PRACTICE REPORT
LABORATORY ANALYSIS
TITRATION
FOS/TAC

Biogas Plant Wambeln
Source: EnerCess

Targeted control of biogas plants with the help of FOS/TAC

A → *biogas plant* functions most efficiently when substrates are added in amounts that are tailored to the → *fermentation process*. For this purpose, the exact status of the fermentation in the digester must be known and documented over a long period of time. This is achieved by regular, easily performed in-house laboratory analyses of the → *FOS/TAC ratio*. The operator is provided with exact information of the biodegradation performance of the digester and therefore of the → *biogas production*. Any interference with the process can be quickly identified and eliminated in a targeted manner. The plant is operated more efficiently and cost-effectively.

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FOS/TAC: Reliable assessment of the fermentation process

Fig. 1: The TIM 840 titrator for FOS/TAC determination at the Wambeln biogas plant.

FOS/TAC
The ratio of volatile organic acids to alkaline buffer capacity is a measure of the risk of acidification of a biogas plant.

What is the FOS/TAC ratio?

The Federal Agricultural Research Centre (Bundesforschungsanstalt für Landwirtschaft/FAL) developed the FOS/TAC analysis from a titration test (Nordmann-Methode) in order to determine the quotient of the acid concentration and the buffer capacity in the fermentation substrate. **FOS** stands for **F**lüchtige **O**rganische **S**äuren, i.e. volatile organic acids, and is measured in mg HAceq/l, while **TAC** stands for **T**otales **A**norganisches **C**arbonat, i.e. total inorganic carbonate (alkaline buffer capacity), and is measured in mg $CaCO_3$/l.

The FOS/TAC ratio has long been recognised as a guide value for assessing fermentation processes. It enables process problems extending as far as the imminent inversion of the digester biology to be detected at an early stage, so that countermeasures can be initiated.

How is the FOS/TAC ratio determined?

Either by manual titration or, more easily and quickly, with a titrator, e.g. the TIM 840 (see Fig. 1 and Fig. 4). Relative to manual titration, this is more accurate and saves several minutes per sample.

Calculation formula (empirical)	
FAL specifications:	Quantity of substrate: 20 ml Sulphuric acid: 0.1 N (0.05 mol/l)

TAC = H_2SO_4-Volume added from start to pH 5 in ml × 250

FOS = (H_2SO_4-Volume added from pH 5 to pH 4.4 in ml × 1.66 – 0.15) × 500

Important: If the quantity of substrate or the strength of the acid differs from the above, the formula must be amended accordingly!
The correct formula is pre-programmed in the HACH LANGE TIM 840/845 titrator and the displayed values can be taken over as they stand, i.e. they do not have to be converted.

Fig. 2: Calculation of the FOS/TAC ratio

Carrying out a FOS/TAC measurement

❶ Take a representative sample of the fermentation substrate.
❷ Remove any coarse components from the sample. It is crucial that sample preparation (filter, tea strainer, kitchen strainer or centrifuge) should always be carried out in the same way.
❸ Weigh out 20 ml substrate and fill up with distilled water if necessary.
❹ Place the sample on a magnetic stirrer and homogenise it continuously during the titration process.
❺ Titration with 0.1 N H_2SO_4 to pH 5; note the volume (ml) of acid added.
❻ Titration with 0.1 N H_2SO_4 to pH 4.4; note the volume (ml) of acid added.
❼ Calculate FOS/TAC using the empirical formula (see Fig. 2).

To measure the FOS/TAC ratio with the TIM 840 titrator, 5 ml prepared sample are introduced into a titration beaker containing a follower bar.
50 ml distilled water are then added, the beaker is positioned under the measuring electrode of the TIM and the measurement is started. The following work steps (❺,❻,❼), which are usually rather tricky in practice, are performed by the titrator. After about 5 minutes the (automatic) titration is complete and the results are displayed.
The TAC and FOS values are calculated directly using a pre-programmed formula.
All measured values can be stored in the autotitrator and/or sent to a printer or PC.

Assessment and use of the FOS/TAC ratio

In practice, a FOS/TAC ratio of 0.3 to 0.4 is normal, although each plant has its own optimal ratio. This can only be determined by long-term observation and regular checks, as there is a strong dependence on the substrate. For example, plants that make use of renewable raw materials usually require a FOS/TAC ratio of 0.4 to 0.6 for stable operation.

The point at which the plant is operating most efficiently – i.e. when gas production is at a maximum and there is no danger that the process will crash can only be determined by testing out a variety of options. Such a crash is extremely costly; several weeks without gas production and the huge amount of work (pumping out, emptying the digester, etc.) required to make the plant operational again can endanger the profitability of a whole year.

