WASTE AND WASTEWATER CHARACTERISATION TO MINIMISE OPEX AND MAXIMISE ENERGY GENERATION

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Abstract

To realise OPEX savings and maximise energy generation it is being increasingly recognised that a detailed understanding of wastewater, feedstock and sludge characteristics is required. The data generated is of most use when inputted in to predictive, calibrated models that allow the user to simulate changes to the configuration of a plant, which alter the characteristics and subsequent running costs and revenues. Once verified the data gathered can be used to drive investment decisions and/or process operational strategies.

In this paper, we outline the standard and specialised test methods for waste and wastewater characterisation that can be used to populate & verify models such as BioWin. These include FRACTIONATION of: 1) Solids, 2) COD, 3) nitrogen and 4) phosphorus, as well as, 5) methods for evaluating total and available energy potential and, 6) the quantification of inhibitory compounds.

Sankey diagrams have been used to demonstrate the potential impact upon energy consumption and generation for a theoretical wastewater treatment works with a PE of 230,000 and taking sludge imports of 15 tonnes of dry solids.

Keywords

Wastewater fractionation Anaerobic digestion, CAPEX, OPEX, Wastewater characterisation, Wastewater treatment.

Introduction

This paper aims to review standard and bespoke test methods utilized for waste and wastewater characterisation to produce data for the population of BioWin models and Sankey diagrams. Such software applications and visual tools can then be used inform process operational strategies to reduce operating costs and maximise energy generation.

Current drivers for optimisation of municipal WwTW assets originate from Ofwat's reform on the expenditure model for investment, moving from a capital investment bias (CAPEX) to Total costs (TOTEX) model. The TOTEX expenditure model considers energy usage, operation and maintenance costs, encouraging a review and optimisation of current assets prior to capital investment.

Drivers for optimisation in the food waste and agricultural anaerobic digestion (AD) sector have been caused by uncertainty over incentives and feedstock competition. These changes have, on occasion, caused operators to switch from processing high volumes of feedstock to obtain a maximum gate fee, to a focus on achieving maximum energy generation for the feedstock available, by more efficient operation of the AD process.

The need for optimisation within the MBT AD sector results from the difficulties associated with preparing the organic fraction and separating recyclables from the bulk waste submitted for treatment.

Understanding waste and wastewater composition throughout the treatment process is vital when investigating the causes of plant down time, energy usage and poor performance. These causative factors which increase OPEX costs include: poorly separated feedstock, energy intensive processes, Inhibition to biological treatment, non-biodegradable substrates, nutrient deficiencies, foaming events, overloading and grit/inorganics deposition.

To maximise energy generation through the AD process, a full energy balance can be used to identify where energy may be being lost and the proportion of energy that is accessible via anaerobic digestion. Secondly, to facilitate the efficient conversion of all available organics to methane, the balance and availability of nutrients is also key.

Interpreting waste and wastewater characterisation data involves a fundamental understanding of: the treatment process in use, the limitations and methodology of standard tests and the value of specialised methods.

Specialised methods applied or reviewed in this paper include the fractionation of solids, COD, nitrogen and phosphorus, as well as methods for evaluating total and available energy potential and the quantification of inhibitory compounds. To illustrate how these methods can be applied on a whole works approach, Sankey diagrams have been used to model specific scenarios, highlighting areas within the process which may be affecting energy generation and contributing to increased OPEX.

Materials & Methods

Analytical methods used for quantifying wastewater characteristics are split in to three main categories. Direct methodologies whereby wastewater characteristics are measured from a direct measurement, for example the total suspended solids test where a certain filter size is used to define the TSS content of a sample.

The second category of methods for determining wastewater characteristics is the bioassay, whereby the response of a biological system is measured to infer the characteristics of a certain wastewater. For example, quantifying the % inhibition that can be caused by a specific wastewater, by measuring the response in terms of oxygen uptake rate (OUR) from a sample of activated sludge, when the influent wastewater is applied.

Thirdly, by direct calculation, for example dissolved solids could be determined by subtracting the total suspended solids value from total solids.

The following table provides a summary of specialised, direct and bioassay methods used for wastewater, feedstock and sludge characterisation, including their nomenclature, method scope, description and limitations. (Furthermore, Appendix 1 outlines the standard methods used for characterisation).

Table 1: Specialised methods for wastewater characterisation

Specialised tests	*Units*	*Method scope*	*Definition*	*Limitations*
Carbonaceous and nitrification inhibition testing	%	Bioassay, respirometry	Determine the % inhibition to carbonaceous and nitrification bacterial populations present in activated sludge	Batch scale test which does not account for accumulating inhibitory effects that may be observed in a continually fed system
BMP	L.CH_4/kg.VS or COD applied	Bioassay	Biochemical methane potential test to determine the suitability of feedstocks for anaerobic digestion	
BCOD biodegradability (OECD 301B)	%	Bio assay	Aerobic batch test to determine the concentration of biodegradable soluble COD	
COD fractionation *N fractionation* *P fractionation* *Solids fractionation*	mg/l mg/l/mg/kg mg/l/mg/kg % w/w, % of DS, mg/l	Multiple colorimetric/Bioassay methods Multiple gravimetric methods	Fractionation to quantify total, available, bound, biodegradable, non-biodegradable, soluble and particulate fractions	characteristics which are inferred from direct measurements rather than bioassays

To allow a further understanding of the individual specialised methods mentioned in the above table the following sections outline the aim of the testing and analytical method summary.

% Carbonaceous and nitrification inhibition

This test provides a % carbonaceous and nitrification inhibition for each provided sample at each dilution if necessary, this test can be used for quantifying the potential risk that can be caused to an activated sludge process by a certain waste stream. It is therefore useful when developing waste acceptance criteria for liquid imp0orted wastes.

