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Instrumentation and control of anaerobic digestion processes: A review and some research challenges

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Abstract

To enhance energy production from methane or resource recovery from digestate, anaerobic digestion processes require advanced instrumentation and control tools. Over the years, research on these topics has evolved and followed the main fields of application of anaerobic digestion processes: from municipal sewage sludge to liquid – mainly industrial – then municipal organic fraction of solid waste and agricultural residues. Time constants of the processes have also changed with respect to the treated waste from minutes or hours to weeks or months. Since fast closed loop control is needed for short time constant processes, human operator is now included in the loop when taking decisions to optimize anaerobic digestion plants dealing with complex solid waste over a long retention time. Control objectives have also moved from the regulation of key variables – measured on-line – to the prediction of overall process performance – based on global off-line measurements – to optimize the feeding of the processes. Additionally, the need for more accurate prediction of methane production and organic matter biodegradation has impacted the complexity of instrumentation and should include a more detailed characterization of the waste (e.g., biochemical fractions like proteins, lipids and carbohydrates) and their bioaccessibility and biodegradability characteristics. However, even if in the literature several methodologies have been developed to determine biodegradability based on organic matter characterization, only a few papers deal with bioaccessibility assessment. In this review, we emphasize the high potential of some promising techniques, such as spectral analysis, and we discuss issues that could appear in the near future concerning control of AD processes.

Key words: Anaerobic digestion, organic matter, characterization, instrumentation, control, diagnosis.

Nomenclature

AD	Anaerobic Digestion
ADM1	Anaerobic Digestion Model N°1
AFM	Atomic Force Microscopy
BCA	Bicinchonic Acid
BD	Ultimate Anaerobic Biodegradability
BMP	Biochemical Methane Potential
BOD	Biochemical Oxygen Demand
CH_4	Methane

CLSM	Confocal Laser-Scanning Microscopy
CO_2	Carbon Dioxide
COD	Chemical Oxygen Demand
Da	Dalton
EPS	Extracellular Polymeric Substances
FOG	Fats, Oils, and Greases
FTIR	Fourier Transform Infrared spectroscopy
GASDM	General Activated Sludge and Digestion Model
GC/MS	Gas Chromatography coupled with Mass Spectroscopy
GISCOD	General Integrated Solid Waste Co-Digestion model
HA	Humic Acids
HPLC	High Performance Liquid Chromatography
HRT	Hydraulic Retention Time
ICA	Instrumentation, Control and Automation
IWA	International Water Association
LCFA	Long Chain Fatty Acids
MPR	Methane Production Rate
MSW	Municipal Solid Waste
NIRS	Near Infra-Red Spectroscopy
NMR	Nuclear Magnetic Resonance spectroscopy
OLR	Organic Load Rate
PLS	Partial Least Square
R^2	Regression coefficient
RI_4	Respiration Index 4 days
SEM	Scanning Electron Microscopy
STP	Standard conditions of Temperature and Pressure
S/X	Substrate to Biomass Ratio
TEM	Transmission Electron Microscopy
TKN	Total Kjeldahl Nitrogen
TOC	Total Organic Carbon

TS	Total Suspended Solids
VFA	Volatile Fatty Acids
VS	Volatile Solids
XPS	X-ray Photoelectron Spectroscopy
3D-EEM	3D Emission Excitation Matrix

1. Introduction

One of the key issues for global sustainable development is the energy consumption, particularly as fossil fuels, which represents up to 80% of the global energy consumption. Moreover, fossil fuels are considered the main source of acidifying contaminants and greenhouse gasses, as well as the main factor contributing to global warming and climate change. Hence, one big challenge for this century is to develop new competitive sources of renewable energy, capable of replacing fossil fuels with a minimum impact on both the environment and society, while maintaining energy (electricity or gas) grid stability (Szarka *et al.* 2013). In this respect, alternative energy sources such as methane from organic residues must be considered.

Anaerobic Digestion (AD) is a biological process in which the organic carbon is converted through oxidation-reduction reactions to both its most oxidized state (CO_2) and its most reduced form (CH_4). The methane produced is an energy source that can be valorized as electricity, heat, biofuel or can be injected into the natural gas grid. In the context of a widely perceived energetic and climatic crisis, AD has become a very interesting alternative for organic waste disposal. For example, in France, wastewater treatment plant (WWTP) energy consumption is about 20 kWh per year per person equivalent, based on a 100,000 person equivalent plant. From these observations and the fact that wastewater sludge potentially contains a high amount of energy that can be recovered, it is clear that WWTPs of the future – or water resource reclamation facilities (WRRFs) as they are now called - should aim at a positive energy balance (Cao and Pawlowski 2012).

1.1 From Municipal Wastewater Solids to Industrial and Agricultural Wastes

AD has been used to stabilize municipal wastewater solids for over 80 years, probably with the first heated, mixed system being employed in Germany in 1927 (Imhoff 1938). During the last 30 years, the total number of papers on AD and industrial applications increased rapidly, mainly due to a favorable environmental policy: the Kyoto protocol (2005), national or international legislation promoting AD, special rates for selling electricity produced from biogas. The evolution of the market also led to a higher complexity of the substrates considered for AD valorization.

In the eighties, industrial wastewater treated by AD began to grow and worldwide, the overall number of anaerobic reactors treating industrial wastewater reached 2266 references in 2007 (van Lier 2008) and kept on increasing since then. The main focus of AD optimization has been about kinetics of soluble substrates, considering acetogenesis and methanogenesis as the limiting steps (Mata-Alvarez *et al.* 2000).

At the end of the eighties, AD applications focused on the conversion of solid waste began to increase. Solid wastes then included mainly municipal solid waste (MSW) and green wastes. The increasing production of solid waste combined with waste management policies aiming at reducing long-term environmental impacts of landfill disposal have created a need for alternative treatment. The use of AD to treat the organic fraction of municipal solid waste became a reality (De Baere 2000; 2008): from 3 plants in 1990 to 55 plants referenced in 2010 in Europe and at least 4 in North America today, for example. From a process control standpoint, the disintegration/hydrolysis step received considerable attention for solid waste since it is the rate-limiting step for substrates containing mainly particulates (Mata-Alvarez *et al.* 2000; Lauwers *et al.* 2013).

Concomitantly, farmers have become increasingly interested in the AD process, both as an additional source of revenue and as an alternative energy source without greenhouse gas emission to the atmosphere. AD is indeed one of the technologies that fulfil European criteria for second generation biofuel production (fuels manufactured from various types of complex organic carbon sources such as lignocellulose biomass or agricultural residues, *e.g.* manure). The case of Germany where more than 7,850 plants generate over 3.5 GW of electricity is an example or in China where more than 35 million household digesters and 25,000 digesters for agricultural residues have been deployed (Fang 2012).

1.2 Biodegradability, Bioavailability and Bioaccessibility

Hydrolysis rate of complex substrates has been identified for a long time as an important factor for AD modelling and process optimization (Vavilin *et al.* 1997), especially considering substrate characterization and hydrolysis kinetics. Modern dynamical models of AD are very useful for optimization of biogas production. For example, the IWA Anaerobic Digestion Model N°1 ADM1 (Batstone *et al.* 2002) has a detailed pathway description, but the model's main drawback is that it also needs detailed input variables and data that may not be available for a specific application (Astals *et al.* 2013a). Indeed, a key-point for the successful description of a bioprocess is appropriate influent characterization data (Huete *et al.* 2006; Buffiere *et al.* 2006; Kleerebezem and van Loosdrecht 2006).

Lately, three major concepts have been shown to be of prime importance to characterize organic matter biodegradation: biodegradability, bioavailability and bioaccessibility (Jimenez *et al.* 2014). Biodegradability is the ability of a substrate to be broken down by a microorganism into simpler compounds but this biodegradation is limited by molecule's bioavailability, complexity and/or toxicity. Bioavailability is defined as the direct access to the molecule to be degraded while Aquino *et al.* (2008) defined bioaccessibility as the possible access to the molecule depending on several factors such as the contact time between the substrate and the microorganism, the efficiency of hydrolytic activity or ultimately any pre-treatment applied to the waste. There is thus a notion of physical accessibility as in the case of the cellulose protection by lignin or vegetal walls acting as a barrier and needing chemical or physical break-up to make cellulose accessible to microorganisms (Motte *et al.* 2014, Reilly *et al.* 2015). Consequently, the bioavailable organic matter is included in the bioaccessible fraction such as the organic fraction able to be degraded by secreted exo-cellular enzymes (Jimenez *et al.* 2014).

In parallel, the control problem associated with anaerobic biological waste or wastewater treatment processes must involve – like in any aerobic processes – process configurations that remain robust against unpredicted perturbations (e.g., physicochemical, mechanical, etc.) and uncertainties in relation to: (a) initial conditions, (b) kinetic and hydrodynamic parameters, (c) yield coefficients, and (d) input concentrations. All these aspects strongly influence the overall objectives of instrumentation and control and are currently profoundly impacting the technical challenges and optimization criteria applied to AD processes.

2. Instrumentation of Anaerobic Digestion Processes

The following section first focuses on classical instrumentation that is very often encountered in practice. On-line instruments that can be used in fast closed-loop control scheme and have proven to be very useful for monitoring any type of digester will be presented first (See also Spanjers and van Lier 2006 for additional information). Next, because of the development of the solid AD process – with long residence time – some techniques that are not yet available in an on-line context will be discussed. They can indeed provide very informative measurements that can help to optimize AD plants with long solid retention time, such as those dealing with municipal or agricultural waste. Sensor dynamics are likely to be less important than static characteristics and other cost benefit considerations in most cases, as the process dynamics are seldom challenging to the sensor technologies used.

2.1 – <u>On-Line Instrumentation</u>

2.1.1 Flow, Temperature, pH and ORP

Instruments to monitor gas and liquid flows are ubiquitous in wastewater treatment. For example, Harremoës *et al.* (1993) provided an extensive overview of liquid flow measurement techniques and pointed out the importance of proper installation for guaranteed accuracy. Measurements are based on pressure differentials resulting from restrictions (venturi, orifice plates, and meshes) placed in the flow path. In addition, electromagnetic and ultrasonic sensors can also be applied.

Temperature is a rather important variable for anaerobic digesters and temperature control is often implemented. Three commonly used types of process measuring instruments are available for measuring temperature: resistance thermometer, thermo-element, and thermistor.

It is normal practice to install pH electrodes in a treatment plant. Immersion of these probes in ‘sticky’ sludge has encouraged the development of different cleaning strategies: hydraulic (water spray), mechanical (brush), chemical (rinsing with cleaning agent) or ultrasonic cleaning. With these techniques, longer periods without maintenance can be attained. Poor or no automatic cleaning may indeed cause problems and self-diagnosis has been integrated in advanced systems. More sophisticated set-ups include automated checks of the impedance of the diaphragm and the glass electrode, while tests performed during (automatic) calibration may be used to indicate other sensor deficiencies. Although pH is a variable that is important in all biological processes, its value is especially critical in anaerobic digestion, eventually leading to acidification and process failure. Hence, its measurement and control are important. However, in the case of wastewaters with high buffering capacity, pH measurements may be rather insensitive to indicate process changes and are therefore not advisable for process supervision and control. In such cases, they may be replaced with bicarbonate and/or alkalinity measuring systems (Di Pinto *et al.* 1990; Hawkes *et al.* 1993 and Guwy *et al.* 1997 – see also section 2.1.3).

Oxidation-reduction potential (ORP) sensors are also sometimes installed since an increase in ORP indicates a possible presence of oxygen in the process. In this respect, it is recommended to maintain an ORP potential below -300 mV relative to a standard hydrogen electrode (depending on the wastewater characteristics) in order to not adversely affect anaerobic methanogenic archaea activity. ORP is also sometimes used to monitor sulfate reduction in digesters and H_2S in the biogas through micro-aeration (Nghiem *et al.* 2014).

As biogas formation rate is one of the most commonly monitored variables in anaerobic digestion processes, gas flow sensors are very often part of digester instrumentation. Pressure measurements can be found in AD plants as well, especially for alarm functions.

2.1.2 Biogas Composition

Gas composition measurements are also required in lab processes and full-scale plants. Typically, specific gas analyzers monitor the content of a component directly and infrared absorption measurements are used to determine carbon dioxide and methane concentrations. There are several of such sensors available today in the market. It has to be kept in mind that, although not always straightforward to predict from measurements in the gas phase, the corresponding concentrations of gasses in the liquid phase are important as they represent the environment the microorganisms operate in. It is possible to use Henry's law to calculate equilibrium aqueous concentration, however it is necessary to know the gas composition and the Henry's constant for each component at the required temperature and in aqueous solutions of variable ionic strength. Also, gas-liquid partitioning in digesters is very dynamic and equilibrium conditions may not be present.

The presence of hydrogen sulphide in the gas and the explosive character of biogas also require careful precautions. Hydrogen sulphide measurement in the gas phase may be performed by monitoring the reaction of sulphide with a Pb-strip. Subsequently, the black PbS that is produced is quantified by colorimetry. No direct on-line measurement of hydrogen sulphide in the liquid phase has been reported though. Membrane inlet mass spectrometry (Ryhiner *et al.* 1992) is another method to directly measure a large number of dissolved gasses and volatile compounds. The MS membrane probe response is often linear over very large concentration ranges. For application of thin membranes – that are required for sufficiently fast response and high sensitivity – the analyzer should be protected because of the rather high risk of membrane rupture. A fast safety shut-off system including fast pressure measurement is thus advised to be installed.

