

**Study of the effect of process parameters on the thermophilic anaerobic digestion of sewage sludge, evaluation of a thermal sludge pre-treatment and overall energetic assessment**

*Estudi de l'efecte dels par metres del proc s en la digesti  anaer bia termof lica de fangs de depuradora, avaluaci  del pre-tractament t rmic dels fangs i valoraci  energ tica global*

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CERTIFIQUEM:

Que l'Enginyera Agrònoma Ivet Ferrer i Martí ha realitzat sota la nostra direcció el treball amb títol **“Estudi de l'efecte dels paràmetres del procés en la digestió anaeròbia termofílica de fangs de depuradora, avaluació del pre-tractament tèrmic dels fangs i valoració energètica global”**, presentat en aquesta memòria, la qual constitueix la seva Tesi per optar al Grau de Doctora per la Universitat Autònoma de Barcelona.

I perquè en prenguem coneixement i consti als efectes oportuns, presentem a l'Escola Tècnica Superior d'Enginyeria de la Universitat Autònoma de Barcelona l'esmentada Tesi, signant el present certificat a:

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## PRESENTACIÓ

Aquesta memòria de Tesi Doctoral s'emmarca dins del projecte de recerca *“Estrategias para la digestión anaerobia termofílica de fangos de EDAR urbana: Estudio del proceso en una y dos etapas. Aplicación de técnicas de pretratamiento del lodo”* del *Plan Nacional I+D+I 2002/2003* finançat pel *Ministerio de Ciencia y Tecnología* (ref. REN 2002-00926/TECNO). Aquest projecte se centrà en l'estudi de la digestió anaeròbia termofílica de fangs d'Estació Depuradora d'Aigües Residuals (EDAR) urbanes i d'estratègies per incrementar l'eficàcia d'aquest procés, així com les propietats dels productes finals (fangs digerits i biogàs).

La finalitat de la recerca duta a terme era la de millorar un dels processos tradicionals de tractament de fangs residuals procedents d'EDARs urbanes, la digestió anaeròbia, per poder optimitzar la conversió d'un residu (els fangs) en productes d'interès i valor, com són, en aquest cas, un gas amb elevat contingut energètic i un biosòlid amb potencial aplicació agrícola o en la restauració de terrenys degradats. L'objectiu final perseguit era, doncs, minimitzar la generació de residus, de manera que en el balanç del procés es pogués parlar de generació de residu igual a zero.

Els fangs de depuradora constitueixen un residu, o subproducte, líquid però amb un contingut relativament elevat en sòlids, per la qual cosa la hidròlisi dels fangs és una fase limitant en el procés de la digestió anaeròbia. D'altra banda, la digestió anaeròbia termofílica (50-55 °C) presenta certes avantatges en comparació al procés mesofílic convencional (35-40 °C), incloent l'acceleració del procés global, que permet reduir el temps de retenció dels fangs (TRF) i per tant reduir el volum del reactor o bé incrementar el cabal tractat; i també la potencial higienització de l'efluent, un factor clau de cara a l'aplicació al sòl dels fangs digerits.

En el marc de l'esmentat projecte d'investigació, els treballs experimentals realitzats per na Mavi Climent (2004) i en Sergio Ponsá (2006), corresponents al Màster d'Iniciació a la Recerca de la Universitat Autònoma de Barcelona, se centraren en l'estudi de la millora de l'etapa hidrolítica; en el primer cas mitjançant l'aplicació de pretractaments mecànics, tèrmics i químics als fangs; i en el segon cas mitjançant la determinació de les condicions òptimes d'operació d'un reactor hidrolític que constituiria la primera etapa en un sistema de tractament anaerobi amb dues etapes.

En el present projecte de Tesi, l'objectiu general se centrà en l'estudi i optimització de la digestió anaeròbia termofílica dels fangs de depuradora. Es varen dur a terme experiments en reactors continus per tal de comparar els resultats obtinguts en processos a diferents temperatures (38-43-50-55 °C), determinar el mínim TRF requerit per a l'operació estable d'un reactor termofílic, i avaluar l'efecte del pretractament dels fangs sobre l'esmentat procés. Cal destacar la importància de l'arranc dels digestors anaeròbics termofílics, un aspecte crític que pot condicionar la posterior evolució del procés.

La primera fase experimental va consistir en el disseny i muntatge d'una planta pilot a escala de laboratori per a la digestió anaeròbia de fangs d'EDAR; i el posterior arranc del procés de digestió anaeròbia mesofílica dels fangs. Aquest treball va constituir la tesina experimental del *MSc. Environmental Diagnostics* cursat a Cranfield University (Anglaterra), que fou equiparat pel Màster d'Iniciació a la Recerca de la Universitat Autònoma de Barcelona.

La planta pilot original fou ampliada per estudiar en paral·lel els efectes de la temperatura dels processos, per un costat, i de la disminució del TRF, per l'altre. Posteriorment, s'hi va incloure un pretractament tèrmic dels fangs a 70 °C, com a pas previ a un digestor termofílic treballant al mínim TRF determinat anteriorment per garantir una operació estable.

L'elecció del tipus de pretractament dels fangs sorgí arrel del treball experimental previ realitzat per na Mavi Climent (2004). D'entre les possibles alternatives, s'avaluaren els pretractaments mecànic amb ultrasons i microones, el tèrmic a elevada temperatura (> 100 °C) i l'alcalí amb hidròxid sòdic. L'objectiu era aconseguir la disrupció de la paret cel·lular del material biològic que integra els fangs secundaris, per tal de millorar el rendiment en la posterior etapa d'hidròlisi biològica. S'observaren millores en termes d'increment de la concentració de matèria orgànica soluble, però no en la producció de biogàs en assajos discontinus o *batch*.

D'acord amb aquests resultats, en el present projecte de Tesi es va optar per realitzar un tractament tèrmic a baixa temperatura (< 100 °C), amb l'objectiu d'accelerar la hidròlisi enzimàtica del material biològic. Proves inicials mostraren increments tant en la solubilització dels fangs com en la producció de biogàs en assajos discontinus i, per tant, es va estudiar l'efecte d'aquest pretractament sobre la digestió termofílica de fangs en un procés continu.

Finalment, s'avaluaren diferents alternatives per al tractament anaerobi de fangs d'EDAR des d'una perspectiva energètica, comparant les produccions d'energia obtingudes a partir del biogàs generat, amb els consums requerits pel funcionament dels digestors sota diferents condicions de treball. L'objectiu final era el de comprovar si increments derivats de majors inversions energètiques en el procés, serien compensats per una major producció neta d'energia.

El projecte d'investigació exposat es va dur a terme a l'Escola Universitària Politècnica del Medi Ambient (EUPMA), pertanyent a la Fundació Estudis del Medi Ambient de Mollet del Vallès, i adscrita a la Universitat Autònoma de Barcelona (UAB), on s'impartien els estudis d'Enginyeria Tècnica Industrial, especialitat Química Industrial, itinerari Medi Ambient. A partir del curs acadèmic 2005/06, aquests estudis es varen traslladar a l'Escola Tècnica Superior d'Enginyeries (ETSE) de la UAB, i en el centre la Fundació Estudis del Medi Ambient va constituir el Centre Tecnològic per la Gestió Integral de Residus Orgànics (GIRO CT), vinculat a la Universitat Politècnica de Catalunya (UPC) i a l'Institut de Recerca i Tecnologies Agroalimentàries (IRTA) de la Generalitat de Catalunya. Aquesta Tesi s'ha realitzat, doncs, a cavall entre l'EUPMA i el GIRO CT, a la Fundació Estudis del Medi Ambient de Mollet del Vallès, Barcelona.



## Agraïments

En primer lloc voldria agrair la direcció, supervisió i suport del meus directors, el Dr. Xavier Font i la Dra. Felicitas Vázquez, i anteriorment la Dra. M<sup>a</sup> Angels Gordillo. Moltes gràcies per la vostra guia, per les idees brillants, i també per la paciència i la calma, especialment en moments en què semblava que el final del camí no s'apropava...

També voldria expressar el meu agraïment a l'Escola Universitària Politècnica del Medi Ambient (EUPMA-UAB), de la Fundació Estudis del Medi Ambient de l'Ajuntament de Mollet del Vallès, que brindà l'oportunitat de dur a terme, entre d'altres, aquesta Tesi Doctoral. A continuació, expressar el meu agraïment al GIRO Centre Tecnològic, per la possibilitat de finalitzar la Tesi mitjançant la incorporació al nou centre.

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I finalment, al Ferran, no sé perquè quan està a prop fa que tot sembli més fàcil...



*Quan surts per fer el viatge cap a Ítaca,  
has de pregar que el camí sigui llarg,  
ple d'aventures, ple de coneixences.  
Has de pregar que el camí sigui llarg,  
que siguin moltes les matinades  
que entraràs en un port que els teus ulls ignoraven,  
i vagis a ciutats per aprendre dels que saben.  
Tingues sempre al cor la idea d'Ítaca.  
Has d'arribar-hi, és el teu destí,  
però no forcis gens la travessia.  
És preferible que duri molts anys,  
que siguis vell quan fondegis l'illa,  
ric de tot el que hauràs guanyat fent el camí,  
sense esperar que et doni més riqueses.  
Ítaca t'ha donat el bell viatge,  
sense ella no hauries sortit.  
I si la trobes pobra, no és que Ítaca  
t'hagi enganyat. Savi, com bé t'has fet,  
sabràs el que volen dir les Ítaques.*

...

*Lluís Llach, Ítaca*



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## ABSTRACT

Energy consumption accounts for some 30 % of the total operating costs of intensive sewage treatment systems. In conventional wastewater treatment plants employing an activated sludge process, around 15-20 % of this energy is used in the sludge treatment line, including sludge pumping, thickening, stabilisation and dewatering. Therefore, optimisation of sludge management can substantially contribute in the reduction of wastewater treatment costs.

Thermophilic anaerobic digestion is more efficient than mesophilic anaerobic digestion, in terms of biogas production, volatile solids (VS) removal and pathogens destruction. The process might be further accelerated by sludge pre-treatment, promoting sludge solubilization and hydrolysis.

The aim of this PhD Thesis was to study the impact of process parameters on the thermophilic anaerobic digestion of sewage sludge, to evaluate the effect of implementing a low temperature pre-treatment step, and to assess alternative processes from an energy perspective.

The experimental results presented were obtained by operating two lab-scale reactors for almost two years. During this period, the effect of process temperature, sludge retention time (SRT), organic loading rate (OLR) and 70 °C sludge pre-treatment on the anaerobic digestion of sewage sludge was studied. The process was evaluated in terms of energy production (i.e. biogas and methane production) and the quality of the effluent sludge (i.e. VS and volatile fatty acids (VFA) content, sludge dewaterability and hygienisation). Focus was put on the stability of the process at decreasing SRT and increasing OLR. Process efficiency during stable performance under each operating condition assayed was compared. Finally, the results were assessed from an energy perspective, by means of theoretical energy balances and ratios; and compared to the results obtained with experimental data from other studies. A first order kinetic model was also used.

The conclusions drawn from the different issues dealt in this work are summarised as follows:

During anaerobic sludge digestion, the transition from a mesophilic (43 °C) to a thermophilic operation (50 °C) may be carried out without disturbing the process, by operating the reactors at high SRT ( $\geq 30$  days) and low OLR ( $\leq 0.5$  kg VS  $\text{m}^{-3}_{\text{reactor}} \text{d}^{-1}$ ). Under such conditions, some VFA accumulation (0.5-2.5 g  $\text{L}^{-1}$ ) and enhanced pathogen destruction (residual *E. coli*  $\leq 10^2$  CFU  $\text{mL}^{-1}$ )

would be the main differences of thermophilic (50-55 °C) compared to mesophilic (38-43 °C) reactors. Thermophilic sludge digestion at 50 °C and 55 °C should be similar in terms of biogas production and effluent stabilisation, hygienisation and dewaterability; provided that other process parameters are the same.

Methane production rate tends to increase proportionally to the OLR, thus to the SRT and VS concentration in the feed sludge. Similarly, the quality of the effluent sludge (VS content, VFA content and sludge dewaterability) is also affected by the OLR. According to the results obtained at 55 °C, methane production rate increased by 2-3 times (from 0.2 to 0.4-0.6 m<sup>3</sup><sub>CH<sub>4</sub></sub> m<sup>3</sup><sub>reactor</sub> d<sup>-1</sup>) by decreasing the SRT from 30 to 15-10 days; increasing the OLR from 0.5 to 2.5-3.5 kg VS m<sup>3</sup><sub>reactor</sub> d<sup>-1</sup>. However, process unbalance resulted from SRT reduction to 6 days, with OLR above 5 kg VS m<sup>3</sup><sub>reactor</sub> d<sup>-1</sup>. The following concentrations might be useful to detect and prevent digester failure during thermophilic sludge digestion: total VFA (2.5 g L<sup>-1</sup>), acetate (0.5 g L<sup>-1</sup>), acetate/propionate ratio (0.5), intermediate alkalinity (1.8 g CaCO<sub>3</sub> L<sup>-1</sup>), intermediate alkalinity/partial alkalinity ratio (0.9), intermediate alkalinity/total alkalinity ratio (0.5), methane content in biogas (55 %).

The 70 °C sludge pre-treatment may initially promote sludge solubilization, increasing the concentration of soluble to total organic matter from 5 to 50 % within 9-24 h; which is followed by a progressive VFA generation after 24 h. Subsequent anaerobic digestion of pre-treated sludge samples (9-48 h) could increase biogas production by 30-40 % working at 55 °C with a SRT of 10 days. Biogas yield is some 30 % higher with pre-treated sludge (0.28-0.30 vs. 0.22 L gVS<sub>fed</sub><sup>-1</sup>) and methane content in biogas is also higher with pre-treated sludge (69 vs. 64 %).

Thermophilic anaerobic sludge digestion would result in net energy production, during cold and warm seasons, provided that digesters with wall insulation and with energy recovery from both the biogas produced and the effluent sludge are used. In this case, the energetic efficiency would be similar for thermophilic digesters working at half the SRT (10-15 days) of mesophilic digesters (20-30 days), meaning that the sludge daily flow rate could be doubled, or the reactor volume reduced, with subsequent savings in terms of sludge treatment costs. Furthermore, two-stage systems (70/55 °C) may result in higher net energy production compared to single-stage systems (55 °C) at 10 days SRT. However, the amount of surplus energy generated increases with digester volume. In spite of the decrease in methane production rate at increasing SRT, energy production is still higher than energy consumption, and therefore the bigger the amount of sludge in the digester, the higher the energy production.

## RESUM

El consum energètic representa un 30 % dels costos d'operació en sistemes intensius de tractament d'aigües residuals urbanes. En depuradores convencionals que utilitzin un sistema de fangs activats, entorn al 15-20 % de l'energia és consumida en la línia dels fangs, que inclou el bombeig, l'espessiment, l'estabilització i la deshidratació. Per tant, la optimització de la gestió dels fangs pot contribuir substancialment en la reducció dels costos de tractament d'aigües residuals.