Oxygen uptake rate (OUR) is measured over a 15-minute period using a dissolved oxygen probe and 275ml BOD bottle. There are 3 bottles, two contain a known volume of the Waste stream and a known volume of nitrifying activated sludge seed, one with and one without nitrification inhibitor, then a 3rd control sample comprising of distilled water. All tests are pH corrected and contain equal proportions of synthetic sewage to ensure maximum respiration rates can theoretically achieved.

To calculate the % inhibition for each sample the % difference between the OUR of the test material and control is measured.

The following chart shows an example of raw data obtained from the analysis for carbonaceous inhibition testing. As the OUR for the sample are less than the control, this indicates that the test material does not reduce microbial activity and is 0% inhibitory, for the sample to have an inhibitory response the OUR of the test material must be greater than the control.

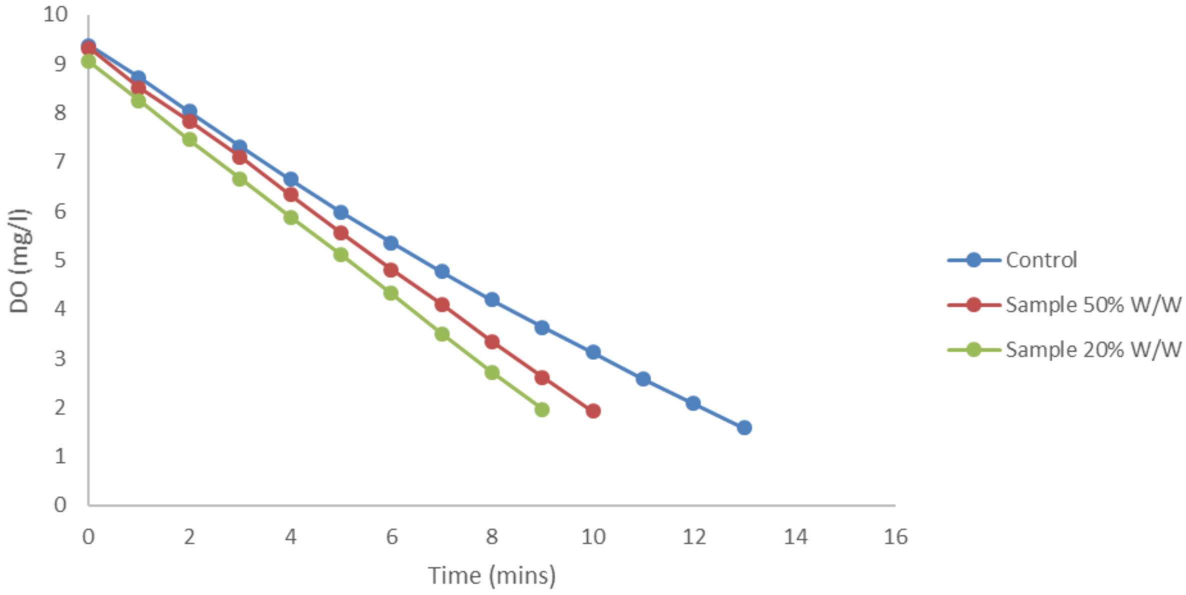

Figure 1: **Chart illustrating DO curves from carbonaceous inhibition test**

Biochemical methane potential

The BMP experimental methodology applied in this study was based on the protocol outlined in the "amenability of sewage sludge to anaerobic digestion 1977, methods for the examination of waters and associated materials" (Standing Committee of Analysts, 1977).

By quantifying the methane volume and concentration from a known mass of anaerobic seed and test material, the potential methane yield in terms of L.CH_4/Kg VS or L.CH_4/Kg COD applied can be determined.

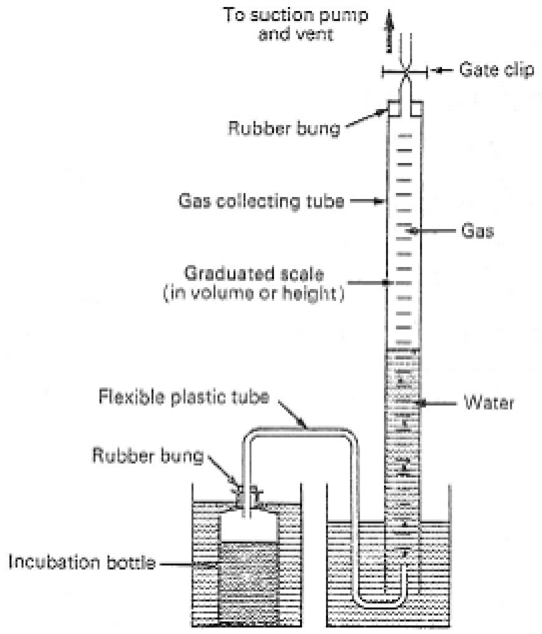

Figure 2: BMP equipment diagram Standing Committee of Analysts. (1978).

Schematic for the BMP equipment used is displayed in figure 1. Vessels are held at mesophilic temperatures at 35^{0}C +/-1 using a water bath over a 14-day test period (longer time periods can be used depending upon the nature of the material tested). Biogas volume is measured via liquid displacement; the aqueous phase corrected to pH 4 to minimize CO_2 solubilisation.