Specific hydrogen (H_2) analyzers have been developed – mainly in laboratories – based for example on electrochemical cells (Mathiot *et al.* 1992). Immersible sensors have been developed to measure dissolved hydrogen concentrations directly in the liquid phase down to partial pressures of 1 Pa (10^{-5} atm). Their reliability and long-term stability have been reported (Pauss and Nyns 1993). An inexpensive amperometric dissolved hydrogen probe has been used to determine the onset of digester failure by substrate overloading (Cord-Ruwisch *et al.* 1997). The measuring principle is based on the oxidation of hydrogen at a platinum black electrode at an adjusted potential. The current flowing to the electrode is directly related to the hydrogen concentration in the bulk liquid but H_2S has to be trapped and removed before the biogas flows into the hydrogen monitor. Björnsson *et al.* (2001a) applied a hydrogen-sensitive palladium–metal oxide semiconductor (Pd-MOS) sensor in combination with a Teflon membrane for liquid-to-gas transfer for the detection of dissolved hydrogen and the monitoring of a laboratory-scale anaerobic digestion process, employing mixed sludge containing mainly food/industrial waste. The sensor gave valuable information about approaching process overload, and can serve as a good alternative for volatile fatty acids (VFA) monitoring. The sensor was stable and robust during 3 months of operation, and therefore it was concluded that hydrogen sulfide, which is known to poison the Pd-MOS sensor, could not penetrate the Teflon membrane.

2.1.3 Alkalinity

The incentive to measure the bicarbonate content of the mixed liquor indeed originates from the fact that imbalance in anaerobic digestion (due to the accumulation of volatile fatty acids, VFA) cannot easily be detected on the basis of pH measurements, especially when the alkalinity of the mixed liquor is high (Hawkes *et al.* 1993). Because the alkalinity is often mainly due to the bicarbonate buffer, it has been proposed since the early sixties that its measurement can be used in control strategies for anaerobic digesters (McCarty 1964). One way to do so is by titration. Such methods involve titrating the sample down to pH 3.5 to determine the bicarbonate content with a correction for the volatile fatty acids present (see for example Ripley *et al.* (1985) or Anderson and Yang (1992)). The method is based on quantifying the gaseous carbon dioxide evolved from the sample as it is acidified. The volume of gas may be measured in two different ways. The overpressure in a closed constant volume vessel can be measured, or the gas volume produced can be measured with a sensitive gas flow meter in a constant pressure system. During titration, interferences from other weak acid/base constituents cannot be excluded and overestimation of VFA may sometimes occur (Purser *et al.* 2014).

2.1.4 Volatile Fatty Acids

Total VFA concentrations have been monitored for a long time as process performance indicators. It gives fast and reliable information of process status compared to other common indicators such as pH, alkalinity, gas production, and gas composition (Ahring *et al.* 1992; Björnsson *et al.* 2001b; Boe *et al.* 2007). Automated bicarbonate and total VFA instruments based on titrimetry have been developed and applied in practice for some years – see for example Feitkenhauer *et al.* (2002) or Ruiz *et al*. (2005).

Compared to total VFA concentration, individual VFA (acetate, propionate, butyrate etc.) can provide more information of the process status. Several studies have highlighted the importance of individual VFA as an early warning of process imbalance (Boe *et al.* 2010; Pind *et al.* 2003; Pratt *et al.* 2003; Pratt *et al.* 2012; Van Ginkel and Logan 2005). Ahring *et al.* (1992) suggested the overall level of n-butyric and iso-butyric was the best indicator of process stress. Boe *et al.* (2010) advised propionate as the most persistent parameter which was effective indicator of stress status of the process. Individual VFA are easily measured off-line using GC or HPLC, provided that all particulate matter has been removed from the sample.

However, only a few studies reported the development of an on-line individual VFA monitoring system because when dealing with anaerobic waste treatment, the presence of particulate matter is often high. Ryhiner *et al.* (1993) used GC for on-line analysis of acetic, propionic, butyric, valeric, and iso-valeric in a UASB reactor treating whey powder solution. The sample was purified by membrane filtration, acidified by phosphoric acid, and injected into the GC column by an auto-sampler with a specially constructed flow-through vial. However, no performance data was shown for this system. Zumbusch *et al.* (1994) used a HPLC for VFA monitoring in a UASB reactor treating baker's yeast wastewater using an ultra-filtration module for sample purification. The main problem of this process was membrane fouling requiring a high level of maintenance of the filtration system. Pind *et al.* (2003) used a GC for on-line analysis of VFA in a CSTR reactor treating manure and sample purification employed a three step filtration; pre-filtration by a rotating filter inside the reactor, ultra-filtration by a membrane cartridge, and a mini-filter for final purification. The system showed good correlation with the off-line measurement. However, membrane fouling was still the crucial problem and the membrane needed to be cleaned every 15–18 h to obtain sufficient flow. Boe *et al.* (2007) developed a new method to measure individual VFA based on headspace gas chromatography (HSGC). The method applies *ex situ* VFA stripping with variable headspace volume and gas analysis by gas chromatography-flame ionization detection (GC-FID). In each extraction, digester sample was acidified with H_3PO_4 and $NaHSO_4$, and then heated to strip the VFA into the gas phase. The system has been tested for on-line monitoring of a lab-scale CSTR reactor treating manure for more than 6 months and has shown good agreement with off-line analysis.

2.1.5 Spectral sensors

Spectral techniques – UV/visisible spectroscopy (UV/vis), Mid InfraRed spectroscopy (MIR), Near InfraRed spectroscopy (NIRS) – are beginning to provide very useful information about the complexity of organic matter.
UV/vis spectroscopic probes in the range of 190 to 750 nm are often used in wastewater treatment plants to measure COD, TOC and NO_3-N (Sarraguça *et al.* 2009). Wolf *et al.* (2013) developed a UV/vis spectroscopic system for VFA measurement (1.1 $g.L^{-1}$ – 3 $g.L^{-1}$) in AD plants. An UV/vis probe from S::CAN was used in combination with a custom-built dilution system to monitor the absorption of fully fermented sludge. To validate the approach, on-line measurements have been taken at a full-scale 1.3 MW industrial biogas plant. Results showed that VFA concentrations can be predicted with an accuracy of 87%. Nevertheless, the necessary dilution system is a disadvantage compared to NIR and MIR spectroscopic systems.

NIRS presents great potential for monitoring the AD process. Holm-Nielsen *et al.* (2008) evaluated the use of NIRS technology on-line (Transflexive Embedded Near Infra-Red Sensor or TENIRS) to monitor a thermophilic digester treating manure and organic food industrial waste. Good correlation was obtained between on-line NIRS measurement of glycerol and VFA content in the anaerobic digester. Further works documented the potential to monitor VFA as well as VS in on-line installations at lab-scale and full-scale plants (Krapf *et al.* 2013, Jacobi *et al.* 2009).

Mid InfraRed (MIR) spectroscopy is another interesting technique to characterize waste organic matter. One major advantage against existing NIR sensors is that process variables such as VFA, total alkalinity (TA), NH_4-N and TS show distinctive peaks in the MIR spectrum between 1,800 and 800 cm^{-1}, which makes it easier to correlate peak intensity to actual concentrations. Provenzano *et al.* (2014) used Fourier Transform InfraRed (FTIR) and fluorescence spectroscopy to characterize the organic matter evolution during AD and composting of pig slurry. Steyer *et al.* (2002) also used for several years a FTIR spectrometer for on-line measurements of COD, TOC, VFA, total and partial alkalinity of an AD fixed bed treating industrial wine distillery wastewater. Spanjers *et al.* (2006) applied the same technique at a full scale plant for the on-line monitoring of VFA, COD, alkalinity, sulphate, and, since aerobic post-treatment was considered, total nitrogen, ammonia and nitrate concentrations. Based on these studies, Wolf *et al.* (2014) developed an on-line MIR system with an FTIR probe using Polychristalline-Infrared (PIR) fibres that allow for higher signal to noise ratio (S/N) ratios as well as longer fibres. Furthermore, a fully automated process interface for cleaning and recalibration was used in order to reduce maintenance to a minimum. Good calibration results were obtained for VFA (R^2=0.97, RMSE 0.372 $g.L^{-1}$), TA (R^2=0.99, RMSE=0.259 $g.L^{-1}$) and NH_4-N (R^2=0.99, RMSE=0.11 $g.L^{-1}$). In spite of all advantages and advances in infrared spectroscopic on-line measurement systems, two main challenges remain: (1) despite the great interest in infrared spectroscopy on organic matter characterization, this technique is not sensitive enough for structural interpretation of complex molecules and does not account for the bioaccessibility of organic constituents; (2) prices for infrared spectroscopic measurement systems, NIR and MIR, are still far too expensive to be widely used in AD plants, so that financial feasibility is mostly not provided.

2.1.6 Other On-line Instrumentation

Other examples of advanced instrumentation can be seen in electronic tongues and noses and microwave or acoustic chemometrics (Madsen *et al.* 2011). A gas chromatograph or mass spectrometer coupled to a sample preparation unit can also be used, but so far no full-scale applications for these methods have been reported.

Liquid phase electrical conductivity is defined as the ability of a solution to conduct electrical current and is directly proportional to ion concentrations. Moreover, it can be easily monitored on-line: a cell formed by two electrodes is placed in the sample and the current between both electrodes is measured by means of the application of a potential difference (Colombié *et al.* 2007). Conductivity measurements could bring very informative measurements for monitoring and control of AD processes since ion concentrations are mainly affected by both VFA and bicarbonate concentrations (Hawkes *et al.* 1994), two of the most reliable indicators of AD process performance. Several studies have been published on the feasibility of electrical conductivity sensors for bioprocess monitoring (see, for instance, Hoffmann *et al.* 2000; Varley *et al.* 2004; Aguado *et al.* 2006; Ellison *et al.* 2007). However, there is still a lack of knowledge regarding its applicability to AD processes, despite some applications in dark fermentation processes for H_2 production (Aceves-Lara *et al.* 2010).

2.2 – Off-Line Instrumentation

With long HRTs or SRTs, off-line characterization of the waste and biomass can be considered as a way to provide operators with useful information to optimize AD plants, even though the data are yet not on-line. Several techniques exist and they are presented below.

2.2.1 Global characterization methodologies

From an analytical point of view, the performance of AD in wastewater or waste treatment is traditionally evaluated using parameters such as chemical oxygen demand (COD), total organic carbon (TOC) and biochemical oxygen demand (BOD). In order to optimize plant design and operation, Raunkjær *et al.* (1994) proposed to link COD fractions and biodegradability. Kayhanian (1995) showed that the content of biodegradable volatile solids (VS) impacted the prediction of biogas production rate and the computation of the organic loading rate and the carbon/nitrogen (C/N) ratio. Since the seventies, the most widely used indicator to assess the performance of digesters has been the amount of methane produced per unit of total solid (TS) or volatile solids (VS) of any given substrate (Chynoweth *et al.* 1993).

2.2.2 Biodegradability and organic matter characterization

One of the key issues in operating and optimizing AD plants is to assess the quantity of methane that can be produced from an organic residue. To this end, the most commonly used method to measure anaerobic biodegradability is the biochemical methane potential (BMP) test (ISO EN 11734 1995).

BMP Data and Use for Process Modeling

The BMP assay is a procedure developed to determine the methane yield of an organic material during its anaerobic decomposition by a mixed microbial community in a defined medium. The procedure was developed for a serum-bottle technique by Owen *et al.* (1979). Angelidaki and Sanders (2004) described the procedure and the calculations. The test ends when the cumulative biogas curve closely approaches an asymptote, usually after 30 days of incubation but it may be much longer for non-easily degradable material such as fibers. Therefore, the main inconvenience of the test is the long time required in its execution. Other negative points are the variability of the results obtained through the BMP tests and their ability to predict continuous digester performances. Concerning the first point, several studies made inter-laboratory assays to compare the BMP test results. Kinetic rates were widely different among different participating laboratories, standard deviations ranged from 57% to 68% (Jensen *et al.* 2009). The relative standard deviation of BMP values ranged from 15% to 24% and decreased to 10% when outliers were not considered (Raposo *et al.* 2011). Currently, only one inter-laboratory (French Inter-laboratory assay 2013-2014) proposes new guidelines and protocol after 2 test rounds achieved on solid substrates. This last study has shown good intra-laboratory repeatability (equal to 4%), reproducibility (between 5 and 7%) and reproducibility (between 13 and 21%) – see Cresson *et al.* (2014).

Concerning the second drawback, according to Jensen *et al.* (2009), the biodegradability and the bioaccessibility of hydrolysis-limited substrates could be defined by the parameters B_0 and k calculated from the Gompertz equation applied to a BMP curve (cumulative methane production versus time), $B = B_0 \times (1 - e^{-kt})$, where B is the cumulative methane production, B_0 is the maximal methane production and k is the hydrolysis rate constant. However, the authors discuss the conservative feature of these parameters measured in a BMP test. Several opinions are found in the literature concerning the use of B_0 and k parameters obtained in batch tests in order to model continuous digesters (see, for example, Val del Rio *et al.* 2011; Nielfa *et al.* 2015; Strömberg *et al.* 2015). Batstone *et al*. (2009) found that the BMP test's parameters should not be used for dynamic modelling of continuous digesters. While the final value of BMP was found to be consistent with continuous data, these authors found that the hydrolysis rate parameter value was lower in a BMP test than in a continuous digester treating thermally a waste activated sludge (*i.e.* 0.15-0.25 d^{-1} versus > $5d^{-1}$). According to Labatut *et al.* (2011), the BMP test is not suitable for predicting methane production kinetics for continuous digesters because it is conducted under diluted conditions, so preventing any inhibition response from being observed. Nevertheless, Jensen *et al.* (2009) found that the batch test was slightly conservative in terms of estimating degradability and rate, when applied to slowly degradable substrates such as waste activated sludge. Fannin *et al.* (1987) concluded that the maximum theoretical methane yield determination was useful to evaluate digester performance and to provide basis for experimental work. On the other hand, biodegradation tests performed sequentially in batch reactors using a slightly different protocol than the one used in BMP tests (Ganesh et al. 2013) were shown to be very informative in assessing the biodegradation kinetics of a broad spectrum of biowaste (García-Gen *et al.* 2015).

