A MINIATURIZED INSTRUMENT TO MEASURE SLOW BIOGAS FLOW RATES

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ABSTRACT

A miniaturized volumetric gasometer, which can be used for the measurement of small biogas flow rates has been. It was called the MilligasCounter® (see www.milligascounter.de). The principle of the gasometer is a wet tipmeter. It measures under non-pressurized conditions (4 – 7 mbar) very slow gas rates in discrete steps of 0.950 to 1.050 ml. It is also suitable for continuously driven laboratory reactors. The linear range of measurements ends at appr. 1.200 ml and is not dependent on the time period. As the instrument records discrete steps the time period could be an hour, a week or even a month. The accuracy depends only on the tightness of tubing material and connectors. The influence by temperature and water vapour can be corrected by a mathematical equation. A great advantage is that the measurements are not dependent on atmospheric pressure changes as it is typical for gasometers based on simple water replacement in connected vessels. In contrast to methods based on pressure transducers no overpressure could be built up. Possibly such an overpressure could interfere with the sensitive anaerobic microbiology.

KEYWORDS

Biogas flow measurement, MilligasCounter®, biodegradation, cofermentation

INTRODUCTION

Anaerobic degradation tests are in common use either as test for the total amount of gas production (biochemical methane potential, BMP) or as anaerobic toxicity assays (ATA) utilizing monomeric substrates and recording the initial rate of gas production (Owen et al., 1979, Scherer 1999, Jörg 2001). However, a nearly pressure free gas flowmeter for small flow rates of humid gases with a varying compostion of $CH_4 + CO_2$ was not available at present (Scherer 1990). The developed MilligasCounter® can also be expanded to online measurements.

MATERIALS AND METHODS

Anaerobic degradation tests were performed to demonstrate the practicability of the MilligasCounter®: 500 ml medium was supplemented adding the following salts as separate stock solutions by stirring. 2.6 mM KH_2PO_4, 3.1 mM Na_2HPO_4 (buffer), 5.0 mM NH_4Cl, 2.0 mM $CaCl_2$ x 2 H_2O, 2.0 mM $MgCl_2$ x 6 H_2O (minerals), 15.0 µM HCl, 0.5 µM $ZnCl_2$, 2.0 µM $MnCl_2$ x 4 H_2O, 2.5 µM H_3BO_3, 0.5 µM $CuCl_2$ x 2 H_2O, 0.1 µM $NaWO_4$ x 2 H_2O, 2.5 µM $CoCl_2$ x 6 H_2O, 25.0 µM $NiCl_2$ x 6 H_2O, 2.5 µM Na_2MoO4 x 2 H_2O, 0.5 µM Na_2SeO_3 x 5 H_2O (trace elements five fold, modified Scherer and Kneifel 1983), 25.0 µM $(NH_4)_2Fe(SO_4)_2$ x 6 H_2O (ferrous iron), 1.0 mM Na_2S x 7 – 9 H_2O (sulfur source, freshly prepared or as a 250 mM solution under N_2). After completing the supplemented medium seed slugde (wastewater plant of the town Geesthacht, near Hamburg) was adjusted to a final dry weight content of 10% and the Avicel cellulose was added as powder. The 2l test flasks were set at temperatures of 20-25 °C in an incubator of 37 °C so that a small pressure increase could increased limited by the MilligasCounter® to 4-7 mbar. The incubator was ventilated to keep the temperature constant at a high level (0.1 °C). Generally anaerobic

cultures had to be degassed with N_2 prior cultivation. This procedure showed in different test series no effect on the mixed sludge culturesculture. Also one time stirring per day by a magnetic stirrer exhibited no effect as measured by the gas production rate.

RESULTS

The measurement principle of the MilligasCounter® can be seen in fig. 1: The gas to be measured flows in via the gas inlet nozzle (1), through the micro capillary tube (2) located in the base of the MilligasCounter and up into the liquid casing (4) which is filled with a special nonaqueous packing liquid (3) (the height of the liquid amounts only 4 cm).The gas rises then as small gas bubbles through the packing liquid, up and into the two measuring chambers (5), which are filled alternatively by the rising gas bubbles. When a measuring chamber is full, the buoyancy of the filled chamber causes the measurement cell to abruptly tip over into such a position that the second measuring chamber begins to fill and the first empties.