Methane concentration is quantified using a Geotech GA5000 instrument capable of measuring CH_4%, CO_2% and O_2%. Seed material volume is 400ml, substrate applied is based on achieving a seed to substrate ratio of 80/20% on a VS or COD basis depending on the solids concentration of the feedstock. The following calculations are used to determine the mass of sample required for the testing and the BMP value of each sample.

$$VS\ of\ sample\ req.g\ =\ \%\ of\ sample\ required\ as\ VS \times (VS\ in\ seed\ g/l\ \times seed\ vol.L) \qquad \textbf{Equation 1}$$

$$Mass\ of\ test\ material\ to\ add\ g = VS\ sample\ req.g\ \times \frac{1000}{VS\ sample\ g/L} \qquad \textbf{Equation 2}$$

$$\text{BMP L.CH}_4\text{/kg.VS applied} = \frac{methane\ produced\ mls}{1000} \times \frac{1000}{VS\ of\ sample\ req.g} \qquad \textbf{Equation 3}$$

The following chart illustrates an example of the BMP evolution, in this case for surplus activated sludge, the chart displays the initial biodegradation of the readily biodegradable fraction from days 0-2, followed by the slow degradable fraction from day 2 onwards, the final BMP value for this feedstock was 153 L.CH_4/kg.VS applied.

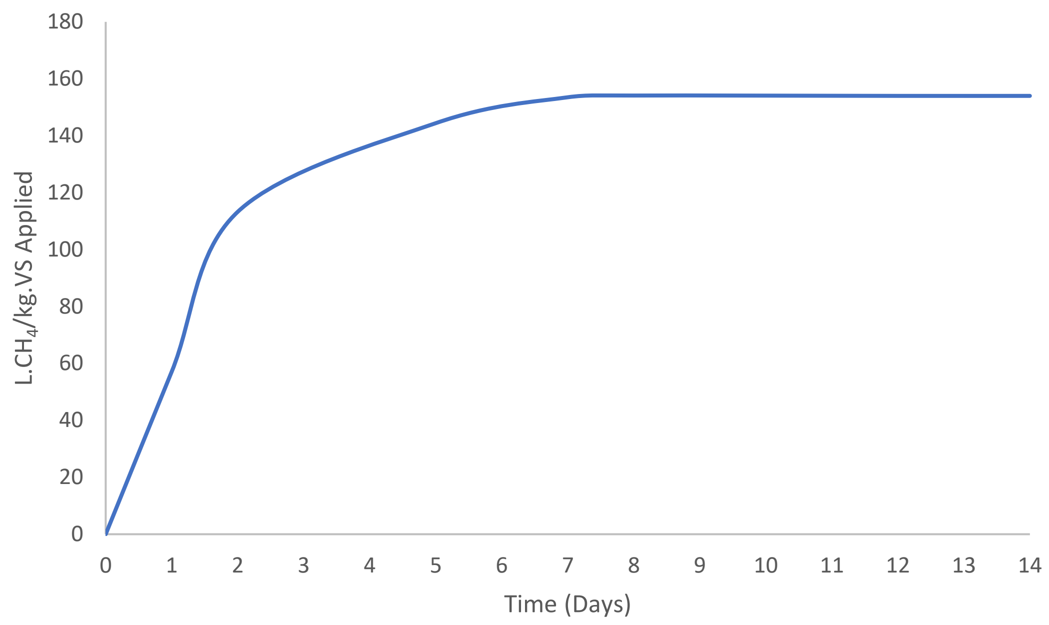

Figure 3: BMP evolution curve for thickened surplus activated sludge

The BMP value gives an indication of the energy available through AD, where calorific value or COD analysis is undertaken it can also be expressed as a percentage availability.

Biodegradable COD % (OECD 301A)

The OECD 301B Zahn Wellens test is used to quantify the proportion of biodegradable COD within samples. A mixture containing a known volume of the test substance, mineral nutrients and activated sludge in aqueous medium is agitated and aerated at 20-25°C in the absence of light for 28 days.

Controls containing ethylene glycol at 1g/l and blanks containing activated sludge and mineral nutrients but no test substance are run in parallel. The biodegradation process is monitored by determination of COD in filtered samples, taken at frequent time intervals.

The ratio of eliminated COD, corrected for the blank, after each time interval, to the initial COD value is expressed as the percentage biodegradation at the sampling time. The percentage biodegradation is plotted against time to give the biodegradation curve fig.4 OECD (1992).

