

## Process Monitoring of Biogas Projects

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### ABSTRACT

Biogas digesters show high potential in the avenue of waste management and renewable energy production. However, the process can only turn out optimum biogas if it is rightfully biologically intrinsic to the digesters. This paper debates the role of process monitoring parameters in optimizing the biogas digester performance. It puts much emphasis on frequent process failures such as an organic overload, hydraulic overload, and ammonia inhibition. This paper calls for a process monitoring strategy that interprets and understands the biological activity going on in the digester. In this way, after measuring some key parameters, the operator will be able to tell instabilities in advance before this wholly results in a crash, hence saving many financial losses related to the restarting of a destabilized system. The paper presents the process monitoring benefits regarding delivery in the context of comprehensive understanding of the biogas process, giving early warnings of instability, successful digester start-up and re-start, and lastly, avoiding the costs of shutdowns and restarts. The economic gains that process monitoring offers with its slightest implementation against the costly shutdown and restart of digesters are huge. It is very informative to operators and biogas researchers because proactive action can be made on improving digester performance for maximum biogas production, which will be possible through the efficient use of process monitoring parameters.

**KEYWORDS:** *Biogas, digester, operator, biological, process monitoring*

### Importance of Process Monitoring

Biogas plants are complex biological systems; they involve diversity in microorganisms that interact with one another in the decomposition of organic materials in the absence of oxygen. The main products are biogas—a CH<sub>4</sub> enriched gas, a renewable fuel usable for car engines and production of local heat or electricity, or through distribution systems. Degradation consists of four consecutive biological steps: hydrolysis, acidogenesis, acetogenesis, and methanogenesis. It means that if any of these processes is adversely affected, the result will immediately tell on the other processes, which may result in instability in the biogas plant. Typical failures in the process include organic overload, hydraulic overload, and ammonia inhibition. Process monitoring is always useful for understanding the activities of a biogas plant and is important for process stability. In case of a highly restrained

microorganism population or even complete failure of the whole plant, the financial consequences for the operator of a biogas plant can be enormous. Process monitoring can enable an overview of the completely biogas process. Detect potential instabilities in anaerobic digesters before a catastrophic failure takes place. Develop or re-start a plant with a high probability of success. In most cases, the costs of basic monitoring are much lower than the Costs plus lost revenues related to the recovery of a biologically upset plant. For instance, if the biogas plant completely fails, it may be necessary that the digester be completely emptied and recommissioned with fresh culture/inoculum. Because of the required start-up time, a lot of time is wasted before the plant reaches full capacity. The plant operator can suffer severe financial repercussions.

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### **What are the steps to develop Process Monitoring in Biogas Projects:**

No biogas plant is like another; proudly, each shows different process circumstances. That means it is not possible to define a generally valid value for every process parameter and state its validity for all plants. This would be of great interest in this context: the obtaining of values for process parameters such as temperature or the pH-value during stable operation at each plant. Since it provides for constant monitoring and recording of the process parameters throughout the life cycle of the plant, deviation from the expected or standard condition can be easily detected and recognized. Other than these characteristics, general process information should also be recorded, relating to the mass of input, organic loading rate, and problems relating to operation if any. Although much of this data is automatically recorded in automated plants, there is always a need to keep a manual operational logbook. Alongside the laboratory-based analysis of parameters, at least some on-line process monitoring equipment should be installed in every biogas plant. Still, how far to go in investing in online equipment needs to be balanced against the economic risks of the biogas plant.

Prior to delving into specifics, it is important to note that the occurrence of several process imbalances can be prevented using effective operational procedures. Consequently, sufficient instruction of the operating personnel is essential.

The personnel of a biogas plant is a significant concern. The following general recommendations are given to help an operator of a biogas plant bypass any irregularity in the process:

1. Rate of feeding that occurs without interruption or pause.
2. A uniform combination of feedstock materials, such as manure and biowaste.
3. Systematic and meticulous alteration of feedstock compositions as needed
4. Minimize exposure to fluctuations in temperature
5. The regularity and strength of stirring or agitation
6. Continuous process monitoring and control

### **Feeding load and interval changes and unstable feed cause problems:**

In the case of large variations in daily organic load applied to the biogas digester, variability in gas production would result. While this is not often a real problem regarding process stability, it might be related to a reduced productivity of the overall biogas project. Further, if the energy contents of two different batches of feed stock are different-for

example, Napier grass types— then biogas production will change, although the nominal feeding rate has not been changed. Another aspect could be interruptions of the feed to a biogas digester. This means that intake disturbances of the order of days for example, irregular feeding for days, sometimes even hours depending on the size of the digester can cause serious problems in process stability. This is, however, very plant-specific depending on feedstock and process.