More Rapid Prediction of Methane Potential

Over the years, several authors developed relationships between the organic matter composition and the methane production or the anaerobic biodegradability. Static models are correlations (obtained by linear regression or partial least square (PLS) regression) where the parameters of interest are expressed as a function of one or more variables based on some analytical composition of the given substrate. Static implies neither kinetic equation nor variation over time. Three kinds of static models appeared in the literature to predict biodegradability of solid organic waste. Table 1 summarizes the comparative analysis, including benefits and drawbacks, of the different characterization methodologies involved in the integrative tools.

- Initial biogas production modelling

Some authors used the initial rate of biogas production modelling in order to predict the final value of BMP (Donoso-Bravo et al. 2011; Strömberg et al. 2015). For example, based on a database, Strömberg et al. (2015) proposed an algorithm to predict the BMP value from incubation experimental data operated during 6 days with an error less than 10%. Donoso-Bravo et al. (2011) used similar technique with incubation during 3-4 days. However, the modelled methane production of a continuous digester was underestimated by 20% with these parameters.

- Organic matter characterization

Over the last two decades, several authors also tried to build other static integrative tools based on organic matter characterization but they were mainly applied to municipal solid waste (Buffiere *et al.* 2006), kitchen, fruits and vegetables wastes (Gunaseelan 2007; 2009). Few studies dealt with municipal sludge although the methodologies used on solid waste can be transposed to sludge. The most recent publications have been presented by Mottet *et al.* (2010), Appels *et al.* (2011) and Jimenez *et al.* (2014).

First, the theoretical BMP obtained from the empirical formula has been calculated since 1930 using the Buswell equation (Neave and Buswell 1930). This stoichiometric equation is based on the elemental composition ($C_nH_aO_b$) where organic matter is reduced to methane and oxidized into carbon dioxide, with the assumption of a total conversion. However, these relationships remain theoretical and they assume that organic matter is fully converted. They did not consider (i) the fraction of substrate used for bacterial growth, (ii) the refractory organic matter (such as lignin) contained in the substrate, (iii) the fraction of the organic matter remains inaccessible due to binding within particles and (iv) the limitation of nutrients (Angelidaki and Sanders 2004). Several authors showed that biodegradability was overestimated using this technique (Shanmugam and Horan 2009, Labatut *et al.* 2011). Additionally, when applied to municipal solid waste, Davidsson *et al.* (2007) showed that theoretical methane potential is more realistic when the calculation is based on biochemical composition (lipids, carbohydrates, proteins) rather than on elemental composition analysis.

From Table 1, correlations obtained depend on the nature of different waste molecules. For example, fiber characterization would be more suitable for lignocellulose-like substrates such as green wastes, fruits and vegetables wastes (Buffiere *et al.* 2006) than for sewage sludge. Indeed, Mottet *et al.* (2010) applied the Van Soest fractionation (Van Soest 1963) to characterize organic matter from municipal sludge in order to build a biodegradability indicator. The error for the validation of the Partial Least Square (PLS) model was about 35%. Van Soest fractionation targets fibers and carbohydrates (i.e. cellulose, hemicellulose, lignin) but sewage sludge are also composed of proteins, humic acids and lipids (Jimenez *et al.* 2013). In the second part of their work, the authors found a better correlation between anaerobic biodegradability and the specific biochemical fractions of organic matter, such as proteins, carbohydrates, lipids and the degree of oxidation of organic molecules. Only Gunaseelan (2007; 2009) considered fibers, carbohydrates, lipids and proteins.

Concerning biomolecules characterization, several methods exist and are summarized in the Table 2. Initially conceived to analyze proteins, lipids and carbohydrates in serum samples, colorimetric methods have been applied in environmental engineering to characterize organic fractions. They are now coupled with analytical improvements such as organic matter extraction techniques (Park and Novak 2007; Ras *et al.* 2008). Table 2 summarizes some of the available methods used to determine the main components of organic matter. Depending on the nature of the substrate (total sludge or EPS solubilized in an extracting agent) the methods are more or less adequate (Jimenez *et al.* 2013). Recently, several reported works used a more advanced methodology: gas chromatography with mass spectroscopy (GC/MS) was used in order to determine the detailed composition of carbohydrates, proteins and lipids present in the sample. Huang *et al.* (2010) used this technology for wastewater characterization.

- Aerobic tests

Indirect correlations between aerobic activity tests and anaerobic tests such as BMP are also often proposed. Aerobic tests are less time consuming than anaerobic tests and they can be easier from a practical point of view (e.g. no need for anaerobic conditions and precautions working in an air environment). Although the respirometric test takes less time than the BMP test, there are some limitations in using it to determine the BMP. First, only the readily biodegradable organic matter is considered (the more complex organic matter, such as cellulose, are degraded more slowly and are not measured in the short-term test) (Lesteur *et al.* 2010). The second limitation is the assumption that the organic matter in sludge presents the same biodegradability under aerobic and anaerobic conditions (Ekama *et al.* 2007). Buendía *et al.* (2008) used long anaerobic and aerobic batch tests in order to estimate readily and slowly biodegradable fractions and found a good correlation between the anaerobic and the aerobic readily biodegradable fraction. However, the slowly biodegradable fraction was underestimated by the aerobic batch testing. In the same way, Park *et al.* (2008) showed some proteins bound to divalent cations were bioaccessible only under aerobic conditions but were not bioaccessible under anaerobic conditions. Higher volatile solids removal was observed under aerobic conditions (48%) compared to AD (39%).

Emerging Techniques for Organic Matter Characterization

Progress in analytical chemistry has led to the development of new instruments and techniques to characterize organic matter. Among them, NIRS and 3D fluorescence spectroscopy are the most promising for instrumentation and biodegradability measurement.

Recently, NIRS is used for BMP assessment following two different approaches. The first approach is to determine the composition of the input material using NIRS and to calculate the BMP value by regression using static models. The second approach to predict the biodegradability uses directly the spectra through a dedicated calibration. Jacobi *et al.* (2012) used both approaches for the determination of the biogas production from maize, which is commonly used in Germany. The calibration allowed errors for volatile solids of 0.74 % fresh matter and for biogas production of 5.26-11.14 l/kg fresh matter. Application of the technique for off-line prediction of continuously gathered data allowed, together with first order degradation kinetics, the prediction of the biogas production of a full-scale biogas plant over several months. Zhang *et al.* (2009) succeeded in building PLS models between NIRS results and ethanol, acetate, propionate and butyrate concentrations in a H_2 producing reactor fed on synthetic wastewater. Lignin concentration has also been correlated to NIRS measurement by Brinkmann *et al.* (2002). However, so far NIRS has not yet found its way into practical implementation at biogas plants. One obstacle seems to be the transfer of calibrations of a given sample set to new samples and the reliability of the predicted values.

Lesteur *et al.* (2011), Doublet *et al.* (2013) and Triolo *et al.* (2011) have successfully developed PLS models for BMP prediction of different waste organic matter BMP values using Near InfraRed Spectroscopy (NIRS). Lesteur *et al.* (2011) and Doublet *et al.* (2013) found a direct correlation between the NIRS analysis and the biodegradability provided by the BMP tests for municipal solid waste. The prediction demonstrated good accuracy (standard deviation of 28 $mLCH_4/gVS$ and relative error of 13% respectively). However, NIRS measurement for biodegradability assessment is still performed on dried-frozen samples and does not consider accessibility of the organic matter.

Another promising technique is the fluorescence spectroscopy. Fluorescence allows the characterization of the analyzed organic material in both liquid and solid phases. The technique gives a topographic map of the organic matter complexity. Identification of molecular-like groups is possible based on the excitation and emission wavelength coordinates (Jimenez *et al.* 2014). It is indeed a selective and sensitive method since fluorescence characteristics are related to the structure and the functional groups in the molecules. Some studies have revealed the potential of fluorescence spectroscopy to link to the complexity of a substrate and its biodegradability (Tartakovsky *et al.* 1996; Reynolds *et al.* 1997) and results on establishing a link between complexity, sludge stabilization degree and accessibility, were encouraging (He *et al.* 2011; Wan *et al.* 2012). Recently, Jimenez *et al.* (2014) proposed a sewage sludge characterization methodology to assess both biodegradability and bioaccessibility needed for modified ADM1 input variables and thus for further optimization of AD plants. These authors combined basic chemical extractions with 3D fluorescence spectroscopy in a 5 days long methodology and predicted successfully both parameters using a PLS regression model. A wide range of biodegradability (0-60%) and of readily/slowly biodegradable fractions (0-46%), representing bioaccessibility, were predicted with errors of 6% for both. However, this technique was specific to sewage sludge, as far as proteins compose the main part of the organic matter in this organic waste.

2.3 - Dynamical Models and Software Sensors

As previously presented, static models have been proposed as an alternative solution to predict biodegradability with several kind of organic matter characterization as explicative variables. However, all the static models were not able to predict simultaneously the bioaccessibility and the biodegradability as the digester dynamics.

Dynamical models accounts for evolution in kinetic equation and biomasses. This leads to more complex models generally based on ordinary differential equations representing mass balance within the process. The first dynamical AD digestion models were proposed in the mid-sixties by Andrews and Pearson (1965) and Andrews and Graef (1971). Only a single stage was considered gathering acidogenesis and methanogenesis. A Haldane kinetic equation was proposed to account for acetoclastic methanogenesis inhibition at high concentration of acetate. Mosey (1983) and Hobson (1985) extended the model with hydrogenotrophic methanogenesis. The models were then extended depending on the different substrates (wastewater, sludge or manure). More than 10 years ago, the IWA Task Group on Mathematical Modelling of Anaerobic Digestion Processes proposed the Anaerobic Digestion Model No1 (ADM1), as a consensual modelling of anaerobic digestion (Batstone *et al.* 2002). The biochemical reactions represented in the model describe: (i) an extracellular disintegration step converting composite particulate matter into carbohydrates, lipids, proteins and inert compounds, (ii) an extracellular enzymatic hydrolysis step that converts the degradation products into their chemical building blocks, *i.e.* LCFA, monosaccharides and amino acids, (iii) acidogenesis or fermentation into hydrogen, acetate and VFA, (iv) acetogenesis of VFA into acetate and (v) acetoclastic and hydrogenotrophic methanogenesis. The extracellular reactions are assumed to be of first-order, while the intracellular biochemical reactions use Monod-type kinetics for substrate uptake and biomass growth. Variants to the ADM1 model given by Batstone *et al.* (2002) are available for plant wide modelling (Rosén and Jeppsson 2006; Grau *et al.* 2007; De Gracia *et al.* 2009; Barat et al. 2012). Many applications of the ADM1 model have been published for a wide variety of substrates (see e.g. Batstone *et al.* 2009; Lauwers *et al.* 2013) and some models account for both the biodegradability and the bioaccessibility of the waste (Mottet *et al.* 2013, García-Gen *et al.* 2015). On the other hand, simpler models have been developed, more suitable to support monitoring or control strategies. For example, the model of Bernard *et al.* (2001a) includes two reactions and turns out to approximate efficiently the ADM1 model (Bernard *et al.* 2005b) for modeling AD processes treating industrial wastewater.

In many occasions, on-line or off-line measurements are not enough to evaluate and to assess the operating conditions of AD plants but, when combined with dynamical models, these measurements can lead to very useful additional information about non measured variables. This methodology leads to the so-called "software sensors". It is possible to distinguish the approaches based on data sets, those founded on expert knowledge (in the broad sense of the term) and those founded upon an analytical – mathematical – description of the system. In this section, we focus particularly on the estimation for the efficient development and implementation of state estimation schemes. These estimation schemes are called estimators, state observers, software sensors, or simply observers, and they can be used for design or optimization strategies in a wide class of biochemical processes. As underlined, these algorithms are able to estimate both state variables, that are normally not measured, and unknown parameters from the available measurements. In biological processes, observers are mainly useful in on-line estimations for control purposes. The most popular approaches used in the past have been the well-known classical extended Kalman filters (EKF) and extended Luenberger observers (ELO). One of the reasons for the popularity of EKF/ELO is that they are easy to implement since the algorithms can be directly derived from the state space model. However, since these estimators are based on a linearized model of the process, the stability and convergence properties are essentially local; it is difficult to guarantee its stability over a wide operating range. As a matter of fact, very few works deal with the observability of nonlinear biochemical processes (e.g., Gauthier and Kupka 1994) and they are usually concerned with particular process applications. Another problem is that the theory for EKF/ELO is developed assuming a perfect knowledge of the system model and parameters, in particular of the process kinetics, and as a consequence, it is difficult to develop error bounds to take into account the large uncertainty of these parameters.