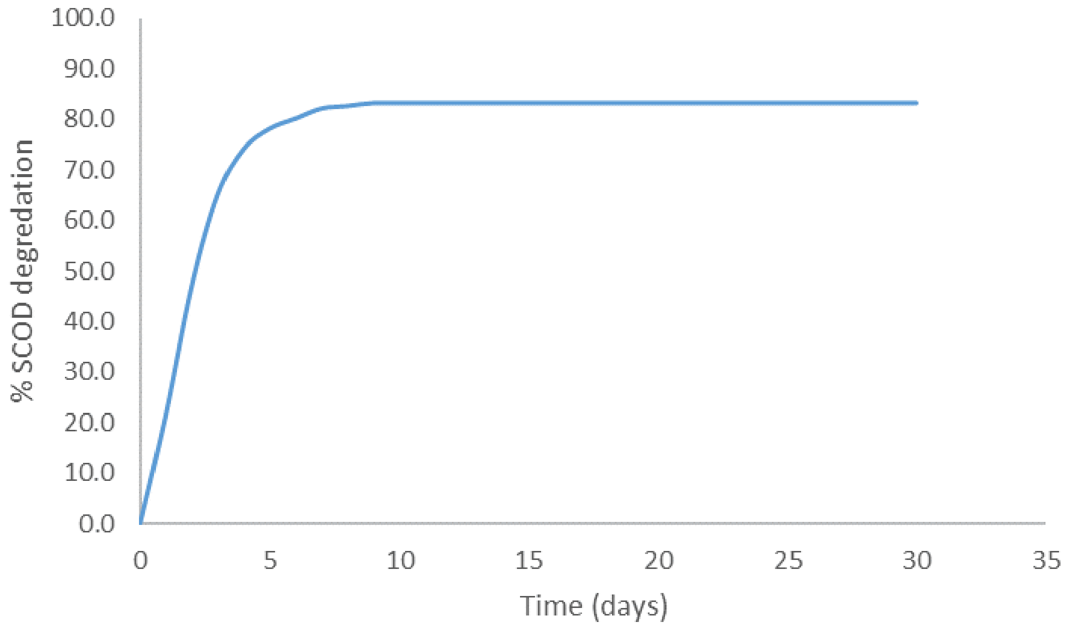

Figure 4: **Chart depicting typical SCOD degradation**

COD fractionation

The COD of a sample is one method of measuring the total energy, fractionation of the COD (figure 5) is useful to understand different removal options. The following procedure is used to measure these fractions and follows guidelines developed by the Dutch Foundation for Applied Water Research (STOWA).

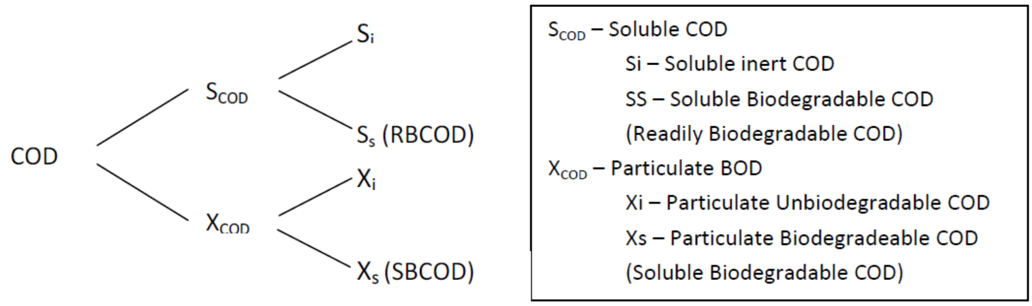

Figure 5: **STOWA COD fractions**

The STOWA method requires a final effluent produced from the waste stream to calculate the various COD fractions. If the waste stream originates from a site without effluent treatment, a 2 day aerobic batch test is undertaken to indicate RBCOD and produce an indicative final effluent for analysis, table 2 illustrates the required analysis and calculation used to complete the COD fractionation.

Table 2: COD fractionation determinants

COD fraction	*Abbreviation*	*Units*	*Method for determination*
Soluble inert COD	Si	mg/l	ffCOD Fe
Soluble Biodegradable COD	Ss	mg/l	ffCOD Ci - ffCOD Fe
Particulate Unbiodegradable COD	Xs	mg/l	BOD ultimate (BCOD) - Ss
Particulate Biodegradable COD	Xi	mg/l	COD Ci -Xs - Ss - Si

Nitrogen fractionation

The TKN of waste / waste water streams tested are split into their constituent fractions (figure 6), following guidelines developed by the Water Environment Foundation.

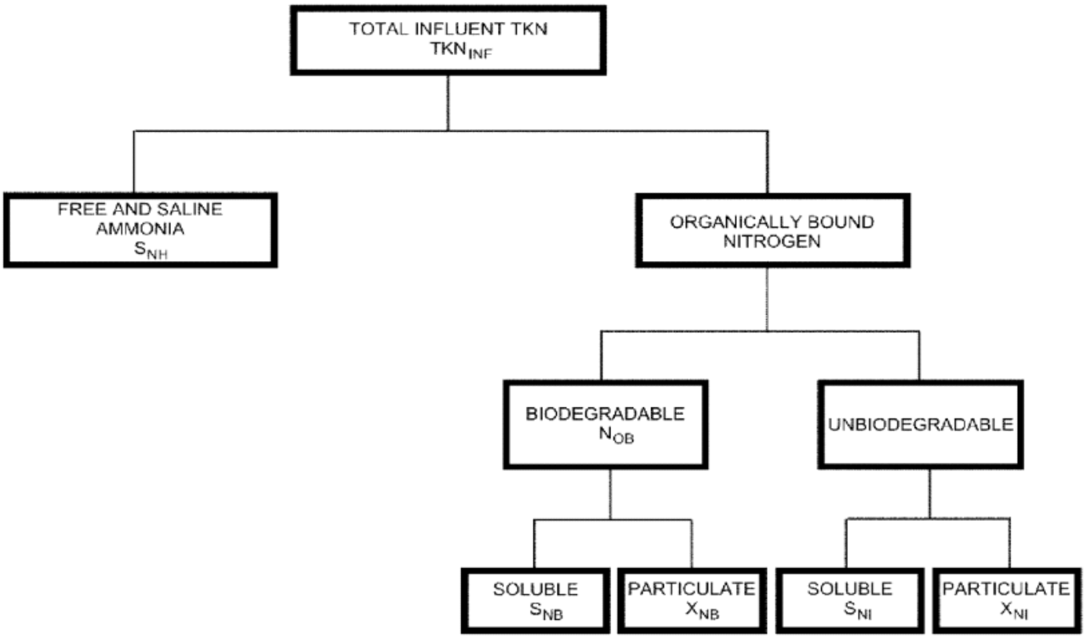

Figure 6: TKN fractions (Melcer 2003)

Table 3 illustrates the required analysis and calculation used to complete the TKN fractionation.