### **Overloading of the feedstock:**

Overload occurs when the amount of organic material being fed into the biogas digester is in excess of the total degradation capacity by microbes for biogas production. Thus, this degrades only partial organic matter into volatile fatty acid, which consequently accumulates in the reactor. In such a case, usually, the methane concentration in the biogas drops. If, in a reactor, the formed VFA concentration were to be higher than what is usually counterbalanced by any buffer capacity, acidification of the digester results, which means a decrease in pH. Unless something is done, acidification will reduce biogas production to zero. In practice, common causes of organic overload include changes in feedstock mixture and composition, inputs that are badly measured or increased mixing that suddenly introduces unreacted material into the digestion process.

### **Hydraulic Overloading of the biogas digester:**

Apart from organic feedstock overload, hydraulic overloading is another possible route to process instability. In the case that the hydraulic retention time is too short to permit the multiplication of anaerobic microbes, their concentration will drop and they will gradually be washed out of the digester; this is a very common problem in most digesters. This will logically cause a problem because biogas production is directly proportional to the concentration of the anaerobic microbes; further, hydraulic overloading of the digester is especially damaging in anaerobic processes. This is because some of the microorganisms involved can have very long reproduction times. Doubling times or replication time of up to 20–30 days have already been shown for methane-forming microbes and, in cases of inhibition, the doubling time will even increase beyond that. Finally, the washing-out of microbes will finally lead to an accumulation of VFA in a manner similar to organic overloading because acidifying microbes grow faster than methanogens. Washing out will finally bring an end to biogas production. It is, therefore, important that all liquid inputs, as well as solid inputs to a digester, be measured and recorded prior to feeding, and once the

liquid slurry comes off the digester, it should also be tested and data recorded.

### Temperature Fluctuations:

Most microorganisms and microbial consortia are optimized for certain temperatures. In mixed culture biogas digesters, the microbial composition will adjust to the applied fermentation temperature. A stable fermentation temperature should always be guaranteed to avoid extreme process fluctuations. Generally, it is advisable not to exceed the following daily temperature changes: 1°C for thermophilic biogas processes and 2-4°C for mesophilic processes. When starting up a Biogas-Digester ensure that the inoculum is already adapted to the expected temperature of the digester to decrease adaptation time and start-up time. In contrast to the factors above, rising process temperature from psychrophilic (< 25°C) to mesophilic conditions (35-40°C) can be of advantage. The reason is that mesophilic processes show better performance. An expected rise in temperature should decrease feeding rate since temperature sensitivity increases with the load rate. Moreover, microorganisms need time to get used to a changed temperature or any cyclic temperature variation. An intentional change in temperature should be an exception rather than a rule and should be followed by constant temperatures.

### Inhibition by Ammonia:

Anaerobic digestion of nitrogen-rich feedstocks leads to protein decomposition and, consequently, formation of ammonium nitrogen (NH<sub>4</sub>-N). In the aqueous environment—like inside a biogas digester—NH<sub>4</sub>-N occurs in the form of NH<sub>4</sub><sup>+</sup> ions and free ammonia NH<sub>3</sub>(aq). The share of NH<sub>3</sub>(aq) free ammonia rises with increasing pH or temperature. Free ammonia, NH<sub>3</sub>(aq), has been described as the main reason for microbial inhibition in the digester, given the fact that this species diffuses through the cell membrane without any problems. The possible mechanisms of inhibition include a change in intracellular pH, increasing metabolic energy, and microbial enzyme reaction inhibition. Other than temperature and pH, the microbial adaptability of high ammonia concentration is also another important factor for the ammonia inhibition phenomenon. High nitrogen feedstocks often cause process instability in biogas facilities; for example, chicken litter. A rapid transition from low to high nitrogen stock might be hard to resist. It is then hard to define the limits of stability due to the mixed culture and its adaptability to ammonia. This explains why literature reviews have different inhibitor doses of ammonium nitrogen. However, the inhibition starts at 1.5 to 3.0 g NH<sub>4</sub>-N L<sup>-1</sup>. Some research, however, indicates that the

inhibition starts at far higher concentrations: 5.0 g – 14.5 g NH<sub>4</sub>-N L<sup>-1</sup>. The difficulty which comes in while establishing NH<sub>4</sub>-N stability limits raises a case that stable anaerobic processes at high concentration of ammonia need adequate time for microbe adaptation time which can also be up to few weeks at low organic loading rate, good trace element availability and low to medium hydrogen sulphide concentrations.