In order to overcome these drawbacks, several other approaches have been proposed from the early seventies (Misawa and Hedrick 1989; Perrier *et al.* 2000; Dochain 2003; Alcaraz-González and González-Álvarez 2007). For example, adaptive observers (Bastin and Dochain 1990, Dochain 2003) belong to the class of observers allowing the estimation of both kinetic parameters and states. As in the EKF, the poorly known (or unknown) parameters are considered to be extra states with no dynamics. One of the original features of the adaptive observer is to consider a nominal process model, *i.e.* a model with nominal values of the poorly known parameters (Chen 1990). The design of nonlinear observers in general has been a very active research area. Most of the nonlinear approaches are placed in the category of ''high gain'' observers (HGO) since they tend to split the dynamics into a linear part and a nonlinear part and to choose the gain of the observer so that the linear part dominates the nonlinear one (Gauthier *et al.* 1992; Gauthier and Kupka 1994; Dochain 2003).

Several linearization methods also have been proposed (Baumann and Rugh 1986). Nevertheless, like EFK/ELO, only local behavior can be guaranteed as they miss practical results on performance and stability. Other approaches are sliding observers based on the theory of variable structure systems (Xiong and Saif 2003) but their design involve conditions that must be assumed *a priori* or that are usually hard to verify (Misawa and Hedrick 1989). All these approaches solve some of the problems described above but in most of the cases, the complexity of the resulting estimating algorithms is a limitation for real time computation. Indeed, monitoring algorithms can prove to be efficient if they are able to incorporate the important well-known information on the process while being able to deal with the missing information in a robust way. They include the lack of on-line measurements and the uncertainty on the process dynamics.

Two relatively new robust nonlinear observers have found a wide acceptation in biological process, including of course anaerobic digestion. Such robust observers are capable of coping simultaneously with the aforementioned problems while remaining easy to implement with a minimum number of straightforward conditions to verify. The first one, the asymptotic observer (Bastin and Dochain 1990; Alcaraz-González and González-Álvarez 2007), although requiring the knowledge of the process inputs, has the main advantage that it permits the exact cancellation of the nonlinear terms of the systems, and so facilitates its design, stability analysis and implementation. The second one, the interval observer, allows for the reconstruction of a guaranteed interval on the unmeasured states instead of reconstructing their precise numerical values assuming that only guaranteed lower and upper limits on the process inputs and model parameters are available (Gouzé *et al.* 2000; Alcaraz-González *et al.* 2005a; Rapaport and Dochain 2005; Moisan *et al.* 2009).

The main disadvantage of the aforementioned asymptotic observer is that the process operational conditions (mainly the hydraulic retention time) establish its convergence properties and it is not possible to modify the convergence rate by choosing a gain like in the classical observers or the HGO. However, adapting the design features of the HGO and adaptive observers, a Tunable Asymptotic Observer (TAO) has been proposed for AD processes (Bernard and Gouzé 2004, Alcaraz-González *et al.* 2005b). Furthermore, in a more diverse sense, super-twisting observers have also been demonstrated recently to be very useful in achieving a very fast convergence without loss of robustness, (Sbarciog *et al.* 2012).

Concerning the drawback of influent uncertainty – very common in AD plants –, the general problem of simultaneous estimation of unmeasured state variables and inputs for nonlinear systems has been addressed from a number of different robust approaches. With respect to AD processes, Theilliol *et al.* (2003) proposed a simultaneous input-and-state concentrations observer that required the full knowledge of the process kinetics. Also, Aceves-Lara *et al.* (2010) simultaneously estimated state space variables and the input concentrations in a biohydrogen production process in which input and state estimations were performed using a state transformation and an asymptotic observer. More recently, Jaúregui-Medina *et al.* (2009) proposed an observer-based estimator, named the “Virtually Controlled Observer” (VCO) because one of the observer's inputs (the hypothetical -unmeasured- influent substrate concentration) is updated by a feedback control that regulates the estimation error of a measured output. In a fixed bed configuration, several of these approaches have also been applied to distributed parameter systems (see e.g., Delattre *et al.* 2004; Aguilar-Garnica *et al.* 2009).

3. Control of Anaerobic Digestion Processes

Because of the inherent complexity and necessity for safety in biotechnological processes, efficient monitoring and decision support systems are required in order to optimize their operation. Indeed, even in normal operational conditions, several types of disturbances may occur with serious consequences in the performance of the process. Fluctuations in the influent to be treated is an illustration and a typical example would be an integrated dairy producing 100 different products that, over the course of a week, result in a wastewater stream with flow/total COD/TSS/FOG/temperature variations of 20x/10x/5x/3x/1.5x, some of these changes taking place in a matter of hours. Hence, the last two decades have seen an increasing interest to improve the operation of AD processes by applying advanced control schemes. Optimized and stable performances are indeed required to be guaranteed consistently and this has major consequences for instrumentation, control and automation (Huntington 1998; Olsson and Newell 1998). Two main factors (which can be interpreted as both, incentives and constraints) have contributed to this new paradigm: (1) the need for optimally controlled plants due to environmental regulatory norms and (2) the need to reduce cost. In order to fulfill these requirements, the optimal control of AD processes faces important uncertainties arising from the intrinsic complexity of plant design. Among others, the main disturbances that can be observed are the following: acidification, inhibition and toxicant exposure (McCartney and Oleszkiewicz 1991; 1993; O’Flaherty *et al.* 1998**;** Hao 2003; Appels *et al.* 2008; Chen et al. 2008; Cirne *et al.* 2008), overload (Waewsak *et al.* 2010; Wijekoon *et al.* 2011), alkalinity, variability of inputs, water content and rheology, foaming, stirring and mixing problems (McMahon 2001; Dalmau *et al.* 2010) and lack of macro- and micro-nutrients (Speece 2008).

By far, the most developed control laws in the literature use the dilution rate as manipulated variable (see Figure 1) but it is mainly in simulation and only few full-scale applications are available. Manipulating the dilution rate is indeed difficult in practice and AD processes are facing the problem of the lack of actuators. Examples for other manipulated variables are liquid recirculation rates and the addition of bases to stabilize the process. In case of a co-digestion plant, only one substrate or a constant substrate mix is usually controlled using the dilution rate as manipulated variable. The other substrates then must be calculated based on boundary conditions such as hydraulic retention time, organic loading rate or restrictions defined by funding schemes (Zhou *et al.* 2012).

Whilst experimentation is required for the tuning of regulators, either on the plant itself or within a simulation environment, design techniques have been developed that allow devising the optimal controller for a particular process model and performance index. Certain constraints imposed on the control action, such as a minimization of the control effort, can be accommodated during the design.

3.1 – Classical control in AD

PID and on/off controllers belong to classical control methods. Table ESM.1 and ESM.2 in Online Resource 1 illustrates some examples of application of these control methodologies in AD.

The first application of on/off control in AD was reported in the 70s (see Table ESM.1 in Online Resource 1), which aimed at setting the manipulated variable to a binary value depending on predefined threshold values. They were followed by PID controls including P, PI, and PID controls. For instance, Marsili-Libelli and Beni (1996) applied PID control for stabilising alkalinity and pH by manipulating the addition of bicarbonate. On the other hand, von Sachs *et al*. (2003) proposed a PI structure for controlling biogas flow rate by modifying the dilution rate in a two-phase AD system.

PID cascade controls (see Table ESM.2 in Online Resource 1) are a simple but effective approach for feed control. Their advantages are that two possibly conflictive set-points can be simultaneously controlled whilst the set-point of the master loop can be set by an expert system. Approaches such as Liu *et al*. (2004a; 2004b), Alferes *et al*. (2008), and Alferes and Irizar (2010) are dedicated to control biogas production at a given set-point or to operate the digester at high organic load. Therefore, these approaches try to maximize the economical benefit of the digester, whereas the set-point is established in order to avoid digester overloads.

As regards adaptive control, Zhou *et al*. (2012), for instance, proposed a PID aimed at controlling the methane flow rate based on measurements of VFA and VFA/TA.

Another control strategy lies on minimizing the COD or VFA content in the effluent (see e.g. Alvarez-Ramirez *et al.* 2002; Mu et al. 2007). The key goal of control strategies of this type is to stabilize digester performance whilst maximizing COD degradation. On the other hand, García-Diéguez *et al*. (2011) proposed an approach capable to maximize methane flow rate whilst tracking a set-point for effluent VFAs.

3.2 - <u>Advanced control in AD</u>

Since classical PID controllers are usually limited to single-input-single-output control loops and to linear, simple cases, different advanced control approaches have been theoretically analyzed and experimentally validated in order to control AD processes.

3.2.1. Expert systems

Expert systems can be classified in rule-based and fuzzy systems (Tables ESM.3 and ESM.4 in Online Resource 1) and systems extended with a surrogate model such as an artificial neural network or special fuzzy systems (Table ESM.5 in Online Resource 1).

Applying nonlinear control methods comes quite natural since biogas plants are nonlinear processes. Such expert systems are quite popular for AD control because of: 1) their intuitive design based on rules, and 2) their non-linearity coping with the non-linearity of the plant. The first approach is performed by rule-based systems such as the well-known fuzzy control, whilst the latter one is performed by the use of neural networks. Furthermore, expert systems can easily incorporate all measured variables and are easily extensible if an additional process value is measured in the future.

Fuzzy logic is a problem-solving tool that can achieve a definite conclusion from imprecise information, allowing intermediate values rather than simple yes/no evaluations (García-Gen 2015). The main benefit of this approach is that it can be used to control non-linear systems. A fuzzy-logic controller (Zadeh *et al.* 1965) is indeed capable of optimizing different kinds of processes under dynamic operating and loading conditions by applying valuable expert knowledge (Verbruggen *et al.* 1997). Moreover, fuzzy-logic control does not require a large amount of data and/or a rigorous mathematical model, and allows for the development of multiple-input-multiple-output control schemes. Hence, fuzzy logic is a powerful tool for AD control (Olsson *et al.* 2005).

Different examples of rule-based and fuzzy-logic-based systems for AD control can be found in literature (see Tables ESM.3 and ESM.4). For instance, Pullammanappallil *et al*. (1991; 1998) developed an expert system aimed to control methane production by switching between different control strategies (set-point control, constant yield control, batch operation and constant dilution rate) based on a t-test. Puñal *et al*. (2003) proposed a PI-based fuzzy logic system for monitoring the effluent VFA concentration in anaerobic wastewater treatment plants, using the dilution rate as manipulated variable. Murnleitner *et al*. (2002) and Grepmeier (2002) proposed expert systems based on fuzzy theory for overload avoidance in AD process. Different inputs were used for such purpose: H_2 concentration, CH_4 concentration, biogas flow rate, pH, and filling level of the buffer tank.

Table ESM.5 in Online Resource 1 summarises different examples of expert systems for AD control consisting of neural networks and special fuzzy systems. For instance, Steyer *et al*. (1997) proposed a hierarchical fuzzy control for VFA concentration which used the control error of pH, temperature and biogas flow rate as input variables. Holubar *et al*. (2002; 2003) used a neural network to maximize methane production and COD degradation by modifying OLR on the basis of different inputs: pH, VFA, and biogas production and composition. Carlos-Hernandez *et al*. (2007) developed a fuzzy supervisory controller to optimise process performance by regulating the addition of base and the dilution rate; whilst this control system was later modified (Carlos-Hernandez *et al.* 2010) following a neural fuzzy structure for estimating methanogenic biomass performance.

3.2.2. Model-based and linearizing control

Linearizing approach is popular for feed control purposes in AD (see Table ESM.6 in Online Resource 1). Moreover, much effort has been applied to develop new model-based control laws that will achieve suitable process performances (Méndez-Acosta *et al.* 2010). In this context, simple models like AM2 (Bernard *et al.* 2001b) are preferred to more complex ones like ADM1 (Batstone *et al.* 2002).

Linearizing control is based on a non-linear controller, which is precisely designed to achieve linear closed-loop dynamics (Isidori *et al.* 1989; Ignatova *et al.* 2008). The main aim of linearizing control is to take advantage of available mathematical models. They allow controlling very efficiently the functioning of a plant and may allow the achievement of finer actions than those controllers that decide only upon the difference between measurements and set points (Olsson *et al.* 2005). Linearizing controllers are designed by a two-step procedure (Kurtz *et al.* 1997). First, a non-linear process model is used in order to synthesize the non-linear state feed-back controller that linearizes the map between a “new” manipulated input and the controlled output. In the second step, a linear pole placement controller is designed for the feed-back linearized system. However, due to the strongly non-linear relationships existing between both inlet and outlet of an anaerobic process, linearizing controllers only attain proper results when the process dynamics are bounded by a defined linear zone (Simeonov and Queinnec 2006).

Applications of adaptive linearizing control have been presented for anaerobic digestion (Renard *et al.* 1988). However, an important problem with adaptive control systems is the necessity for on-line identification of the process model while the plant is in closed-loop operation. An approach to deal with the identification problem consists of considering that the process model belongs to a bounded class of possible models with fixed parameters. The identification is then reduced to the choice of the correct model, or, as in the Model Weighting Adaptive Control (MWAC) approach (Gendron *et al.* 1993), by weighting the different models into a composite process model.

Another method in this category is the interval-based approach. Concerning Interval Observers, a recent control approach that uses the partial information provided by this kind of observers has been designed to exponentially stabilize a regulated variable in a neighborhood of a predetermined set-point (Rapaport and Harmand 2002). As for observers, these approaches have been also applied to distributed parameter systems applied to fixed-bed bioreactors (e.g., Dochain *et al.* 1997; Babary *et al.* 1999; Antoniades and Christofides 2001; Aguilar-Garnica *et al.* 2009).