Table 3: Nitrogen fractionation (Melcer 2003)

Nitrogen fraction	**Abbreviation**	**Units**	**Method for determination**
Free and saline ammonia	S_{NH}	mg/l	NH_3-N
Organically bound nitrogen	O_{NH}	mg/l	TKN - NH3-N
Particulate organically bound nitrogen	$X_{NB} + X_{NI}$	mg/l	Filtered TKN - TKN
Soluble organically bound nitrogen	$S_{NB} + S_{NI}$	mg/l	Filtered TKN - NH3-N
Soluble biodegradable organically bound nitrogen	S_{NB}	mg/l	(Filtered TKN - NH_3-N)*0.97 (0.97 indicative factor)
Soluble unbiodegradable organic nitrogen fraction	S_{NI}	mg/l	(Filtered TKN - NH_3-N)*0.03 (0.03 indicative factor)

Phosphorus fractionation

Fractionation follows WEF guidelines.

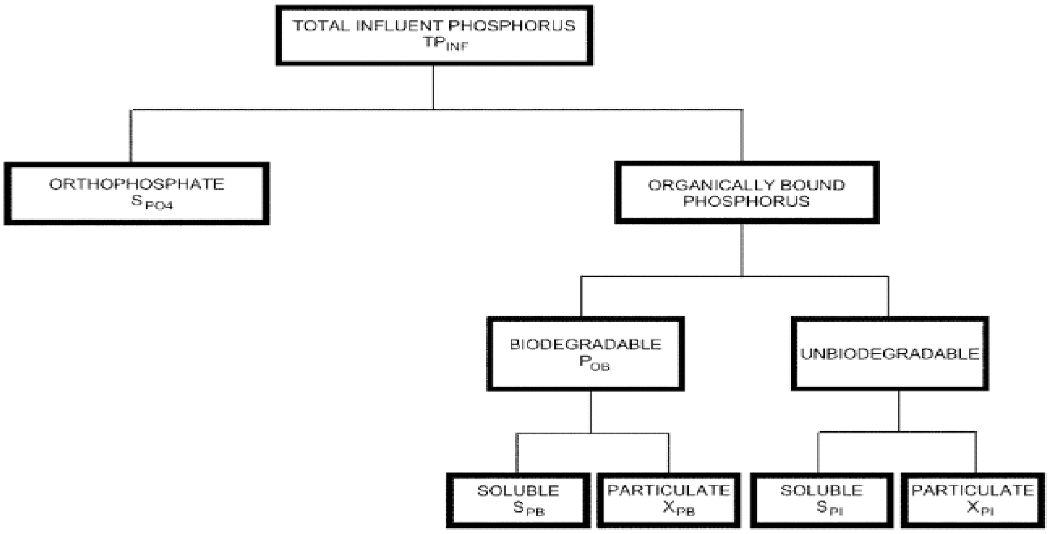

Figure 7: P Fractions (Melcer 2003)

The following method is applied to calculate the various P fractions, table 4 illustrates the required analysis and calculation used to complete the P fractionation.

Table 4: P Fractionation

Phosphorus fraction	Abbreviation	Units	Method for determination
Orthophosphate	S_{PO4}	mg/l	PO_4^{3-}P
Organically bound Phosphorus	O_{BP}	mg/l	P - PO_4^{3}-P
Particulate organically bound Phosphorus	$X_{PB} + X_{PI}$	mg/l	Filtered P - P
Soluble organically bound Phosphorus	$S_{PB} + S_{PI}$	mg/l	Filtered P – PO_4^{3}-P

Results

Maximising energy generation

BMP and COD fractionation

Biochemical Methane Potential (BMP) and biodegradable COD (BCOD) analyses provide measures of the potential energy generation and losses (e.g. filtrate). Samples can be taken directly from sites, or where new technologies/processes are proposed developed through bench scale treatability trials, or through a combination of both.

Sample points include the digester feeds and blend, pre and post pre-treatment (biological hydrolysis), digester, digestate holding tank and dewatered fractions.

Results are displayed on a fresh weight and COD basis to allow a direct comparison between samples of liquid, sludge and semi-solid matrix, the results displayed in fig. 8 include the following fractions.

- Theoretical maximum methane yield L.CH4/kg.COD applied
- Actual methane yield L.CH4/kg.COD applied
- Actual methane yield L.CH4/kg.fresh applied

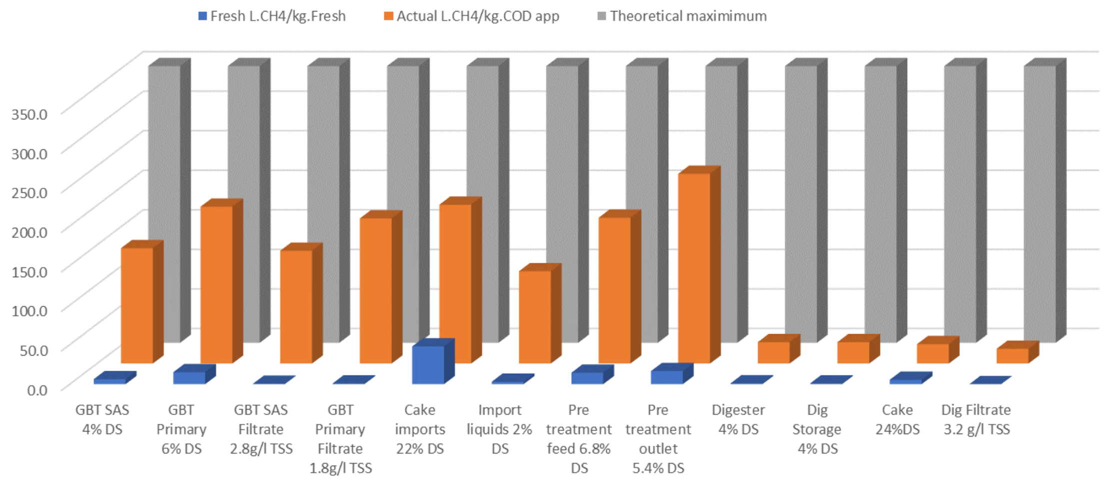

Figure 8: Chart illustrating BMP fractionation

This BMP assessment allows a greater understanding of the potential methane yield achievable for each sample, to highlight where energy may be recovered and/or maximised. Of particular interest are the losses in the system, which include: 1) filtrate from the gravity belt thickeners (GBTs) for Primary sludge, 2) Filtrate from the Surplus Activated Sludge (SAS) GBTs, and 3) 'open' storage and mixing of the digestate.

