TESTING WASTEWATER FOR ITS REVENUE POTENTIAL

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ABSTRACT

The wastewater treatment design team of CPG NZ Ltd (formerly Waste Solutions) pioneered the design and value engineering of industrial and agricultural anaerobic wastewater treatment by introducing the novel high rate Covered In Ground Anaerobic Reactor (CIGAR) digester system. These CIGAR™ systems match existing tank based digester systems in performance and biogas yield but at approx. 1/3rd of the construction and operating costs for the comparable scale. The first CIGAR™ system was commissioned in 2003 and since then more than 30 full scale systems have been designed/installed in SE Asia and South America with a 100 % success record. All these applications are commercially successful and typically achieve payback periods of less than 2 years when the biogas is used as quality fuel for factory boiler and power production plan (genset) operation. To be able to confirm the expected financial and technical performance of biogas digester projects at the project development step it is critical to offer the investors the use of credible, proven, robust and accurate test and analysis methodology for (A), for the biogas yield; (B), the achievable wastewater treatment efficiency; and (C), the typical digester sludge stability (metabolic activity) under realistic digester operation conditions. Here we present an overview of the innovative testing and process demonstration methodology and the systems that CPG have developed in the last decade to determine the biogas production potential and biogas process stability for a wide range of solid and liquid industrial waste materials.

KEYWORDS

Wastewater, Anaerobic, Digestion, Biogas, Biological Methane Potential, Sludge Activity Test,

1 INTRODUCTION

The wastewater treatment design team of CPG NZ Ltd (formerly Waste Solutions) pioneered the design and value engineering of industrial and agricultural anaerobic wastewater treatment by introducing the novel high rate **C**overed **I**n **G**round **R**eactor (CIGAR) digester system. These CIGAR™ systems match existing tank based digester systems in performance and biogas yield but at approx. 1/3rd of the construction and operating costs for the comparable scale. The first CIGAR™ system was commissioned in 2003 and since then more than 30 full scale systems have been designed/installed in SE Asia and South America with a 100 % success record (Thiele, 2011).

These operating anaerobic digestion plants produce together every day more than 5 million kwh biogas that is used for heat and power production and production of carbon credits in agro-industrial processing and in bio-refinery applications. All these applications are commercially successful and typically achieve payback periods of less than 2 years when the biogas is used as quality fuel for factory boiler and power production plan (genset) operation. To be able to confirm the expected financial and technical performance of biogas digester projects at the project development step it is critical to offer the investors the use of credible, proven, robust and accurate test and analysis methodology for (A), for the biogas yield; (B), the achievable wastewater treatment efficiency; and (C), the typical digester sludge stability (metabolic activity) under realistic digester operation conditions.

Here we present an overview of the innovative testing and process demonstration methodology and the systems that CPG have developed in the last decade to determine the biogas production potential and biogas process stability for a wide range of solid and liquid industrial waste materials. We give examples for the use of the methodology for a number of different wastewater types. We show the application of this methodology for the rapid anaerobic sludge activity test (SAT) in sludge samples from operating full scale digester plants and give examples for the use of the methodology in fully automated remote controlled anaerobic digester pilot plants industrial wastewater treatment plants.

2 MATERIALS AND METHODS

2.1 BIOCHEMICAL METHANE POTENTIAL (BMP) AND ANAEROBIC SLUDGE ACTIVITY TEST (SAT)

Biochemical Methane Potential Test (BMP): An aliquot of wastewater is placed in a 160 ml serum together with a defined amount of mesophilic anaerobic seed sludge and pH buffer. The pH buffer is typically a phosphate or carbonate / CO_2 buffer. Strict anaerobic culturing techniques are applied to ensure obligate anaerobic bacteria in the inoculum do not get exposed to air, as oxygen is toxic for them. The headspace of the serum bottle is purged with nitrogen gas and carbon dioxide is added to give around 35% CO_2 (V/V). The presence or absence of oxygen traces is indicated using an oxidation-reduction potential indicator (Resazurin).