Some other recent approaches for control of this kind of processes have been derived from the theory based on differential geometry (Isidori 1989; Henson and Seborg 1997). Control approaches based on differential geometry allow for the transformation of a nonlinear system into a partially or totally linear one, by means of a nonlinear state transformation, which is obtained from directional derivatives of the output. It is important to remark that geometric control differs totally from the linear approximation of dynamics by calculation of the Jacobian. Either state-space (Hunt and Su 1983) or input-output linearization (Méndez-Acosta *et al*. 2004; 2005; 2008) have been employed.

More recently, sliding mode approaches have been also used mainly to control Anaerobic Sequential Batch Reactors (ASBR), (Vargas *et al.* 2008), as well as in continuous bioreactors (Lara-Cisneros *et al.* 2015). In general, the sliding mode approaches are widely used due to robustness with respect to uncertainties.

3.2.3. Other advanced controllers

Table ESM.7 in Online Resource 1 summarizes other advanced control approaches, including, for instance, disturbance monitoring, non-linear, adaptive, and robust control.

A nonlinear adaptive control law for bioreactors which is robust in the face of unknown kinetics has been proposed recently for the global stabilization of bioreactors and then applied to the regulation of anaerobic digestion processes (Mailleret *et al.* 2004). Similar to linearizing control, different interval-based approaches have been used to exponentially stabilize a regulated variable in a neighborhood of a predetermined set-point (Alcaraz-González *et al.* 2005a).

On the other hand, most of the controllers reviewed before were developed to regulate known set-points or to track well defined trajectories. However, in AD operation, the control objective could be to optimize a criterion that is dependent of unknown parameters in order to keep a performance criterion at its optimal value. Also, it is well known that the explicit form of the performance function in AD processes is highly uncertain (e.g., the growth rate of methanogenesis or growth rate of acidogenesis) (Lara-Cisneros *et al.* 2015). Extremum-Seeking-Control (ESC) and probing control are two techniques to handle these kinds of dynamic optimization problems (Dochain *et al.* 2011; Guay *et al.* 2004; Liu *et al.* 2006; Marcos *et al.* 2004a; 2004b; Steyer *et al.* 1999). The goals of ESC schemes and probing control is to find the operating setpoints, *a priori* unknown, such that a performance function reaches its extremum value. Steyer *et al.* (1999) developed a probing control approach based on the analysis of disturbances added on purpose to the influent flow rate. By increasing the influent flow rate for a short period of time, the increased biogas yield was compared to the expected one. Overloading or inhibition could be interpreted as a negative effect of the disturbance (i.e. an unsatisfactory gas yield). Liu *et al.* (2006) developed a cascade controller system that is embedded into a rule-based supervisory system based on ESC. This controller was applied to intensify biogas production in an anaerobic up-flow fixed bed reactor at laboratory scale and achieved good performance, especially during the early startup and during rejection of disturbances. In particular, the process was operated at maximum productivity and had safety margins adequate to ensure reliable operation, react fast on disturbances and avoid unstable process conditions. Lara-Cisneros *et al.* (2015) proposed an ESC scheme with sliding mode to achieve the dynamic optimization of methane outflow rate in anaerobic processes. The control law was designed to regulate VFA concentration at the optimal value whilst maximizing methane production. However, only numerical experiments illustrated the performance and robustness of the proposed control approach.

Concerning the need of sensors for control purposes, even if there now exists a large variety of devices for measuring almost all key variables, they still remain relatively expensive for medium and small enterprises, mainly in developing countries. In this sense, the challenge to control AD processes is to do it with a minimum of information, even if it is obtained off-line. In this context, discrete control approaches are beginning to be used (Méndez-Acosta *et al.* 2011).

3.2 - Control in Anaerobic co-Digestion (AcoD)

Anaerobic co-digestion (AcoD) presents higher potential energy recovery than conventional single substrate AD. Therefore, high effort has been focussed on AcoD in order to: 1) enhance process performance thus maximising biogas production; 2) navigate into the use of new co-substrates; and 3) increase process feasibility by the application of digestates for agricultural purposes (Mata-Alvarez *et al.* 2014).

For instance, biogas production has been classically improved by co-digesting manure and organic waste (see, for instance, Ahring *et al.* 1992; Tafdrup 1994). Since manures are often associated with poor methane yields, AcoD of manure with other organic wastes has been identified as a cost-effective alternative for improving process efficiency (Mata-Alvarez *et al.* 2011; Frigon *et al.* 2012; Astals *et al.* 2013b)). This co-digestion process is usually optimised when biogas yield is above 30 m^3 biogas per m^3 biomass treated, which normally requires a 25% organic waste ratio (Boe 2006). Nevertheless, lower ratios may be enough when treating concentrated wastes (Gregersen 2003).

Other classical AcoD process is the co-digestion of sewage sludge with the organic fraction of municipal solid waste (OFMSW). Besides the biowaste composition (food waste, market waste, etc.), biogas production during the co-digestion of sewage sludge and biowaste highly depends on several factors such as sewage sludge composition (primary, secondary or mixed), OLR, reactor configuration, operating temperature or mixing conditions (Mata-Alvarez *et al.* 2011). For instance, Silvestre *et al.* (2015) assessed the effect of OFMSW loading rate and particulate size on sewage sludge mesophilic anaerobic co-digestion in a CSTR operating at 20 days of SRT. This study revealed that sewage-sludge–OFMSW mixture composed by 54% of inlet volatile solids (OLR of 3.1 kg COD m^{-3} d^{-1}; 1.9 kg VS m^{-3} d^{-1}) resulted in an increased in volumetric methane production and methane yield of up to 200 and 59%, respectively.

Recent literature has reported increasing interest by the scientific community on the applicability of AcoD to new biowastes. For instance, co-digesting sewage sludge and microalgae is considered one promising technology for energy production, whilst representing a key step for recycling nutrients for algal cultivation (Ward *et al.* 2014). Recent research has shown that AcoD can increase anaerobic degradability of algae by improving substrate composition. Nevertheless, further research is needed since the quantity and quality of the produced biogas vary considerably depending on anaerobic inocula, waste composition and operating conditions (Ajeej *et al.* 2015).
The control of AcoD processes can be addressed following the same strategies used for classical AD processes. However, it is crucial to characterise comprehensively the co-substrates and to choose adequately the blend of substrates to be treated (García-Gen 2015).

Alvarez *et al*. (2010) developed a methodology for optimising feed composition in AcoD of agro-industrial wastes. This optimisation protocol was based on a linear programming method aimed to set up different blends for maximising the total substrate biodegradation potential ($L_{CH4} \cdot kg^{-1}$ substrate) or the biokinetic potential ($L_{CH4} \cdot kg^{-1}$ substrate $\cdot$ d^{-1}). To this aim, the controller defined restrictions on several characteristics of the mixture, such as NH_4^+, lipids or C/N ratio. The methodology was validated using three types of agro-industrial biowaste: pig manure, fish waste and biodiesel waste. Validation results were related to the mixture of biowaste to be feed to the AcoD process in order to maximise biodegradation potential and methane production. Linear programming was proved to be a powerful, useful and easy-to-use tool to estimate methane production in co-digestion units where different substrates can be fed (Alvarez *et al.* 2010).

Wang *et al.* (2012) proposed optimizing the feeding composition and the carbon/nitrogen (C/N) ratio for improving methane yield during AcoD of multi-component substrates (dairy, chicken manure and wheat straw). The results showed that co-digestion performed better than individual digestion in terms of methane potential. Maximum methane productions were achieved with a dairy/chicken manure ratio of 40:60 and a C/N ratio of 27:1 (after optimization using response surface methodology). The results suggested therefore that better performance of AcoD can be fulfilled by optimizing feeding composition and C/N ratio.

Wang *et al*. (2013a) evaluated two statistical methods for optimizing feeding composition in AcoD systems. To this aim, a simplex-centroid mixture design (SCMD) and central composite design (CCD) were evaluated using methane potential as response variable. Each co-substrate (dairy manure, chicken manure, swine manure and rice straw) served as an independent variable in SCMD and CCD, involving two factors: the manure and C/N ratios together with the C/N ratio of the blend. Experiments demonstrated that co-digestion of three-component substrates resulted in higher methane potentials, as well as on better fitted models to predict the response based on selected variables. In response surface plots, SCMD showed the interactions among each component in the co-substrates and CCD presented the interaction between the ratio of manures and the C/N ratio. SCMD and CCD were both suitable methods for optimizing feeding composition during anaerobic co-digestion.

Jiménez *et al*. (2014) optimised methanogenic activity using the response surface methodology during the AcoD of agriculture and industrial wastes. This optimisation accounted for microbial community performance, taking into account the effect of each substrate concentration and their interactive effects on specific methanogenic activity and microbial community diversity. The results showed a significant interaction among the substrates and an enhancement of the methane production and specific methanogenic activity. The optimization allowed identifying substrate interaction effects in a concentration range with a reduced number of experiments. The model validation proved to be useful for defining optimal combination of wastes in AcoD systems.

García-Gen *et al.* (2015) proposed a control strategy for optimising AcoD in terms of methane productivity, digestate quality and process stability. To this aim, a linear programming approach was adopted to calculate the feeding of multiple substrates for maximum methane productivity, taking into account restrictions based on experimental and heuristic knowledge. Alkalinity ratio measurements against reference values were used for quantitatively assessing process stability by using an empirical diagnosis function. A second empirical diagnosis function was defined to compare methane flow rate measurements against a reference value of maximum capacity. The quantitative change applied to the most active constraint of the substrate blend optimisation problem (leading to a new set-point of feeding substrates blend) was calculated by a variable-gain control function derived from the previously commented diagnosis functions. This closed-loop control architecture was successfully validated in a 1 m^3 hybrid Upflow Anaerobic Sludge Blanket – Anaerobic Filter (UASB-AF) reactor, treating blends of substrates (gelatine, glycerine and pig manure supernatant) at OLR values between 0.71 and 6.33 $gCOD \cdot L^{-1} \cdot d^{-1}$. The proposed controller was capable to increase methane productivity whilst recovering the system from transient acidifications.

3.3 - <u>Sulphide Control</u>

Different control strategies can be applied to minimize problems related to sulphide in the system (Cirne *et al.* 2008). The monitoring of sulphate in the influent cannot be considered as a realistic option since sulphate concentration in the influent cannot be predicted nor monitored. Final removal of sulphide (e.g. desulphuration of biogas) is based on the application of different physico-chemical or biological techniques sometimes requiring additional treatment units:

- selective inhibition of SRB using compounds such as nitrite, antibiotics, or molybdate. However, these actions are not very effective when operating continuous AD processes and they also present a negative effect on MA.
- pH increase in order to move the H_2S/HS^- equilibrium towards less toxic HS^-.

- sulphur precipitation using organic or inorganic compounds (mainly iron salts). The main drawbacks of this technique are the reagent cost, the increase in sludge production and possible pipes obstructions from precipitates.
- H_2S stripping by high stirring in the reactor, recycling the produced biogas after scrubber or other H_2S removal technologies.
- oxidation of sulphide with oxygen or nitrate using chemical or biological processes. This process consists of introducing small amounts of these compounds without affecting process performance (van der Zee *et al.* 2007; Cirne *et al.* 2008; Fdz-Polanco *et al.* 2009a; 2009b).

3.4 - Control of Anaerobic Membrane Bioreactors (AnMBR)

Several operating strategies to control membrane fouling in anaerobic or aerobic membrane reactors have been experimentally validated. For example, Jeison and van Lier (2006) developed an on-line cake-layer management protocol that monitored critical flux constantly and prevented excessive cake-layer from building up on the membrane surface; Smith *et al*. (2006) developed a control system to optimize back-flushing which reduced the water needed for back-flushing by up to 40%; Vargas *et al*. (2008) established a control algorithm for fouling prevention which regulated back-flushing and Park *et al.* (2010) studied how membrane fouling could be reduced by successively increasing and decreasing membrane gas sparging intensities, and recorded the effectiveness in reducing membrane fouling.

Anaerobic membrane bioreactors (AnMBR) can be very efficiently used to treat urban wastewater but they require more sophisticated process control systems than for aerobic MBR systems or other conventional anaerobic systems – such as up-flow anaerobic sludge blanket (UASB); expanded granular sludge blanket (EGSB); or anaerobic filters (AF). For example, Robles *et al.* (2014) implemented a model-based supervisory controller to optimize filtration in an AnMBR demonstration plant. Energy savings of up to 25% were achieved when using gas sparging to scour membranes and the downtime for physical cleaning was about 2.4% of operating time. The operating cost of the AnMBR system after implementing the proposed supervisory controller was about €0.045/m^3, 53.3% of which were energy costs. In another application, Robles *et al.* (2013; 2015) obtained similar results using a 2-layer control system measuring the treatment flow rate (controlling the HRT), the sludge wasting volume (controlling the SRT), the temperature, and the gas sparging intensity in the anaerobic reactor and controlling the permeate flow rate, the trans-membrane pressure (TMP), the sludge flow-rate recycled through the membrane tanks, and the gas sparging intensity in the membrane tanks.

4 – What is next?

Many ideas and many perspectives arise from all the above details about current scientific and technical achievements.

Instrumentation

With respect to instrumentation, it is indeed believed that (1) more and more advanced sensors will be soon available (2) confidence index associated to the measurements will provide human operators with the ability to decide on the best actions based on the quality of the measurements (3) sensors network will allow the human operator to anticipate future problems, (4) software sensors and (5) use of large data base and all of this will improve by far the information content currently retrieved from AD plants. The simultaneous use of a sensor network (Steyer *et al.* 2004) and of numerical models will clearly help in extending and qualifying the available measurements.

