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Continuous Analysis of Liquid Pig Manure with Near-infrared Spectroscopy (NIRS)

Near-infrared spectroscopy (NIRS) is a quick and timely chemometrical method of slurry analysis, which requires no extensive sampling. Data immediately available on specific contents and features is advantageous, e.g. for utilising feed protein, for optimising feeding, for process control in biogas technology, as well as for monitoring and balancing substance flows. The requirements on the measuring personnel are low. Measurements can be carried out without chemical additives and hence high costs for personnel, materials and waste disposal can be saved.

Liquid manure is a mixture of faeces, urine, feed residues and water. Composition and quantity are dependent on animal species, animal age and feeding as well as on farm specific factors. Liquid manure has the characteristics of a suspension, where by spectroscopic methods the liquids can be measured in the mode of transmission and the solids in the mode of reflection [1]. The measuring method presented here combines both by using the mixed transmission or transflection (transmission with partial reflection). Specific demands to the measuring cell attached to the spectrometer result from that.

At the Institute for Agricultural Process Engineering/University Kiel a flow-through measuring cell has been developed and tested in a feasibility study with liquid pig manure. One special feature of this test cell is the small path length of 3 mm, where the sample to be tested is presented to the spectrometer. Further the cell is equipped with a ceramic disc, attached opposite to the spectrometer sensing head, which reflects 100% irradiated energy. *Figure 1* shows the configuration of the measuring cell. The liquid manure sample flowing through the measuring cell is irradiated with near infrared light. A part of the NIR-radiation hits the particles of the sample, there it is absorbed partially and partially diffuse reflected, which is recorded by the detectors of the spectrometer. That is the way to analyse the solids in liquid manure. Another part of the radiation passes the particles of the liquid manure and is partially absorbed in the liquid phase. That part of radiation that still passes the sample, hits the ceramic disc and there it is reflected completely, passes the sample a second time on the way back and is recorded then analogical to the diffuse reflection of the particles. Since the measurement of transmission comprises limitations in the path length, some restrictions to the thickness of the sample presented to the spectrometer have to be considered [2]. However, this is still the way to detect the liquid and solid phase of the liquid manure in one measurement by transflection.

NIRS - flow throw prototype

Figure 2 shows the complete measuring device for the NIR prediction of liquid manure, consisting of diode array spectrometer (Zeiss CORONA 45 NIR), measuring cell, pump, sample bottle holder (1 l samples), valves, power supply and computer. The impeller pump conveys the liquid manure with constant velocity in the system cycle and guarantees a homogeneous and representa-

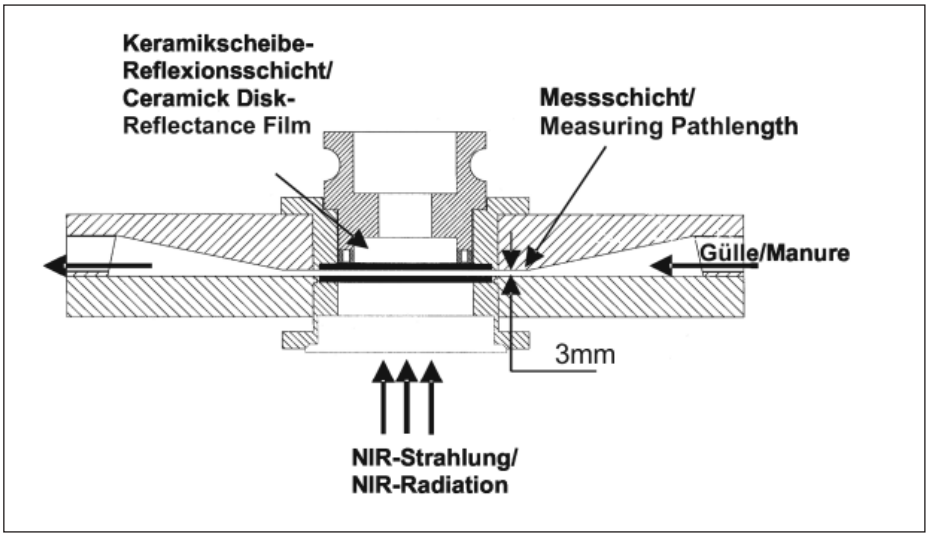

Fig. 1: Schematic presentation of measuring cell

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Keywords

Online-liquid-manure-analysis, near-Infrared-spectroscopy (NIRS), transflectance, through-flow measuring method

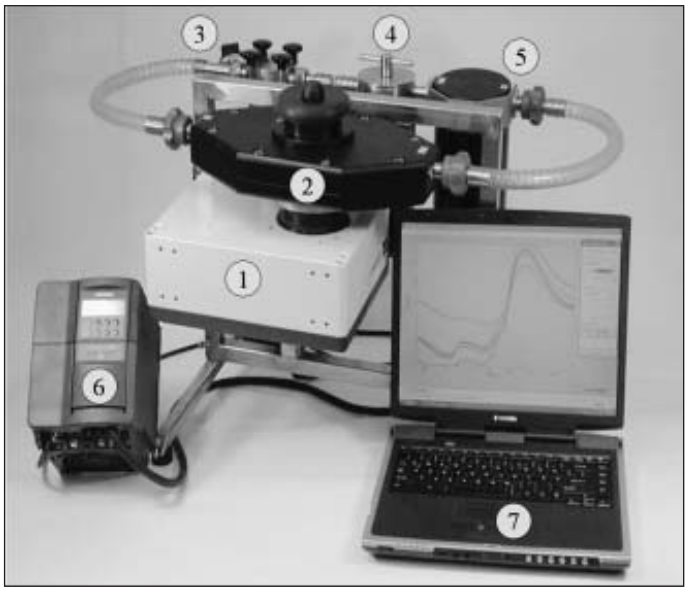

Fig. 2: NIR measuring system: NIR-Spectrometer ZEISS-CORONA 45 (1), measuring cell (2), electric pump (3), multi way valve (4), sample compartment unit (1l sample) (5), frequency inverter (6) and PC (7)

tive flow through the test cell. The spectrometer scans the liquid manure flowing through the measuring cell and delivers spectral data to the computer. By means of water and compressed air, delivered from the valves, the system can be cleaned and dried between the measurements of different samples.