In order to obtain meaningful results, the seed culture must be acclimatized to the pollutants in the wastewater. While some of this acclimatization can and will take place during the incubation period in the BMP test, it is best to have pre-adapted seed sludge available. CPG holds in its process testing culture collection a number of mesophilic and thermophilic seed sludge cultures maintained with different carbon sources to accommodate for the rapid and effective testing of a wide range of wastewater types as soon as a sample has arrived in our laboratory.

The volume of the produced biogas is measured by penetrating the butyl rubber septum of the serum bottle (*Photograph* 1) with a hypodermic needle attached to a graduated syringe. The methane content of the produced biogas is measured by gas chromatography (GC) using a molecular-sieve separation column and a thermal conductivity detector. Together with the amount of methane, the nitrogen, carbon dioxide and oxygen content in the headspace of the serum bottles is quantified in one GC run.

To accurately determine the background methane production from the anaerobic seed culture, three or more negative controls (seed sludge without substrate) are incubated together with the treatments. The methane produced in the treatments is corrected for the methane produced in the negative controls.

To verify that the results obtained in the treatments are correct, a set of positive controls receives a defined amount of chemical oxygen demand (COD), typically in the form of ethanol. The added COD represents a methane equivalent that allows the assessment of the completeness of the methane producing reaction under "ideal conditions" with an ideal test waste. In the case of ethanol, 95% of the added COD should be converted to methane; the other 5% are consumed in the buildup of new bacterial biomass.

Photograph 1: Two 160 ml serum bottles with butyl rubber septum as used in BMP tests

Anaerobic Sludge Activity Test (Anaerobic SAT): Anaerobic Sludge Activity **(**Methanogenic activity) refers to the rate at which methanogenic bacteria utilize their food sources to produce methane and carbon dioxide. Since about 60-70 % of the methane formed is channeled through acetic acid, determination of the activity of acetoclastic methane formers present in a sludge sample presents a fairly good indication of the overall methanogenic activity of the sludge (Isa et al., 1993).

The Methanogenic Activity Test can be used for:

- Reactor Performance Studies
- Stimulation and Inhibition Studies
- Biodegradability and Adaptability Studies

A regularly monitored anaerobic reactor will typically display similar levels of methanogenic activity. Any discernible or rapid drop in methanogenic activity indicates disturbance of the microbiological consortium, highlighting the need for counteracting measures. A change in loading rate or the supplementation with trace elements or other nutrients might be necessary.

The stimulating or inhibiting effect of a substance or waste component on methanogenic activity can be studied by adding different amounts of the substance in question to the test bottles. The measured specific methane producing activities (g methane COD/g bacterial sludge VSS/day) are then compared with a control (serum bottle free of investigated substance) and the degree of stimulation/inhibition is evaluated. Apart from the effect of different substances on the sludge activity, the effect of changes to the process (mixing, sludge thickening, recycle rate, etc.) can also be evaluated using this test.

The methanogenic activity test can also be used to assess the adaptability of a sludge cultured on a particular waste to anaerobic digestion of other wastewaters. For this purpose the test bottles are first fed with a simple substrate like acetate or ethanol to activate the methanogens. In a second feed dose the tested wastewater is added. This dosing serves to acclimatize the sludge to the wastewater. In a third dosing of the wastewater, the methanogenic activity is determined. The highest observed activity gives an indication of the cross-acclimatization and adaptability of the sludge to the wastewater. This can be a useful tool when deciding on what sludge to use as seed culture during the startup of an industrial anaerobic digester system.

The anaerobic SAT is set up the same way as the BMP test (Section 2.1). Other than with the BMP test, where the final amount of produced methane is recorded, the activity test records the rate of methane production over time; this can usually be done within one day, whereas the BMP might take 6 weeks or longer

2.2 PARAMETRIC BMP

The parametric BMP assay is a variation of the regular BMP test. Instead of a single condition for the treatment, a whole array (typically in a 4 x 4 matrix format) of different test conditions is assessed in the parametric BMP test. This allows for the rapid screening for suitable digestion parameter combinations that offer suitable reaction/process conditions. Parameters that are varied usually include loading rate, salt concentrations, and temperature or headspace composition. Many other parameters could be included in such a testing array and test array is typically specified by the process design engineers. The result of the parametric BMP test is a three dimensional (3D) response surface that allows for fast identification of suitable operational parameters as well as evaluation of the process robustness under the chosen conditions.