As pointed out earlier in the paper, the analysis of individual VFA species has often been proposed as an important measurement parameter for the diagnosis, optimisation and control of anaerobic processes. Most of this information is today collected off-line and are mainly based on either GC or HPLC analysis and have been benchmarked comprehensively in Raposo *et al.* (2013). As off-line monitoring of VFAs is likely to have a significant lag in measuring VFA and inputting the data into a feedback control loop would have significant draw backs due to the time delay in analysis and inputting the data. There is a significant challenge to overcome in producing an instrument for on-line or at-line species specific VFA analysis that is relatively easy to operate at low capital and operational cost. There has been a significant amount of activity directed automating off-line techniques in particular GC headspace techniques (Boe *et al.* 2007 and Boe *et al.* 2008) but there has been limited uptake for this method beyond the initial publications. An alternative approach has been the use of Near Infra Red Spectroscopy (NIRS) for acetate, propionate and TVFA analysis but the NIR analyser despite requiring relatively little maintenance was found to have a too high error of prediction for accurate quantification (Ward *et al.* 2011). An alternative approach to the traditional analytical techniques of GC, HPLC or IR spectroscopy may be to use biosensors as the measurement system. This offers the potential of a relatively low cost sensor system, with high specificity and sensitivity and no requirement for continuous supply of a chemical or gaseous mobile phase as required by GC or HPLC techniques. An approach based on microbial fuel cells (Kaur *et al.* 2013; 2014) and genetically engineered light emitting bacteria (Li and Yu 2015) have been proposed as possible solutions to develop a more effective on-line VFA instrument. A microbial fuel cell based biosensor was able to discriminate between acetate, propionate and butyrate, with a response time of 1-2 minutes with a sensitivity of 5 $mg.L^{-1}$ when cyclic voltammetry analysis was utilised (Kaur *et al.* 2013). The sensor linearity was limited to 5-40 $mg.L^{-1}$ but this could be addressed with appropriate sample dilution. An alternate biosensor approach using a genetically engineered *E. coli* based biosensor with light emitting response to propionate has been demonstrated with a linear response of 1-10 mM (Li and Yu 2015), however other VFAs such as acetate and butyrate are important species in anaerobic digestion require measurement. Despite a number of innovative approaches taken to measuring individual VFA species, an effective and low cost instrument for the on-line or at line measurement has yet to be identified.

In the first section dedicated to the instrumentation, the lack of "sensors" for monitoring biodegradability and bioaccessibility has been highlighted. As pointed out by the substrate evolution, agricultural and municipal solid wastes are more and more used. This kind of complex substrates need long HRT and the off-line option can be acceptable in order to drive their digestion or co-digestion.

Despite the fact that several tools are promising like NIRS for biodegradability prediction, this technique is, until now, applied on dried-frozen samples and the impact of drying samples on the BMP values obtain with not prepared sample has not been studied. As previously mentioned, NIRS technology has a great potential as sensor, and work has to be followed to develop a probe able to predict BMP value on raw samples. However, this technique does not give bioaccessibility or biodegradation rate parameters. In the case of co-digestion for example, these parameters are crucial. Other study does it in a faster way than BMP test (for example, Jimenez *et al.* 2014) but it needs advanced knowledge of the methodology used and advanced and expensive material (i.e. 3D fluorescence spectroscopy). Therefore, more efforts have to be done on how to transpose these promising but complex techniques into a cheap and practical "sensors". For example, research on multi-excitation wavelength fluorescence probes would be done, and, associated with an optimized chemical extraction protocol would be able to predict both biodegradability and bioaccessibility. These kinds of information would be very valuable in order to predict the optimal mixture to do during co-digestion for example.

As previously mentioned, spectroscopic on-line sensors are of particular interest to the AD industry and research as they allow the on-line monitoring of crucial process variables. Nevertheless, high prices and complex calibration routines hinder commercial success. Newly developed tunable Micro-Electronic-Mechanical-System (MEMS) based Fabry-Pérot interferometers for the UV/vis, NIR and MIR wavelength ranges provide a very promising solution. Not only are these spectrometers on a chip very small 5 x 10 cm but also relatively cheap, if manufactured in big numbers. Currently, two different system designs exist. Neumann *et al.* (2010) introduced a tunable MEMS interferometer for the middle- and long-infrared range using a pyro-detector. The different wavelengths can be generated by two bragg reflectors whose distance can be changed by a spring suspension. Although, the presented performance results are good, the spring suspension is considered to be a weakness as it makes the spectrometer sensitive to vibration and wear. Therefore, the Technical Research Centre of Finland (VTT) developed an interferometer design with piezo-effect based tuning of the gap between the reflectors (Antila *et al.* 2014, Mäkynen *et al.* 2014). In general, these MEMS systems allow for completely new probe designs where the spectrometer is directly integrated into the probe so that the fibre length can be reduced significantly, increasing the S/N ratio. Thus, not only the sensitivity of a sensor is increased but also the size of the whole sensor system is reduced. This particularly important for MIR sensors where a short fibre length is crucial to guarantee a high S/N ratio. Malinen *et al.* (2014) gives a broad overview of the possibilities in various applications. In high quantities, prices for MEMS spectrometers are expected to drop to 70-100€ per piece, which makes spectroscopic sensors attractive for the use in AD plants.

Confidence indexes are information about the way measurements are obtained. One important lesson from applying ICA in AD plants is that some sensor technologies are more useful than other ones. Indeed, if all on-line sensors provide numerical values of the measured variables, some (e.g. spectrometer or titrimeter) also provide information on how the measurements have been obtained (Steyer *et al.* 2006). This information can then be used as a confidence index on the measurement and is of great help to decide – in a closed loop context – if a control law can rely or not on the obtained measurements. In order to guarantee a safe operation of the plant, the controller can indeed be turned off in case of sensor fouling or any other dysfunctionning in the instrument.
However, this increase in complexity in the management of the sensor data and the automation of the process may involve dedicated highly qualified operators for permanently recalibrating and adapting the complex implemented algorithms. Indeed, most of the monitoring, diagnosis or control advanced strategies which are described on Table 4 have been tested (when they have been experimented) on short time periods (generally less

than a few weeks), with a precalibrated set of parameters and initial conditions. These additional degrees of freedom, which are rarely clearly stated, must be managed on the long term for operational perspectives. Better accounting for such degrees of freedom, automating these aspects to reach robust autoadaptive algorithms, or allowing a remote expert to manage them (Bernard *et al.* 2005a; 2005b) is thus a challenge for the future years.

Models and virtual sensors

Even if modeling AD has been an active research topic these last two decades, improving the models supporting monitoring and control strategies is also very challenging. Due to the increasing complexity of the substrates, hydrolysis was considered as the limiting step introducing the notion of bioaccessibility. Based on the degradation kinetics of the sludge, new variables appeared by taking into account the bioaccessibility of the substrate. A better knowledge of the sludge composition indeed leads to more realistic although more complex models. However, despite the techniques described the literature until now, input variables of ADM1 are still difficult to characterize. Advanced analytical techniques could provide a higher degree of information on the composition of any given substrate. Promising new tools can be used for direct measurement, such as NIRS, 3D-EEM SPF and LIF probes in order to describe the biodegradability of a waste. However, with the biodegradability, the bioaccessibility is a key concept of the model input variables characterization. Some studies proposed bioaccessibility assessment specific to sewage sludge. Further investigations need to be performed in order to find a relevant and rapid tool for organic matter characterization of more solid wastes in order to obtain reliable parameters for the biological processes models. ADM1 is sensitive to the substrate composition, and a methodology providing characterization rules based on substrate type using either upstream knowledge, chemical analysis (for simple substrates), or biochemical testing would greatly improve the predictability potential of the models (Batstone 2013), and then their further efficiency in monitoring and control strategies.

A more accurate description of the physicochemical models, and especially of the precipitation related to calcium and phosphorus (Batstone *et al.* 2012) is a difficult yet necessary step to better under the cycle of phosphorous. Even if it may strongly increase the model complexity, considering sulfur reduction and oxidation processes are also challenges for the future. Also, the spatial distribution of the chemicals and biomasses within the reactor should now be accounted for and integrated in the models. These points should be seen in a larger context than AD, and a plant wide approach (see e.g., Olsson *et al.* 2014) must prevail. For example, physicochemical models must describe phosphate speciation and release under aerobic and anaerobic conditions, while micropollutants must be tracked along the full treatment plant.

Soft sensing or virtual sensing is the use of models to predict process parameters that are expensive or difficult to measure from more accessible process measurements. They are an effective method of providing in-line estimates of quantities that are difficult to measure on-line, and as such offer the possibility of providing enhanced monitoring of processes, both in terms of providing additional process information and acting as a reference for sensor fault detection. They have previously been demonstrated for estimating parameters such alkalinity, chemical oxygen demand, inorganic carbon, and volatile fatty acids in waste water treatment plants (Bernard 2011) and more recently for total alkalinity in biogas plants (Ward *et al.* 2011). However, development and updating of soft sensor models requires expert knowledge due to the complex modeling techniques required and the need for tailored training data, putting them beyond the research of most small scale biogas plant operators. Newly developed powerful Machine Learning methods facilitate soft sensor development because of their ability to learn vastly complex and nonlinear relationships (Gaida *et al.* 2012). Further research in this area is necessary to tap the full potential of the existing methods with regard to AD processes.

Control

With respect to both observer and control design, one may also expect the development of high power computation capacity will fundamentally change our way of thinking. Modern control techniques usually necessitate the use of a limited order model to be able to guarantee stability and performance robustness. Techniques based on particulate filters (Cf. for instance Goffaux and Van de Wouwer (2005) and Benyahia *et al.* (2012) for applications to chemostat models) coupled with the use of nonlinear optimal controllers present the advantage of being able to use complex model while dealing with uncertainty. Of course, the price to pay is a relatively less degree of guarantee but the higher the on-line computer capabilities, the higher the state space to be investigated and the lower the probability to push the process towards a dangerous functioning zone. Another promising route concerns the use of innovative passive control approaches in which control objectives are considered at the initial conception step of the process. In terms of performances, it is for instance well known that series of reactors perform better than single processes. However, this design may penalize both investment costs and the stability of the process since reducing the size of the first reactor. The introduction of alternative configurations of the different reactors and the judicious choice for their respective volumes may lead to a more robust global system with respect to specific uncertainty and disturbances (if compared to a single tank reactor), cf. for instance the work by Rapaport *et al.* (2014) on the stabilization of chemostats with substrate-inhibited kinetics.

It is usually expected that a controller using a more complex model would lead to better performances. Assuming the on-line computation capability is available, it may be true. But the use of very simple models from which a control may "really" be optimal with respect to a given performance index, from a mathematical viewpoint, may be helpful to think of new control strategies. For instance, the work by Sbarciog *et al.* (2010) allowed us to propose a new control strategy able to guarantee sub-optimal performances while preserving the stability of the whole process (Rodriguez *et al.* 2013).

Microbial management of bioprocesses is another emerging topic with a great potential. This is particularly true for AD which involves a huge biodiversity (Carballa *et al.* 2015). Thanks to the development of molecular analytical tools (denaturing gradient gel electrophoresis, single-strand conformation polymorphism…), the anaerobic microbiome has been more and more characterized (Vanwonterghem *et al*. 2014, Sundberg *et al.* 2013). Considering the biodiversity can give raise to a new paradigm for the control and optimization of AD. Until now, the principal objective of control was to stabilize the digester. Nonetheless, a stable process tends to reduce the biodiversity through the section of the fittest species in the imposed environment. Although this selection process could increase the steady-state performance, it could seriously alter the resilience of the process (Ramirez *et al.* 2009). Dynamical feeding has been proposed in order to select a microbiome with a high ability to adapt to disturbances (De Vrieze *et al*. 2013). Bioaugmentation have been also applied, in particular in response to stress (e.g. Schauer-Gimenez *et al.* 2010; Tale *et al.* 2011). Concerning the model-based control laws, most of them are designed assuming one population for one function. Recently, Mairet and Bernard (2014) have proposed to evaluate the performances of such control laws when several species are present. Using the control law proposed by Mailleret *et al* (2004) as an example, they have shown that a slow-growing species can lead to reactor shutdown. This framework can be used to design robust control laws which better tame biodiversity. Rapaport and Harmand (2002) also proposed a "biocontrol" strategy using biotic microbial ecosystem capabilities to select certain species. Although attractive, these approaches remain studied in simulations only. The control of the microbiome involved in AD is an exciting challenge for the future, but the lack of on-line instrumentation for biodiversity monitoring can limit process implementation. Recently, on-line flow cytometers have been proposed for AD (Koch *et al.* 2014) and can open new directions for closed-loop microbial control strategies.

Recently, novel potential actuators emerged to control methanogenic pathways (Liu *et al.* 2013; Lin *et al.* 2013). Indeed, methanogenic pathways (i.e. acetoclastic or hydrogenotrophic) have been analyzed using stable carbon isotope signature. This analysis is made on the biogas phase and thanks to an isoprime mass spectrometer linked with a gas chromatography, a carbon fractionation can be performed. This information is very valuable because it points out the contribution of the different methanogenic pathways producing methane and carbon dioxide. For example, Liu *et al.* (2013) made cartography of the methanogens type depending on ammonium and acetate high concentrations. In the same way, Lin *et al.* (2013) showed the impact of the addition of bicarbonate on the methanogenic biodiversity. This kind of information would be very valuable in order to drive a digester in case of acid or/and ammonia inhibition, without loose energetic performance.

