Parameter Identifiability using Gas Flow Rate and pH Measurements from Anaerobic Batch Reactor Experiments

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Abstract
This paper is part of a wider study into model-based control of an anaerobic digester with off-line parameter estimation using batch or sequencing batch experiments. This paper considers parameter estimation and identifiability from off-line batch experiments. A parameter identifiability study based on ethanol degradation is presented; it was found that a maximum of five parameters can be determined when gas flow rate and pH are measured in a laboratory-scale batch experiment in which brewery sludge was fed with ethanol. The identifiable parameters were the ethanol dose, carbon dioxide mass transfer coefficient, any one parameter concerned with the acetate degradation, any one parameter related to the ethanol degradation and any parameter linked to dissolved hydrogen usage. The influence of including and excluding pH data on the parameter estimation was investigated and it was found that inclusion of pH data gave more precise parameter estimates (smaller confidence limits) on the estimated parameters, but did not influence the number of parameters that were identifiable.

Keywords
Anaerobic Sequenced Batch Reactor; Parameter Identifiability; Anaerobic Digestion Modelling; Co-digestion; Weijers and Vanrolleghem (1997) Method

INTRODUCTION

Anaerobic digestion offers a number of potential advantages as an alternative to aerobic digestion for the treatment of high strength and toxic industrial wastes, due to low energy input, potential power generation via methane, and long residence time, which increases the possibility of degrading refractory organic substances. However, its major drawback is the possibility of the system failing, which can be mitigated by co-digesting the industrial wastes with ordinary municipal waste, and by advanced control of the process. This study was part of a pilot project to evaluate model-based control of a co-digestion process. The success of the co-digestion modelling approach hinges on the acquisition of key parameters. It has been suggested that acetoclastic methanogenesis is key to the stability of the digester thus needs to be characterised accurately(van Rensburg et al., 2001).

The anaerobic sequenced batch reactor (AnSBR) has been selected as the experimental system for determining parameters required for the co-digestion model. This selection was based on the relatively low cost of the set-up, as well as the ease of operation (Batstone et al., 2004). There is also flexibility in the manner in which AnSBR experiments can be run, in terms of multiple doses of various substrates. The laboratory-scale AnSBR had continuous gas flow rate and pH measurements. The focus of this investigation was on determining how many and which kinetic parameters could be determined reliably from gas flow rate and pH measurements.

MATERIALS AND METHODS

Modelling

Modelling of an anaerobic system can be divided into three main categories: the biological reactions, the physico-chemical processes, and the reactor unit model. In this instance, the UCT ADM2 model was used to describe the biological and physico-chemical system(Ikumi, 2011).UCT ADM2 uses differential equations to describe the kinetics of the biological reactions, but describes

aqueous ionic reactions using an algebraic equilibrium speciation model(Brouckaert et al., 2010). The gas phase model, assumes that the methane is insoluble and carbon dioxide is soluble. Thus carbon dioxide transfer to the gas head space is modelled using non-equilibrium liquid-gas transfer (Batstone et al., 2002). Since ethanol is not a standard state variable in the UCT ADM2, this study, extended the model base with the addition of ethanol degradation stoichiometry and kinetics.

Ethanol was selected as the reference substrate as it has been shown to give batch experiment gas flow rate and pH curves with two distinct regions corresponding to predominantly acetogenic and predominantly methanogenic activity in the experiment(Batstone et al. 2004). It was anticipated that kinetic constants and possibly active micro-organism concentration data might be identifiable for the two different micro-organism populations from a single substrate under such conditions.

The unit model for the AnSBR was developed from Artan et al. (2002) and Bagley and Brodkorb (1999). Each phase; filling (a few seconds long), reacting (eight and a hours), settling (thirty minutes) and decanting (ten minute); were modelled independently. Biological reactions took place during the filling and reacting phase. The settling in the unit was modelled as a point-settler with no reaction taking place as it is assumed that the substrate concentration is negligible at the end of the reaction.

WEST (www.mikebydhi.com) was used as the modelling environment for these models.

Parameter Identifiability

The method proposed by Weijers and Vanrolleghem (1997)was used to rank the parameters according to their identifiability. The reliability with which the parameters could be estimated was studied by first using a base set of parameters to generate gas flow rate and pH data for a simulated experiment, and then perturbing each of the parameters in the parameter subset selected from the identifiability ranking study from their base value and then regressing the same parameters using the simulated experimental data generated using the base set of parameter values. This exercise was performed twice, once using *clean* data from the base simulation, and again using the same data but with noise artificially added such that the statistical characteristics of the artificial noise were similar to those of experimentally observed noise in batch tests. This procedure was repeated with subsets with increasing number of parameters until the cut-off criterion; this criterion defined the maximum subset of parameters that is identifiable by observing the percent deviation of the regressed parameter values from their nominal values as a function of number of parameters regressed and selecting as the cut-off point, the number of parameters at which a sharp increase in deviation was observed (Figure 1).