"At the end of 2006 we started up our cofermentation plant. The fermentable material is a selective mixture of food residues, pig slurry and chicken droppings. We control the fermentation process by carrying out regular FOS/TAC measurements with the TIM 840 from HACH LANGE. Usually the ratio is between 0.3 and 0.4 in the digester and between 0.2 and 0.3 in the post-digester. We can recognise a threat of acidification immediately, because the ratio increases, and can take appropriate corrective action. In such a case we change the composition of the biomass input, e.g. by increasing the proportion of pig slurry or chicken droppings, which increases the buffer capacity (TAC). Thanks to this simple monitoring method, we need have no fear that the process will crash."

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Rules of thumb for the assessment of FOS/TAC ratios (empirical values provided by DEULA-Nienburg).

FOS/TAC ratios	Background	Measure
>0.6	Highly excessive biomass input	Stop adding biomass
0.5–0.6	Excessive biomass input	Add less biomass
0.4–0.5	Plant is heavily loaded	Monitor the plant more closely
0.3–0.4	Biogas production at a maximum	Keep biomass input constant
0.2–0.3	Biomass input is too low	Slowly increase the biomass input
<0.2	Biomass input is far too low	Rapidly increase the biomass input

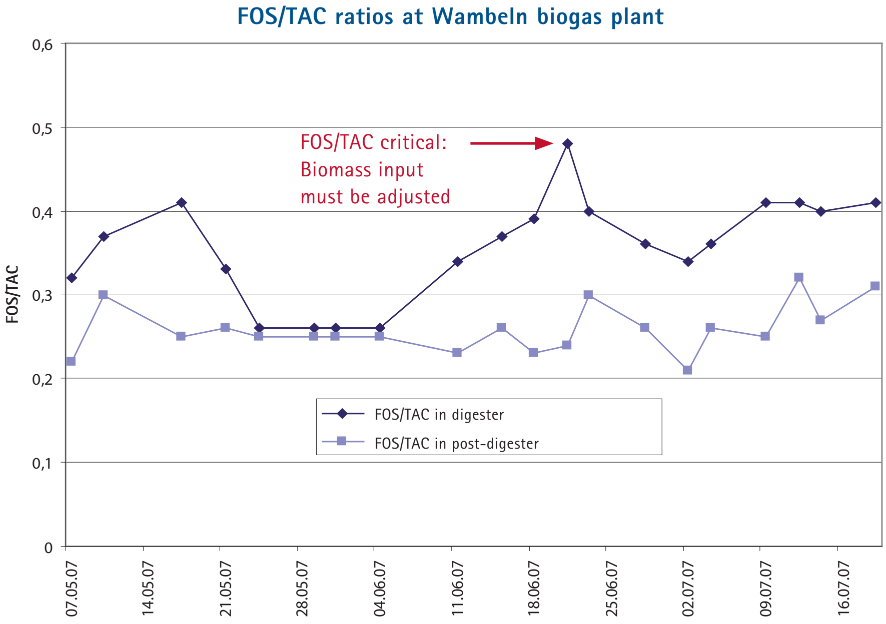

Fig. 3: FOS/TAC curve of the Wambeln biogas plant over a period of 3 months

Technical data

TITRALAB Modell	TIM 840/845
Burettes	
Number of burettes	1/2
Burette extension	Up to a maximum of 6 burettes
Techniques	
pH/mV measurements	●
Endpoint titration	●
Turning point titration	●
FOS/TAC measurements	●
Dosage techniques	
Continuously dynamic	●
Incremental monotonic/dynamic	●
Peripherals	Sample changer, balance, printer, PC software
Electrode inputs for	
Indicator electrodes	1
Reference electrodes	1
Polarised electrode	1

Table: Technical data, TIM 840/845

Fig. 4: HACH LANGE TIM 840 titrator for the determination of FOS/TAC, pH and redox potential

Literature

- Prof. Dr. Peter Weiland, Christa Rieger, Institut für Technologie und Biosystemtechnik, Abt. Technologie, Bundesforschungsanstalt für Landwirtschaft (FAL): „Prozessstörungen frühzeitig erkennen“, BIOGAS Journal 4/06
- Dr. Jürgen Wiese EnerCess GmbH, Ralf König HACH LANGE GmbH: „Prozessbegleitende Fermenterüberwachung auf Biogasanlagen“, DVGW energie | wasser-praxis 09/2006
- HACH LANGE Information „Routine-Analytik für Biogas“, Art.Nr. DOC032.72.20007.APR07
- Kurzbedienungsanleitung: FOS/TAC-Messung mit dem HACH LANGE TITRALAB

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