The setup for a parametric BMP follows the same procedures as a normal BMP test (see Section 2.1).

3 RESULTS AND DISCUSSION

3.1 ANAEROBIC SLUDGE ACTIVITY TEST

Figure 1 gives an example of a typical anaerobic sludge activity test (SAT). A CPG customer was concerned about the effect of air and chemical dosing applied during normal sludge dewatering on the viability of the methanogenic bacteria in mesophilic digester sludge in their municipal digester plant. A sample of the feed sludge to the dewatering treatment station and one of the dewatered treated sludge was obtained at the CPG laboratories by refrigerated courier, On arrival the sample was immediately tested using the procedures described in section 2 above. The results were obtained within 24 hours. The test data produced the consistent results that the relative methanogenic activity of the sample after the dewatering treatment was within 90 % of the activity of the untreated sludge control (Figure 1). The methane production rate was linear indicating that no changes in sludge activity occurred during the test. The test showed also that the sludge bacteria had sufficient carbon and energy source available from within the sludge material for effective anaerobic metabolism (good rate of methane production in the controls without ethanol addition). This result allowed the client to defer a significant amount of capital works and continue with the existing sludge dewatering practice.

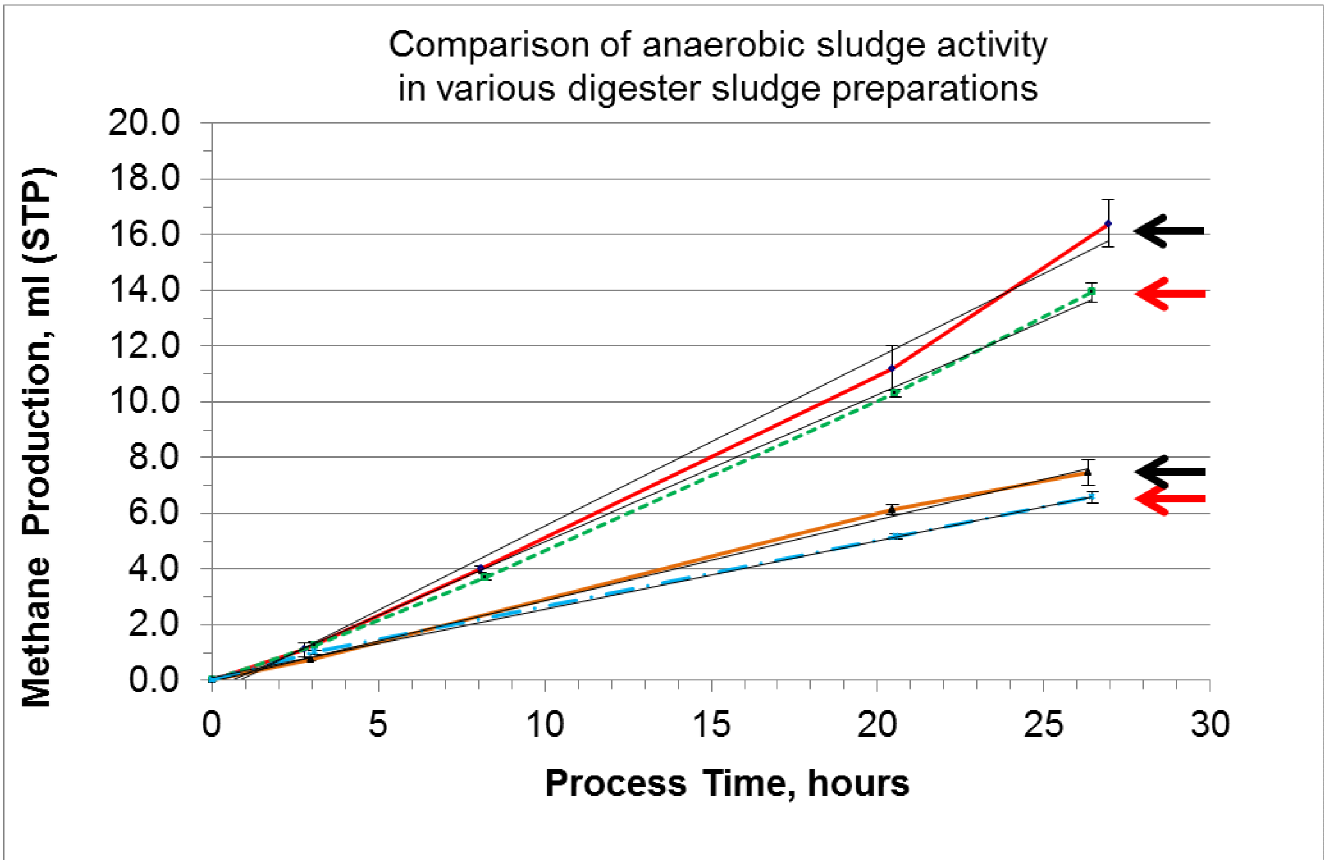

Figure 1: Example of the methanogenic activity comparison of mesophilic anaerobic sludge at 35 ^{o}C between matched samples. The samples were either taken from the mixed liquor of an anaerobic digester (about 1 % TSS, ←) or the thickened sludge recovered from the mixed liquor (5-6 % TSS) and exposed to polymer flocculants and air (←) . Top set of lines are incubations with 2000 mg COD/L added carbon source, bottom curve endogenous methane production without added carbon source). Each curve is the result of triplicate tests (error bar: standard deviation).