RESULTS AND DISCUSSION

A local parameter sensitivity analysis was performed to identify the parameters that have the most influence on the gas flow rate measurements. These parameters were then used in the parameter ranking. Batstone et al. (2004) only considered the parameters pertaining to the ethanol and acetate consuming microorganisms. In the local sensitivity analysis, it was found that the estimation of hydrogen consumption parameters were significantly sensitive to the gas flow rate and pH measurements. Results for the parameter ranking are displayed in Table 1.

Table 1. Ranking of Parameters according to identifiability using the Weijers and Vanrolleghem (1997) Method for either *gas flow rate data* only and for *both gas flow rate and pH data*. The *most* identifiable parameter is listed *first*. [M – mass; Y – yield; K_{CO2} – Carbon Dioxide gas transfer coefficient; X – biomass concentration; k_s – half saturation constant; μ – maximum growth rate; et – ethanol; am – acetoclastic methanogens; hm – hydrogenotrophic methanogens]

Gas	Rank:1 – 9	M_{et}	Y_{hm}	Y_{am}	Y_{et}	K_{CO2}	$k_{s\text{-}am}$	$k_{s\text{-}hm}$	$k_{s\text{-}et}$	X_{hm}
	Rank: 10 - 17	μ_{am}	X_{et}	X_{am}	K_{et}	μ_{hm}	μ_{et}	K_{am}	K_{hm}	
Gas & pH	Rank :1 – 9	M_{et}	Y_{et}	Y_{am}	Y_{hm}	K_{CO2}	$k_{s\text{-}et}$	$k_{s\text{-}am}$	μ_{am}	μ_{hm}
	Rank :10 - 17	X_{et}	$k_{s\text{-}hm}$	μ_{et}	K_{et}	X_{am}	X_{hm}	K_{am}	K_{hm}	

Both cases ranked the mass of ethanol added to the system as the most identifiable parameter. This

value should be known in a batch experiment, but because there was a significant probability of gas leaks and calibration inaccuracies with the gas flow meters used in the associated experimental study, it was taken as a variable parameter. (Thus a deviation between added ethanol and regressed ethanol mass would indicate unreliable gas flow rate measurement calibration). The yields of each micro-organism group were ranked next followed, by the carbon dioxide mass transfer coefficient. Decay constants were ranked as the least identifiable parameters considered for both cases. Each subset was investigated until the cut-off criterion was reached (Figure 1).

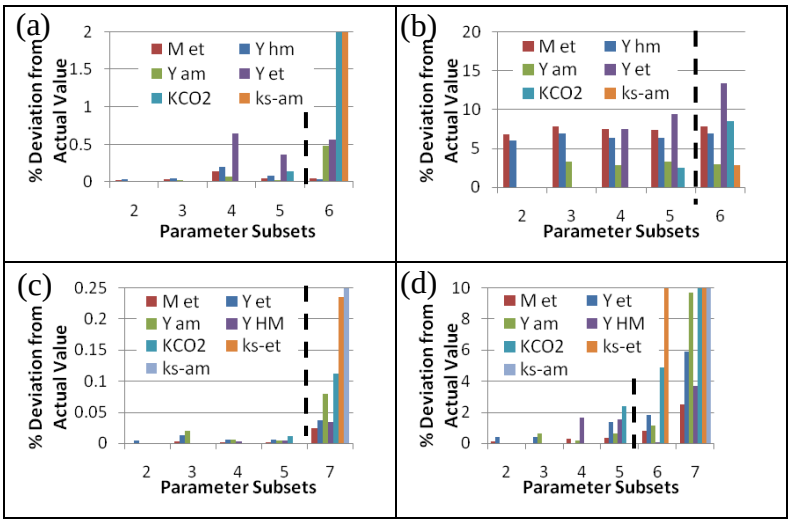

Figure 1. Results for the selection of the maximum number of parameters that are identifiable (a) Gas flow rate ranked parameters fitted to simulated gas flow rate data (b) Gas flow rate ranked parameters fitted to simulated gas flow rate data with noise (c) Gas flow rate and pH ranked parameters fitted to simulated gas flow rate and pH data (d) Gas flow rate and pH ranked parameters fitted to simulated gas flow rate and pH data with noise. The dotted line represents the cut-off for the maximum number of parameters that are identifiable from the available data.