Analysis is undertaken on each of these streams to evaluate the methane potential and readily biodegradable COD, the latter is an opportunity in the feed to the digesters and a cost in any liquors that would require aerobic treatment.

Opportunities for further energy generation can also be identified from the biodegradability of the material by undertaking a COD fractionation on each sample, fig. 8 displays the biodegradable and unbiodegradable and total COD concentration of each sample.

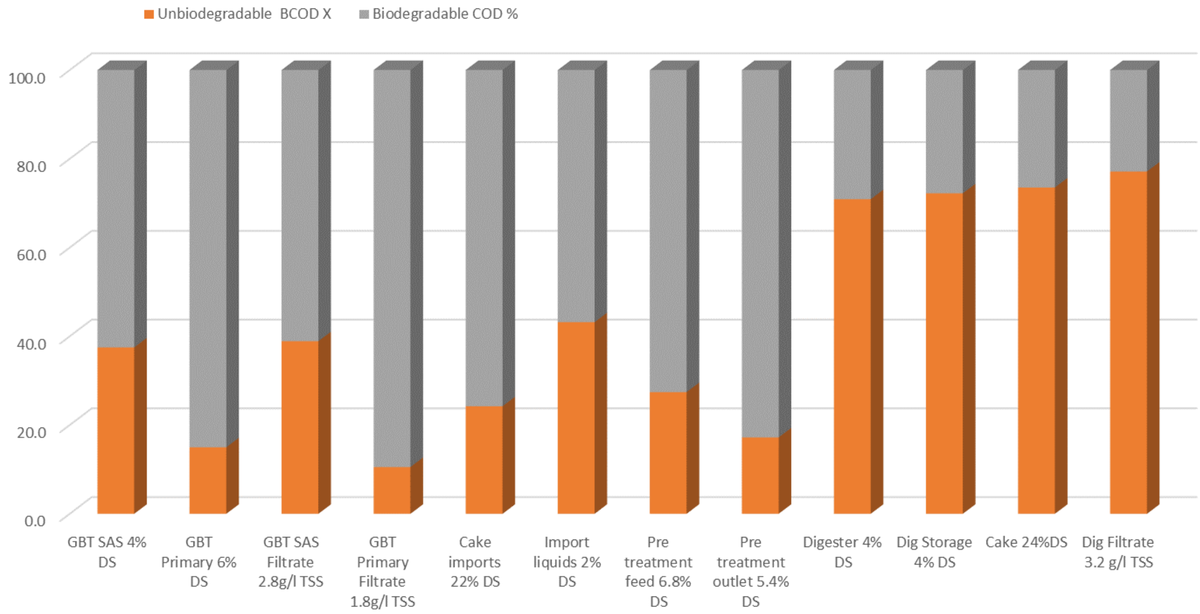

Figure 9: Chart illustrating COD fractionation

This chart allows the Samples with a relatively high unbiodegradable fraction to be identified for further pre-treatment, or potentially isolated and removed from the process.

Minimising OPEX

Biodegradability, toxicity and inhibition

Screening trade effluents and industrial sludge imports is vital in reducing operational costs relating to plant downtime, caused by overloading, inhibition and toxic shock to biological processes.

Five samples of potential sludge imports and 5 samples of trade effluents to illustrate the potential risk to treatment are shown in figure 10.

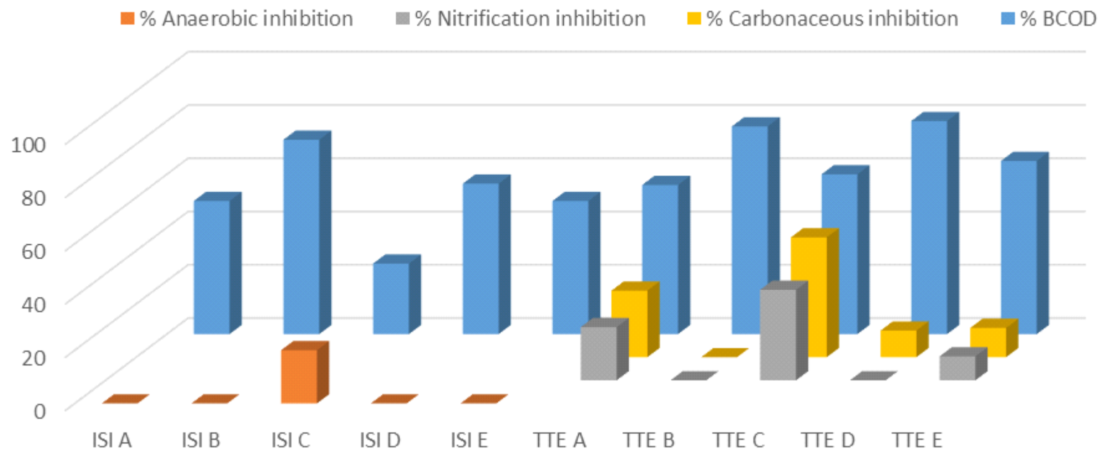

Figure 10: Chart illustrating BCOD, inhibition/toxicity potential of a variety of trade effluents and sludges

This chart allows the Samples with inhibitory properties and high unbiodegradable COD content to be identified for further pre-treatment, or potentially isolated and removed from the process.

