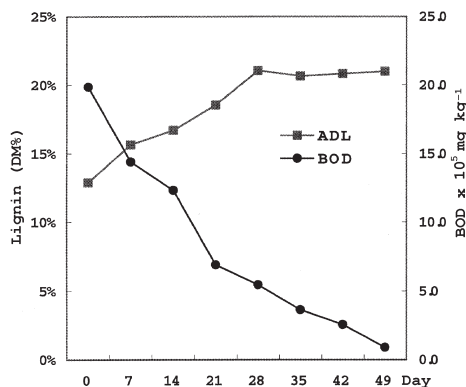
**Table 2. Relationship between BOD value and chemical compositions value in manure.**

	<i>r</i>	Se
NDF	–0.887	3.70
ADF	–0.909	2.53
ADL	–0.988	1.25
OCW	–0.955	2.37
Ob	–0.789	4.92

## Results and discussion

### Chemical composition

Table 1 shows the chemical composition of manure. The values of the major components in manure were 54.5–65.7% for OCW, 12.9–21.2% for ADL and  $0\text{--}19.9 \times 10^{-5}$  mg kg<sup>-1</sup> for BOD, respectively. During the four weeks of processing, the amount of ADF and ADL increased, while the amount of NDF and OCW decreased. After this period each composition reached a constant value. The BOD rapidly decreased until the third week and then it decreased slowly. The relationship between the BOD and each composition of manure is shown in Table 2 and Figure 1. The correlation coefficients (*r*) between BOD and ADL, ADF or OCW content were negatively high and ranged from –0.909 to –0.988, i.e. manure having a large amount of ADL tended to show a lower BOD. In other words, the BOD decreased as the ADL increased according to the progression of the decomposition of manure by fermentation. The remarkably high negative correlation between BOD and ADL can be attributed to the

**Figure 1. Development of the amount of ADL and BOD during the making of manure.**

**Table 3. Results of calibration and validation for determining chemical composition in manure.**

	Wavelength (nm)	Calibration		Validation		
		<i>r</i>	<i>SEC</i>	<i>r</i>	<i>SEP</i>	<i>RPD</i>
NDF	1570	0.971	0.96	0.955	1.03	4.12
ADF	1766, 484, 1546	0.983	0.61	0.979	0.60	4.90
ADL	1770	0.996	0.29	0.994	0.39	7.59
OCW	492	0.976	1.07	0.783	2.62	1.46
Ob	2118, 512	0.943	1.07	0.959	0.97	3.35
Nitrogen	2386, 596, 1558	0.998	0.08	0.914	0.28	2.71
ASH	566	0.988	0.91	0.866	3.05	1.57
BOD	468	0.997	0.58	0.928	2.44	3.04

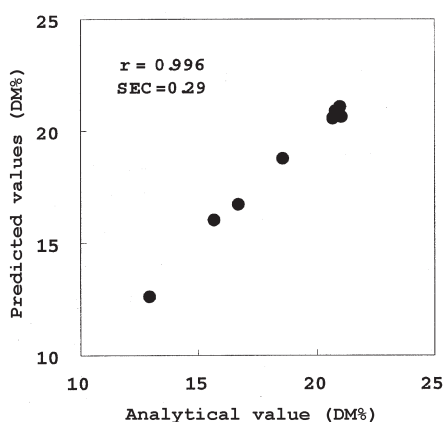
decomposed quantity of hard materials such as lignin. The same phenomenon may account for the high correlation between the BOD and OCW as well as ADF.

These facts suggest that the ADL content in manure is one of the factors which shows the degree of fermentation and, therefore, the ADL content can be used to estimate the BOD of manure.

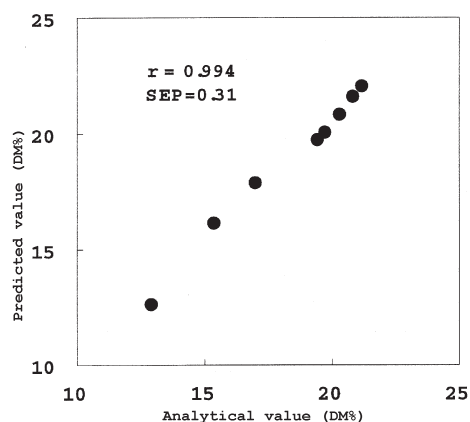
### NIR reflectance analysis

#### Calibration

Table 3 shows calibration results for determining various components in manure, i.e. the correlation coefficient (*r*) and standard error of calibration (*SEC*). The *r* and *SEC* values were 0.996 and 0.29



**Figure 2.** Relationship between the laboratory analysis and predicted content using an multiple reference data employing one wavelength.



**Figure 3.** Relationship between the laboratory analysis and predicted content using an NIR calibration of ADL.

for ADL (Figure 2), 0.971 and 0.96 for NDF, 0.983 and 0.61 for ADF, 0.976 and 1.07 for OCW, 0.943 and 1.07 for Ob, 0.997 and 0.58 for BOD, respectively.

#### **Validation of calibration**

Table 3 and Figure 3 show validation results for determining various components in manure, i.e. the correlation coefficient, standard error of prediction (*SEP*) and the ratio of standard deviation of the reference data in the prediction sample set to *SEP* (*RPD*). The *r*, *SEP* and *RPD* values were 0.99, 0.39 and 7.59 for ADL, 0.96, 1.03 and 4.12 for NDF, 0.98, 0.60 and 4.90 for ADF, 0.96, 0.97 and 3.35 for Ob and 0.93, 2.44 and 3.04 for BOD, respectively. The results indicated that organic compositions in manure samples are highly predictable using NIR.

#### **References**

1. H. Morimoto, *Experimental method of animal nutrition*, (in Japanese). Yokendo, Tokyo, Japan, pp. 280–304 (1971).
2. A. Abe, S. Horii and K. Kameoka, *J. Anim. Sci.* **48**, 1483 (1997).
3. A. Abe and S. Horii, *J. Jpn Grassl. Sci.* (in Japanese with English summary) **25**, 70 (1979).
4. P.J. Van Soest, *J. Assoc. Off. Anal. Chem.* **46**, 825 (1963).
5. P.J. Van Soest and R.H. Wine, *J. Assoc. Off. Anal. Chem.* **50**, 50 (1967).
6. Association for Utilization of Sewage Sludge, *J. Jpn Sewage Works Assoc.* (in Japanese) Tokyo, 146 (1996).