La digestió anaeròbia termofílica és més eficient que la mesofílica i psicrófílica, en termes de producció de biogàs i metà, eliminació de sòlids volàtils (SV) i destrucció de patògens. El procés es pot accelerar mitjançant el pre-tractament dels fangs, afavorint la seva solubilització i hidròlisi.

L'objecte d'aquesta Tesi Doctoral fou estudiar l'impacte dels paràmetres del procés en la digestió anaeròbia termofílica dels fangs de depuradora urbana, avaluar l'efecte del pre-tractament tèrmic dels fangs a baixa temperatura, i valorar processos alternatius des del punt de vista energètic.

Els resultats experimentals presentats s'obtingueren mitjançant l'operació de dos reactors de laboratori durant prop de dos anys. En aquest període es va estudiar l'efecte de la temperatura del procés, del temps de retenció dels fangs (TRF), de la velocitat de càrrega orgànica (VCO) i del pre-tractament a 70 °C en la digestió anaeròbia dels fangs de depuradora. El procés fou avaluat en termes de la producció d'energia (biogàs i metà) i de la qualitat del fang digerit (contingut de SV i d'àcids grassos volàtils (AGV), facilitat de deshidratació i higienització). S'analitzà l'estabilitat del procés a mesura que es reduïa el TRF i s'incrementava la VCO, i es comparà l'eficiència en períodes d'estabilitat corresponents a les diferents condicions operacionals. Finalment, s'avaluaren els resultats des del punt de vista energètic, mitjançant el càlcul de balanços i ratis energètics teòrics, que es compararen amb els resultats obtinguts a partir de dades experimentals d'altres estudis. També s'utilitzà un model cinètic de primer ordre.

Les conclusions que es desprenen d'aquest treball es resumeixen a continuació:

Durant la digestió anaeròbia dels fangs, la transició d'un reactor mesofílic (43 °C) a termofílic (50 °C) es podria dur a terme sense alterar el procés, treballant a TRF elevats ( $\geq 30$  dies) i VCO baixes ( $\leq 0.5 \text{ kg SV m}^{-3}_{\text{reactor}} \text{ d}^{-1}$ ). En aquestes condicions, les principals diferències entre reactors

termofílics (50-55 °C) i mesofílics (38-43 °C) fan referència a una certa acumulació d'AGV (0.5-2.5 g L<sup>-1</sup>) i millora de la destrucció de patògens (*E. coli* ≤ 10<sup>2</sup> UFC mL<sup>-1</sup>). La digestió termofílica a 50 °C i 55 °C dóna lloc a resultats similars pel que fa a la producció de biogàs, estabilització, higienització i facilitat de deshidratació de l'efluent, si no varien els altres paràmetres operacionals.

La producció de metà tendeix a incrementar proporcionalment a la VCO, és a dir al TRF i el contingut de SV als fangs alimentats. Així mateix, la qualitat de l'efluent (contingut de SV i AGV, facilitat de deshidratació dels fangs) també depèn de la VCO. D'acord amb els resultats obtinguts a 55 °C, la producció de metà s'incrementà 2-3 vegades (de 0.2 a 0.4-0.6 m<sup>3</sup><sub>CH4</sub> m<sup>3</sup><sub>reactor</sub> d<sup>-1</sup>) en disminuir el TRF de 30 a 15-10 dies, incrementant la VCO de 0.5 a 2.5-3.5 kg SV m<sup>3</sup><sub>reactor</sub> d<sup>-1</sup>. En canvi, el procés es desestabilitzà amb la reducció del TRF a 6 dies i VCO per sobre de 5 kg SV m<sup>3</sup><sub>reactor</sub> d<sup>-1</sup>. Les següents concentracions poden ser útils per detectar i prevenir la desestabilització d'un digester termofílic de fangs: AGV totals (2.5 g L<sup>-1</sup>), acetat (0.5 g L<sup>-1</sup>), rati acetat/propionat (0.5), alcalinitat intermèdia (1.8 g CaCO<sub>3</sub> L<sup>-1</sup>), rati alcalinitat intermèdia/alcalinitat parcial (0.9), rati alcalinitat intermèdia/alcalinitat total (0.5), contingut de metà al biogàs (55 %).

El pre-tractament a 70 °C afavoreix la solubilització dels fangs, incrementant la proporció de matèria orgànica soluble respecte la matèria orgànica total del 5 % al 50 % en 9-24 h; seguit d'una progressiva generació d'AGV després de 24h. Durant la subseqüent digestió anaeròbia de fangs pre-tractats (9-48 h), s'incrementà la producció de biogàs en un 30-40 %, treballant a 55 °C i 10 dies de TRF. El rendiment de producció de biogàs fou un 30 % superior amb fangs pre-tractats (0.28-0.30 vs. 0.22 L gVS<sup>-1</sup>) i el contingut de metà al biogàs també fou superior (69 % vs. 64 %).

La digestió anaeròbia termofílica de fangs pot donar lloc a una producció neta d'energia, durant estacions fredes i càlides, si s'utilitzen reactors amb aïllament tèrmic de les parets i amb recuperació energètica a partir del biogàs i dels fangs digerits. En aquest cas, l'eficiència energètica de reactors termofílics treballant a la meitat de TRF (10-15 dies) que reactors mesofílics (20-30 dies) seria similar, per la qual cosa el cabal diari podria ser doblat, o el volum del reactor reduït, amb el consegüent estalvi en el cost de tractament dels fangs. A més, un sistema en dues etapes (70/55 °C) produiria més energia neta que un sistema en una sola etapa (55 °C) amb un TRF de 10 dies. De totes maneres, la quantitat d'energia neta generada augmenta amb el volum del digester donat que, malgrat la disminució en la producció de metà a TRF creixents, la producció d'energia segueix essent superior al consum, i per tant com més quantitat de fangs hi hagi al digester, més energia es produirà.

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## ABBREVIATIONS AND SYMBOLS

A	Surface area of the reactor wall	$m^2$
AR	Alkalinity ratio	
ADM1	Anaerobic digestion model N° 1	
CFU	Colony forming units	N° 100 mL <sup>-1</sup>
COD	Chemical oxygen demand	g L <sup>-1</sup>
CST	Capillary suction time	s
CSTR	Continuous stirred tank reactor	
DAS	Data acquisition system	
HRT	Hydraulic residence time	d
IA	Intermediate alkalinity	g CaCO <sub>3</sub> L <sup>-1</sup>
LCFA	Long chain fatty acid	
k	Heat transfer coefficient	W m <sup>-2</sup> °C <sup>-1</sup>
k <sub>h</sub>	First order rate coefficient	d <sup>-1</sup>
K	Potassium	
N	Nitrogen	
OFMSW	Organic fraction of municipal solid wastes	
OLR	Organic loading rate	kg VS m <sup>-3</sup> <sub>reactor</sub> d <sup>-1</sup>
P	Phosphorus	
PA	Partial alkalinity	g CaCO <sub>3</sub> L <sup>-1</sup>
PC	Personal computer	
P <sub>biogas</sub>	Biogas production rate	m <sup>3</sup> <sub>biogas</sub> m <sup>-3</sup> <sub>reactor</sub> d <sup>-1</sup>
P <sub>CH4</sub>	Methane production rate	m <sup>3</sup> <sub>CH4</sub> m <sup>-3</sup> <sub>reactor</sub> d <sup>-1</sup>
PS	Primary sludge	
Q	Sludge daily flow rate	m <sup>3</sup> d <sup>-1</sup>
S <sub>e</sub>	Effluent VS concentration	% or g L <sup>-1</sup>
S <sub>i</sub>	Influent VS concentration	% or g L <sup>-1</sup>
SP <sub>0</sub>	Maximum specific methane production	m <sup>3</sup> <sub>CH4</sub> kg VS <sup>-1</sup> <sub>fed</sub>
SP <sub>biogas</sub>	Specific biogas production	m <sup>3</sup> <sub>biogas</sub> kg VS <sup>-1</sup> <sub>fed</sub>
SP <sub>CH4</sub>	Specific methane production	m <sup>3</sup> <sub>CH4</sub> kg VS <sup>-1</sup> <sub>fed</sub>
SRT	Sludge retention time	d
T	Process temperature	°C

*Abbreviations and Symbols*

$T_{\text{sludge}}$	Influent sludge temperature	$^{\circ}\text{C}$
$T_{\text{amb}}$	Ambient temperature	$^{\circ}\text{C}$
TA	Total alkalinity	$\text{g CaCO}_3 \text{ L}^{-1}$
TDS	Total dissolved solids	$\%$ or $\text{g L}^{-1}$
TS	Total solids	$\%$ or $\text{g L}^{-1}$
TSS	Total suspended solids	$\%$ or $\text{g L}^{-1}$
V	Reactor working volume	$\text{L}$ or $\text{m}^3$
VDS	Volatile dissolved solids	$\%$ or $\text{g L}^{-1}$
VFA	Volatile fatty acids	$\text{g L}^{-1}$
VS	Volatile solids	$\%$ or $\text{g L}^{-1}$
$VS_{\text{removal}}$	Percentage of VS removed from the influent	$\%$
VSS	Volatile suspended solids	$\%$ or $\text{g L}^{-1}$
$Y_{\text{biogas}}$	Biogas yield	$\text{m}^3_{\text{biogas}} \text{ kg VS}^{-1}_{\text{removed}}$
$Y_{\text{CH}_4}$	Methane yield	$\text{m}^3_{\text{CH}_4} \text{ kg VS}^{-1}_{\text{removed}}$
WAS	Waste activated sludge	
WWTP	Wastewater treatment plant	
$\gamma$	Specific heat of sludge	$\text{kJ kg}^{-1} \text{ }^{\circ}\text{C}^{-1}$
$\eta$	Efficiency of the CHP unit for electricity generation	$\%$
$\lambda$	Heat recovery from effluent sludge	$\%$
$\rho$	Specific density of sludge	$\text{kg m}^{-3}_{\text{sludge}}$
$\xi$	Lower heating value of methane	$\text{kJ m}^{-3}_{\text{CH}_4}$
$\Psi$	Efficiency of the CHP unit for heat generation	$\%$

## **Chapter 1. Introduction**

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## Chapter 1. INTRODUCTION

### 1.1. INTEREST OF SEWAGE SLUDGE ANAEROBIC DIGESTION IN THE CONTEXT OF CATALONIA

#### 1.1.1 Sludge production and management

The organic solid waste obtained as a result of municipal and industrial wastewater treatment is known as sludge. The amount of sludge produced in Catalonia increased from 522,296 t in 1994 to 1,177,693 t in 2004, which corresponds to an increase of 125 % over a period of ten years; according to data published by the Catalan Waste Agency (*Agència de Residus de Catalunya (ARC)*).

Sludge management is regulated by the Catalan Waste Law (*Llei 15/2003, de 13 de juny, de modificació de la Llei 6/1993, del 15 de juliol, reguladora dels residus*). According to the so-called Waste Management Hierarchy, the priority of waste management alternatives is as follows: waste minimisation, waste recycling, waste valorisation and ultimately waste disposal. Valorisation alternatives may include material valorisation, energetic valorisation or both, depending on the process.

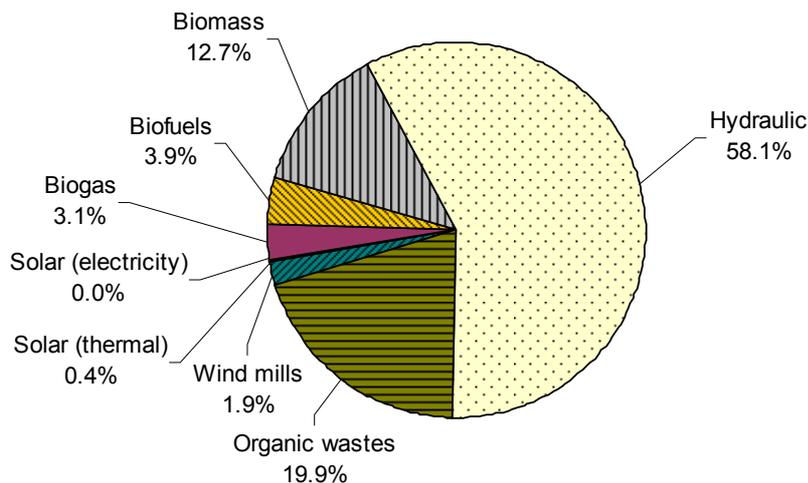
Due to its origin, sludge belongs to the category of industrial wastes, which are regulated by the Industrial Waste Management Program of Catalonia (*Programa de Gestió de Residus Industrials de Catalunya (PROGRIC 2001-2006; 2007-2012)*). In the Catalan Waste Catalogue (*Catàleg de Residus de Catalunya*), which is equivalent to the European Waste Catalogue, valorisation and/or final disposal options for wastes of sewage treatment and specifically for sewage sludge obtained after thickening or dewatering processes in wastewater treatment plants (WWTP) include: construction use; agricultural use; composting and landfilling.

In municipal WWTP, sewage is treated in the so-called wastewater treatment line and the resulting sewage sludge is treated in the so-called sludge treatment line. Treated sludge is commonly regarded as biosolids. Its potential use on land is restricted by the heavy metals content (Council Directive 86/278/EEC of 12 June 1986 on the protection of the environment, and in particular of the soil, when sewage sludge is used in agriculture; in Spain *Real Decreto 1310/1990, de 29 de octubre, por el que se regula la utilización de lodos de depuración en el sector agrario*).

According to the Catalan Water Agency (*Agència Catalana de l'Aigua (ACA)*, 2007), the amount of biosolids produced in Catalonia increased from around 200,000 t in 1998 to nearly 550,000 t in 2006; out of which 88-95 % could potentially be used on land. Up to 95.1 % of sewage sludge is treated through: aerobic and anaerobic digestion (25.9 %), composting (39.1 %) and thermal drying (35 %); its final disposal being: use on land (85.6 %), energetic valorisation (2.5 %) and landfilling (11.8 %).

### 1.1.2 Renewable energy production

Renewable energy production in Catalonia is still very scarce. According to the Catalan Energy Institute (*Institut Català de l'Energia (ICAEN)*), in 2003 only 3.6 % of primary energy consumption in Catalonia corresponded to renewable energy (740,348 tonne oil equivalent (toe)), distributed as shown in Figure 1.1.