3.2 BIOLOGICAL METHANE POTENTIAL (BMP) TEST

The BMP test is a very powerful tool to determine a robust estimate of the amount of methane that can potentially be produced from a given waste material under optimum digestion process conditions. It is a batch test under well defined laboratory conditions and thus cannot fully represent the process conditions that prevail in a given full scale anaerobic digestion system for the given type and nature of the waste. BMP tests can be offered for solid waste with a TS concentration range from 20 % to > 90 % TS in the waste wet matter and for liquid waste with a concentration range of 0 – 20 % TS in the wet matter. CPG has successfully conducted BMP tests for materials with very high salt content (> 2 % salt), very high ammonium-N content (> 1 % ammonium-N) and very high fat content (up to 90 % fat, oil and grease in the dry matter).

The key to the application of this testing tool is the experience base that CPG have accumulated over more than 20 years of practice with this test in a large number of industries and anaerobic digestion systems types. BMP tests have been very successful to estimate the practically achievable methane yield on full scale in CIGAR™ reactor systems (Thiele JH, December 2011; webinar by the New Zealand Bioenergy Association). Typically between 80 % and 90 % of the BMP test result (m^3 methane/ t COD loaded) can be expected as practical methane yield in a full scale digester system. Table 1 below gives an example of a typical BMP methane yield calculation and Figure 2 an example of one BMP test run for a factory wastewater example. Calculations of the applicable methane yield from a BMP test are typically based on at least triplicate parallel BMP test runs.

