



## Use of near infrared spectroscopy in online-monitoring of feeding substrate quality in anaerobic digestion

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### ABSTRACT

In order to keep the anaerobic process stably and uniformly producing biogas it needs to be supplied with either an even amount of substrate of stable quality or varying amounts according to variations in quality. Feeding amounts are usually adjusted manually as a reaction to changing rates of biogas production. Continuous information about the actual substrate quality is not available and feedstuff analyses are costly. Aim of this study was to assess the feasibility of near infrared spectroscopic (NIRS) online monitoring of substrate quality in order to find ways towards more exact control of biogas plant feeding. A NIRS sensor system was designed, constructed and calibrated for continuous monitoring of (RMSECV in brackets) dry matter (DM) (0.75 %fresh matter (FM)), volatile solids (0.74 %FM), crude fat (0.09 %FM), crude protein (0.22 %FM), crude fiber (1.50 %DM) and nitrogen-free extracts (0.93 %FM) of maize silage.

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### 1. Introduction

Biogas production by anaerobic digestion (AD) of biomass is of growing importance in response to environmental and future energy supply concerns. In Germany this sector has been particularly promoted by legislative changes in the years 2000 and especially 2004. By the end of the year 2010, about 5700 biogas plants have been established.

Modern biogas plants ferment liquid and/or solid substrates, mainly from agricultural sources. A typical liquid substrate is manure while the most abundant solid substrates are specifically grown energy crops, such as maize and grass, which are being stored as silages. More and more sugar beets are also being considered a future energy crop. Monofermentation based solely on maize silage has become very common. Furthermore organic wastes from different industries are used.

Agricultural substrates are relatively complex in composition and – due to locally and temporarily diverging conditions of growth and storage – varying in quality. Contrary to these variations the biogas process itself calls for constant conditions regarding dosage and composition of the substrates. This can only be achieved by continuous and representative supervision of the substrates applying adequate sensors. To date control of substrate dosage is mostly accomplished by regular automatic feeding based on gravimetric or volumetric assessment of substrate amounts,

without substrate quality control. Changes in substrate quality, leading to altered biogas production, can therefore not be controlled in-time and have to be buffered by the mostly flexible gas storage system until the plant operator reacts. Beyond this buffer they lead to over- or underproduction of biogas, resulting in monetary losses. To date feeding strategies are adapted *following* changes in gas quality and quantity instead of reacting *in advance* to changes and according to actual substrate quality.

Biomass composition for use as feedstuff for feedstock as well as for biogas plants is usually described by Weender analysis (Jeroch et al., 1999), differentiating between dry matter (DM), crude ash, volatile solids (VS), crude protein (XP), crude lipids (XL), crude fiber (XF) and nitrogen-free extracts (NFE). Additional information about the less digestible or indigestible fiber and lignin fractions can be provided by Van Soest analysis (Van Soest, 1966).

Hence to date there is no tool for online supervision of substrate this study aimed at applying near infrared spectroscopy (NIRS) to this end.

NIRS is being used in a wide range of applications in agriculture and has already been proposed as a process analytical tool for the supervision of the anaerobic digestion process (Hansson et al., 2002; Jacobi et al., 2009; Lomborg et al., 2009; Nordberg et al., 2000) as well as for determination of manure composition (Dolud et al., 2005; Saey, 2006; Sorensen et al., 2007; Zimmermann, 2009). Concerning the application for the monitoring of feedstuff quality it has been a standard analytical tool for a long period. Initially and most commonly until today it is applied only to dried

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samples, but at least since [Abrams \(1988\)](#) tried estimation of several parameters of fresh grass and legume silages it has been used on undried samples in a couple of laboratory studies as well. The results from studies applying NIRS on fresh matter samples aiming at some of the Weender analysis parameters relevant for the biogasification process are cumulated in [Table 1](#).