**Figure 1.1.** Distribution of renewable energy in Catalonia in 2003 (according to the Catalan Energy Program 2006-2015)

One of the aims of the Catalan Energy Program (*Pla de l'Energia de Catalunya 2006-2015*) is to reinforce renewable energy production. On the whole, over 50 % of the total renewable energy is to be obtained from biomass (Table 1.1), through different processes which are grouped into four categories:

- Physicochemical processes (homogenization, densification)
- Thermochemical processes (combustion, pyrolysis, gasification)
- Biochemical processes (anaerobic digestion, alcoholic fermentation)
- Chemical processes (esterification)

**Table 1.1.** Objectives of the Catalan Energy Program 2006-2015

Energy source	Objective value	Saved ktoe	% renewable energy	Capital investment
Wind mills	3,000 MW	619,481	25.2	2,790 M€
Solar (thermal)	1,250,000 m <sup>2</sup>	86,040	3.1	346 M€
Solar (electricity)	100 MW	10,277	0.4	482 M€
Biogas	121.5 MW	205,570	8.3	285 M€
Biofuels	15 % substitution	680,480	27.5	220 M€
Biomass (thermal + electricity)	100 MW	444,683	16.3	260 M€

Anaerobic digestion is a biochemical process which consists of the degradation of organic materials, like sewage sludge, organic fraction of municipal solid wastes (OFMSW), cattle manure, pig slurries, etc.; into biogas. This process takes place in enclosed anaerobic environments, for example in landfillsights, in storage tanks or in anaerobic reactors. Comparing biogas production in 2003 (Figure 1.1) and the predictions for 2015 (Table 1.1), this value should increase from 3.1 to 8.3 % of the total renewable energy production in Catalonia. On the other hand, stringent regulations on final disposal of organic wastes in landfillsights will result in decreased biogas production and energy recovery from such installations in the future. This means that more anaerobic digesters for the treatment of organic wastes should be implemented and its efficiency in terms of energy recovery optimised.

### 1.1.3 Sludge treatment through anaerobic digestion

Traditionally, anaerobic digestion has been used in WWTP for the stabilisation of sewage sludge, its major advantage with respect to alternative treatments being the potential net energy production. In Catalonia, sludge anaerobic digestion is implemented in some municipal WWTP like Sant Feliu de Llobregat, Gavà, El Prat, Granollers, La Llagosta, Montornès or Lleida, amongst others. However, in most cases energy recovery is not optimised. The use of biogas for digester heating is a common practise; but combined heat and power generation (or cogeneration) with the biogas produced is still very scarce, in spite of successful implementation of this technology, for example, in Sant Feliu de Llobregat where the electricity generated from biogas covers some 40 % of the total electricity demand of the WWTP.

Furthermore, all sewage sludge digesters in Catalonia operate in the mesophilic range of temperatures (35-40 °C), while thermophilic digestion (50-55 °C) is more efficient in terms of

energy production and has long been used for sewage sludge treatment in other countries (Buhr and Andrews, 1977). For example, in Moscow WWTP thermophilic sewage sludge digesters have successfully been working for more than 50 years, and currently 18 reactors (5000 m<sup>3</sup> each) are operating at 55 °C (Pakhomov *et al.*, 2006). In Central Prague WWTP, sludge stabilisation is achieved by means of a two-stage system formed by 6 pairs of reactors (4823 m<sup>3</sup> each) connected in series; in which only the first digester is heated and stirred. The original mesophilic process (38/35 °C) was swapped to thermophilic (55/52 °C) as a result of increased sludge daily flow rate. Mean biogas production was around 0.48 and 0.61 m<sup>3</sup><sub>biogas</sub> m<sup>-3</sup><sub>reactor</sub> d<sup>-1</sup> with mesophilic and thermophilic operation, respectively; and methane content in biogas around 66 %; thus methane production was increased by 27 % (Zábranská *et al.*, 2000a). Additionally, the implementation of mechanical and thermal sludge pre-treatment processes, increased biogas yield by 15-26 %, from 0.46 to 0.53 m<sup>3</sup><sub>biogas</sub> kg VS<sup>-1</sup> (Zábranská *et al.*, 2000b; Zábranská *et al.*, 2006).

This scenario suggests a potential for anaerobic digestion implementation and improvement within the Catalan context. With regards to sewage sludge treatment, the enhancement of biogas production and energy recovery in WWTP may contribute to increase renewable energy production, with subsequent decrease in fossil fuel consumption. This should help reducing green house gases emissions resulting from the energy sector, which is essential bearing in mind its effect on Climate Change.

## **1.2. AN OVERVIEW ON SEWAGE SLUDGE ORIGIN, TREATMENT AND COMPOSITION**

### **1.2.1. Origin of sewage sludge**

Wastewater treatment consists of a series of operations through which pollutants are gradually removed, yielding a cleaner aqueous effluent and a relatively high-solids waste known as sewage sludge. Sewage composition depends on its origin; this is to say on the effluents discharged to sewers. In general, effluents from industrialised areas are more likely to have high concentrations of organic and/or inorganic pollutants compared to those from urban areas. Factors like sewage composition, the size of the population served or the emplacement of each WWTP will determine the sequence of processes designed to purify the effluent prior to its discharge in the environment. While most WWTP are based on biological processes; chemical treatments, pre-treatments or complementary treatments are sometimes required. Consequently, sewage sludge composition will be influenced by these factors.

In general wastewater treatment consists in the following stages:

- Pre-treatment
- Primary treatment
- Secondary treatment
- Tertiary treatment

The **pre-treatment** of wastewater consists of a series of physical operations, basically focused on screening and grit removal, as well as fat separation. The wastes separated through these processes have to be disposed of.

The **primary treatment** includes physical and sometimes chemical operations. The main process is the primary sedimentation in primary sedimentation tanks. The settlement of suspended solids towards the bottom of the tanks enables the removal of a fraction known as primary sludge, which has around 2-6 % solids, mainly composed of flocculated organic matter requiring further treatment.

The **secondary treatment** is usually biological and consists of the biodegradation of organic compounds dissolved in the wastewater. Depending on each process, biological reactors may operate under aerobic or anaerobic conditions, in continuous or batch mode, with suspended or fixed biomass, etc. In the activated sludge process, which is by far the most common in WWTP, suspended aerobic microbes degrade organic matter either in completely mixed or in plug flow reactors with aeration. In other systems like percolating filters, microbes are fixed forming biofilms, yielding less waste biomass compared to suspended growth processes.

The standard design of an activated sludge unit consists of two tanks: a bioreactor and a secondary clarifier (similar to the primary sedimentation tank), where microbial biomass settles and is removed from the purified flow. This is the fraction known as secondary or waste activated sludge (WAS), which is basically composed of biomass and, contrary to primary sludge, it is partially stabilised.

**Tertiary treatments** are additional processes aimed to provide a final purification; typically resulting from nutrients removal, especially nitrogen (nitrification/denitrification) and phosphates. The removal of nutrients might be included in the secondary treatment or alternatively be enhanced in extensive systems like reed beds.

In WWTP, sewage is treated in the so-called wastewater treatment line, and the resulting mixture of primary and secondary sludge is treated in the so-called sludge treatment line. The treatment given to the sludge may vary according to its final destination. While biological processes are most appropriate if it is to be used as an organic fertiliser on agricultural land; chemical and/or thermal processes might be used with highly polluted sludges that need to be disposed of.

It is generally estimated that sludge production as a result of biological wastewater treatment is around 50-70 g dry matter per inhabitant per day, corresponding to an approximate annual production per inhabitant of 18-25 kg of dry matter or 90-125 kg of dewatered sludge requiring treatment, according to data from La Llagosta WWTP and Metcalf and Eddy (2003).

### 1.2.2. Sewage sludge treatment

Sewage sludge treatment typically involves a series of four steps, namely:

- Sludge thickening
- Sludge stabilisation
- Sludge conditioning
- Sludge dewatering

#### a) Sludge thickening

The aim of sludge thickening is to increase the solids concentration in the sludge and to reduce the volume of sludge requiring further treatment. By means of gravity thickening or by flotation, the sludge volume is typically decreased by 2-3 % of the original volume.

**Gravity thickening** is used either prior to sludge stabilisation, enhancing such process while reducing the capital cost of the sludge treatment plant (i.e. smaller reactor); or following sludge stabilisation to concentrate the product obtained. It is usually carried out in circular tanks, with full diameter pickets mounted on arms to form a fence that moves at very slow speed, promoting conglomeration and accelerating settling. The concentrated sludge is extracted from the bottom, whereas the supernatant overflows and is returned to the wastewater treatment line. The residence time has to be sufficient but not excessive, because raw sludge tends to produce offensive odours, and also because it can lead to sludges excessively concentrated for pumping and transportation.

**Thickening by flotation** can be performed in two different ways, which are natural flotation and dissolved air flotation. In both cases, the clarified effluent is returned to the wastewater treatment line.

## **b) Sludge stabilisation**

The objective of this process is to reduce, inhibit or eliminate the putrefaction potential of sludge, potential offensive odours emissions and the levels of pathogens. It is also aimed to reduce the sludge volume, hence the sludge handling costs. It can be achieved by means of biological methods, including anaerobic and aerobic digestion, and composting; chemical methods, such as stabilisation with lime and chloride oxidation; and thermal methods, like thermal heating and incineration (see Section 1.2.2.d). Amongst the biological ones, anaerobic digestion is perhaps the most used.

**Anaerobic digestion** is a fermentation process by which anaerobic microbes degrade organic sludge in an enclosed reactor, yielding partially stabilised sludge and biogas as by-product, which is a renewable source of energy. More detailed information on anaerobic digestion is given in Section 1.3.

**Aerobic digestion** is a biodegradation process by which aerobic microbes degrade organic sludge in an open air reactor. It is actually similar to an activated sludge process, but in this case microbial growth is limited by soluble organic matter, and microbes use their own protoplasm to satisfy their energy requirements. This is known as the endogenous stage in microbial growth kinetics. The main advantage of aerobic compared to anaerobic digestion is that it is faster (i.e. 35-45 % volatile solids removal with residence times around 10 days). On the other hand, high energy requirements are its major disadvantage.

**Composting** can be used as a stabilisation process itself or following aerobic or anaerobic digestion in order to improve the quality of digested sludge, which is partially stabilised. It consists of the decomposition and stabilisation of organic solids by aerobic thermophilic (50-70 °C) and mesophilic (30-40 °C) biological processes. Sludge composting requires the mixture of dewatered sludge with some organic support, like wood shavings or sawdust. The final product is a stabilised organic material known as compost, its quality depending on the composition of materials in the mixture. Compared to anaerobic digestion, composting is less sensitive to

variations in environmental conditions and the capital cost of the treatment plants is much lower, but it tends to be far more labour intensive.

### c) Sludge conditioning

The aim of sludge conditioning is to improve sludge characteristics to enhance subsequent dewatering.

**Chemical conditioning** consists of the addition of coagulants such as iron chloride, lime, aluminium sulphate and inorganic polymers, which leads to the coagulation of solids, with the corresponding desorption of water contained in the sludge.

**Thermal conditioning** is achieved by heating the sludge during brief periods and under pressure, which results in solids coagulation, the rupture of the gel structure and the reduction of sludge affinity for water; together with sludge sterilisation.

### d) Sludge dewatering

The purpose of sludge dewatering is to reduce the moisture content, in order to ease sludge handling and decrease transportation cost. Dewatering technologies include simple drying beds or reed beds; as well as thermal and mechanical processes, which are usually preceded by sludge conditioning.

**Drying beds** are shallow vessels with a gravel layer up to 0.5 m, where stabilised sludge is spread to be drained and dried by filtration and evaporation. The final water content depends on the sludge characteristics, weather conditions and duration of the process, which might be reduced by previous sludge conditioning. Drying beds are simple in operation and useful for small throughputs when high levels of solids are not required; its major inconvenient being the requirement of large surface area.

**Centrifugation** consists of the separation of conditioned sludges into a liquid phase and a sludge cake. In centrifuges continuous dewatering is obtained by incorporating a horizontal axis cylinder or a conical bowl in compact enclosed premises, preventing offensive odours emissions. Centrifuges allow high throughputs and achieve solids concentrations up to 45 %. The main inconveniences are high energy requirements and maintenance costs.

**Filtration** processes include vacuum filtration or pressure filtration of conditioned sludge. Sludge dewatering by vacuum filtration involves a rotary drum filter which is semi-submerged in an open tank, and results in a thickened sludge and a supernatant. In pressure filtration sludge cakes are filtered by applying high pressures, obtaining the sludge cake filtrate as a waste effluent. This system allows high throughputs and results in solids contents as high as 30 %.

**Thermal drying** consists of the evaporative removal of interstitial water in sludge and is capable of removing up to 98 % of the water content in dewatered sludge. Under conditions of high temperature and pressure, proteins are hydrolysed causing cell destruction, organic compound solubilization and free ammonia emissions. This method is not sludge sensitive and results in a highly concentrated product; but depending on the sludge origin the heavy metal concentration might be too high for its use as fertiliser.

### 1.2.3. Sewage sludge composition

Sludge composition is characterised by four major parameters:

- High contents of water and organic matter
- Variable concentration of nutrients
- Presence of organic and inorganic micropollutants
- Presence of pathogens

High organic matter content (up to 80 %), together with a certain amount of macronutrients (N, P, K) plus some micronutrients, give sludge the potential to be used as an organic fertilizer; but its use may be limited by the presence of contaminants and pathogens. In Spain, it is regulated by the *Real Decreto 1310/1990, de 29 de octubre; por el que se regula la utilización de lodos de depuración en el sector agrario*.

The organic loading of sewage results from the presence of natural and synthetic organic compounds. Recalcitrant compounds, which resist biodegradation, remain in the treated water flow and waste sludge. Some examples include hydrocarbons and pesticides. Apart from those, high concentrations of heavy metals are typically found in wastewaters, and these too tend to cumulate in the waste sludge. A consequence of periodical spreading of such sludge onto agricultural fields might be the accumulation of heavy metals in the soil, which could then be absorbed by crops and get into the food chain. Due to the potential toxic effect of certain heavy

metals, limit concentrations are stringently regulated (Council Directive 86/278/EEC of 12 June 1986 on the protection of the environment, and in particular of the soil, when sewage sludge is used in agriculture; in Spain *Real Decreto 1310/1990, de 29 de octubre, por el que se regula la utilización de lodos de depuración en el sector agrario*).

The same situation applies to pathogenic microorganisms (including bacteria, viruses, and protozoa), which have an inherent health risk potential if spread onto agricultural fields. This risk can be reduced if disinfection techniques are used in the sludge treatment process. For this reason, advanced treatments providing effluent hygienisation prior to land application are proposed in the 3<sup>rd</sup> Draft EU Working Document on Sludge (Environment DG, EU, 2000), with limit values proposed for *Salmonella* spp. (absence in 50 g) and *Escherichia coli* (6 log<sub>10</sub> reduction to less than 5×10<sup>2</sup> CFU g<sup>-1</sup>).

As already discussed, sewage sludge composition depends on a range of factors, including the origin of the wastewater, the presence and type of industries discharging to sewers, the wastewater and sludge treatment system, etc. (Pomares, 1982). The consequent variability in chemical and physico-chemical properties can be seen in Table 1.2, showing sewage sludge composition (Pomares and Canet, 2001).

The moisture content is strongly affected by treatment processes, especially sludge thickening and dewatering, which may explain the difference between the maximum and minimum value (2.7 and 95.2 %). Despite its variability, organic matter content is generally high, ranging from 36 to 80 %, within the range of cattle manure or compost obtained from the organic fraction of municipal solid wastes (OFMSW).