### Inhibition by Hydrogen Sulphide:

Hydrogen Sulphide is a product of the anaerobic degradation of sulphur compounds. Like ammonia, the undissociated form of free hydrogen sulphide, H<sub>2</sub>S (aq), is known to inhibit. In addition, hydrogen sulfide precipitates metal ions decreasing trace element bioavailability, e.g. iron. Monitor gas phase concentration, reactor temperature and pH to calculate H<sub>2</sub>S (aq) concentration. For example, 1 % H<sub>2</sub>S (10000 ppm) in gas phase correspond to 26 mg H<sub>2</sub>S (aq) L<sup>-1</sup> when at 35°C and pH 6.9. Inhibition by hydrogen sulphide commences at 30 mg H<sub>2</sub>S (aq) L<sup>-1</sup>. It was observed that the inhibition typically occurs above 100 mg H<sub>2</sub>S (aq) L<sup>-1</sup>, and even 200 mg H<sub>2</sub>S(aq) L<sup>-1</sup> can be tolerated after sufficient adaptation time. Literature values are also available for a wider range of inhibitory limits for H<sub>2</sub>S (aq): 40 to 400 mg L<sup>-1</sup>. However, H<sub>2</sub>S (aq) at much lower concentrations have been frequently found to be a problem in the field, especially in the presence of other inhibitors like ammonia or low iron concentrations.

### Inhibition by Heavy Metals:

Here the heavy metals can be compared to other living organisms. At small quantity, they will be essential to the microbial activity, but at the same time also detrimental at higher concentrations. Heavy metals can be detected in samples from the digester either by ICP-MS or AAS techniques. Normally, low amounts are well-tolerated because the bioavailability of insoluble precipitates formed by these elements with sulphide and carbonate is reduced. Only in higher concentration feedstocks, such as biowaste, that means toxicity monitoring is relevant. In most biowaste treatment plants, heavy metal analysis has been realized to control the digestate quality but not the process stability. It was also determined that the lowest reported inhibition concentrations for heavy metals are Cu 40 mg L<sup>-1</sup>, Cd 20 mg L<sup>-1</sup>, Zn 150 mg L<sup>-1</sup>, Ni 10 mg L<sup>-1</sup>, Pb 340 mg L<sup>-1</sup>, and Cr 100 mg L<sup>-1</sup>. Braun 1982 determined that the concentrations for 20% at pH 8 are Cd 157 mg L<sup>-1</sup>, Ni 73 mg L<sup>-1</sup>, Cu 113 mg L<sup>-1</sup>, and Zn 116 mg L<sup>-1</sup>.



**Micro Nutrients Limitations:**

The lack of micro nutrients in biogas digester may result in reduced performance, known as "trace element limitation". Low availability of one of the important critical trace elements may reduce microbial activity. Examples of trace elements needed for stable biogas process are Ni, Co, Mb, Se, and Fe. Trace elements are needed to build up enzymes and hence become a crucial element for micro-organisms. Similar to the heavy metal assays, physical presence in the digester is not enough; bacteria require more than that. The bioavailability of trace elements requires that they have to be soluble and not precipitated or adsorbed for trace element detection in fermentation broth. For biological availability calculation, scientists have come up with a technique whereby sequential usage for a series of solvents can be used on digester samples. Then the traces of those elements in different solvents are checked regarding their bioavailability. On the other hand, trace element deficiency might very likely occur more in mono-digestion. At the same time, one can never completely exclude that it may also happen in co-digestion-based biogas projects. Increased manure usage as feedstock hardly results in a trace element deficiency. Testing of trace element limitation is not routine and thus not a part of the criteria for process monitoring. In the case of instability in a plant process and with regard to VFA, if the concentrations increase, then investigate the main causes of these process imbalances and eliminate them. If there is no decline in symptoms, then trace elements availability and add elements as appropriate. Inhibitive effects are more proved by excessive addition of trace elements. Furthermore, land application of digestate becomes very problematic when the quantities of trace elements become higher than permissible limitations.