The following table summarises the opportunities for reducing OPEX, energy maximisation and recovery, including potential improvement actions for optimisation.

Table 5: Summary of optimisation opportunities identified from the data and potential improvement actions

Optimisation opportunity	Improvement action
Solids lost in dewatered GBT and SAS liquor	Improve dewatering performance and solids recovery
low methane yield for SAS and sludge imports	Further pre-treatments to increase BCOD fraction
Residual biogas in digestate	Increase retention time, feed DS or capture biogas from storage
Digester performance	Apply nutrients, optimise OLR and improve feedstock balance
Inhibitory feedstocks	Identify, isolate and remove or charge elevated fee

Estimating costs & revenues

COD, Fractionation and BMP data can be used to develop basic cost/revenue models and identify, for further investigation, potential changes to a site's operation or even to outline areas for capital expenditure. It is particularly useful where either a waste digestion or water company operates similar treatment processes across its asset base, in this case it can be used to develop baseline data and identify the reasons for similarities/differences in performance.

Energy (expressed as MWh) is a common metric used by businesses for output from CHP engines or gas-to-grid systems, with the key performance indicator of MWh/tonne of dry solids processed being used across the water industry. It is less common in the measurement of energy, however, to express the feed inputs to the digestion system, the cake produced or the liquors generated as energy flows.

Calorific value analysis, by bomb calorimeter, for solid/semi-solid samples that cannot be effectively diluted or pipetted, or COD analysis for liquid samples both provide repeatable and accurate measure of the energy value.

Table 3: Calorific value of sludges (adapted from Smith, 2014 & Mills, 2015)

Parameter	Mills	Smith	
	Primary & SAS	Primary	SAS
Volatile solids %	77	70	80
CV (MJ/kg DS)	19	25	21
CV (MJ/kg VS)	24.7	35.7	26.25
COD: VS	1.93	2.79	2.05

The former provides values in MJ/kg, the latter in mg/l, both are easily converted in to kWh or MWh: 1) 1 kWh = 3.6 megajoules, 2) 1 kg of COD = 3.56 kWh. Therefore, where the COD or CV is measured and the associated flow is known the energy flow can be calculated.

Taking the data shown in the examples and developing an energy diagram for a site treating ~230,000 PE (population equivalent) and importing 15 tonnes of dry solids per day, the energy flows can be mapped.

Table 3: Input data to Sankey diagram

Input	MWh per day
GBT thickened SAS	56.4
GBT SAS filtrate	2.5
GBT thickened primary	31.4
GBT primary filtrate	8.5
Liquid imports	18
Cake imports (diluted with final effluent for pumping)	46.2
CHPe	32.2
Digester biogas (other, including losses, flare, boiler)	48
Digester storage/secondary digestion (not collected, measured through BMP analysis)	10
Digested cake	60.3
Centrate	1.5
TOTAL	216.2

The total tonnes of dry solids equivalence of the site is ~35, the table and Sankey diagram show:

- 216.2 MWh total energy in the sludge, equivalent to 6.2 MWh/TDS
- 2.29 MWh/TDS in the form of energy in the biogas; a 37% conversion.
- 0.91 MWh/TDS after CHP; equivalent to 14.7% of the total energy in the sludge.
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- 10 MWh (4.6%) is lost to atmosphere
- 12.5 MWh (5.8%) returned to the works in the form of filtrates & centrate

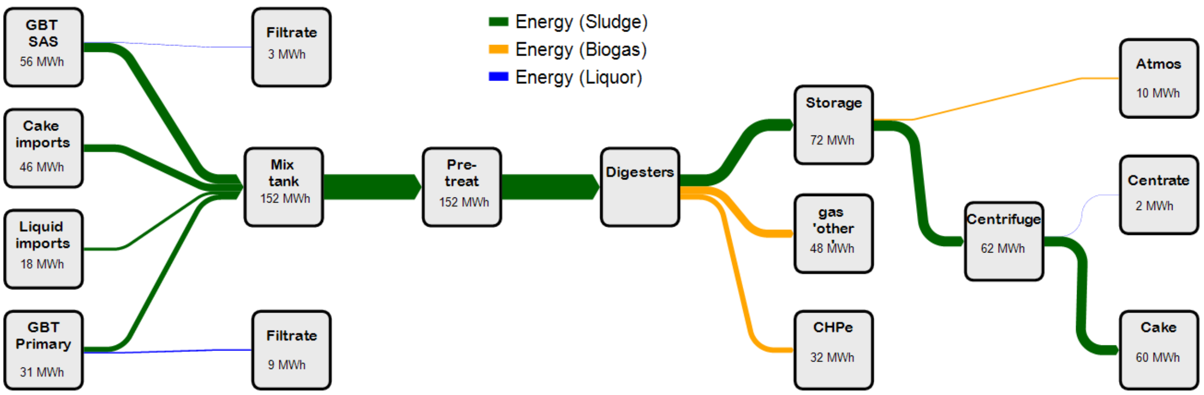

Figure 11: Energy diagram

Of the losses in the system, capturing gas from digestate storage would require capital upgrades, however, increasing the hydraulic retention time of the digesters (potentially possible by increasing the concentration of dry solids fed) would reduce this loss.

Returns in the centrate from cake dewatering have a relatively low proportion of biodegradable COD, however returns from the GBT primary and SAS have a biodegradable COD content of >60% and methane yields of 140-180 litres of methane per kilogram of COD applied. Options therefore to reduce energy lost in the filtrate (e.g. reduced retention time in primary tanks) or to blend raw primary/SAS with cake imports could offset, in part, these losses. The potential increase in biogas production is equivalent to ~7 MWh per day.