A relatively high concentration of nitrogen (2-7 % N) is also common in sewage sludge. However, the total N content and its organic and mineral forms are very much dependant on the origin of the wastewater, together with the wastewater and sludge treatment. For example, municipal wastewater typically has a high concentration of urea that is rapidly hydrolysed yielding ammonia N, which might then be oxidised to nitrate and subsequently to N gas if nitrification and denitrification processes are incorporated in the wastewater treatment line. With regards to the sludge, the mineral fraction (ammonia N and nitrate) can be as high as 50 % in liquid sludges, whereas the major fraction is organic N in dewatered sludges. This parameter is particularly important upon sludge use as organic fertilizer for agricultural crops, because the mineral fraction

is readily available for the crops, but only a portion of the organic N is available within the same year.

**Table 1.2.** Composition of the sludge from wastewater treatment plants in the Region of València (Spain) in 1997 (Pomares and Canet, 2001)

Parameter	Unit	Amount *
Moisture	%	2.7 – 95.2
Total solids (TS)	%	4.8 – 97.3
Volatile solids (VS)	%	36.5 – 79.4
Oxydable organic matter	%	27.6 – 74.5
Nitrogen (N)	%	2.0 – 7.4
Phosphorus (P <sub>2</sub> O <sub>5</sub> )	%	0.82 – 5.25
Potassium (K <sub>2</sub> O)	%	0.08 – 1.24
Calcium (CaO)	%	3.25 – 19.80
Magnesium (MgO)	%	0.42 – 2.42
Cadmium (Cd)	ppm	< 0.5 – 10
Chromium (Cr)	ppm	< 0.5 – 4479
Copper (Cu)	ppm	78 – 912
Iron (Fe)	ppm	2,485 – 98,592
Mercury (Hg)	ppm	< 0.5 – 1.4
Manganese (Mn)	ppm	51 – 402
Nickel (Ni)	ppm	4.4 – 567
Lead (Pb)	ppm	23 – 2,804
Zinc (Zn)	ppm	195 – 5,098
pH		6.2 – 7.5
EC (1:5 extract)	dS m <sup>-1</sup>	1.23 – 9.35

\* Data referred to dry matter, except the moisture content

With reference to the other macronutrients, the major proportion of phosphorus and almost all potassium is usually found in mineral form; with concentrations ranging from 0.82 to 5.25 % and 0.08 to 1.24 % expressed as P<sub>2</sub>O<sub>5</sub> and K<sub>2</sub>O, respectively.

Although some heavy metals are also essential microelements for plant nutrition (i.e. iron, cuprum, zinc or manganese), they are needed in very low concentrations. As already discussed, high concentrations of heavy metals could restrict land spreading of sewage sludge.

In general, the pH of sewage sludge is around neutrality, although it can be slightly acidic or slightly basic. As far as salinity is concerned, its variability from 1 to 9 dS m<sup>-1</sup> (expressed as electric conductivity of a 1:5 extract) results from different sludge origins and treatments. This means that, at least in some cases, there would be a potential risk of soil salinisation upon periodical land spreading of sludge. Furthermore, in the case of sludges flocculated with iron or calcium chloride, there is a potential toxicity to crops sensitive to chlorides (Pomares and Canet, 2001).

### 1.3. FUNDAMENTALS OF ANAEROBIC DIGESTION OF SEWAGE SLUDGE

Anaerobic digestion is a microbiological process that occurs naturally in the environment, for example in lagoons or in the stomach of ruminants. Under anaerobic conditions, organic materials are biodegraded through a complex microbiological process leading to the production of a more stabilized organic material and biogas with high methane (CH<sub>4</sub>) content.

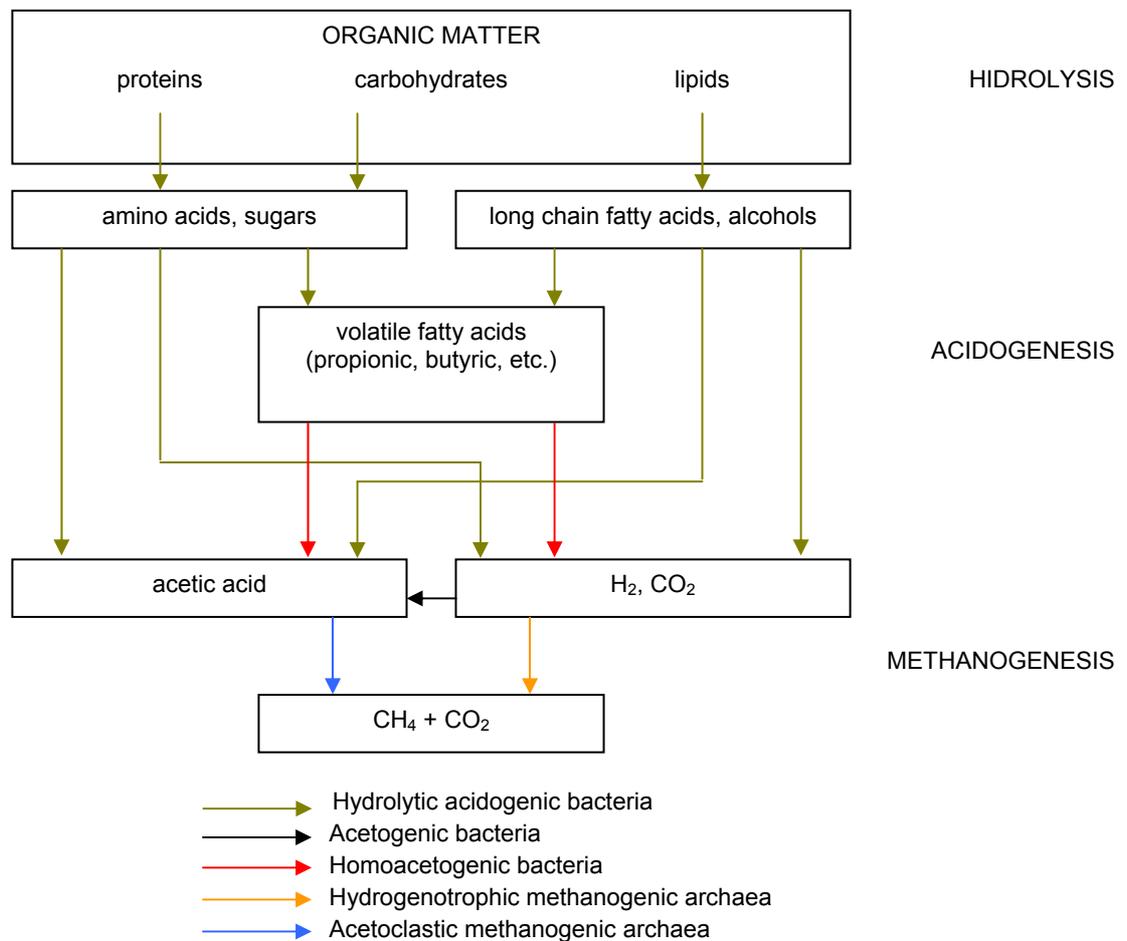
The technological application of this process in bioreactors gives an appropriate solution for the treatment of organic wastes and by-products, such as sewage sludge. The effluent of bioreactors can be used as an organic fertiliser as long as it meets current legislation for land application. Biogas production depends on the composition of the raw materials treated and operational conditions (reactor design, process temperature, sludge retention time (SRT), etc.), being typical values 0.5-1 m<sup>3</sup><sub>biogas</sub> m<sup>-3</sup><sub>reactor</sub> d<sup>-1</sup>, with 60-70 % methane. As methane energetic value is 10 kWh m<sup>-3</sup>, it can be used for heating and/or electricity production, giving an energetic valorisation of the organic materials treated. Hence, technological strategies based on anaerobic digestion may allow for both sustainable waste management and renewable energy production. This is the major advantage of anaerobic digestion with respect to aerobic treatment alternatives, like aerobic digestion or composting.

#### 1.3.1. Description of the process

The process takes place in an enclosed reactor in absence of oxygen, where degradation of organic materials occurs through 3 consecutive stages, namely hydrolysis, acidogenesis and methanogenesis (Figure 1.2).

In the first stage, facultative hydrolytic bacteria using extracellular enzymes hydrolyse particles and complex molecules (proteins, carbohydrates and lipids) to soluble compounds (amino acids,

sugars, long chain fatty acids (LCFA) and alcohols). During the acidogenic fermentation, these compounds are firstly transformed to short chain or volatile fatty acids (VFA), like propionic and butyric acid, and subsequently into acetic acid and other VFA, hydrogen and carbon dioxide. Finally, methanogenic archaea produce methane from acetic acid and from carbon dioxide and hydrogen.



**Figure 1.2.** Stages and bacterial populations involved in anaerobic digestion (Pavlostathis and Giraldo-Gómez, 1991)

Most anaerobic systems consist of a single-stage digester, which means that all stages take place in the same reactor. In such situation, environmental conditions (i.e. pH, redox potential, temperature, etc.) may favour the development of a certain group of bacteria, but it is important to maintain equilibrium to ensure a balanced degradation process. For this reason, the control of environmental conditions is a key factor, especially regarding methanogenic microorganisms, which are strict anaerobes, with the lowest growth rate and are the most sensitive to sudden changes in environmental conditions. In effluents with mostly soluble organic compounds (like wastewater), hydrolysis and acidogenesis are pretty straightforward, and methanogenesis tends to

be the most critical stage. On the other hand, the hydrolysis of particulate organic materials is rate limiting with substrates like sewage sludge, manure or the organic fraction of municipal solid waste.

Some treatment plants have implemented two-stage systems in which hydrolysis-acidogenesis and methanogenesis are separated. This allows for different environmental conditions in each reactor, promoting the development of different microbial population in each reactor, which is reported to guarantee more stable process performance, its major inconvenience being that it is a costly solution.

### 1.3.2. Process and control parameters

Process parameters can be split into the so-called environmental parameters (pH, redox potential, alkalinity, concentration and nature of organic and inorganic compounds) which are summarized in Table 1.3; and operating parameters (temperature, sludge retention time and cellular retention time, organic loading rate and stirring). Most operating parameters will depend upon system configuration and design.

**Table 1.3.** Anaerobic process and control parameters

Parameter	Optimum range	Potential risk
pH	6.5-7.5	Digester acidification Requires external control if the substrate has low buffer capacity
Alkalinity	1.5-3 g CaCO <sub>3</sub> L <sup>-1</sup>	Ensures buffering capacity Allows for indirect detection of digester acidification
Redox potential	< -300 mV	Indicates reductive atmosphere in the system
C/N	~ 30 (15-45)	> (N deficiency) may decrease reaction rate < (N excess) may cause inhibition, especially due to ammonia N
C/P	~ 150	< (P excess) do not cause inhibition

Control parameters are required for monitoring and control process performance, in order to maintain optimum and steady operating conditions. Stability is particularly important in anaerobic systems, because they are quite sensitive to chemical and physico-chemical inhibitions. Moreover, as anaerobic degradation of particulate organic matter is a slow process (compared to aerobic degradation, for instance), it requires high SRT and stability recovery might take long period (Soto *et al.*, 1993a). Stability loss may result from:

- Organic overloading (influent strength) or hydraulic overloading (washout risk).
- Thermal shock, caused by a rapid increase/decrease of the temperature.
- Presence of toxic or inhibitory substances, either coming with the influent or formed during the fermentation process (i.e. free ammonia).
- Changes of physico-chemical conditions in the system: pH, redox potential, temperature, etc.

Basic parameters for appropriate control of an anaerobic system include: temperature, biogas production rate, pH, alkalinity and organic matter content (determined as volatile solids (VS), chemical oxygen demand (COD) or biochemical oxygen demand (BOD)). These data enables the calculation of the main operating and efficiency parameters. However, if possible, analyses of intermediate species (VFA) and reaction products (biogas composition) gives direct information on process performance. An early detection of process unbalance should help avoiding an eventual digester failure.

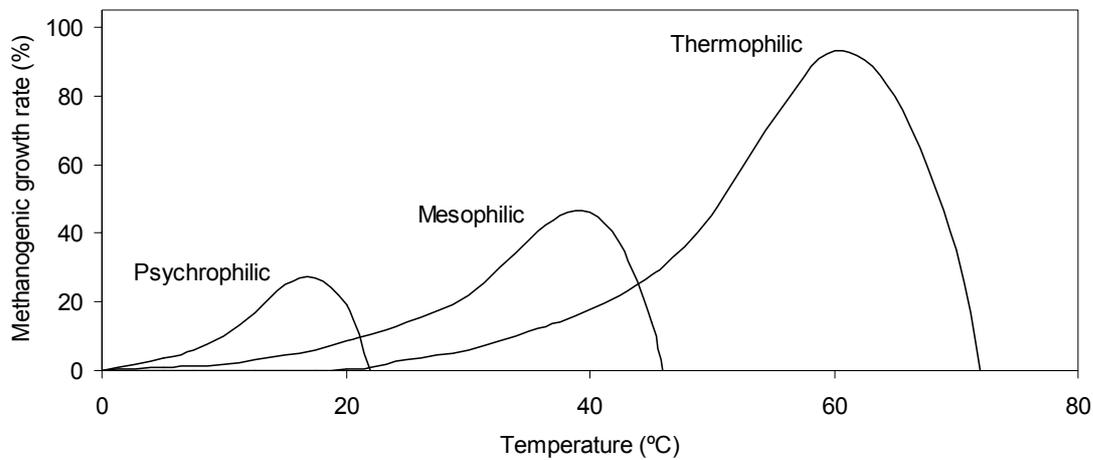
### 1.3.3. Digester design

Digester design depends on (1) substrate composition, especially regarding the solids concentration, and flow rate; and (2) economical constraints, both for the implementation of the system and, most importantly, for its operation and maintenance. Some basic design parameters are:

- Type of flow: batch, intermittent or continuous.
- Stirring system (mechanical stirrers; gas or sludge recirculation), if any.
- Biomass retention mechanism (if any), suspended or fixed biomass.
- Temperature range: psychrophilic (< 25 °C), mesophilic (30-40 °C) or thermophilic (50-60 °C).
- SRT: 10-50 days, depending on process temperature and flow rate.
- Volume, according to SRT and flow rate, and number of units.

Process temperature has to be set within the above mentioned ranges (psychrophilic, mesophilic or thermophilic), to promote maximum growth rates of the corresponding microbial populations, thus maximum substrate degradation rates and process efficiency (Figure 1.3). In general, the higher the temperature, the faster the reaction rate and the lower the SRT and volume required.

Treating organic wastes like sewage sludge, the SRT can be as low as 10-15 days at 55 °C, typically 15-20 days at 35 °C and up to 40-50 days at ambient temperature.

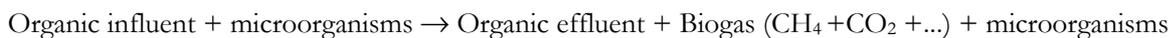


**Figure 1.3.** Dependence of methanogenic microorganisms specific growth rate ( $\mu$ ) on temperature (adapted from Van Lier *et al.*, 1993)

Typical digesters for the stabilisation of sludge in conventional wastewater treatment facilities are continuous stirred tank reactors (CSTR), with suspended biomass through mechanical stirring or biogas recirculation, operated in continuous or semi-continuous mode. Digester volume depends on sludge flow rate and SRT, which in turn depends on the organic loading rate (OLR) and process temperature. It also varies from one-stage and two-stage systems. In two-stage systems, with two reactors connected in series, the operating conditions (temperature and SRT) may be the same or may vary to enhance each stage separately.