Measuring fresh, untreated silage samples containing plant particles in the range of mm–cm calls for special care concerning the representativeness of the spectral measurements. Commonly various spectra of different fractions of the same sample are averaged in order to receive one representative spectrum. [Reeves et al. \(1989\)](#), found that calibrations of ground silages lead to superior results compared with unground samples. Similar results are presented by [Baker et al. \(1994\)](#) and [Gordon et al. \(1998\)](#) using undried samples and by [Lovett et al. \(2005\)](#) with dried material.

Influences of sample, ambient and spectrometer temperature on NIR spectra are an issue well known to NIRS users and are well described in literature ([Williams, 2001b](#)). There are different approaches to avoid temperature effects, most of which depend on knowledge of sample temperature. PLS-regression is supposed to be able to overcome temperature effects. To this extend samples from the complete range of temperatures to be expected for an application need to be included into the calibration data set ([Hageman et al., 2005](#); [Williams, 2001b](#)). Since the aim of working in-process in real-life applications calls for measuring devices that do not need to interfere with the process, special treatment of the substances to be analyzed is to be avoided. This excludes comminution as well as special temperature control. Therefore this study aimed at evaluating the possibilities of silage characterization using NIRS, avoiding special sample treatment or temperature control.

## 2. Methods

### 2.1. Linnau biogas plant

The Linnau biogas plant (Linnau, Schleswig–Holstein, Germany) is operated in the thermophilic temperature range (50–55 °C) and consists of three process stages: primary and secondary heated fermenter and an unheated repository for slurry storage. The plant is

fed only with maize silage. Cumulated input (and loading rates) varied during the observed period from 25–50 t FM d<sup>-1</sup> (2.0–4.0 kg VS m<sup>-3</sup> d<sup>-1</sup>) with an average of 37 t FM d<sup>-1</sup> (3.0 kg VS m<sup>-3</sup> d<sup>-1</sup> calculated for the first two stages).

### 2.2. Near infrared spectroscopy

A measuring-head and a matching flange were designed and constructed. The flange was designed to be screwed onto any type of conveyor or pipe where direct contact with the material to be observed is given and to withstand mechanical stress. It held a sapphire window and separated the sample from the measuring head. The aluminum structure of the measuring head was designed to hold the light source, the light intake, the optic fiber cable uptake and the power supply unit ([Fig. 1, right](#)). Light source and light uptake each were equipped with a lens system (PSS-H-117 & PSS-L-105, Polytec, Waldbronn, Germany) to focus onto the measuring spot behind the sapphire window. The light source was installed at an angle of 90° to the sapphire window and the light uptake was installed at 45° to both window and light source. The light source included a tungsten-halogen-bulb and illuminated a circle with a diameter of 1.8 cm and a surface of 2.54 cm<sup>2</sup> of the substrate. It was installed into the sidewall of a hopper at the feeding bunker in a position where silage passed prior to being pressed into the slurry stream of a passing pipe by means of an auger ([Fig. 1, left](#)). The position was above the auger, where at all times during feeding silage was present and moving slowly (appr. 1 cm/s). Maize silage passed the site of measurement during eighteen feeding events per day, with maximum intervals of 1.5 h between feedings. Feeding events lasted 30 min on average. During feeding the silage passed the measuring head with a speed of approximately 1 cm/s. The silage moved directly alongside the sapphire window with no space in between. Via a fiber optic cable (FC-IR-400, Mikropack, Ostfildern, Germany) the measuring head was connected to an NIR-spectrometer (Polytec 1720, Polytec, Waldbronn, Germany) covering the 850–1650 nm region with a 256-diode indium gallium arsenide detector. An optical switch (mol 1 × 4 S400 NIR, Leoni Fiber Optics GmbH, Neuhaus-Schiernitz, Germany) was installed between measuring head and spectrometer to