In addition, the readily biodegradable filtrate will, without intervention, require treatment in the aerobic treatment plant. The returned readily biodegradable load is equivalent to ~2 tonnes per day. Assuming: 1) a COD: BOD of 2:1; 2) 2 kg of oxygen per kilogram of BOD oxidised; 3) 3 kg of oxygen per kWh; and 4) £0.10 per kWh; the daily treatment cost for aeration alone is £67/d or ~£24k p/a.

Conclusions

In understanding a wastewater treatment and/or digestion plant on a variety of feedstocks it's essential to understand where and how energy is transformed and lost from a closed system. specialised methods are an invaluable diagnostic tool to review and optimise the waste and wastewater treatment process.

When the aim of the investigation is to maximise energy generation throughout the process, biochemical methane potential analysis coupled with COD fractionation allows a full energy balance to be completed factoring in the total and biodegradable fractions of each waste stream.

Once the fractionation is completed this allows areas for potential optimisation to be identified, these could include solids lost in dewatered GBT and SAS liquor, low methane yields for SAS and sludge imports, residual biogas in digestate and digester performance under design capacity.

Improvement actions to maximise energy generation from the example data included improving dewatering performance and solids recovery, further pre-treatments to increase BCOD fraction of SAS and improved digester performance (nutrients, OLR, and feedstock balancing).

Using the data obtained for a STW treating 230,000 PE and receiving imports of 10 TDS/d, the cost associated with treating GBT and SAS liquor aerobically was ~£24,000 per annum. It also must be noted that this is energy that is lost and not recovered via anaerobic digestion. In addition, making use of the energy in these liquors (which is largely BCOD), either through diluting cake or reducing the retention time in storage tanks/processes could lead to an increase of 7MWh of biogas per day.

Specialised methods for the determination of biodegradability, toxicity and inhibition can be used to reduce operational costs associated with plant downtime, this analysis allows inhibitory, toxic or non-biodegradable sludge imports or trade effluent to be identified for removal, to reduce plant downtime associated with overloading, inhibition and toxic shock to biological processes.

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Appendix 1

Table 5: Standard tests for wastewater characterization (Clesceri 1998).

Standard tests	*Units*	*Test method*	*definition*	*Limitations*
Chemical oxygen demand	mg/l	Direct, Manganese digestion, Colorimetric	Total Organic contaminants	Affected by Chloride interference
Soluble Chemical oxygen demand	mg/l	Direct, Manganese digestion, Colorimetric	Soluble Organic contaminants	Affected by Chloride interference
Flocculated and filtered COD	mg/l	Direct, flocculation and precipitation of colloidal matter using zinc sulphate and pH correction 10.4, followed by COD analysis of filtrate (0.45µm)	Influent readily biodegradable COD by subtracting ffCOD of influent from ffCOD of effluent	Purely indicative as it is assumed all compounds < 0.45µm are RBCOD
Biochemical oxygen demand 5 day/ BOD ultimate 10 day	mg/l	DO uptake Manometric / Respirometry	Aerobically Biodegradable organic contaminants	Seed material required for sterile samples
Soluble Biochemical oxygen demand	mg/l	Bioassay, DO uptake Manometric / Respirometry	Soluble Aerobically Biodegradable organic contaminants	
Calorific value	mj/kg	Bomb calorimetry	Total Energy value	Homogenous sample is required to obtain an accurate result
Phosphorus	mg/l/mg/kg	Acid persulphate digestion,		Interference can be caused with

		Colorimetric / MS or OES		samples of high colour conc, when undertaking colorimetric methods, specific metals can also cause interference if at sufficient concentration
Orthophosphate	mg/l	Ascorbic acid, Colorimetric		
Nitrogen	mg/l/mg/kg	Persulfate digestion, Colorimetric / TKN		
Ammonia	mg/l	Nessler method, Colorimetric		
Nitrite	mg/l	Ferrous sulfate, colorimetric		
Nitrate	mg/l	Cadmium reduction colorimetric		
Dry solids	% w/w	Dried 105ºC, Gravimetric	Total solids	method does not account for volatile organics lost in the evaporation process, not suitable for samples containing Oil
Volatile solids	% of DS	Ignited 1550ºC, Gravimetric	organic fraction of total solids	
Ashed solids	% of DS	Ignited 1550ºC, Gravimetric	inorganic fraction of total solids	
Dissolved solids	mg/l	1.2µm filter, Gravimetric	solids in solution	
Total suspended solids	mg/l	1.2µm filter, Gravimetric	Solids in suspension	
Volatile suspended solids	mg/l	1.2µm filter ignite 550^{0}C , Gravimetric	organic fraction of total suspended solids	
Specialised tests	Units	Method scope	Definition	Limitations
Carbonaceous and nitrification inhibition testing	%	Bioassay, respirometry	Determine the % inhibition to carbonaceous and nitrification bacterial populations present in activated sludge	Batch scale test which does not account for accumulating inhibitory effects that may be observed in a continually fed system
BMP	L.CH_4/kg.VS or COD applied	Bioassay	Biochemical methane potential test to determine the suitability of feedstocks for anaerobic digestion	

SCOD biodegradability (OECD 301B)	%	Bio assay	Aerobic batch test to determine the concentration of biodegradable soluble COD	
COD fractionation	mg/l	Multiple colorimetric/Bioassay methods	Fractionation to quantify total, available, bound, biodegradable, non-biodegradable, soluble and particulate fractions	characteristics which are inferred from direct measurements rather than bioassays
N fractionation	mg/l/mg/kg			
P fractionation	mg/l/mg/kg			
Solids fractionation	% w/w, % of DS, mg/l	Multiple gravimetric methods		