**Quantity and Quality of Feedstock to the biogas digesters:**

Quantity and quality of feedstock to a biogas digester matter because of the process instabilities through changes in feeding—that is, quantity—and composition, that is, quality, to the biogas digester. For solid feedstock, there may be an automatic feeding system with weighing cells and data recorders; on a daily basis, these data can be noted and can be quite easily monitored. A small dam in front of the feeding system should be made to protect it from loading machines/vehicles. Less accurate but daily shovel loads, for example, from a wheel loader, may deliver meaningful data in less sophisticated biogas plants. Reduced feed means reduced biogas production. Increased feed leads to acidification and process instability. Liquid feedstock should not be reported together with solid feedstocks for two

reasons: Main reason: high organic matter content contributes towards daily biogas digester feed. High liquid content in feedstock, for instance rain-water, may reduce retention time leading to hydraulic overloading as observed in the above paragraph. Queries revealed that recently constructed biogas projects in India are provided with weighing scale/equipment to quantify solid feedstock input. Quantification regarding liquid feed stock is usually not available. It was, therefore, observed that nearly 45% of German biogas plants do not record the liquid feedstock or process water intake. Since a majority of biogas plants are CSTR, sans microbe retention systems, daily feedstock intake affects the microorganism retention time. Since the input of liquids to the digester is most often not documented, biogas plant operators often do not know what the actual retention time truly shall be. It is advisable to have flow meters that can measure the quantity of liquid feedstock fed to the digesters, and yes, recent new biogas projects in India indeed have flow meters. Levels of storage/feeding tanks may also be good to keep a track record of. Another way is by recording pumping time, though it is not sure if the information derived will be very accurate as it will depend on the feedstock composition and pump wear n tear.

Besides quantification, characterization of feedstock is important. This is particularly applicable on the organic fraction of municipal solid waste treatment plants, where the kinds of feedstocks vary. Tracking the individual feedstock that arrives at the facility is essential. In case a biogas project is run successfully with a constant feedstock, say cow dung or any animal manure, then characterizing feedstock does not become much significant. In this regard, the plant operator and owner have to be aware of the full list of feedstock properties and methodologies for required analyses for biogas plant monitoring. Among those specific function areas is knowledge regarding the feedstock's pH value, considering that too acidic or alkaline feedstock may depart from the ideal pH range in a digester, pH 7-8. In such cases, caustic or acid will be required; however, biogas digesters can bear a rather large pH range of feedstocks due to their buffering ability. One key measurement would be volatile solids, VS, for feedstocks since they represent the source of organic matter for the production of biogas. Since the ash level is typically low in feedstocks, simply knowing the total solids content may be adequate where TS is used to represent total solids and VS as the volatile solids, such that  $TS = VS + \text{ash}$ . For liquid type feedstocks, like wastewater, VS or even TS measurements may not be reliable since volatile chemicals can be present, such as acetic acid and ethanol. In such cases, one measures COD,

or chemical oxygen demand. COD is not normally measured for solid feedstocks due to their complexity and poor reproducibility compared with VS measurements. Feedstock nitrogen content is measured by the TKNS (total Kjeldahl nitrogen). Tracking TKN content in feedstocks can become important because a shift from nitrogen-poor to nitrogen rich mixtures can result in process instability. The accumulation of ammonia in the digester due to nitrogen-rich feedstocks can limit ammonia production. A BMP test, the biochemical

methane potential, learners determine biogas production and velocity of degradation for a feedstock. As BMP tests are time-consuming, they are rarely put into action.

For example, at the time of introducing a new feedstock. However, it is advisable to regularly conduct BMPs on feedstock feed to the digester and the results should be recorded and tabulated. This will also yield information about the performance of the digester.