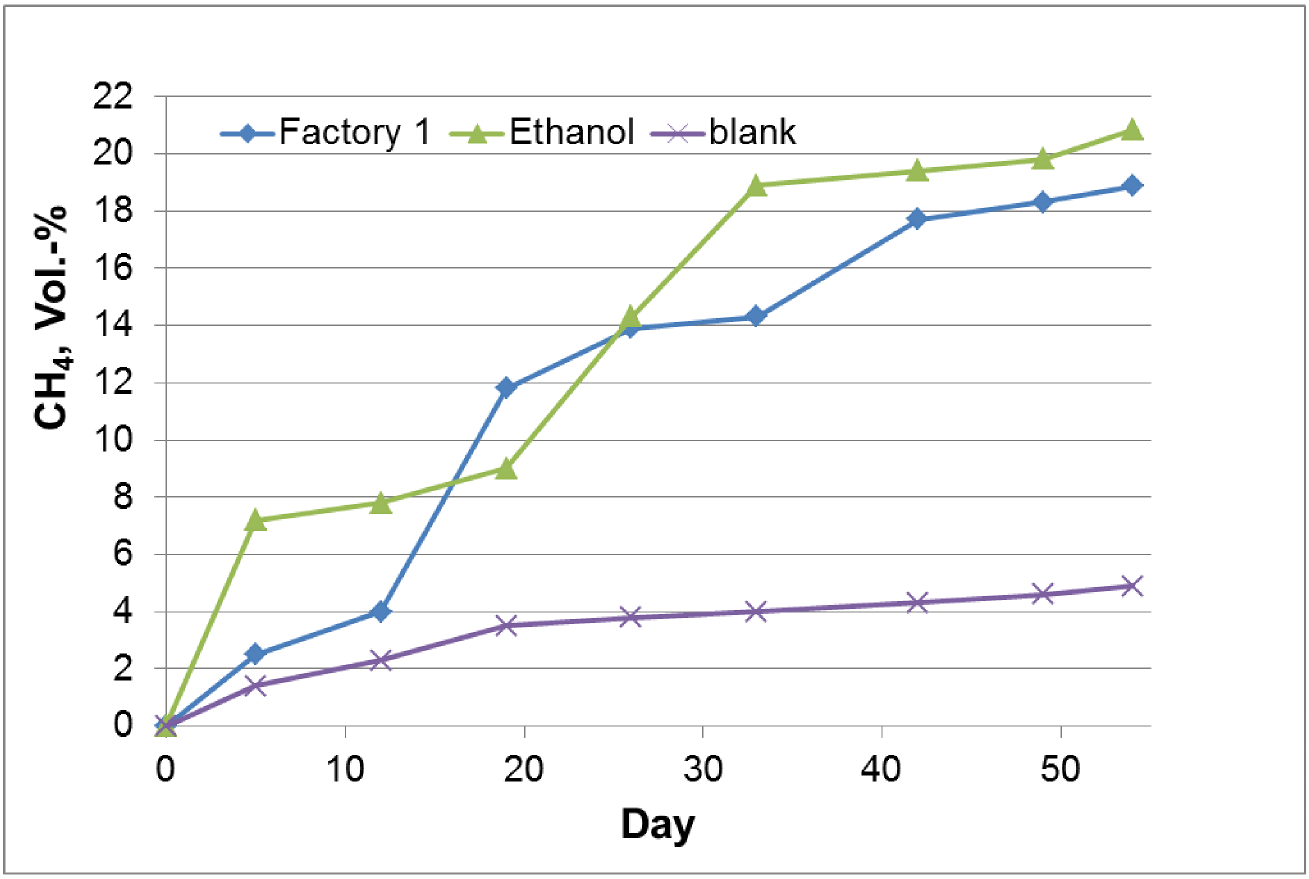

Figure 2: Example of one BMP test run at 35 °C for cassava starch wastewater using mesophilic anaerobic sludge as seed sludge (blank). The positive controls (Ethanol) demonstrate viability and validity of the test with a known amount of added ethanol as external COD source and the treatment (Factory 1) contains a known COD amount of the wastewater for which the tests determine the BMP. The actual BMP value for a given wastewater is the difference between triplicates of factory wastewater samples and triplicates of the blank after 50 days of testing.

Table 1: Sample calculation of the measured methane yield in the Biochemical Methane Potential (BMP) test. The theoretical methane yield for 1 t COD added is 315 – 330 m^3 methane/t COD_{added} if one conservatively allows for a 5-10 % conversion of the COD that is added to bacterial biomass in the test procedure. % RSD: relative standard deviation, % of the mean

Starch Factory BMP	Initial COD Concen-tration	Total Methane		Waste Specific Methane Yield		Waste Specific Methane Yield		COD conversion	
	(g COD/L)	(ml)		(m³ CH_4/t COD)		(m³ CH_4/t waste water)		%	
		Mean	% RSD	Mean	%RSD	Mean	%RSD	Mean	%RSD
Blank	0.0	6.0	2.5	N/A[b]	N/A[b]	N/A[b]	N/A[b]	N/A[b]	N/A[b]
Positive Control	2.1	31.4	4.9	309.8	6.1	508.4	6.1	91.1	6.1
Waste (Factory X)	2,0	31.7	4.2	320.7	3.5	3.4	3.5	94.3	3.5

3.3 PARAMETRIC BIOLOGICAL METHANE POTENTIAL (BMP) TEST

The parametric biochemical methane potential test was developed by CPG as a response of the large amount of exploratory enquiries about the potential for methane production from complex and difficult to treat solid and liquid waste materials. The test method gives the option to custom design a matrix of digestion system condition parameters against which the performance of typical anaerobic seed sludge cultures can be evaluated with the methane output as the key evaluation parameter.