The measurement of gas volume therefore occurs in discrete steps with a resolution of 0.950-1.050 ml (= content of measuring chambers).

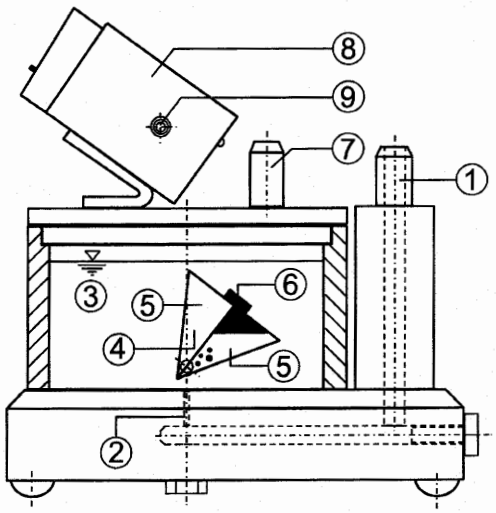

Figure 1. Scheme of the developed MilligasCounter®
1.gas inlet nozzle, 2. micro capillary tube, 3. packing liquid (height only 4 cm), 4. liquid casing, 5. two measurement chambers, 6. permanent magnet, 7. gas outlet nozzle, 8. Counter mechanism, 9. socket reed contact

Through the combination of a permanent magnet (6) and a magnetic sensor (reed contact), the tilting procedure creates a pulse which is registered by the counter mechanism (8). The measured gas escapes through the gas outlet nozzle (7) and the switching pulses of the reed contact can be obtained via the socket (9).
15 MilligasCounters® can be deposited on a 115 l incubator and the impulses can be counted online by an "impulse-data logger" to transfer the data to a personal computer.

An example of an experimental series as measured by MilligasCounters® is given in fig. 2.

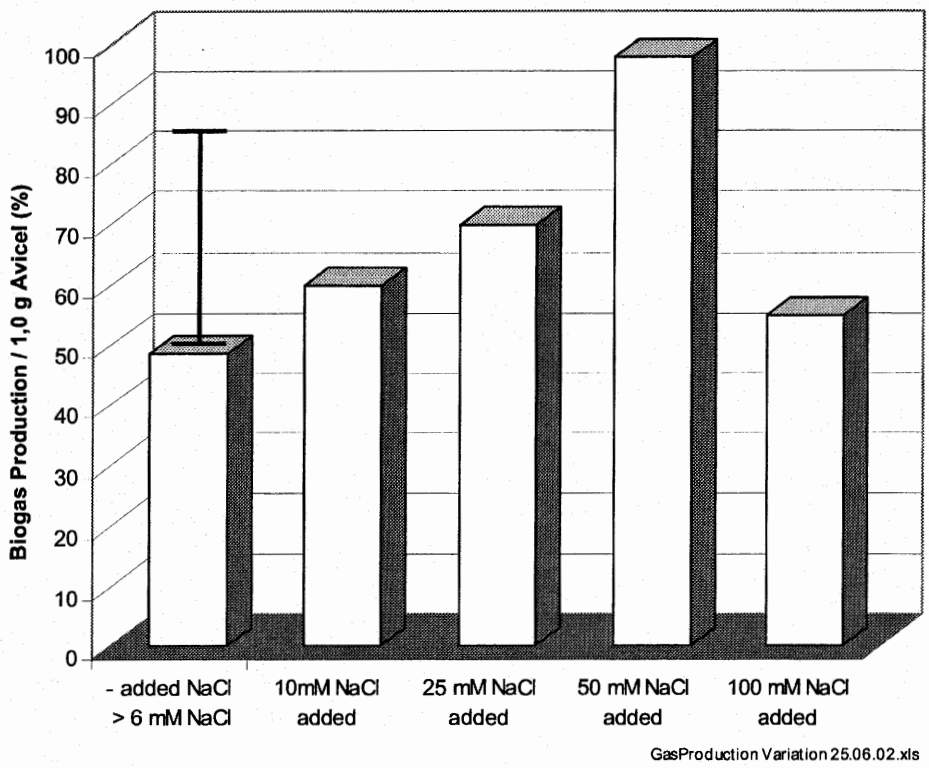

Figure 2 . Anaerobic degradation of microcristalline cellulose (Avicel) in test flasks at different NaCl-concentrations (added). The medium was supplemented as described in the text and contained ≥ 6 mM Na^+ (the bar at the left is explained in text).The netto gas production is shown (the reference gas production without cellulose was already substracted). 100% gas production was set equivalent to 854 ml per 1.0 g Avicel at 25°C (gas temperature in the MilligasCounters). Water pressure of 25°C in the gas phase was taken into consideration.