**Table No: 1 Overview of biogas feedstock classification and standard and methods**

PARAMETERS	USED FOR	APHA STANDARD METHOD
pH	➤ Measure of the acidity or alkalinity of the feedstock.	➤ 4500-H+ B: Electrometric Method
Total Solids (TS)	➤ Measure of the total amount of solid material in the feedstock, including both organic and inorganic matter.	➤ 2540 B: Total Solids Dried at 103-105°C
Volatile Solids (VS)	➤ Measure of the amount of organic matter in the feedstock, which can be degraded to produce biogas.	➤ 2540 E: Fixed and Volatile Solids Ignited at 550°C
Chemical Oxygen Demand (COD)	➤ Measure of the total quantity of oxygen required to oxidize all organic material into carbon dioxide and water.	➤ 5220 B: Open Reflux Method ➤ 5220 C: Closed Reflux, Titrimetric Method ➤ 5220 D: Closed Reflux, Colorimetric Method
Total Kjeldahl Nitrogen (TKN)	➤ Measure of the total concentration of organic nitrogen and ammonia in the feedstock.	➤ 4500-Norg B: Macro-Kjeldahl Method 4500-Norg C: Semi-Micro Kjeldahl Method
Bio Methane Potential (BMP)	➤ Measure of the potential biogas yield from the feedstock under anaerobic conditions.	➤ No direct APHA method; generally assessed using protocols from other standards like ISO 11734, VDI 4630, or similar guidelines



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### **Biogas Volume and Biogas Composition:**

As usual with biotechnology, the detailed monitoring the fermentation product, such as biogas, provides useful insights. Therefore, it is recommended to monitor gas production volume and composition. Changes in gas output or composition can indicate process imbalance in process monitoring.

Volume of biogas produced: Many devices can be used to measure biogas reduction/volume, including:

1. Ultrasonic flowmeters
2. Fluidistor oscillation
3. The turbine flowmeter
4. Vortex flow meters
5. Dynamic pressure probes
6. Thermal flowmeters
7. Diaphragm/bellows gas meters

The measuring of the volume of biogas in biogas plants is difficult due to its variable composition, dirty and corrosive, wet, and low-pressure. Table 2 summarizes the advantages and disadvantages of various measuring systems. Gas flowmeters should, in general, be installed for easy removal and cleaning. Moreover, the complexity of the sensor must only be as large as necessary—depending on the effort needed for data transfer, calculation work, etc.—because of the purpose of the plant. It is also advised to use ultrasonic flow meters and fluid oscillator meters in the measurement of raw biogas since they have the abilities to handle water and corrosion and, in addition provide reliable results at low pressures of gases. A turbine flow meter, vortex flow meter, or dynamic pressure probe may also be used in the measurement of the raw biogas. Long-term problems are likely to be caused by deposits or biofilms unless regularly maintained and cleaned. In small or non-automated biogas plants, most commonly used conventional diaphragm or bellows meters are used. Mechanical gas meters can, in the long term, generate problems due to corrosion, fouling, or wear while measuring raw biogas. One can measure biogas after washing and drying to avoid humidity-related or corrosion-related issues. However, this means a considerable loss of online direct information on gas production for process monitoring. Some flow meters require drying and cleaning of the gas; these are used in preparation for. Besides monitoring fermentation products, biogas volume measurement can deliver the biogas yield per unit of organic material input, one of the most critical parameters, for example,  $\text{Nm}^3 \text{ t}^{-1} \text{ VS}$ . For correct measurements of biogas yields, sampling over one week with constant feedstock mix and OLR is recommended. This parameter gives a full picture of performance of the degradation process. Biogas from cow dung, maize silage, and food waste produce between 200–500  $\text{Nm}^3 \text{ t}^{-1} \text{ VS}$ . Maximum biogas formation potentials are 746  $\text{Nm}^3 \text{ t}^{-1} \text{ VS}$  for carbohydrates, 1390 for lipids, and 800 for proteins. Online measuring systems as well as portable gas composition measuring systems for determination of the gas composition are applied by various biogas plant operators. Tests for gas composition typically feature infrared or thermal conductivity sensors for  $\text{CH}_4$  and  $\text{CO}_2$ , while  $\text{H}_2\text{S}$  and  $\text{O}_2$  are measured using electrochemical sensors. Biogas composition is a very good parameter for process control. A drop in methane content could point at organic overload if the mix of feedstock has not changed significantly during the recent past. Similarly, a sudden surge of  $\text{H}_2\text{S}$  may lead to process instability. Changes in biogas production and composition should, however, be considered together with other early indicators of process imbalance, such as the alkalinity ratio and VFA concentrations. In some cases, gas composition measurements also include  $\text{H}_2$ . Since  $\text{H}_2$  concentration is a forerunner to process imbalance.