A first calibration was carried out on 25 liquid manure samples (fattening pigs, piglets and sows), depicted in *figure 3*. In contrast to static measurements, the flow through measurement realised here, allows the determination of larger sample volumes. This increases the representativeness of the measurement. By permanent pumping the sample in the system cycle, the homogeneity of the sample is assured at any time of the measurement. The integration time of a single measurement is below 1s, so that during a single measurement cycle several hundred spectral scans can be recorded. These are averaged and allow the quantitative prediction of the interesting analytes. Precondition for this prediction is to carry out a calibration process prior to the measurement. In this calibration step reference samples have to be analysed with classical chemical methods for the substance classes and analytes to be examined.

The calibration of the spectral data with reference data from the laboratory is performed with multivariate statistical methods by detecting variations in the principal components by means of PCA (principal component analysis) and PLS (partial least squares regression). The quality of the calibration model has been estimated with cross validation. The coefficient of determination (R^2) for total nitrogen, ammonium nitrogen, dry matter and pH was 0.96, 0.98, 0.94 and 0.95, respectively (*Fig. 3*).

Currently the NIRS flow-throw measuring system is used in a pig fattening trial for the examination of liquid manure. The accuracy of prediction in the fattening trial is even better than in the preliminary tests with different types of liquid manure samples presented here. This is due to more uniform liquid manure samples in the fattening trial compared to the first feasibility test with different manure types from different stalls with different animal feeds.

Conclusion

It was the intention of this study to use the NIR-spectroscopy for quantitative determination of liquid manure constituents. It could be shown, that the developed NIR-method can be applied to analyse liquid pig manure. In comparison to conventional laboratory analyses the advantages arise particularly from the quickness, the high measurement density and the immediate availability of the results. The entire measuring device for the flow through measurement of liquid manure with NIRS has been proven as reliable and robust. Basically the system provides the possibility for online-applications, using it in a bypass mode to monitor and control processes.

Literature

Books are identified by •

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[2] *Malley, D. F., L.Yesmin and R.G. Eilers*: Rapid Analysis of Hog Manure and Manure-amended Soils Using Near-infrared Spectroscopy. Soil Sci. Soc. J. 66 (2002), pp.1677-1686

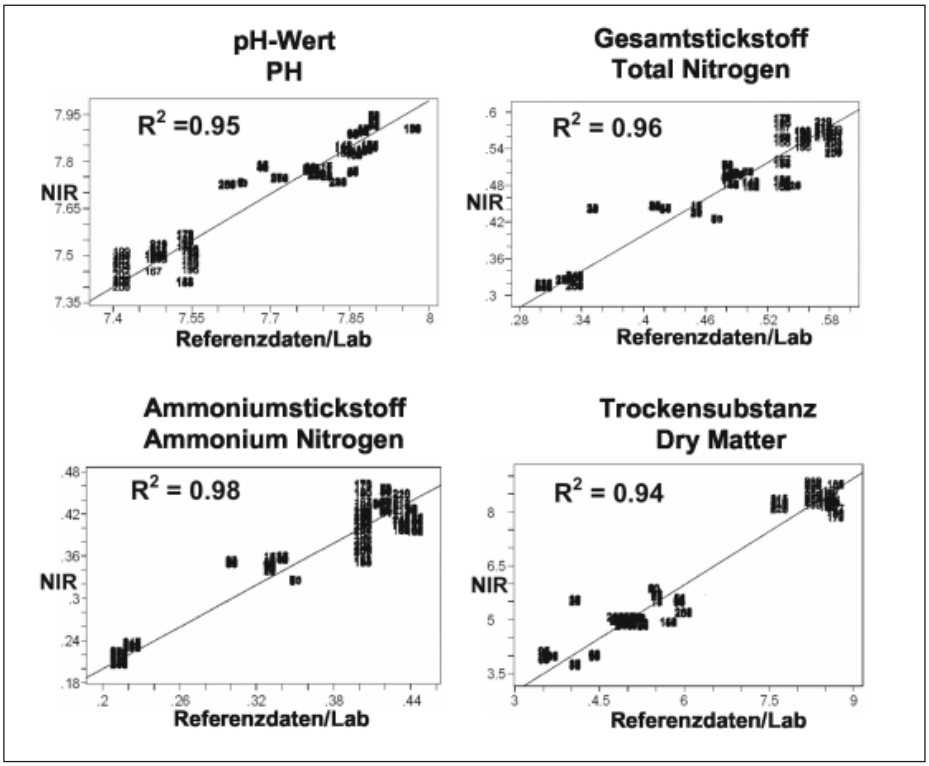

Fig. 3: Linear regression for pH, total nitrogen, ammonium nitrogen and dry matter (n=25)