**Table 1**  
Literature survey: Statistical parameters of reviewed NIR-calibrations of undried silages.

Para-meter	Authors	Range(nm)	Regr. type	Pre-treatment	Derivative	R <sup>2</sup> (%)	VE	Slope (%)	n <sup>a</sup>	PC	RPD	Unit	Min	Max	Mean	SD	Material
<b>DM</b>	<a href="#">Abrams (1988)</a>			smooth	1st	98						%FM			39.4	13.7	H
	<a href="#">Reeves et al. (1989)</a>	1100–2500	MRP			99	1.51	98	40(20)	8.7	%FM	24.1	76.0	51.8	13.2	A	
	<a href="#">Reeves et al. (1989)</a>	1100–2500	MRP			96	1.32	90	40(19)	5.6	%FM	19.6	57.0	37.3	7.4	M	
	<a href="#">Sorensen (2004)</a>	400–2500	PLS	SNVD	2nd	99	0.89		43	6	%FM	22.0	79.0	40.0	14.0	G	
	<a href="#">Sorensen (2004)</a>	400–2500	PLS	SNVD	2nd	93	0.97		37	6	%FM	28.0	44.0	35.0	3.3	M	
	<a href="#">Park et al. (2005)</a>	400–2500	MPLS	SNVD	1st	83(70)	1.34		86(27)	2.5	%FM	21.8	44.4	28.7	3.8	M	
	<a href="#">Cozzolino et al. (2006)</a>	400–2500	MPLS	SNVD	2nd	85	2.74		90	6	%FM	22.8	52.8	34.5	6.5	M	
	<a href="#">Liu and Han (2006)</a>	1100–2500	PLS	SC	1st	90	1.72	99	94(31)	8	%FM	13.4	43.5	25.1	5.9	M	
<a href="#">Gibaud (2007)</a>	960–1690	PLS	MSC		98	1.05	96	158	13	6.1	%FM			36.5	8.6	G	
<b>XF</b>	<a href="#">Gibaud (2007)</a>	960–1690	MPLS	none	2nd	70	1.46	86	169	12	2.2	%VS			27.1	3.2	G
<b>XP</b>	<a href="#">Reeves et al. (1989)</a>	1100–2500	MRP			88	1.59	117	40(20)	2.5	%DM	5.4	26.1	19.9	1.4	A	
	<a href="#">Reeves et al. (1989)</a>	1100–2500	MRP			83	0.58	114	40(19)	1.1	%DM	6.5	12.4	8.6	4.0	M	
	<a href="#">Sinnaeve et al. (1994)</a>	400–2500	MPLS	SNVD	2nd	97	0.49		56	6	%DM	7.3	16.5	11.0	2.9	R, T	
	<a href="#">Cozzolino et al. (2006)</a>	400–2500	MPLS	SNVD	2nd	91	0.65		90	7	%DM	3.8	24.3	7.7	32	M	
	<a href="#">Liu and Han (2006)</a>	1100–2500	PLS	SC	1st	82	0.74	78	94(31)	4	2.4	%DM	4.8	11.6	8.2	1.8	M
	<a href="#">Gibaud (2007)</a>	960–1690	MPLS	SNVD	2nd	49	1.59	100	159	6	1.8	%VS			16.8	2.9	G

*Italic* and normal letters within one column mark differences within the calculation of a parameter. Values were rounded and units were modified if appropriate, missing values were calculated by the author if possible, otherwise left blank.

<sup>a</sup> Test set in brackets; A, alfalfa; CV, variation coefficient; DM, dry matter; FM, fresh matter; G, grass; H, various hay-silages; M, maize; MPLS, modified PLS; MRP, multiterm regression procedures; n, number of samples; PLS, partial least squares regression; R, rye-grass; R<sup>2</sup>, coefficient of determination (*calibration/validation*); RMSECV, root mean square error of cross validation; RMSEP, root mean square error of prediction; RPD, ratio of standard error of performance to standard deviation (SD)/SECV or SD/SEP or SD/RMSECV or SD/RMSEP; SC, unspecified scatter correction; SD, standard deviation; SECV, standard error of cross validation; VE, validation errors (RMSECV or RMSEP or SECV or SEP); SNVD, standard normal variate with detrend; T, Trifolium; VS, volatile solids; XF, crude fiber; XP, crude protein.

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