#### 1.3.4. Biogas production

Anaerobic biodegradation of organic matter yields a mixture of gases, known as biogas, and biomass. Biogas is composed mainly by methane (60-70 % CH<sub>4</sub>), but also by carbon dioxide (30-40 % CO<sub>2</sub>) and trace amounts of other gases like H<sub>2</sub> and H<sub>2</sub>S. A simplified equation of the process may be written as follows:



According to a theoretical mass balance for an anaerobic digester operating under steady state conditions, the organic matter removed from the system is converted to methane. Expressed as COD, the methane produced as a result of COD conversion is  $0.35 \text{ m}^3_{\text{CH}_4} \text{ kg COD}_{\text{removed}}^{-1}$  (at

standard conditions) and, expressed as VS, it is approximately  $0.5 \text{ m}^3_{\text{CH}_4} \text{ kg VS}_{\text{removed}}^{-1}$  (at standard conditions). The latter is calculated assuming a theoretical conversion coefficient of  $1.425 \text{ kg COD kg VS}^{-1}$ ; which is obtained by approximating the composition of organic solids in the sludge to the formula  $\text{C}_{18}\text{H}_{19}\text{NO}_9$ . Bearing in mind that methane content in biogas is around 60-70 %; in terms of biogas such values would be higher. The values  $0.35 \text{ m}^3_{\text{CH}_4} \text{ kg COD}_{\text{removed}}^{-1}$  and  $0.5 \text{ m}^3_{\text{CH}_4} \text{ kg VS}_{\text{removed}}^{-1}$  correspond to the maximum specific methane production. Since methane energetic value is  $10 \text{ kWh m}^{-3}$ , they are equivalent to  $0.35 \text{ kWh kg COD}_{\text{removed}}^{-1}$  and  $0.5 \text{ kWh kg VS}_{\text{removed}}^{-1}$ .

The fuel gas produced might be burned in heaters or fuelled in combined heat and power generation (or cogeneration) units. The technology known as cogeneration is based on the simultaneous production of electricity (originally mechanic energy) and heat (thermal energy), from a primary energy source (a fuel); like natural gas, diesel oil or biogas. In this way, in cogeneration units a fuel is used to produce electricity, while the waste heat is recovered; optimising the use of primary energy.

The efficiency of heat and power units, in terms of electricity and waste heat production, depend on the equipment used, which can be a gas turbine, a vapour turbine or an internal combustion engine. Maximum power efficiencies are in the range of 30-40 %, whereas waste heat accounts for 50-60 %. Most of the waste heat is actually a flow of hot water or vapour at some  $90 \text{ }^\circ\text{C}$ , which is not always easy to use (Claramunt, 1997). It should be used to cover the heat demand on-site or elsewhere, otherwise excess waste heat may be a limiting factor for the implementation of this technology.

In an anaerobic digestion plant, heat requirements are mainly those for the maintenance of process temperature in the bioreactor. Hence, they may vary depending on, amongst others, the type of reactor (surface, material, insulation, etc.), the substrate (specific heat) and environmental temperature. A balance between the heat requirements of the digester and the waste heat from the cogeneration unit, would determine the surplus energy to be used elsewhere in the process.

#### **1.4. AN APPROACH TO THE IMPROVEMENT OF ANAEROBIC SLUDGE DIGESTION**

As already discussed, mesophilic anaerobic sludge digestion is widely used for the stabilisation of sewage sludge WWTP. The conventional process ( $35\text{-}40 \text{ }^\circ\text{C}$ ;  $>20$  days SRT) is efficient in terms

of solids reduction, but requires high SRT, thus large reactors; and it is not as efficient in terms of biogas production (i.e. renewable energy production). Sludge digestion is a slow process mainly due to the disintegration and hydrolysis of particulate compounds, but also as a result of low growth rates of methanogens. Additionally, a considerable proportion of solid compounds are recalcitrant, which leads to poorer efficiencies in organic solids removal and methane production.

For this reason, continuous attempts to improve process performance, either by accelerating the reaction rate or by increasing the amount of biodegradable compounds, can be found in the literature. Most of them require intensive use of energy for sludge pre-treatment through mechanical or high temperature/pressure processes, or even the use of additional chemicals, which may affect final sludge disposal. The use of waste heat from heat and power generation units brings a sustainable way of improving the process (Bonmatí, 2001). Some approaches might be:

1. Thermophilic operation (50-60 °C), either in one or two-stage systems.
2. Low temperature (< 100 °C) sludge pre-treatment.

Thermophilic operation in one and two-stage systems has long been implemented in some countries (Buhr and Andrews, 1977). However, it is still not clear which are the optimum conditions (temperature and SRT) to maximise methane production, while enabling sufficient organic solids removal to guarantee a minimum quality of the effluent sludge, and ensuring a stable operation of the thermophilic process. Such issues are addressed in Chapter 4.

Regarding low temperature sludge pre-treatment, it has been extensively studied with the aim of determining optimum pre-treatment conditions to enhance sludge solubilization, and in some cases anaerobic biodegradability under mesophilic conditions. But little work has been done under thermophilic conditions (Gavala *et al.*, 2003; Climent *et al.*, 2007), especially in a continuous process (Skiadas *et al.*, 2004; Lu *et al.*, 2007). Optimum conditions for a low-temperature pre-treatment of the mixture of primary sludge and WAS in order to improve subsequent thermophilic anaerobic digestion have not been determined. Chapter 5 is focused on this aspect.

Finally, energy consumption as a result of such processes should be taken into account. In theory, heat requirements for the operation of thermophilic sludge digestion, which are about twice those of mesophilic digestion, should be covered with the waste heat from a heat and power generation with biogas; together with heat regeneration from the sludge outflow

(Zupančič and Roš, 2003). Similarly, extra energy requirements for the operation of a pre-treatment step (70 °C) of primary sludge should be covered by the extra methane production (Lu *et al.*, 2007). On the whole, it depends on the digester volume, sludge flow rate, process and environmental temperature, methane production, etc. Therefore, a study in detail of the energy balance of each process is required. This study is covered in Chapter 6.



## **Chapter 2. Objectives**

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## **Chapter 2. OBJECTIVES**

The aim of this PhD Thesis was to study the thermophilic anaerobic digestion of sewage sludge and some strategies for its enhancement by sludge hydrolysis optimisation. For this purpose, an experimental set-up was designed to carry out semi-continuous experiments. The lack of thermophilic inoculum made it necessary to start-up the process with mesophilic inoculum and acclimate the digesters to thermophilic conditions. Process performance was studied at different operating temperatures and at decreasing sludge retention time (SRT). The effect of low temperature sludge pre-treatment was thereafter evaluated. Finally, all processes were assessed from an energy perspective.

The specific objectives of the present work were:

1. To study the effect of process temperature on the anaerobic degradation of sewage sludge, in terms of gas production and quality of the effluent sludge (Chapter 4).
2. To study the thermophilic anaerobic digestion of sewage sludge focused on process stability and efficiency at decreasing sludge retention time (Chapter 4).
3. To evaluate the effect of a low temperature pre-treatment (70 °C) on the thermophilic anaerobic digestion of sewage sludge (Chapter 5).
4. To assess alternatives for the enhancement of conventional sewage sludge digestion from an energy perspective (Chapter 6).



## **Chapter 3. Materials and Methods**

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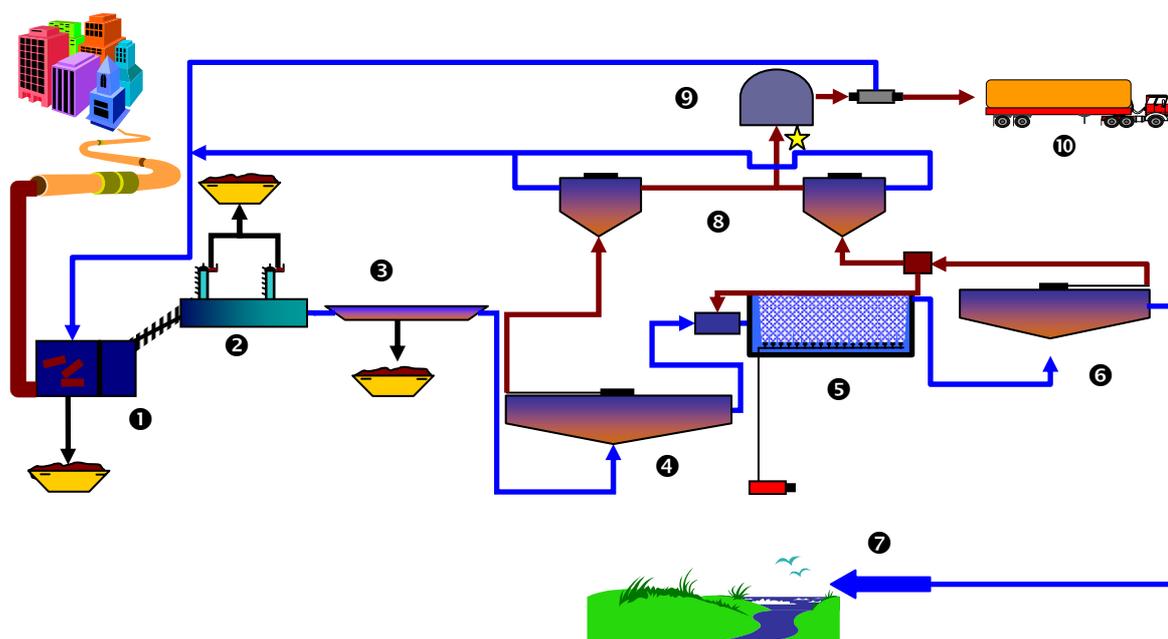
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## Chapter 3. MATERIALS AND METHODS

### 3.1. SEWAGE SLUDGE

The sludge used for this work was obtained from two municipal wastewater treatment plants (WWTP), La Llagosta and Granollers, near Barcelona (Spain). These WWTP serve an equivalent population around 130,000 equivalent inhabitants (EI). The conventional wastewater treatment used in these plants consists of preliminary and primary treatment and secondary treatment in the activated sludge unit. Primary sludge (PS) and secondary waste activated sludge (WAS) are thickened and mixed (this is the sampling point shown in Figure 3.1), before undergoing mesophilic (38 °C) anaerobic digestion at very high sludge retention time (SRT ~ 40 days) aimed to reduce the solids content and improve dewatering in a centrifuge prior to final disposal. Most of it is applied in agricultural crop fields and some minor proportion is sent to landfill.



**Figure 3.1.** Schematic diagram of the wastewater treatment plants of La Llagosta and Granollers. Wastewater treatment line: pre-treatment (1, 2, 3), primary treatment (4), secondary treatment (5, 6), effluent discharge (7); sludge treatment line: thickening (8), stabilisation by anaerobic digestion (9), treated sludge to final disposal (10); sludge sampling point (yellow star)

The inoculum used to seed the digesters was mesophilic digested sludge from Granollers WWTP. The substrate was the mixture of thickened PS and WAS (75 / 25 % v/v), which was collected weekly and stored at 4 °C until use. Sludge from Granollers WWTP, namely low-solids sludge, was used for the first 14 months; whereas sludge from La Llagosta WWTP, namely high-solids sludge, was used thereafter.

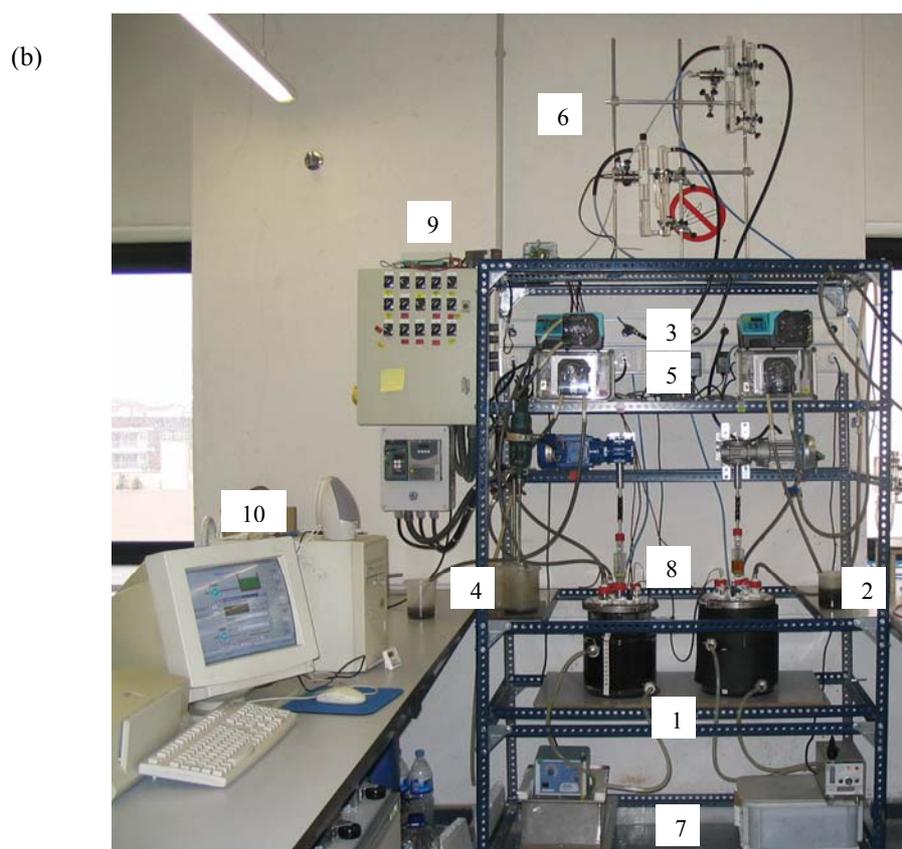
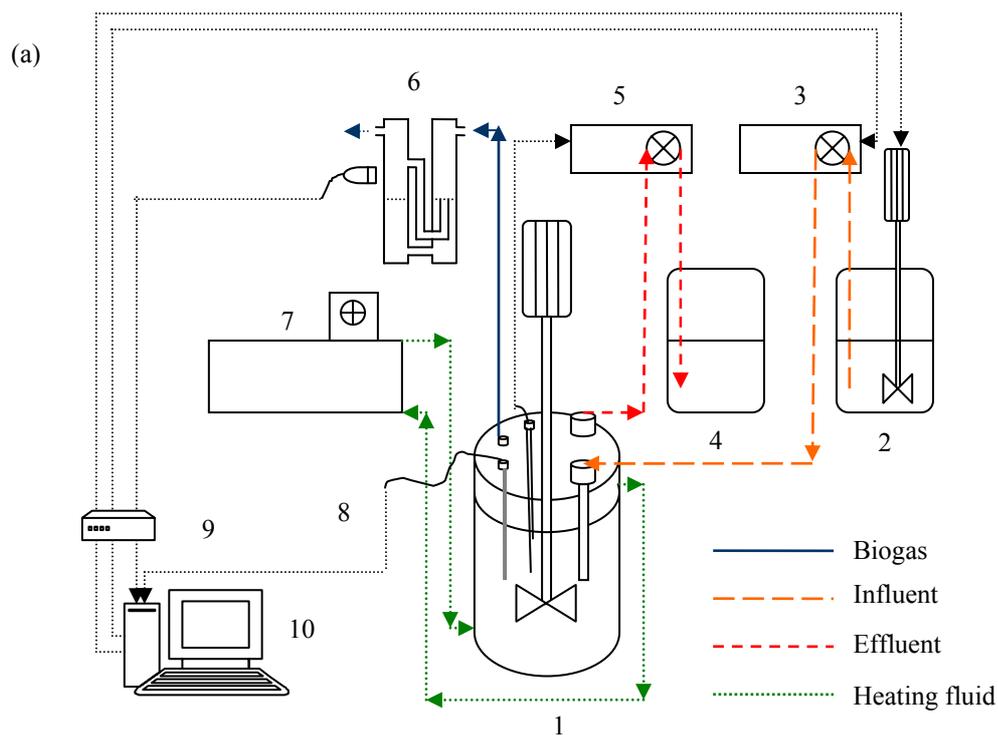
### 3.2. EXPERIMENTAL SET-UP