**Table 2 Comparison of Gas Volume sensors/measurement units with Pros and Cons**

SENSOR TYPE	APPLICATION	PROS	CONS
Dynamic Pressure Probes	<ul style="list-style-type: none"> <li>➤ Aerodynamic testing</li> <li>➤ High-speed gas flow monitoring</li> </ul>	<ul style="list-style-type: none"> <li>➤ Direct measurement of flow velocity</li> <li>➤ High sensitivity to small changes in flow</li> <li>➤ Suitable for high-speed gas flows</li> </ul>	<ul style="list-style-type: none"> <li>➤ Complex installation and alignment</li> <li>➤ Sensitive to pressure and temperature fluctuations</li> <li>➤ High maintenance</li> </ul>
Diaphragm Gas Meters	<ul style="list-style-type: none"> <li>➤ Residential and commercial gas metering</li> </ul>	<ul style="list-style-type: none"> <li>➤ High accuracy for small to medium flows</li> <li>➤ Long service life</li> <li>➤ Mechanical operation, no power required</li> </ul>	<ul style="list-style-type: none"> <li>➤ Bulky and heavy</li> <li>➤ Slow response to changes in flow</li> <li>➤ Not suitable for high flow rates or rapid changes</li> </ul>
Fluidistor Oscillator	<ul style="list-style-type: none"> <li>➤ Gas chromatography</li> <li>➤ Environmental monitoring</li> </ul>	<ul style="list-style-type: none"> <li>➤ High precision at low flow rates</li> <li>➤ Low power consumption</li> <li>➤ Compact design</li> </ul>	<ul style="list-style-type: none"> <li>➤ Limited to low to medium flow rates</li> <li>➤ Can be affected by temperature and pressure changes</li> </ul>



Ultrasonic Flow Meter	<ul style="list-style-type: none"> <li>➤ Natural gas metering</li> <li>➤ Industrial gas flow measurement</li> </ul>	<ul style="list-style-type: none"> <li>➤ High accuracy and reliability</li> <li>➤ No moving parts, low maintenance</li> <li>➤ Suitable for a wide range of gases and pressures</li> </ul>	<ul style="list-style-type: none"> <li>➤ Expensive upfront cost</li> <li>➤ Requires clean, bubble-free gas</li> <li>➤ Sensitive to installation conditions</li> </ul>
Vortex Flow Meters	<ul style="list-style-type: none"> <li>➤ Steam and gas flow measurement</li> <li>➤ HVAC systems</li> </ul>	<ul style="list-style-type: none"> <li>➤ Good for a wide range of flow rates</li> <li>➤ No moving parts, low maintenance</li> <li>➤ Suitable for various gases</li> </ul>	<ul style="list-style-type: none"> <li>➤ Accuracy can be affected by pipeline vibrations and gas density changes</li> <li>➤ Less accurate at low flow rates</li> </ul>
Thermal Flow Meters	<ul style="list-style-type: none"> <li>➤ Thermal Flow Meters</li> </ul>	<ul style="list-style-type: none"> <li>➤ Affected by changes in gas composition</li> <li>➤ Limited to clean, dry gases</li> <li>➤ Not suitable for high-pressure applications</li> </ul>	<ul style="list-style-type: none"> <li>➤ Laboratory gas flow measurement</li> <li>➤ Leak detection systems</li> <li>➤ Laboratory gas flow measurement</li> </ul>
Turbine Flow Meters	<ul style="list-style-type: none"> <li>➤ Custody transfer of natural gas</li> <li>➤ Process gas flow monitoring</li> </ul>	<ul style="list-style-type: none"> <li>➤ High accuracy for steady flows</li> <li>➤ Suitable for a wide range of flow rates and pressures</li> <li>➤ Quick response time</li> </ul>	<ul style="list-style-type: none"> <li>➤ Moving parts subject to wear</li> <li>➤ Sensitive to impurities and particulate matter</li> <li>➤ Requires regular calibration</li> </ul>

### Digester Temperature:

This becomes the reason for maintaining a constant temperature inside the biogas digester for optimal microbial performance. The Optimum temperature of fermentation ranges from 36-43°C for mesophilic degradation and 50-65°C for thermophilic degradation depending upon the bacteria involved. Moreover, the other factors are influenced by fermentation temperature, for example, ammonia dissociation and inhibition. Thermophilic fermentation is not as good in degrading protein-rich feed stocks because  $\text{NH}_3(\text{aq})$  concentration increases with an increase in temperature. Temperature measurement is normally done by Pt100 thermometers, always applied in food and biotech industries. It is recommended to measure temperature at different levels in a biogas digester to avoid the risk of incorrect temperature reading.