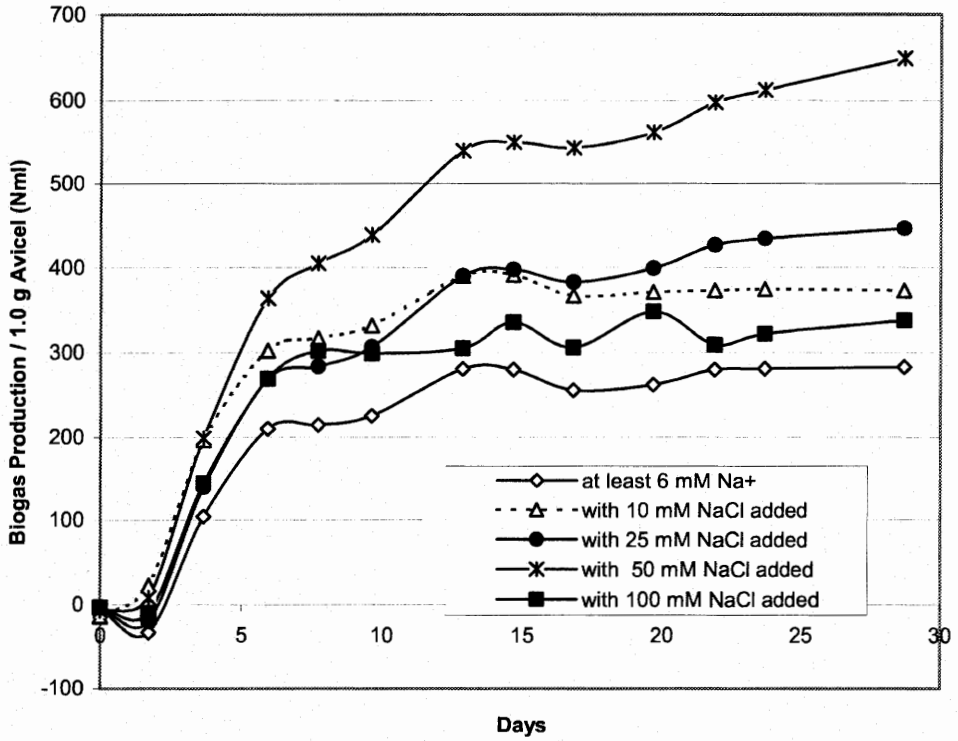

Figure 3 : Biogas production from microcristalline cellulose (Avicel) in test flasks at different NaCl-concentrations (added). For further conditions see figure 2 and text.

◇ Gas production without external supplementation of Na^+ to the basis medium (≥6 mM Na^+)

Figure 2 and 3 indicate that the sodium content of sewage sludge for anaerobic biodegradation tests could be limiting so that the netto gas production from cellulose as test substrate was decreased to 48% of the theoretical substrate conversion (fig. 2), but apparently not the gas production rate over the time, see fig. 3. The behaviour of the gas production curve was at all added NaCl concentrations nearly equal. However, a too high NaCl concentration retarded the cellose conversion to biogas, e.g. 100 mM in fig. 3. This sodium effect wasn't seen so clear as shown in fig. 2/3 in different test series and varied by the used seed sludge. In general the gas production without addition of external NaCl reached 50-80% of the calculated theoretical degradation rate. This is indicated by a bar at the left in fig. 2.

Sodium can be used by many anaerobic bateria and methanogenic archaea as an energy source (Perski et al. 1982, Daniels et al. 1984). This may be responsible for the increased netto gas production at 50mM added NaCl to nearly 100 % in the test, s. fig. 2.

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