The cut-off criterion value was different for each of the four categories (with and without pH data, with and without added noise) and was identified when the addition of another parameter to the regression sub-set resulted in an increase in deviation in regressed parameter values from the base set values that was significantly larger than for previous increases in regressed parameter number. When the parameters were only fitted to gas flow rate, the cut-off criterion was 1% for the simulated data fitting and 10% for the simulated data with noise. A maximum of five parameters can be identified from gas flow rate data only before the deviation increased above this value. A smaller absolute value for cut-off criterion was established when pH data was included in the parameter estimation with values of 0.05% for the simulated data only and 4% for the simulated data with noise. This implies the use of both gas flow rate and pH data in parameter estimation from batch tests would improve the precision (reduce the confidence limits) of the estimated parameter values. However, despite the increased precision, the same number of parameters (five) were found to be identifiable when pH data was included.

Generic literature data for yield co-efficient can be obtained, while micro-organism population concentration is experiment-specific, making these concentrations more useful as regression parameters. It was hypothesised that if the yield values were considered to be constant, fixed at values proposed from literature, the corresponding biomass concentrations would become identifiable (Figure 2).

The fitting to the simulated data showed that all the parameters deviations from the actual value were below 0.05%. Deviations from the actual value for the fitting against simulated data with noise were all below 4%. These results indicated that biomass concentration *or* yields were interchangeably identifiable for each micro-organism population stimulated in the experiment, but that they were not simultaneously identifiable from this type of experiment. This is linked to model structural identifiability. This means that only combination of parameters can be identified.

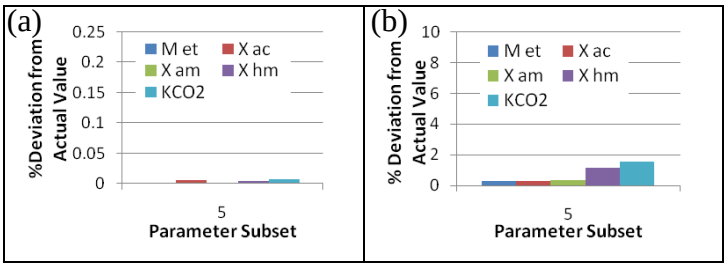

Figure 2. Results for determining whether the biomass concentrations can be identified instead of the yields (a) Parameters fitted to the simulated gas flow rate and pH data (b) Parameters fitted to gas flow rate and pH simulated data with noise

To test these conclusions, the model was fitted to real experimental data, by regressing the ethanol dose, biomass concentrations, and CO_2liquid vapour transfer coefficient. It was found that the model was very sensitive to the estimate of initial total carbonate concentration. The eventual fit of model to data is shown in Figure 3.

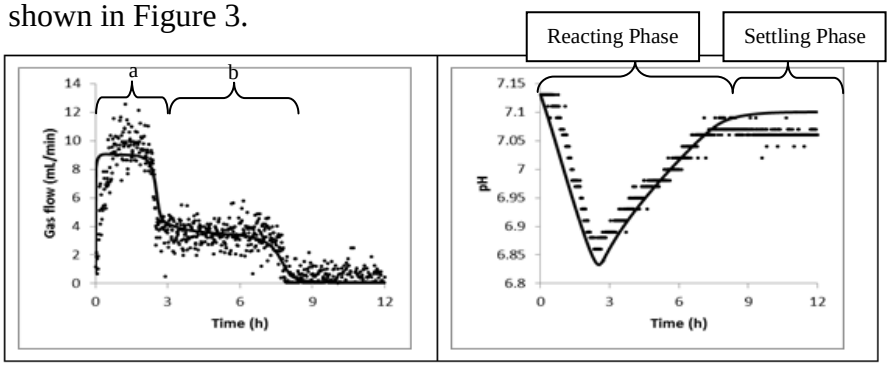

Figure 3. Fit of model to experimental data for ethanol degradation in an anaerobic batch reactor. a is the predominately the acetogenic activity. b is the predominately the methanogenic activity

CONCLUSION

When an AnSBR is fed with ethanol, only five parameters are identifiable from gas flow rate and pH data. Of this parameter set, the mass of ethanol and the carbon dioxide mass transfer coefficient can be determined. The other parameters which are identifiable are related to the ethanol, acetate and hydrogen consuming micro-organism populations. Any one of their kinetic parameters could be identified if the others were set at literature values. It would be advisable to estimate the biomass concentration and set the other parameter values to literature values, since concentration is experiment-specific. Including pH data into the regression gives more precise parameter estimates then regression to gas flow rate data only.

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