### Total Solids Concentration:

Use the Total Solids (TS) content in a digester to measure the viscosity of the fermentation broth in the reactor. CSTR reactor should not have huge fluctuations in TS to avoid stirring/agitation issues or inability to pump digester content, viscosity should not exceed a particular level. For wet fermentation systems, which are the bulk of biogas operations, TS concentration should not exceed 10%. This simplifies pumping and mixing digester contents. Increased TS concentration might cause stirring issues in fibrous feedstocks like Napier grass or even with paddy

straw. Often, dilution with fresh water, digestate, liquid feedstock, or process water is required. Monitoring the TS in the digester provides plant operators with data on dilution levels. Comparing TS and VS of feed stock and digestate can help assess the percentage of degraded feed stock. Residual analysis always helps the operator to understand the difference in the TS and VS of the feed and the outlet slurry. The liquid portion of digestate can be used as process water, such as after separation by screw press separators or centrifuges or decanters only if it is advisable to use monitoring the levels of the salts and the solids within the digester.

### pH of the Feed and the outlet slurry:

The process of fermentation or digestion status can be estimated very well by measurement of pH values. The dependence of the buffer capacity in biogas plants is, however, based on dissimilarities. A change in pH will only then be shown with  $\text{CO}_2$ , carbonate, and ammonia solutions when instability in a process starts to occur. Even though a pH measurement cannot present an indication early enough of a process imbalance, it still delivers useful information for process control. Normally, biogas digesters measure pH offline with a laboratory pH-meter after sampling from the digester. Online pH measurement is awkward due to fouling of the electrode, which requires frequent cleaning and calibration. Moreover, special adapters are needed to withdraw the electrode without leakage. Off-line pH measurements are less

accurate with respect to online measurements of pH due to sampling, storage, and temperature-related uncertainties. For better comparison, perform off-line pH measurements if possible at the same temperatures.

#### **Ammonium Nitrogen (NH<sub>4</sub>-N):**

Anaerobic digestion produces several compounds, including ammonium nitrogen (NH<sub>4</sub>-N). Inhibition by ammonia is common for nitrogen-rich feed sources is the reason for process imbalance. Monitoring NH<sub>4</sub>-N concentrations in the digester can indicate if ammonia inhibition is creating process imbalance. NH<sub>4</sub>-N analysis can be performed using automated lab systems following the US-American standard "APHA 4500-NH<sub>3</sub>-Nitrogen" (APHA, 1998) or the German DIN 38406-5:1983-10 (1983). Free ammonia (NH<sub>3</sub>(aq)), the inhibitory form of NH<sub>4</sub>-N, can be calculated based on its concentration. For calculating NH<sub>3</sub>(aq) using a standard formula, digester pH and temperature are required: For monitoring it is suggested using the immediately quantifiable NH<sub>4</sub>-N, not NH<sub>3</sub>(aq) as the parameter. The estimation of NH<sub>3</sub>(aq) relies on precise pH determination within the biogas digester. The pH is generally sampled off-line, making it challenging to determine the actual pH inside the biogas digester. Consequently, even a small pH fluctuation (e.g., 0.2–0.3 pH units) can significantly impact NH<sub>3</sub>(aq) calculations. Conversely, NH<sub>4</sub>-N measurement in the biogas digester is precise. Skilled biogas plant operators can create their own monitoring system by calculating NH<sub>3</sub>(aq) from NH<sub>4</sub>-N.

#### **Volatile Fatty Acids (VFA):**

Volatile fatty acids (VFA) are short-chained organic acids, such as acetic, propionic, butyric, and valeric acids, or their branched isomers. They are intermediate metabolites formed in the process of acidification in anaerobic digestion and are precursors for methane. Their inhibition from methanogenesis, the biological conversion to methane, can occur if they accumulate. Several methods are used to measure VFA; steam distillation, colorimetric, chromatographic, titrimetric methods, and others. This review focuses on commonly used methods. The main VFA parameters that are used for monitoring the biogas process are individual VFA concentration and total VFA concentration.

#### **External high-performance laboratory measures individual VFA:**

Monitoring of the individual VFA concentrations in the digester gives very good indications of the process. Their research offers direct feedback on the different microorganism groups interacting and being inhibited in the reactor. Because acetic acid is the

ultimate precursor to methane, small accumulation in the digester is usual. Small quantities of propionic acid are acceptable. The acetic acid/propionic acid ratio has turned out to be an excellent predictor of process. The formation of butyric or valeric acid, mainly their branched isomers, indicates severe process instability. Individual VFA are measured using either HPLC or GC analysis. Chromatography equipment is relatively expensive; thus, these studies have to be usually conducted in external laboratories. Proper handling, transport, and storage of the samples are very important for correct results.