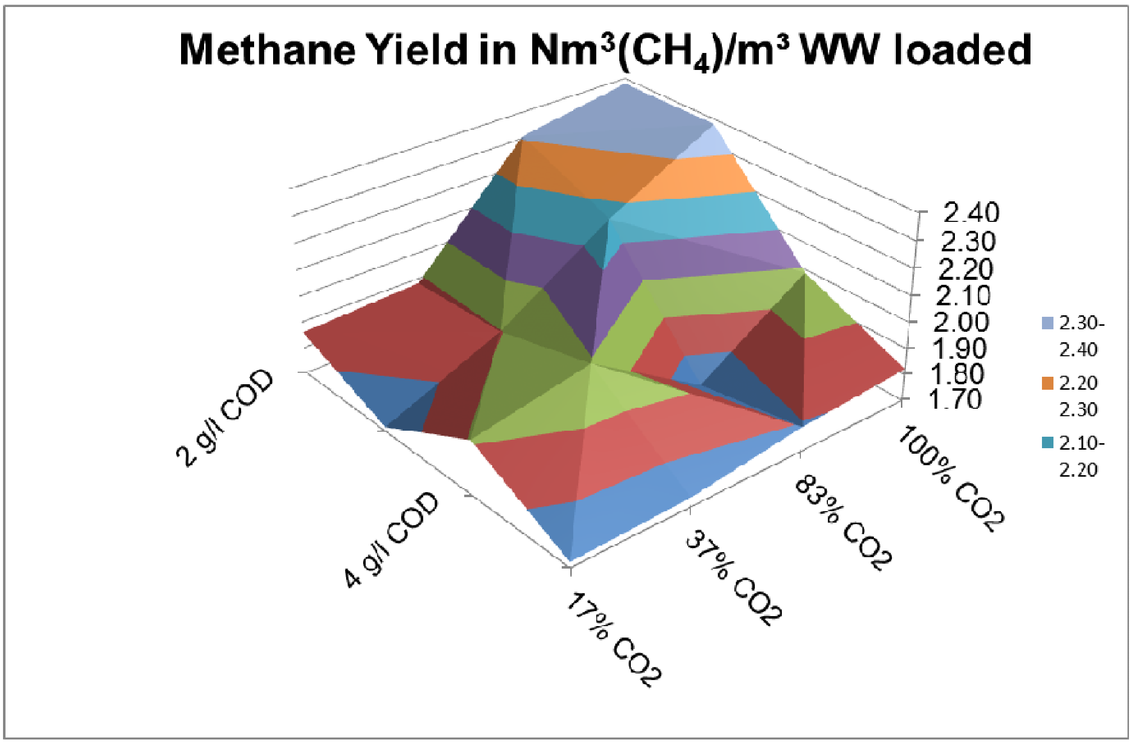

Figure 3: Example of a parametric BMP test at 35 °C for pharmaceutical factory wastewater using mesophilic anaerobic sludge as seed sludge. The negative controls (3 x blank) have already been subtracted from the presented data. The actual BMP value for a given wastewater treatment condition is the value on the Y axis of the parametric response surface. The actual test condition for each point on the parametric response surface is indicated by the X,Z axis

parameter combination for the test conditions and the Z axis value for the response. In this case the BMP is expressed on the Y axis as the m^3 methane produced/ m^3 wastewater loaded.

Typical a time series of three dimensional preliminary methane yield response series is obtained. Examples of response surfaces are shown in Figures 3 – 5. Each response surface is a snap shop in time of the dynamics of the anaerobic digestion of this wastewater. The laboratory technician determines by a quantitative test progress algorithm the relative degree of completion of the test for a given response surface (data not shown) .