The balance between the synergistic production and consumption of VFA intermediates in the AD with respect to process stability is important and has been outlines in section 3.1. Disagregation of the trophic groups in the AD process by physically separating them into an acidogenic stage reactor and a methanogenic stage reactor is not a new idea. Two stage AD, often with the intention of improving hydrolytic processes, has been studied by many researcher over the last 40 years e.g. Ghosh *et al.* (1975). However, increased scope for control actuation may be available by such stage separation especially with the ability to monitor and manage microbial populations more effectively in recent years. Furthermore, the stages can be integrated with each other and other processes to improve gas yields as reported by Massanet-Nicolau *et al.* (2013). Guwy *et al.* (2011) described how the integration of multi stage bioprocesses can be used to extract or utilize the products. The extraction of VFAs for example may simultaneously deliver valuable chemicals and controlled supply of substrate for methanogenesis to a subsequent stage. This VFA extraction may be achieved by conventional electrodialysis, as proposed by Jones *et al.* (2015) in an acidogenic stage also generating hydrogen. VFAs are also an appropriate substrate for bioelectrochemical systems as has been demonstrated by many researchers and reviewed by Pant *et al.* (2010). The application of multivariable control strategies as described in this paper may deliver optimal system performance, although control of each of the stages or sub-processes may be independently controllable under a system level supervisory regime.

The capacity of ADs to utilize additional CO_2 was demonstrated by several authors, which could provide a potential solution for on-site sequestration of CO_2 streams while enhancing methane production by CO_2 sparging. CO_2 could then become an efficient actuator to improve AD performances. Few studies have indeed considered the potential of CO_2 biological conversion in anaerobic processes, reporting benefits both in terms of carbon uptake and renewable energy production (Salomoni and Petazzoni 2006; Salomoni *et al.* 2011). Interestingly, microorganisms operating under CO_2 saturated conditions continue to synthesize CH_4. Alimahmoodi and Mulligan (2008) stated a 69–86% CO_2 uptake when dissolving this gas in the influent of an upflow anaerobic sludge blanket (UASB) reactor. Francioso *et al.* (2010) and Salomoni *et al.* (2011) further confirmed the potential of CO_2 biological conversion in two phase anaerobic digestion (TPAD), and observed 25% methane (CH_4) yield enhancement when sparging CO_2 into the first stage. Moreover, the net production of CO_2 in CO_2-recirculating AD units can be reduced by a factor of 4. Fernández *et al.* (2014) addressed the reduction of CO_2 emissions and enhancement of biogas production associated with CO_2 enrichment of anaerobic digesters (ADs). The benefits of CO_2 enrichment were examined by injecting CO_2 at 0, 0.3, 0.6 and 0.9 M fractions into batch ADs treating food waste or sewage sludge. Daily specific methane (CH_4) production increased 11–16% for food waste and 96–138% for sewage sludge. Potential CO_2 reductions of 8–34% for sewage sludge and 3–11% for food waste were estimated. Mohd Yasin *et al.* (2015) used CO_2 as the substrate to generate methane by enriched methanogens after anaerobic enrichment of waste activated sludge (WAS) and they demonstrated that methanogens from WAS have significant potential for converting the greenhouse gas CO_2 into the fuel methane. Moreover, methane production was increased 70 fold by active methanogens in the enriched methanogens culture after 3 days in the presence of H_2 and CO_2.

Indeed, the addition of H_2 into an anaerobic digestion has been performed in several studies (Luo et al. 2012; Luo and Angelidaki 2013; Wang et al. 2013b; Díaz et al. 2015) in order to remove CO_2 from biogas while methane production increased, through the hydrogenotrophic pathway. For example, Luo et al. (2012) showed that increasing both hydrogen partial pressure and mixing intensity would give 22% of methane production. One main barrier highlighted was the gas-liquid mass transfer of H_2 because of the low solubility of this gas.

Conclusions and perspectives

Over the years, knowledge on anaerobic digestion has increased and several instruments are now available to monitor efficiently the AD processes. Global parameters for organic matter characterization can indeed be used and biodegradability, bioavailability and bioaccessibility of complex solid substrates can be assessed. Modelling, especially through the development and consolidation of the ADM1 model, has successfully proven its ability to translate the biological steps occurring in the AD. Since its creation, many improvements have been carried out, and ADM1 has been tailored to a broad variety of substrates. But there are still progresses to be accomplished to better manage the influent composition, and further represent physicochemical processes such as precipitation. There is still a gap between these more and more accurate models, but also involving higher degrees of freedoms, and simpler models which support most of the monitoring, diagnosis and control algorithms. Bridging this gap, combining these theoretical approaches with information provided by innovative sensors, and reducing expert needs to run these algorithms will probably significantly improve the attractiveness of the approach together with its efficiency.

These developments will also contribute to improve emerging processes such as thermophilic and ultra-high rate processes (Ge *et al.* 2011), or supporting co-digestion strategies (Mata-Alvarez *et al.* 2011). Modelling, monitoring and control are also expected in the objective of recovering nutrients (Mehta and Batstone 2013) and for tracking micropollutants, trace organics, pathogens and recalcitrant (Fountoulakis et al. 2008).

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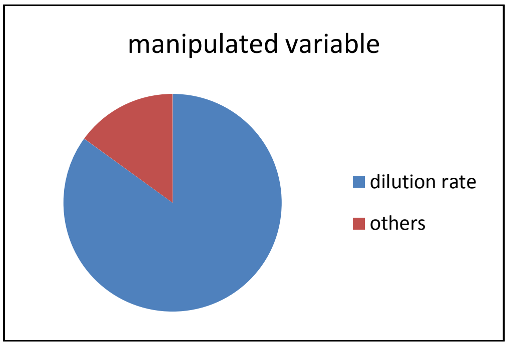

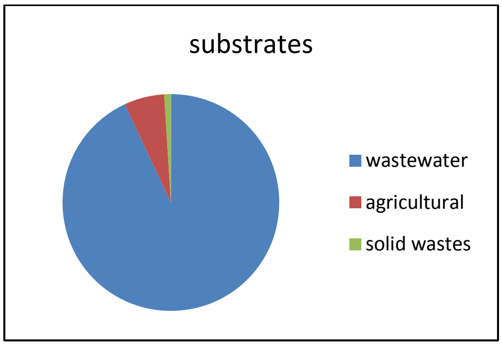

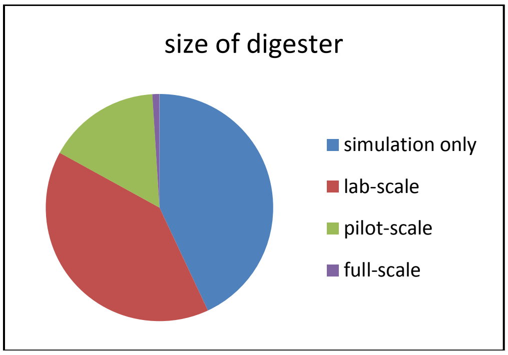

Figure 1: Percentage distribution of manipulated variable (121 publications), size of digester (134 publications) and substrates (109 publications) of the reviewed publications.

Table 1: Summary of the different methodologies used in integrative tools found in the litterature

Integrative tools	Characterization methods	Benefits	Drawbacks	References
Static model PLS, correlations, Stoechiometric reaction	Biochemical characterization Proteins, carbohydrates, lipids, COD/TOC, TOC soluble	Analytical simple and rapid methods	Model validation not yet achieved Based on one type of sludge (secondary) Care to be taken of the accuracy of methods used Not take into account complexity and accessibility	*Mottet et al. (2010)*
	CHNOS elemental analysis	Fast and practical method	Consideration of the whole organic matter degradation: the biodegradable fraction is not used Over-estimation of BMP tests	*Shanmugam and Horan (2009)*
	Van Soest and fibers analysis	Faster and practical method Validation on several solids wastes Accessibility taking into account with growing extraction power	Not suitable for sewage sludge in terms of protocol (porosity) Model validation not conclusive	*Chandler et al. (1980)* *Gunaseelan (2007)* *Mottet et al. (2010)*
	Aerobic respiration rate	Faster than a BMP test (4 days instead of 21-30 days) Promising on solid wastes	Only readily substrate taken into account No accessibility taken into account Assumption on the same biodegradability under aerobic and AD	*Cossu and Raga (2008)* *Scaglia et al. (2010)*
	Initial rate technique	Faster method than BMP Maximum production rate and affinity constant determined	Extrapolation in continuous digester underestimate methane production Not information on substrate bioaccessibility	*Donoso-Bravo et al. (2011)* *Strömberg et al. (2015)*
	Biochemical characterization bioaccessibility compartiment	Bioaccessibility taken into account Biochemical fractions calculated from practical analysis	Necessity of long batch test for fractions assessment	*Yasui et al. (2006, 2008)* *Mottet et al. (2010)*
	NIRS	Biodegradability assessment Fast Various type of substrates	Necessity of drying and freezing the sample Bioaccessibility not tkan into account	*Lesteur et al. (2010)* *Doublet et al. (2013)*
	3D fluorescence spectroscopy combined with accessibility characterization	Bioaccessibility taken into account Both biodegradability and bioaccessibility predicted Fast method	Calibrated on sludge-like samples	*Jimenez et al. (2014)*

Table 2: Analytical protocols for biochemical compounds determination

Organic fraction	Method type	Concentration (mg/L)	Reagent used	Standard	Reference
Proteins	Colorimetric	0-200	Folin reagent Copper sulfate 0.5% (w/w)	Bovine albumin serum	Lowry et al., 1951 Frølund et al., 1996
	Colorimetric	0-200	Bicinchonic acid		Smith et al., 1985
	Colorimetric	0-100	Gornall biuret reagent and NaCl		Gornall et al., 1949
	Colorimetric	2-120	Coomassie brilliant blue G-250 reagent		Bradford, 1976
	Standard method for TKN assessement	N content x 6.25 g proteins/gN	Mineralisation and ammonia dosage	None	Kjeldahl, 1883
Humic acids like	Colorimetric	0-200	Folin Reagent	Humic acids (Aldrich)	Frølund et al., 1996
Polysaccharides	Colorimetric	0-100	Phenol 5% (w/w) Sulfuric acid 95%	Glucose	Dubois et al., 1956
	Colorimetric	0-100	Anthrone 0.125% (w/v) Sulfuric acid 95%		Dreywood et al. 1946 Raunkjær et al., 1994
Fibers	Extractions	-	Weende method Van Soest	None	Henneberg and Stohmann, 1860; Van Soest, 1963
Lipids	Colorimetric	0-1000	Vanillin 0.6% (w/w) Phosphoric acid 85% Sulfuric acid 95%	Commercial olive oil	Frings and Dunn 1970
	Extraction Infrared spectroscopy	-	CCl_4, Uvasol, Al_2O_3, Na_2SO_4, HCL 6M	cornoil	APHA, 2005
	Extraction Gravimetry		Organic solvent	-	APHA, 2005

Table ESM: Examples of AD controllers found in the literature

Table ESM.1: Classical Control of Biogas Plants: on/off controls & PID controls

Control type	Authors	Description	Manipulated variable	Control variable
on/off	Rozzi (1984)	proposal of three controllers (1, 2, 3) purpose of stabilization application: simulation only	alkaline solution	1) pH 2) bicarbonate 3) pH, pCO2
on/off	Andrews (1974)	application: CSTR, simulation only, wastewater	recirculation	CH4 flow rate
P deadband	Denac et al. (1990)	based on alkaline consumption Application: lab-scale FBR, wastewater	- dilution rate - alkali addition	- effluent VFA - pH
I deadband	Feitkenhauer et al. (2002)	application to an acidic phase reactor, goal: max. VFA application: lab-scale CSTR, wastewater	dilution rate	VFA
PI	Batstone and Steyer (2007)	proposal of two controls (1, 2) application: simulation only (ADM1), wastewater	dilution rate	1) VFA 2) alkalinity
PID	Marsili-Libelli and Beni (1996)	purpose: stabilization application: simulation only	bicarbonate addition	bicarbonate alkalinity

Table ESM.2: Classical Control of Biogas Plants: adaptive PID and PID cascade control

Control type	Authors	Description	Manipulated variable	Control variable
adaptive PI	Perrier and Dochain (1993)	proposal of three controllers (1, 2, 3) application: simulation only	dilution rate	1) effluent COD 2) dissolved H2 3) propionate
cascade P	Liu et al. (2004)	inner loop: pH; outer loop: gas flow rate setpoint of outer loop given by rule-based supervisory system lab-scale AFB reactor, wastewater, mesophilic	dilution rate	OLR
cascade P	Boe and Angelidaki (2012)	inner loop: VFA; outer loop: gas flow rate rule-based system as in Liu et al. (2004) application: pilot-scale CSTR, manure, thermophilic	dilution rate	CH4 flow rate
cascade PI	Alvarez-Ramirez et al. (2002)	inner loop: VFA; outer loop: COD application: lab-scale UASB, wastewater	dilution rate	effluent COD
cascade PID	García-Diéguez et al. (2011)	inner loop: methane flow rate; outer loop: VFA application: pilot-scale UASB-AF, wastewater, mesophilic	dilution rate	- CH4 flow rate - effluent VFA