#### **In-house lab measurement of total VFA:**

The total VFA parameter characterizes the concentration of all VFA's presented. There are options for determining total VFA: either by titration or photometric techniques, or summing of each VFA. Titration methods are advisable to detect total VFA since it is inexpensive, reliable, and fast processes. According to literature, the titration method is advisable. Before the measurements, the sample should be filtered through a 0.45 µm membrane filter or centrifuged for 10 min at 10,000 g to remove suspended particles. Following that, a three-point titration of 20 mL of the sample should be conducted using 0.05 mol L<sup>-1</sup> sulphuric acid at pHs of 5.0, 4.3, and 4.0. The total VFA calculation formula is as follows: There are photometric test kits that could measure the total VFA on-site, but they may not be reliable for some feedstocks due to the intrinsic colour of digester content. A distillation pre-treatment can be applied to photometric test kits to evaporate and then condense VFA and remove interference. This must, however, be considered with the losses that occur during distillation. Theory suggests using a measure of an intermediate alkalinity in a twelve-step process called the alkalinity ratio analysis for total VFA. Measured IA values in the alkalinity ratio were also observed to deviate appreciably from actual VFA concentrations that were evaluated by HPLC. Thus, IA cannot be relied upon as a valid measure of the total VFA concentration.

#### **Alkalinity:**

The alkalinity ratio is the two-point titration test relating the intermediate to partial alkalinity. The first one is the intermediate alkalinity, which expresses accumulation of volatile fatty acid and becomes a potential indication of problems in the process. The second metric, for the partial alkalinity, would denote the buffer capacity in the digester through the expression of bicarbonate alkalinity. The bicarbonate buffer capacity in biogas production is important to avoid pH reduction due to moderate accumulation of volatile fatty acids, which might bring biogas



production to a standstill. Alkalinity ratio is also expressed as IA/PA ratio, VFA/bicarbonate, Ripley ratio, or VFA/ALK. In German literature, the parameter is abbreviated as FOS/TAC. The most common titration method applied is the FOS/TAC method. In this titration, the pH is first titrated to 5.0 — bicarbonate alkalinity — and then to 4.4. The titration was performed in 20 mL of filtered or centrifuged digester sample using 0.05 mol L<sup>-1</sup> sulphuric acid. Two-point titration similar to FOS/TAC but with different pH values of pH 5.75 and pH 4.3 is mentioned in English literature as the Ripley ratio.

The FOS/TAC-alkalinity ratio can be estimated using the formula –

$$\text{FOS/TAC} = [(Y*1.66) - 0.15] * 500 / X * 250$$

X - Volume of added acid until pH 5.0 in mL

Y - Volume of added acid from pH 5.0 to 4.4 in mL.

A small on-site laboratory can determine alkalinity ratios with normal laboratory equipment or with an automated instrument. All techniques to determine alkalinity ratios at biogas digesters produce project/digester specific results that cannot be used to compare across other projects/digesters. Even when using the same methodology, plant variations due to feedstocks, sample pre-treatment—for example, centrifuging or filtering—and staff carrying out the titration occur. Nevertheless, the determination of the alkalinity ratio is a low-cost option for laying control of a particular process of a biogas plant.

### Recommendations:

Biogas is a complex biological process, operated by a diversity of microorganisms, composed of sequential reaction steps. Process monitoring is important to ensure stable anaerobic digestion. Variables which may influence the intensity of needed process monitoring include, but are not limited to, the size of biogas digesters or projects, the economic risk due to process instability, and the frequency of change in feedstock types. Process monitoring parameters in this area can basically be divided into two groups. One group of characteristics is indicative of a forthcoming imbalance in the process and thus enables the biogas plant operator to act quickly before an actual process imbalance takes place. Another group contains process-describing parameters which are normally helpful in finding and correcting the cause of a possible process imbalance. Furthermore, many biogas plants will require detailed process optimization beyond merely process monitoring, which unfortunately could not be covered within this article. Nevertheless, regular monitoring of a biogas plant should be implemented before any process

optimization is considered. And finally, another element that may secure reliable operation is having highly educated operators for biogas plants.

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