The key for this test is that it can introduce the organic loading rate of a given digester system and the type of a given digester system (CSTR tank, UASB digester system) into the design of the parametric matrix in the X/Z plane of the test system

In the example shown in Figure 3 above, the X axis represents the loaded COD concentration faced by the seed sludge for a significant amount of time in the actual test and allows to determine the treatment process optimum parameter combination for the concept design of a treatment system for this particular wastewater. The "optimum" in this case would be estimated from the test results as a CO_2 concentration in the gas phase of > 83 % CO_2 and a COD concentration in the liquid not exceeding 3.2 g COD/L. This test is a very powerful tool for the feasibility assessment of the anaerobic treatment of inhibitory industrial wastewater.

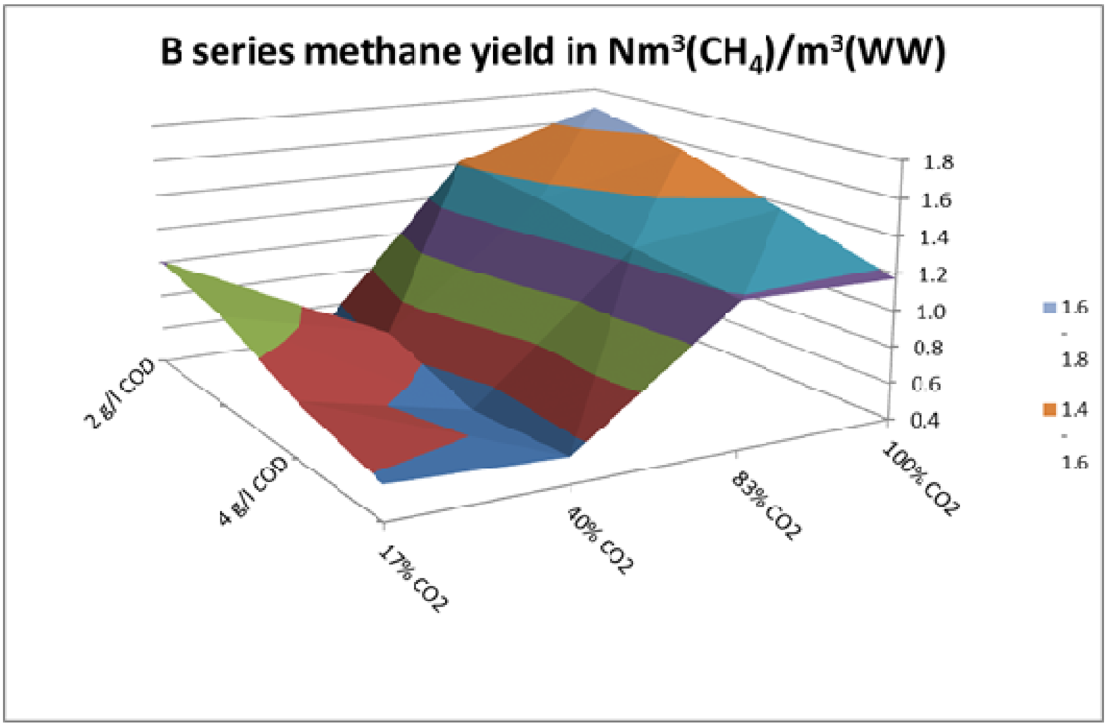

Figure 4: Example of a parametric BMP test at 35 °C for pharmaceutical factory wastewater. Units on X axis and Z axis as for Figure 4. In this case (for a different pharmaceutical wastewater), a secondary treatment optimum is indicated towards lower CO_2 concentrations. Note that the test predicts that digester loading with high COD concentrations is tolerated at high CO_2 concentrations above 83 % CO_2.

Figure 4 shows that the response surface results are specific for the actual nature of the tested wastewater and the method is thus suitable to detect wastewater characteristics and their effects on the anaerobic bacterial seed sludge that cannot be easily seen using a traditional BMP test (see 3.2). In this case the seed sludge, incubation time, COD loading rate and conditions were identical to the materials used for preparation of the response surface in Figure 3, but the nature & type of the tested wastewater was different in salt content and salt composition.