Table ESM.3: Expert Systems Control of Biogas Plants: rule-based

Control type	Authors	Description	Manipulated variable	Control variable
expert system	Boe et al. (2008)	if propionate …, then in-/decrease feed high fluctuations in biogas flow rate, because propionate is too persistent application: lab-scale CSTR, cow manure, thermophilic	dilution rate	propionate
expert system	Barnett and Andrews (1992)	rules implemented with fuzzy logic inputs: a lot; output: a few next to dilution rate application: simulation only	dilution rate	normal operation
expert system	Chynoweth et al. (1994)	rules based on CH4 flow rate, its derivative, dilution rate and its derivative able to distinguish between overloading, underloading and inhibition application: lab-scale CSTR, wastewater, mesophilic	dilution rate	CH4 flow rate
expert system	Moletta et al. (1994)	inputs: pH, biogas flow rate, H2 content of biogas application: lab- and pilot-scale FBR, wastewater, mesophilic	dilution rate	normal operation
expert system	Ehlinger et al. (1994)	decision tree: pH, gas and H2 flow rate application: lab-scale FBR, mesophilic, wastewater	dilution rate	normal operation
expert system	Flores et al. (2000)	application: start-up of pilot-scale UASB-AF reactor, wastewater	dilution rate	normal operation
expert system	Pullammanappallil et al. (1991, 1998)	bumpless switch between four different control strategies based on a t-test: 1) set-point control, 2) constant yield control 3) batch operation, 4) constant dilution rate application: lab-scale CSTR, wastewater, mesophilic	dilution rate	CH4 flow rate
expert fuzzy system	Müller et al. (1997)	H2 and CH4 flow rate; uses Fuzzy C-Means Clustering of Marsili-Libelli and Müller (1996) application: lab-scale FBR, wastewater, mesophilic	- bypass - storage - dilution	normal, overload, inhibition, toxicity
expert fuzzy system	Puñal et al. (2001, 2002), Carrasco et al. (2002)	many input variables application: pilot-scale UASB-AF, wastewater	flow rates	over-, underload recovery

Table ESM.4: Expert Systems Control of Biogas Plants: fuzzy controls

Control type	Author	Description	Manipulated variable	Control variable
fuzzy P	Bernard et al. (2001)	inputs: TA, VFA/TA application: pilot-scale FBR, wastewater	dilution rate	VFA/TA
fuzzy P	Scherer et al. (2009)	inputs: pH value, CH4 content and specific gas flow rate application: lab-/pilot-scale CSTR, agricultural, meso-/thermophilic	dilution rate	OLR
fuzzy I	Boscolo et al. (1993)	inputs: nine variables application: pilot-scale CSTR, OFMSW, thermophilic	- feed rate - TS of feed - recycling rates	normal operation
fuzzy P + PI	Murnleitner et al. (2002)	inputs: H2, CH4, biogas flow rate, pH, filling level application: lab-scale FBR, two-stage, wastewater, mesophilic	- different flows (PI) - pH (P) - temperature (P)	overload avoidance
fuzzy PI	Estaben et al. (1997)	inputs: error to setpoints of gas flow rate and pH value and the derivatives of the errors; output: change of feed rate application: lab-scale FBR, wastewater	dilution rate	- gas flow rate - pH value
fuzzy PI	Puñal et al. (2003)	inputs: error of VFA to its setpoint and its derivative output: change of feed rate application: pilot-scale AFB, wastewater	dilution rate	effluent VFA
fuzzy PI	Garcia et al. (2007)	inputs: CH4 flow rate; H2 content of gas; VFA/TA output: change of feed rate application: ADM1, lab-scale UASB-AF, wastewater	dilution rate	OLR
fuzzy PI cascade	Martinez-Sibaja et al. (2007)	- inner loop (conventional PI): pH - outer loop (fuzzy PI): gas flow rate application: simulation only	dilution rate	- gas flow rate - pH value

Table ESM.5: Expert Systems Control of Biogas Plants: neural networks and special fuzzy systems

Control type	Author	Description	Manipulated variable	Control variable
hierarchical fuzzy	Steyer et al. (1997)	inputs: control error of pH, T and biogas flow rate for a small rule-set a hierarchical fuzzy structure is chosen application: lab-scale FBR, wastewater, mesophilic	dilution rate	VFA
neural network	Holubar et al. (2002, 2003)	ANN models for: pH, VFA, biogas production and composition optimal COD loading rate is solution of max. CH4 flow rate and COD degradation; application: lab-scale CSTR, primary sludge	COD loading rate	CH_4 flow rate
neural	Wilcox et al. (1995), Guwy et al. (1997)	ANN model for bicarbonate alkalinity (BA) out of past BA values application: lab-scale FBR, ice-cream and baker's yeast WW	BA dosing pump	bicarbonate alkalinity
neural network	Emmanouilides and Petrou (1996)	adaptive on-line trained neural networks application: simulation only	dilution rate	- CH4 flow rate - effluent COD
neural fuzzy	Yordanova et al. (2004)	fuzzy PI, fuzzy tuning control application: simulation only, wastewater	dilution rate	Biogas flow rate
neural fuzzy	Waewsak et al. (2010)	ANN models for: pH, TA and VFA, predicted out of past values application: lab-scale UASB-AF, synthetic WW, mesophilic	dilution rate	- high performance - stability
fuzzy supervision	Carlos-Hernandez et al. (2007)	Takagi-Sugeno supervisor switches between: 1) open loop, 2) base addition (fuzzy PI), 3) dilution rate (fuzzy PI) application: FBR, wastewater, simulation only	- base addition - dilution rate	high performance
fuzzy supervision	Carlos-Hernandez et al. (2010a)	as in Carlos-Hernandez et al. (2007) PCA and Takagi-Sugeno estimate biomass and substrate application: CSTR, wastewater, simulation only	- base addition - dilution rate	CH_4 flow rate
fuzzy supervision	Gurubel et al. (2013)	as in Carlos-Hernandez et al. (2010a), additional using PSO to improve setpoint tracking	- base addition - dilution rate	CH_4 flow rate
neural fuzzy	Carlos-Hernandez et al. (2010b)	as in Carlos-Hernandez et al. (2007) neural observer trained by EKF estimates methanogenic biomass application: FBR, abattoir wastewater, simulation only	- base addition - dilution rate	high performance

Table ESM.6: Linearizing Control of Biogas Plants

Control type	Author	Description	Manipulated variable	Control variable
linearizing	Alvarez-Ramirez et al. (1996), Monroy et al. (1996)	adaptive, no need for measuring biogas flow rate application: lab-scale UASB, wastewater, mesophilic	dilution rate	effluent COD
linearizing	Petre et al. (2007)	adaptive, asymptotic state observer application: simulation only	dilution rate	effluent COD
feedback linearization	Angulo et al. (2007)	derivation using AM1 (Bernard et al., 2001a), model-based application: simulation only, AFB reactor, wastewater	dilution rate	effluent VFA
external linearization	Renard et al. (1988)	adaptive control, influent COD needs to be measured application: lab-scale CSTR, WW (citric acid), mesophilic	dilution rate	effluent COD
external linearization	Johnson et al. (1995)	Renard et al. (1988) approach used application: lab-scale AFB, wastewater, mesophilic	dilution rate	effluent COD
linearizing	Dochain and Perrier (1993)	direct adaptive linearizing application: CSTR, simulation only	dilution rate	propionate
linearizing	Bernard et al. (2001)	adaptive control, influent COD estimated by soft sensor application: pilot-scale FBR, wastewater	- dilution rate - alkalinity	VFA/TA
linearizing	Rincon et al. (2009)	adaptive control, normal form of fold bifurcation application: simulation only, wastewater	dilution rate	effluent VFA
linearizing	Simeonov and Queinnec (2006)	model-based, organic wastes and acetate application: simulation only, CSTR, mesophilic	acetate addition	biogas flow rate
robust linearizing	Rapaport and Harmand (2002)	interval observer application: simulation only, CSTR	dilution rate	effluent COD
geometric	Méndez-Acosta et al. (2005)	to avoid overshooting fuzzy-based gain-scheduling and antiwindup scheme are used, high-gain observer application: simulation only, AFB, wastewater	dilution rate	effluent COD
geometric robust	Méndez-Acosta et al. (2008)	model-based: extended Luenberger observer application: pilot-scale AFB, wastewater	dilution rate	effluent VFA
geometric robust	Méndez-Acosta et al. (2007)	model-based: extended Luenberger observer; proposal of two controls (1, 2); TOC: total organic carbon application: pilot-scale AFB, wastewater,	dilution rate	1) VFA 2) TOC

		mesophilic		
geometric robust	Méndez-Acosta et al. (2010)	model-based: extended Luenberger observer, antiwindup structure application: simulation only, wastewater	- dilution rate - alkali solution	- VFA - TA
linearizing	Dochain and Bastin (1985)	nonlinear adaptive application: CSTR, simulation only	dilution rate	effluent VFA
generic model control	Costello et al. (1989)	improvement of Dochain and Bastin (1985) application: CSTR, simulation only, wastewater	dilution rate	effluent COD
linearizing	Petre et al. (2013)	three controls: 1) adaptive (asymptotic observer), 2) robust, 3) robust-adaptive (interval observer, both) application: CSTR, simulation only, wastewater	dilution rate	effluent COD
VSM	Tartakovsky et al. (2002, 2005)	variable structure model (VSM) containing three linear submodels, for each submodel one linearizing control application: lab-scale UASB, synthetic wastewater, mesophilic	influent COD	effluent COD
decoupled linearizing	Aguilar-Garnica et al. (2009)	two-phase AD system, modeled by PDE, observer-based estimator application: simulation only, two AFBs, wastewater	recycle flow rates	- effluent VFA - effluent COD

Table ESM.7: Other Advanced Controls for Biogas Plants

Control type	Authors	Description	Manipulated variable	Control variable
disturbance monitoring	Steyer et al. (1999)	increased biogas yield caused by an impulse in feed is compared with expected. Overloading/inhibition reflected by an unsatisfactory gas yield. application: lab-scale FBR, wastewater, mesophilic	dilution rate	biogas flow rate
disturbance accommodating	Harmand et al. (2000)	ARMAX model with bias estimation application: lab-scale FBR, wastewater	dilution rate	biogas flow rate
nonlinear adaptive	Polihronakis et al. (1993)	proposal of three controls: 1), 2) and combination of both combination switches between both control objectives application: full-scale, wastewater	dilution rate	1) effluent COD 2) CH4 flow rate
adaptive robust	Hilgert et al. (2000)	ARMAX model with uncertain part, estimated by kernel estimator application: lab-scale FBR, wastewater, mesophilic	dilution rate	biogas flow rate
adaptive	Harmon et al. (1993)	taken from Pind et al. (2003) application: lab-scale CSTR, glucose	temperature	CH4 flow rate
nonlinear	Harmon et al. (1990)	constant reactor yield control application: lab-scale CSTR, synthetic WW, thermophilic	dilution rate	CH4 flow rate
sampled delayed control	Méndez-Acosta et al. (2011)	nonlinear, robust, delayed measurements, COD measured daily application: lab-scale AFB, wastewater, mesophilic	dilution rate	effluent COD
robust output feedback	Antonelli et al. (2003)	nonlinear; only measured variable: CH4 flow rate application: pilot-scale AFB, wastewater, mesophilic	dilution rate	CH4 flow rate
robust output feedback	Mailleret et al. (2003)	CH4 flow rate and input COD needed application: pilot-scale AFB, wastewater	dilution rate	effluent COD
nonlinear adaptive	Mailleret et al. (2004)	CH4 flow rate needed application: pilot-scale AFB, wastewater	dilution rate	effluent COD
nonlinear adaptive	Dimitrova and Krastanov (2009)	extremum seeking algorithm to maximize CH4 production application: simulation only	dilution rate	- effluent COD - CH4 flow rate
adaptive	Seok (2003)	recursive system identification, convex optimization problem application: lab-scale FBR, wastewater, mesophilic	dilution rate	propionate
extremum seeking	Marcos et al. (2004)	adaptive; substrate concentration kept at setpoint application: CSTR, AFB, simulation only	dilution rate	CH4 flow rate
extremum seeking	Simeonov and Stoyanov (2011)	application: CSTR, simulation only, mesophilic	dilution rate	CH4 flow rate
LQT	Mu et al. (2008)	linear quadratic tracking (LQT) and error integral action application: simulation only, lab-scale UASB, distributed	- recirculation-to-feed ratio - bypass-to-	effluent COD

		model, wastewater	feed ratio	
NMPC	Aceves-Lara et al. (2010)	asymptotic observer estimates influent, effluent and some product concentrations; dark fermentation application: lab-scale CSTR, diluted molasses, mesophilic	dilution rate	H2 flow rate
EPSAC-MPC	Ordace et al. (2012)	Extended Prediction Self-Adaptive Control (EPSAC) application: simulation only (ADM1), wastewater sludge	feed flow rates	CH4 flow rate
variable-gain	Rodríguez et al. (2006)	indirect COD control by controlling H2 in gas phase application: pilot-scale UASB-AF, wastewater	dilution rate	effluent COD
composed	Wang et al. (2013)	algebraic differential estimator, model-free application: CSTR, simulation only, agricultural, mesophilic	dilution rate	CH4 flow rate
adaptive optimization	Ryhiner et al. (1992)	steepest descent finds optimal operating point application: FBR, wastewater	dilution rate	- CH4 flow rate - VFA
saturated proportional	Grognard and Bernard (2006)	no input COD measurement needed; attracts to a region application: simulation only, wastewater	dilution rate	effluent COD
H_∞	Flores-Estrella et al. (2013)	application: simulation only, wastewater	dilution rate	effluent COD
dynamic compensator	Simeonov and Stoyanov (2003)	linear model with interval parameters; proposes two controls (1, 2) application: simulation only	dilution rate	1) biogas flow rate 2) effluent COD
robust adaptive	Rincón et al. (2012)	Lyapunov-like function application: simulation only, wastewater	dilution rate	effluent VFA
robust interval	Alcaraz-González et al. (2005)	interval observers application: pilot-scale AFB, wastewater	dilution rate	effluent COD

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