The experimental set-up used in this work (Figure 3.2) consists of two jacketed continuous stirred tank reactors (CSTR), connected to a thermostatic bath through which temperature is controlled (35-55°). The total volume of each reactor is 6.5 L; corresponding to 5 L working volume (sludge) and 1.5 L headspace volume (biogas). Each reactor is composed of a glass vessel with a stainless steel top cover, in which continuous mixing is achieved by means of an anchor shaped stainless-steel impeller rotating by the action of a small industrial engine (SITI MI-40). Semi-continuous feeding is automated via a Data Acquisition System (DAS, by STEP S.L.) which activates the feeding and extraction peristaltic pumps (Watson Marlow 501 FAC/RL2) twice a day, giving a total volume (Q) corresponding to the SRT. The volume of biogas produced is measured with a device designed by Mata-Álvarez *et al.* (1986). The detector is a capacitive sensor (Carlo Gavazzi M18) connected to the DAS. Process temperature is also monitored on-line by means of a thermal sensor (DESIN) submerged in the liquor and connected to the DAS. Real time data from the DAS is displayed in a PC (software by STEP S.L.), as shown in Figure 3.3. The design and set-up of the lab-scale pilot plant is fully described in Ferrer (2003a) and Ferrer *et al.* (2004a).

### 3.3. EXPERIMENTAL PROCEDURES

#### 3.3.1. Process monitoring

Process performance was followed by on-line measurement of biogas production and process temperature, together with the measurement of sludge daily flow rate. Analyses of influent and effluent sludge samples (total and volatile solids (TS and VS), volatile fatty acids (VFA), pH and alkalinity) and biogas samples (% CH<sub>4</sub>) were carried out with the periodicity shown in Table 3.1. Physico-chemical parameters were routine analyses, whereas microbiological determinations (*E.coli* and *Salmonella* spp.) and capillary suction time (CST) were only determined for initial or final characterisation of influent and/or effluent sludge samples. Analytical procedures were based on Standard Methods (see Section 3.6).



**Figure 3.2.** Schematic diagram (a) and caption (b) of the experimental set-up used for mesophilic and thermophilic anaerobic digestion of sewage sludge. 1) Continuous stirred tank reactors (R1 and R2); 2) Influent storage; 3) Feed pump; 4) Effluent storage; 5) Extraction pump; 6) Gas meter; 7) Thermostatic bath; 8) Temperature sensor; 9) Data acquisition system; 10) Personal computer

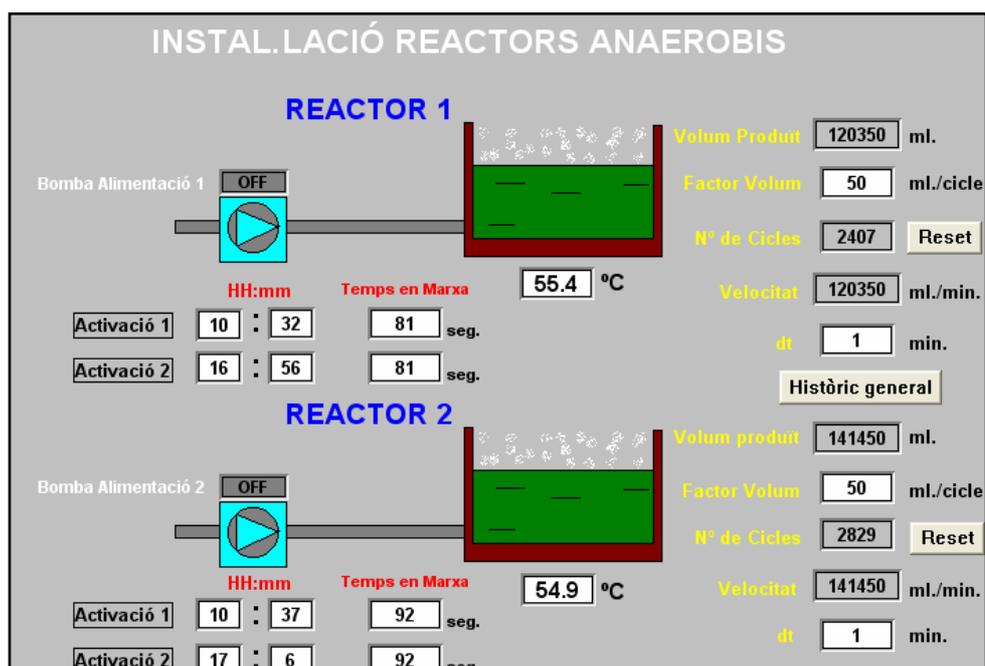


Figure 3.3. Display of real time data from the data acquisition system in a personal computer

Process efficiency under stable conditions for each treatment assayed was evaluated in terms of biogas and methane production rates ( $\text{m}^3 \text{m}^{-3} \text{reactor} \text{d}^{-1}$ ), specific productions ( $\text{m}^3 \text{kg VS}^{-1}_{\text{fed}}$ ) and yields ( $\text{m}^3 \text{kg VS}^{-1}_{\text{removed}}$ ), as well as the quality of the effluent sludge (i.e. concentration of VS and VFA, sludge dewaterability and hygienisation, etc.). Process parameters were calculated according to Section 3.3.2.

Table 3.1. Periodicity of analyses for monitoring process performance (adapted from Soto *et al.*, 1993a)

	Parameter	Periodicity
Influent and effluent	Total solids and volatile solids	2-3 times per week
	pH	2-3 times per week
	Alkalinity	2-3 times per week
	VFA concentration	2-3 times per week
	<i>E. coli</i> and <i>Salmonella</i> spp.	For characterisation
	Capillary Suction Time	For characterisation
Digester	Temperature	Daily
	Sludge flow rate	Daily
	Biogas production	Daily
	Biogas composition	2-3 times per week

### 3.3.2. Calculation of parameters

Process parameters were calculated as follows:

#### Sludge retention time (SRT)

In completely mixed reactors without recycling, like the CSTR used in these experiments, the hydraulic residence time (HRT) is the same as the cellular retention time, also known as the age of the sludge. In this work, since the CSTR were treating sewage sludge, they are referred to sludge retention time (SRT), calculated from the reactor working volume (V) and the sludge daily flow rate (Q):

$$SRT = \frac{V}{Q} \quad (\text{d}) \quad \text{Eq.3.1.}$$

#### Organic loading rate (OLR)

The OLR is the amount of organic matter added per day, referred to the reactor working volume. It depends on the SRT and organic matter (i.e. VS) concentration in the influent ( $S_i$ ).

$$OLR = \frac{Q}{V} S_i = \frac{S_i}{SRT} \quad (\text{kg VS m}^{-3} \text{ reactor d}^{-1}) \quad \text{Eq. 3.2.}$$

#### Volatile solids removal (VS removal) and total solids removal (TS removal)

VS removal is estimated as the difference between the VS concentration in the influent and effluent, with respect to the VS concentration in the influent. For a given SRT, the mean value of influent VS ( $\hat{S}_i$ ) during that SRT can be used, and compared to the daily measurement of effluent ( $S_e$ ). TS would be analogously calculated.

$$VS_{\text{removal}} = \frac{\hat{S}_i - S_e}{\hat{S}_i} 100 \quad (\%) \quad \text{Eq. 3.3.}$$

#### Biogas production rate ( $P_{\text{biogas}}$ ) and methane production rate ( $P_{\text{CH}_4}$ )

Biogas production rate is the volume of biogas produced per day, referred to the reactor working volume ( $\text{m}^3_{\text{biogas}} \text{ m}^{-3} \text{ reactor d}^{-1}$ ). Knowing its composition, it is possible to estimate methane

production rate, as a product of biogas production rate and methane content (% CH<sub>4</sub>) in biogas ( $\text{m}^3_{\text{CH}_4} \text{m}^{-3}_{\text{reactor}} \text{d}^{-1}$ ).

### Specific biogas production ( $SP_{\text{biogas}}$ ) and specific methane production ( $SP_{\text{CH}_4}$ )

The specific biogas and methane productions are calculated by referring biogas ( $P_{\text{biogas}}$ ) and methane ( $P_{\text{CH}_4}$ ) production rates to the organic loading rate (OLR).

$$SP_{\text{biogas}} = \frac{P_{\text{biogas}}}{\text{OLR}} \quad (\text{m}^3_{\text{biogas}} \text{kg VS}^{-1}_{\text{fed}}) \quad \text{Eq. 3.4.}$$

$$SP_{\text{CH}_4} = \frac{P_{\text{CH}_4}}{\text{OLR}} \quad (\text{m}^3_{\text{CH}_4} \text{kg VS}^{-1}_{\text{fed}}) \quad \text{Eq. 3.5.}$$

### Biogas yield ( $Y_{\text{biogas}}$ ) and methane yield ( $Y_{\text{CH}_4}$ )

Biogas and methane yields are calculated by referring biogas ( $Y_{\text{biogas}}$ ) and methane ( $Y_{\text{CH}_4}$ ) yield to the VS removed.

$$Y_{\text{biogas}} = \frac{P_{\text{biogas}}}{V \text{ OLR } VS_{\text{removed}}} \quad (\text{m}^3_{\text{biogas}} \text{kg VS}^{-1}_{\text{removed}}) \quad \text{Eq. 3.6.}$$

$$Y_{\text{CH}_4} = \frac{P_{\text{CH}_4}}{V \text{ OLR } VS_{\text{removed}}} \quad (\text{m}^3_{\text{CH}_4} \text{kg VS}^{-1}_{\text{removed}}) \quad \text{Eq. 3.7.}$$

## 3.4. ANAEROBIC BATCH TESTS

Anaerobic batch tests were used to determine the anaerobic biodegradability of sludge samples under thermophilic conditions (55 °C). Biogas production was measured manometrically, with a device designed for the purposes of this study (Ferrer, 2003b; Fornés, 2004; Ferrer *et al.*, 2004b), which is shown in Figure 3.4. Batch tests were based on Soto *et al.* (1993b).

The inoculum was thermophilic sludge from the effluent of a lab-scale 5 L continuous stirred tank reactor (CSTR), maintained at 20 days SRT and 55 °C. This digester was fed with sludge mixture (PS and WAS) from the same WWTP as that used for the anaerobic batch tests. The substrate was either raw sludge (control treatment) or pre-treated sludge (at 70 °C for 9, 24, 48

or 72 h). A blank test with only inoculum was used to determine biogas production by the inoculum itself. Each treatment was performed in triplicate.

Each bottle-reactor (300 mL, SIGG<sup>®</sup>) was filled with 100 g of inoculum and 50 g of substrate (the blank test only with 150 g of inoculum) and was subsequently purged with N<sub>2</sub> and sealed. The bottles were incubated at 55 °C and biogas production was followed by the pressure increase in the headspace by means of a SMC Pressure Switch manometer (1 bar, 5 % accuracy), until biogas production ceased (Figure 3.4). Biogas samples were taken periodically for the analysis of methane content by gas chromatography.

Accumulated volumetric biogas production (mL) was calculated from the pressure increase in the headspace volume (150 mL) at 55 °C and expressed under normal conditions (20 °C, 1 atm). The net values of biogas production were obtained by subtracting biogas production of the blank treatment to biogas production of each treatment.



**Figure 3.4.** Incubation and biogas measurement during anaerobic batch tests (Ferrer *et al.*, 2004b)

### 3.5. LOW-TEMPERAURE (70 °C) SLUDGE PRE-TREATMENT

The low temperature pre-treatment was carried out at 70 °C in order to enhance thermal solubilization of particulate material, as well as enzymatic hydrolysis.

Beakers containing 0.5 L of sludge were submerged in a thermostatic bath at 70 °C during 9, 24, 48 and 72 h. The beakers were covered with plastic film, to avoid water evaporation, and gently

stirred (Heidolph RZR1) to ensure temperature homogeneity. Samples of raw and pre-treated sludge were analysed for total solids (TS), volatile solids (VS), total dissolved solids (TDS), volatile dissolved solids (VDS), volatile fatty acids (VFA) and pH.

The effect of pre-treatment time was assessed by the increase in VDS and VFA, comparing the initial concentration of VDS and VFA in the raw sludge with those obtained after each pre-treatment time assayed. Sludge solubilization was also evaluated by the increase in the ratio soluble to total volatile solids (VDS/VS), calculated as shown in Eq. (5.1), where the sub-indexes refer to raw (o) and treated (t) sludge samples.

$$VDS/VS = \frac{(VDS/VS)_t - (VDS/VS)_o}{(VDS/VS)_o} \quad (\%) \quad \text{Eq. 3.8.}$$

### 3.6. ANALYTICAL METHODS

#### 3.6.1. Sample preparation

Total solids, volatile solids, microbiological analyses (*E.coli* and *Salmonella* spp.) and capillary suction time were determined directly from fresh influent and effluent sludge samples.

Soluble constituents were determined from the supernatant of samples centrifuged (HERAEUS Biofuge Primo) at 7000 rpm for 30 minutes. Supernatants underwent vacuum filtration through 1.2 µm nominal pore size glass fiber filters (Albet FVC047, Spain). The soluble fractions of total solids and volatile solids, pH and alkalinity and volatile fatty acids (acetic, propionic, iso-butiric, n-butiric, iso-valeric and n-valeric acids) were analysed from the filtrate supernatant. Samples for VFA analysis were further filtered through a 0.45 µm nylon syringe filter.

#### 3.6.2. Total and volatile solids

Total solids (TS) and volatile solids (VS) contents were analysed according to the methodology described in the Standard Methods procedure 2540G (APHA, 1999).