Figure5 below gives a good example how the parametric BMP test can be used to prevent major risks for the digestion system process design for full scale treatment systems. In this case an industry that produces about 1000 m^3/day of a wastewater with a very high COD and salt content needed assurance of the correct wastewater dilution prior to treatment. Typically, the VS content in the waste is used to size the reactor vessel for an anaerobic digester system. The results from the parametric BMP test however showed that for this particular application the potassium content of the waste was the more important process design parameter.

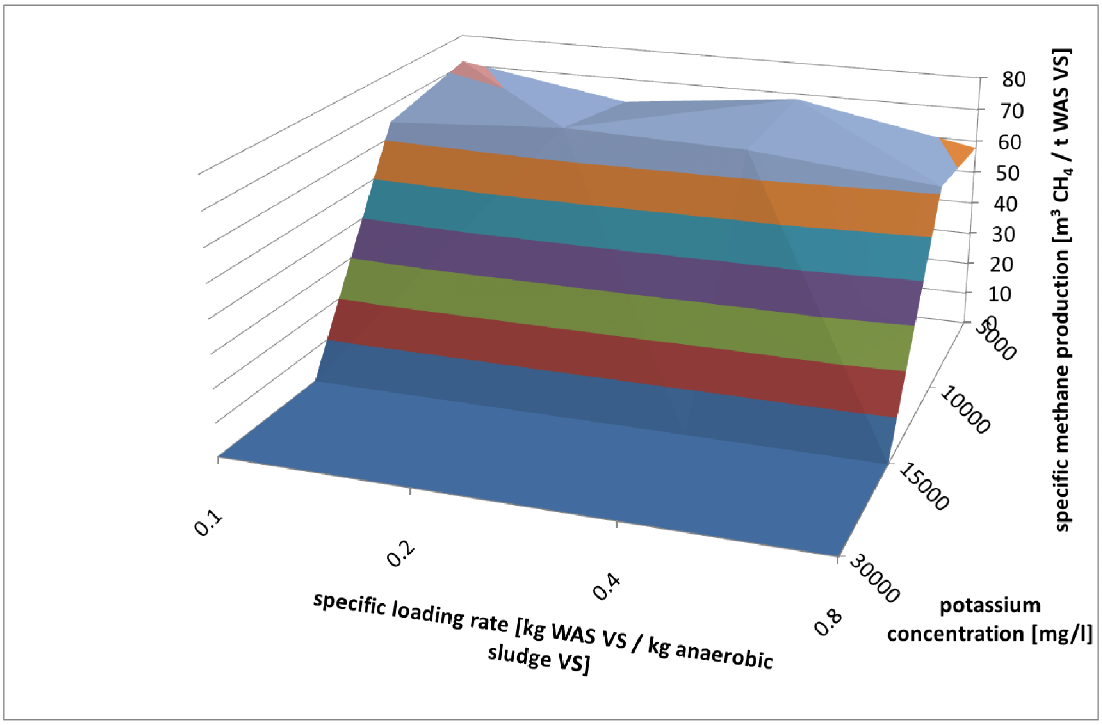

Figure 5: Example of a parametric BMP test at 35 °C for a fermentation wastewater. The actual BMP value for a given wastewater treatment condition is the value on the Y axis of the parametric response surface. In this case the potassium concentration in the wastewater has been identified as extremely inhibitory at a level of > 10,000 mg K/L whereas the organic content in the wastewater was not inhibitory in the tested range.

4 CONCLUSIONS

In conclusion, this paper has demonstrated that the tools available to the designer of modern cost effective anaerobic digestion systems on large scale are now much further advanced than the simple BMP test practices in the industry since several decades (Farooqi et al, 1993). Complex bioprocess design parameters like the "nature of the waste", toxicity to a given anaerobic digester seed sludge type and acceptable COD loading rate can be evaluated in a short period of time by high throughput screening techniques of the new parametric BMP test.

Once the suitable parameter space is defined and suitable parameter ranges are known, s traditional BMP test can then establish the precise revenue potential of a given complex solid waste or wastewater and a traditional anaerobic sludge activity test can be used to precisely quantify the robustness and activity of the anaerobic bacteria in the particular chemical and physical conditions that are set by the chemical composition of a particular solid or liquid waste material.

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