TS and VS were analysed from 10 g (~10 mL) samples of fresh influent or effluent sludge. Total dissolved solids (TDS) and volatile dissolved solids (VDS) were analysed from 10 g (~10 mL) samples of filtered supernatant.

Total solids (or dry matter) correspond to the material remaining after water evaporation from a sample placed at 105 °C (Rottermann 2711) for at least 24 hours. Volatile solids (or organic matter) correspond to the loss of weight caused by the ignition of a sample (previously dried at 105 °C) at 550 °C for 2 hours in a muffle furnace (Heron 12-PR/200 Series 8B). The remaining residue is the ash (or mineral matter).

$$TS = \frac{\text{weight}_{105^{\circ}\text{C}}}{\text{weight}_{\text{fresh sample}}} 100 \quad (\% \text{ or } \text{g L}^{-1}) \quad \text{Eq. 3.9.}$$

$$VS = \frac{\text{weight}_{105^{\circ}\text{C}} - \text{weight}_{550^{\circ}\text{C}}}{\text{weight}_{\text{fresh sample}}} 100 \quad (\% \text{ or } \text{g L}^{-1}) \quad \text{Eq. 3.10.}$$

### 3.6.3. pH

pH was measured from the filtered supernatant with a pH-meter (with a glass electrode), previously calibrated by applying commercial buffer solutions at pH 7.02 and 4.00.

### 3.6.4. Alkalinity

Alkalinity measurement according to the Standard Methods procedure 2320B (APHA, 1999), consists of a titration of the sample with a strong acid until the pH decreases to 4.3. At this point, more than 99 % of bicarbonates ( $\text{HCO}_3^-$ ) are already converted into carbon dioxide ( $\text{CO}_2$ ). But titration is affected by more than 80 % of VFA, which are typically abundant in anaerobic systems. To avoid this phenomenon, an alternative is to titrate down to pH 5.75 (Hill and Jenkins, 1989). The value obtained is a better indicator of the real alkalinity that relies on  $\text{HCO}_3^-$  species.

Based on the methods above, Ripley *et al.* (1986) proposed a two step titration: a first one down to pH 5.75, which is due to  $\text{HCO}_3^-$  species and is known as partial alkalinity (PA); and a second one down to pH 4.3, which corresponds to the total alkalinity (TA). The intermediate alkalinity (IA), which is related to VFA concentration, is then estimated as the difference between TA and PA. It can be used as an indirect measurement of VFA concentration. The alkalinity ratio (AR), defined as the ratio between intermediate and total alkalinity (IA/TA), or between intermediate and partial alkalinity (IA/PA); may be a useful indicator of the concentration of VFA in the sample.

TA and PA were analysed from 10 g (~10 mL) samples of filtered supernatant. The titrant used was hydrochloric acid (HCl) of known concentration. pH was measured in continuous mode during titration, until the values of pH 5.75 and 4.3 were reached. Total, partial and intermediate alkalinities are calculated as follows:

$$TA = \frac{V_{4.3} N_{HCl}}{V_{sample}} 50 \quad (\text{g CaCO}_3 \text{ L}^{-1}) \quad \text{Eq. 3.11.}$$

$$PA = \frac{V_{5.75} N_{HCl}}{V_{sample}} 50 \quad (\text{g CaCO}_3 \text{ L}^{-1}) \quad \text{Eq. 3.12.}$$

$$IA = TA - PA \quad \text{Eq. 3.13.}$$

where:  $V_{4.3}$  = Volume of HCl used for titration down to pH 4.3 (L)  
 $V_{5.75}$  = Volume of HCl used for titration down to pH 5.75 (L)  
 $V_{sample}$  = Volume of filtrate supernatant (L)  
 $N_{HCl}$  = Concentration of HCl (eq L<sup>-1</sup>)  
 50 = transformation factor to convert eq CaCO<sub>3</sub> L<sup>-1</sup> into g CaCO<sub>3</sub> L<sup>-1</sup>

### 3.6.5. Volatile fatty acids

Volatile fatty acids, or short chain fatty acids, are intermediate products of anaerobic biodegradation of complex organic compounds into CH<sub>4</sub> and CO<sub>2</sub>. Therefore, they are very useful indicators of process performance, stability or unbalance. In this study, VFA were indirectly determined by measuring the intermediate alkalinity and alkalinity ratio; and directly quantified by gas chromatography. VFA quantified were: acetic acid; propionic acid; iso-butyric acid; n-butyric acid; iso-valeric acid and n-valeric acid.

Samples of filtered supernatant were further filtered through a 0.45 µm nylon syringe filter, as previously explained. The subsequent extraction (1/1) was done by mixing 0.6 ml of filtered sample and 0.6 ml of chloroform, after previously acidifying the sample with 50 µL of concentrated sulphuric acid (H<sub>2</sub>SO<sub>4</sub> 95 %). The mixture was hand stirred and left for a few minutes to let the two phases separate again.

For VFA analysis, the chromatograph (Perkin-Elmer AutoSystem XL Gas Chromatograph) was equipped with a capillary column (HP Innowax 30 m × 0.25 mm × 0.25 µm) and a flame ionisation detector (FID). Helium (He) was used as carrier gas, with a split ratio of 13 (column flow: 5 mL min<sup>-1</sup>). The oven was kept at an initial temperature of 120 °C for 1 min, it was subsequently increased at a constant ratio of 10 °C min<sup>-1</sup> to 245 °C and maintained for 2 min. The temperatures of the injector and detector were 250 °C and 300 °C, respectively. The system was calibrated with dilutions of commercial (Scharlau, Spain) VFA (acetic, propionic, iso-butyric, n-butyric, iso-valeric and n-valeric acids) with concentrations in the range of 0-1000 mg L<sup>-1</sup>. Detection limit of VFA analysis was 5 mg L<sup>-1</sup>. The total time of each run was 15 minutes.

### 3.6.6. Biogas composition

Biogas composition is a key parameter to evaluate process performance and efficiency. Firstly, because methane content in biogas allows calculating methane production and yield through anaerobic biodegradation of an organic substrate under the studied conditions; secondly, because it may be indicative of process unbalance. For instance, inhibition of methanogenic microorganisms would result in lower CH<sub>4</sub> and higher CO<sub>2</sub> content in biogas.

Biogas composition was determined by gas chromatography, based on Standard Methods procedure 2720C (APHA, 1999). The chromatograph (Perkin-Elmer AutoSystem XL Gas Chromatograph) was equipped with a thermal conductivity detector (TCD) and a packed column (Hayesep 3 m 1/8 in. 100/120) into which biogas samples from the headspace of the reactors were injected. The carrier gas was He in splitless mode (column flow: 19 mL min<sup>-1</sup>). The oven was maintained at a constant temperature of 40 °C. Injector and detector temperatures were 150 °C and 250 °C, respectively. The system was calibrated with pure samples of methane (99.9 % CH<sub>4</sub>) and carbon dioxide (99.9 % CO<sub>2</sub>). Retention time was 1.5 and 3 minutes for CH<sub>4</sub> and CO<sub>2</sub>, respectively. The total time of each run was 10 minutes.

### 3.6.7. Microbiological analyses (*E. coli* and *Salmonella* spp.)

Microbiological analyses were ordered to an official accredited laboratory (Laboratory of Food Analyses Dr. Ferrer Rovira, in Esplugues de Llobregat, Barcelona). Samples of influent and effluent sludge were only analysed for characterisation, but not as routine analyses. *Escherichia coli* were quantified by the methodology ISO 16649:2000 and the results were expressed as colony forming units per mL (CFU mL<sup>-1</sup>). In the case of *Salmonella* spp., only presence or absence was

determined by the methodology NF-V08-052 and the results were presence / absence per 50 mL of sample.

### **3.6.8. Dewaterability tests**

Sludge dewaterability was determined using the Capillary Suction Time (CST) test. As described in the Standard Methods procedure 2710G (APHA, 1999), the CST test determines the rate of water release from sludge, and provides a quantitative measure (in seconds) of how readily a sludge releases its water. The results can be affected by sludge temperature, sample volume and sludge solids concentration. Therefore, it is recommended to divide the sludge's CST value by its solids concentration.

The CST model used was a Triton CST filterability tester, model 200, Triton Electronics Ltd., Essex, UK. Standard filter papers (Part No. 815095) were supplied by Triton Electronics. Sludge temperature was measured before each test. 5 mL sludge samples were analysed in triplicate. The results were expressed as CST (s), and also as CST (s) standardised to 1 g TS kg<sup>-1</sup> and 1 g VS kg<sup>-1</sup>.

**Chapter 4. Study of single-stage anaerobic digestion  
of sewage sludge. Effect of process temperature,  
sludge retention time and organic loading rate**

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## **Chapter 4. STUDY OF SINGLE-STAGE ANAEROBIC DIGESTION OF SEWAGE SLUDGE. EFFECT OF PROCESS TEMPERATURE, SLUDGE RETENTION TIME AND ORGANIC LOADING RATE**

### **Abstract**

The anaerobic biodegradation of sewage sludge is faster in thermophilic reactors, which enables the reduction of sludge retention time (SRT) and reactor volume. Both temperature and SRT have direct influence on sludge treatment costs, with respect to capital investment, operation and maintenance of the reactor. The enhancement of sludge dewaterability may also contribute in the reduction of treatment costs. Regarding sludge final disposal, thermophilic digestion should help preventing the spread of pathogens in the environment upon land application of digestates.

The aim of this Chapter was to study the effect of process temperature, SRT and organic loading rate (OLR) on methane production, effluent stabilisation, hygienisation and dewaterability; during semi-continuous anaerobic digestion of sewage sludge in two lab-scale reactors (5 L).

The transition from a mesophilic (43 °C) to a thermophilic (50 °C) operation was carried out without disturbing the process, working at high SRT ( $\geq 30$  days) while feeding low-solids sludge (i.e. low OLR). Under such conditions, the main difference between mesophilic and thermophilic processes referred to volatile fatty acids (VFA) and effluent hygienisation. Thermophilic digestion at 50 °C and 55 °C behaved similarly; provided that other process parameters were the same. A linear correlation was found between methane production rate and OLR, as well as between effluent characteristics (volatile solids (VS) and VFA contents, and sludge dewaterability) and OLR, during thermophilic digestion at 55 °C. Methane production rate was increased (from 0.2 to 0.4-0.6  $\text{m}^3_{\text{CH}_4} \text{m}^3_{\text{reactor}} \text{d}^{-1}$ ) by decreasing the SRT from 30 to 15-10 days, while increasing the OLR from 0.5 to 2.5-3.5  $\text{kg VS m}^3_{\text{reactor}} \text{d}^{-1}$ . Although it was further improved at the lowest SRT of 6 days, with an OLR higher than 5  $\text{kg VS m}^3_{\text{reactor}} \text{d}^{-1}$ , progressive VFA accumulation and reduced methane content in biogas suggested process unbalance. The following concentrations might be useful to detect and prevent digester failure during thermophilic sludge digestion: total VFA (2.5  $\text{g L}^{-1}$ ), acetate (0.5  $\text{g L}^{-1}$ ), acetate/propionate ratio (0.5), intermediate alkalinity (1.8  $\text{g CaCO}_3 \text{L}^{-1}$ ), intermediate alkalinity/partial alkalinity ratio (0.9), intermediate alkalinity/total alkalinity ratio (0.5), methane content in biogas (55 %).

## 4.1. INTRODUCTION AND OBJECTIVES

### 4.1.1. Introduction

#### 4.1.1.1. *Insight into the effect of process parameters on anaerobic digestion performance*

In anaerobic digesters, biogas production depends on the amount of organic matter degraded by anaerobic microorganisms. This in turn is influenced by the composition of the substrate, presence and equilibrium between anaerobic consortia, and process parameters like sludge retention time (SRT), organic loading rate (OLR), temperature and reactor design, amongst others.

Sludge hydrolysis is the rate limiting stage of the overall process; it affects the total amount of solids converted into soluble compounds and ultimately to biogas. However, soluble substrates utilization rates for fermentation and methanogenesis play a key role on process stability. The concentration of intermediate products like volatile fatty acids (VFA) is a common indicator of process unbalance (Marchaim and Krause, 1993; Pind *et al.* 2002). An accumulation of VFA in the digester may result from either insufficient methanogenic population to utilize all VFA produced or insufficient retention time for this process to take place.

According to their optimal growth temperature ranges, bacteria and archaea can be classified into psychrophiles (0-20 °C), mesophiles (10-50 °C) and thermophiles (40-110 °C) (Tolner *et al.*, 1997). Since the growth rates of methanogenic archaea are lower than those of fermentative bacteria, they determine the minimum (or washout) SRT for methanogenesis. At 20, 25 and 35 °C the washout SRT are 7.8, 5.9 and 3.2 days, respectively, which turn into 40, 30 and 15 days design values by taking a safety factor of 5 for suspended growth processes (Metcalf and Eddy, 2003). Because the growth rates of thermophilic methanogens are 2-3 times higher than those of mesophilic homologues (Van Lier *et al.*, 1993; Mladenovska and Ahring, 2000), the minimum and design SRT would be in the range of 1-2 and 5-8 days, respectively.

Process temperature not only affects the reaction rate and required SRT to achieve a certain process efficiency (i.e. solids removal and methane production), but also plays a key role regarding process stability. Methanogenic archaea are especially sensitive to temperature fluctuations, even to changes around 1 °C d<sup>-1</sup> (Metcalf and Eddy, 2003). This can be particularly critical for thermophilic processes, since they are reported to be less stable than mesophilic ones (Buhr and Andrews, 1977). For this reason, a number of studies have focused on the effect of

temperature fluctuations on thermophilic anaerobic digestion (Van Lier *et al.*, 1993; Ahring *et al.*, 2001a; Bouallagui *et al.*, 2004; El-Mashad *et al.*, 2004; Kim *et al.*, 2006).

Both temperature and SRT have direct influence on treatment costs, with respect to initial capital investment (i.e. digester volume depends on the SRT), as well as operation and maintenance costs (i.e. digester heating). Hence, interest has also been put on studying the effect of the SRT on process performance (Lin *et al.*, 1986; Zhang and Noike, 1994; Miron *et al.*, 2000; De La Rubia *et al.*, 2006; Ponsá *et al.*, 2008). From an economical point of view, it would be most interesting to operate at a minimum SRT allowing optimising methane production and solids removal, whilst assuring process stability.

Considering the whole sludge treatment line in WWTP, sludge stabilisation in anaerobic digesters is followed by sludge conditioning and dewatering steps. Since solids dewatering accounts for 7 % of energy requirements in WWTP (Metcalf and Eddy, 2003), the reduction of dewatering costs by enhancing sludge dewaterability is of major importance. However, from the literature it is not clear whether the anaerobic process improves or degrades sludge dewaterability; and whether mesophilic or thermophilic effluents are easier to dewater is not clear either (Houghton *et al.*, 2000; Houghton and Stephenson, 2002; Neyens and Baeyens, 2002; Novak *et al.*, 2003).

#### 4.1.1.2. *Thermophilic anaerobic digestion*

Traditionally, mesophilic digesters working at 35-40 °C with SRT of over 20 days have been used for the treatment of sewage sludge in large WWTP. Mesophilic digestion brings an intermediate solution combining process efficiency and energy consumption, in between psychrophilic (< 25 °C) and thermophilic systems (50-55 °C). Nevertheless, the thermophilic process is the most efficient in terms of organic matter removal and methane production (Buhr and Andrews, 1977; Záborská *et al.*, 2000a; Ahring *et al.*, 2001b). The reason for this is that the growth rates of thermophilic methanogens are higher than those of mesophilic methanogens; whereas biomass yield is much lower (El-Mashad *et al.*, 2004). As a result, by accelerating the overall reaction rate it is possible to reduce the SRT and consequently the digester volume; whilst yielding fewer amounts of biosolids to be disposed of.

According to the 3<sup>rd</sup> Draft EU Working Document on Sludge (Environment DG, EU, 2000), thermophilic digestion should enable effluent hygienisation for its use on land, which is strongly recommended whenever it is possible in order to recycle the nutrients and organic matter

contained in the sludge, improving soil fertility and minimising the amount of waste going to incineration or landfill. Consequently, there has been a growing interest upon this technology.

#### 4.1.1.3. Start-up of thermophilic anaerobic digestion

The start-up of an anaerobic digester is a slow and critical stage affecting subsequent process operation (Soto *et al.*, 1993). Factors affecting its duration include: the source of inoculum used to seed the digester; the composition of the substrate initially fed to promote bacterial growth; the OLR and SRT; the digester design and configuration; and the start-up strategy; amongst others.

The easiest way to start-up a thermophilic anaerobic reactor is by seeding it with digested effluent from an existing thermophilic facility, since excess anaerobic sludge represents an almost ideal seed material for starting up new installations (Lettinga, 1993). This is a major drawback in regions or countries where still no such facilities exist.

Thermophilic digesters may then be seeded with primary and waste activated sludge (Bolzonella *et al.*, 2003a), where anaerobic microorganisms are always present to some extent; or preferably with mesophilic digested sludge (De la Rubia, 2003; Záborská *et al.* 2000a; Kim *et al.*, 2002; Bousková *et al.*, 2005), in which some 10 % of thermophiles are already present (Chen, 1983). The latter implies that following digester seeding there is a transition period in which mesophilic microorganisms are to be replaced by thermophilic homologues (Van Lier *et al.*, 1993). The key point is how to perform such transition, whilst promoting the growth of a minor thermophilic population present in the bioreactor. Besides, during the conversion of a full-scale digester from mesophilic to thermophilic operation, side effects should not compromise treatment efficiency and quality of the effluent discharged.

Two main strategies to start-up thermophilic anaerobic digesters with mesophilic inoculum are reported in the literature. The first one consists of a straight temperature increase from 35 to 55 °C, usually accompanied by an initial drop of the OLR. In this case, the OLR is progressively increased according to process performance (Ahring *et al.*, 2001b; Bolzonella *et al.*, 2003b). It results in short transition periods of less than one month (Krugel *et al.*, 1998; Bousková, 2005; Palatsi *et al.*, 2006). However, thermal shock caused by such a sudden temperature increase is likely to reduce microbial activity and process efficiency, which is a major inconvenience for full-scale facilities. Furthermore, heating systems may not be capable of rising up sludge temperature by 20 °C in a single step.

Alternatively, digester temperature can be gradually or step-wise increased, whilst keeping a constant OLR. A long cautious period of over 20 months has been successful in the conversion of full-scale digesters without affecting process efficiency during transient conditions (Zábranská *et al.*, 2000a). In laboratory studies it has been shown that the required time for adaptation of the reactor to thermophilic temperature in step-wise increase is about twice of the time needed for one-step increase (Bousková, 2005). Some authors point out that the transition period might be shortened by avoiding temperatures between 43 and 50 °C, which neither favour mesophilic nor thermophilic microbial growth rates (De la Rubia, 2003; Palatsi *et al.*, 2006).

#### 4.1.2. Objectives

The aim of this Chapter was to study the impact of process temperature (43, 50 and 55 °C), SRT and OLR on the anaerobic digestion of sewage sludge. Process performance was monitored at decreasing SRT, while the influence of the solid content in the feed sludge, hence the OLR and its variability, were evaluated. The combined effect of all these process parameters on biogas and methane production, as well as effluent stabilisation, hygienisation and dewaterability, were assessed. The transition from a mesophilic to a thermophilic operation and the effect of temperature fluctuations on the stability of the process were also studied.

## 4.2. METHODOLOGY

The experiments were carried out in the experimental set-up described in Chapter 3 (Section 3.2). The sludge was obtained from municipal WWTP, as explained in Chapter 3 (Section 3.1). Analytical methods are detailed in Chapter 3 (Section 3.6).

#### 4.2.1. Start-up of thermophilic anaerobic sludge digestion

Since there are no full-scale thermophilic reactors in Barcelona Metropolitan Area or elsewhere in Catalonia, the lab-scale reactors were seeded with 5 L of digested sludge from a mesophilic full-scale reactor and two strategies were followed to start-up thermophilic anaerobic digestion. In Reactor R<sub>1</sub>, during a step-wise temperature increase (38-43-50-55 °C) a constant OLR was maintained; whereas in Reactor R<sub>2</sub>, a single-step temperature increase from 38 to 55 °C was followed by a gradual increase of the OLR.

### 4.2.2. Reactor R1

In reactor R<sub>1</sub> the initial conditions were similar to those of the digester from which inoculum was obtained (38 °C and 37 days SRT). Process temperature was increased to 43, 50 and 55 °C, only after stable performance was observed. In this way, the effect of process temperature (43, 50 and 55 °C) and temperature fluctuations on process efficiency was studied at 30-35 days SRT. After reaching stable operation at 55 °C, the SRT was gradually decreased down to 10 days. In addition, the solids content of feed sludge was increased, by changing from low-solids to high-solids sludge. This digester was operated for 18 months, under the conditions summarised in Table 4.1.

**Table 4.1.** Operating conditions in Reactor R<sub>1</sub>

Reactor	Period	Days (n°)	Temperature (°C)	SRT (d)	Solids content in feed sludge *
	I	1-21	38	35	low-solids
	II	22-59	43	35	low-solids
	III	60-203	50	30	low-solids
R <sub>1</sub>	IV	204-402	55	30	low-solids
	V	403-439	55	25	low-solids
	VI	440-476	55	15	high-solids
	VII	477-557	55	10	high-solids

\* low-solids: total solids < 4 %; high-solids: total solids > 4 %

### 4.2.3. Reactor R2

In Reactor R<sub>2</sub>, a single-step temperature increase from 38 to 55 °C was followed by a gradual increase of the OLR, resulting from decreasing the SRT to 30, 25, 20, 15, 12.5, 10, 8, 7 and 6 days. The OLR was also increased by changing from low-solids to high-solids sludge. Each subsequent SRT decrease was to be made once the digester had reached stable operation. This digester was operated for 21 months, under the conditions summarised in Table 4.2.

### 4.2.4. Definition of stable periods

Stable periods were defined as those in which the process showed a fairly constant performance in terms of biogas production, VFA concentration and pH in the reactor (Angelidaki and Ahring, 1994; Hansen *et al.*, 1999; El-Mashad *et al.*, 2004); without showing symptoms of process unbalance or failure (i.e. cease in biogas production, VFA accumulation or pH drop) for at least

one SRT. In order to simulate full scale operation, sludge was collected weekly from the WWTP, with the consequent variability in composition, especially regarding the solids content. In general, process parameters were strongly affected by variations in influent sludge composition. For this reason, we compared the longest stable periods obtained under each condition assayed, in order to minimise the variability of measurements.

**Table 4.2.** Operating conditions in Reactor R<sub>2</sub>

Reactor	Period	Days (n°)	Temperature (°C)	SRT (d)	Solids content in feed sludge *
R <sub>2</sub>	I	1-77	55	> 30	low-solids
	II	78-161	55	30	low-solids
	III	162-203	55	25	low-solids
	IV	204-256	55	20	low-solids
	V	257-331	55	15	low-solids
	VI	332-437	55	10	low-solids
	VII	438-464	55	15	low-solids
	VIII	465-483	55	15	high-solids
	IX	484-529	55	10	high-solids
	X	530-568	55	8-7	high-solids
	XI	569-606	55	6	high-solids
	XII	607-653	55	10	high-solids

\* low-solids: total solids < 4 %; high-solids: total solids > 4 %

## 4.3. RESULTS AND DISCUSSION

### 4.3.1. Sludge composition

Two types of sludge were used in order to assess the effect of solids concentration in the influent. The threshold for the so-called high-solids digestion in CSTR was 4 % total solids (TS), corresponding to moisture contents of 96 % as suggested by Lay *et al.* (1997). According to this, the mixture of thickened primary sludge (PS) and waste activated sludge (WAS) used initially had a low-solids concentration (TS < 30 g L<sup>-1</sup>), during periods I-V (Reactor R1) and I-VII (Reactor R2); whereas the solids concentration was in general high (TS > 40 g L<sup>-1</sup>) during periods VI-VII (Reactor R1) and VIII-XII (Reactor R2), as indicated in Tables 4.1 and 4.2. The sludge composition is shown in Table 4.3 (Section 4.3.2) and Table 4.6 (Section 4.3.3) for Reactors R1 and R2, respectively.

Generally speaking, in the low-solids sludge TS and VS were in the range of 20-32 and 14-24 g L<sup>-1</sup>, respectively; and the ratio of VS to TS between 68-77 %. The pH was always below neutrality (< 7). In the high-solids sludge, TS and VS were around 40 and 30 g L<sup>-1</sup>, respectively; with 74 % VS/TS ratio. The pH was slightly higher (≥ 7). Towards the end of the experimental period, TS and VS eventually increased up to 55 and 35 g L<sup>-1</sup>, respectively; and VS/TS ratio decreased to 58 %. The pH ranged between 6.5 and 7. In general, the values are typical of sludge from conventional activated sludge WWTP entering digestion, with TS below 5 % and VS/TS around 70 % (Speece, 1988).

However, it is worthwhile mentioning that the composition of sewage sludge was never constant (see standard deviations in Tables 4.3 and 4.6). As already explained, in order to simulate full scale operation, sludge was collected weekly from the WWTP, which resulted in enormous variations; especially regarding solids concentration after storm episodes (typical from Mediterranean climate) that resulted in extremely diluted sludge, with TS concentration even lower than 20 g L<sup>-1</sup>. In general, the results obtained were strongly affected by variations in influent sludge composition, especially solids content, as will be discussed later.

### **4.3.2. Anaerobic sludge digestion at different process temperature (Reactor R1)**

#### *4.3.2.1. Process performance*

Process performance during the long term operation of Reactor R<sub>1</sub> (557 days) is illustrated in Figures 4.1 to 4.6. In such Figures, periods corresponding to the different operating conditions shown in Table 4.1 are separated by vertical lines. Mean values of operating and efficiency parameters during stable periods under each condition assayed are summarised in Table 4.3.

#### *Periods I and II: mesophilic anaerobic digestion at 38 °C and 43 °C, feeding low-solids sludge*

Initially, the conditions in Reactor R1 were similar to those of the full-scale digester used as inoculum source (38 °C and 37 days SRT). At day 22 process temperature was increased to 43 °C without causing major process disturbance, although the pH rose from 7.5-8 to 8-8.4, and remained like this from that moment onwards (Figure 4.6). Similarly, during a step-wise temperature increase from 38 to 45, 50 and 55 °C, other authors have not detected any disturbance after the temperature increment from 38 to 45 °C, while they did notice a severe drop in methane production rate after the other temperature increments (Van Lier *et al.*, 1993).

**Table 4.3.** Average feed and digested sludge characteristics and operational parameters during semi-continuous anaerobic digestion of low- and high-solids sludge in Reactor R1

Parameter	Period						
	I	II	III (a)	III (b)	IV	VI	VII
<b>Working conditions</b>							
Temperature (°C)	38.25 ± 1.87	43.25 ± 0.32	49.38 ± 4.73	50.87 ± 1.48	55.38 ± 0.37	54.84 ± 0.47	53.09 ± 0.63
SRT (d)	37.06 ± 1.12	35.54 ± 1.20	30.29 ± 2.93	32.08 ± 4.52	30.87 ± 1.83	15.04 ± 1.40	9.97 ± 0.58
OLR (kg VS m <sup>-3</sup> reactor d <sup>-1</sup> )	0.47 ± 0.01	0.44 ± 0.02	0.48 ± 0.06	0.75 ± 0.21	0.64 ± 0.17	2.06 ± 0.19	3.03 ± 0.33
<b>Feed composition</b>							
TS (g L <sup>-1</sup> )	22.75	20.78 ± 0.79	21.60 ± 3.12	32.54 ± 9.74	27.23 ± 7.16	41.41 ± 1.63	39.19 ± 6.43
VS (g L <sup>-1</sup> )	17.44	15.31 ± 0.53	14.42 ± 1.99	24.38 ± 7.57	20.58 ± 4.92	30.78 ± 0.72	30.39 ± 2.08
VS/TS	76.66	70.70 ± 0.72	68.65 ± 3.76	72.29 ± 5.40	77.03 ± 4.27	74.72 ± 1.78	73.57 ± 1.31
Total VFA (g L <sup>-1</sup> )	0.00	0.59 ± 0.05	1.39 ± 0.47	2.57 ± 0.63	2.23 ± 0.54	2.01 ± 0.22	2.21 ± 0.37
pH	6.65 ± 0.09	6.59 ± 0.03	6.67 ± 0.60	5.83 ± 0.22	6.12 ± 0.21	7.24 ± 0.34	6.95 ± 0.19
<b>Effluent composition</b>							
TS (g L <sup>-1</sup> )	15.06 ± 0.48	12.14 ± 0.58	14.44 ± 1.66	16.03 ± 1.63	20.28 ± 2.07	20.98 ± 2.41	29.45 ± 1.53
VS (g L <sup>-1</sup> )	9.16 ± 0.39	7.49 ± 0.29	9.00 ± 1.04	10.48 ± 1.03	13.46 ± 1.35	14.03 ± 1.13	19.65 ± 1.09
VS/TS	60.82 ± 1.28	61.91 ± 0.67	62.35 ± 0.81	65.35 ± 1.43	66.17 ± 2.81	66.51 ± 4.02	66.72 ± 0.31
VFA (g L <sup>-1</sup> )	0.00	0.00	0.85 ± 0.41	1.17 ± 0.38	1.21 ± 0.29	1.79 ± 0.33	2.50 ± 0.16
Acetate (g L <sup>-1</sup> )	0.00	0.00	0.29 ± 0.19	0.22 ± 0.07	0.13 ± 0.05	0.21 ± 0.06	0.42 ± 0.06
Propionate (g L <sup>-1</sup> )	0.00	0.00	0.31 ± 0.14	0.54 ± 0.20	0.66 ± 0.13	0.91 ± 0.16	1.18 ± 0.06
iso-Butyrate (g L <sup>-1</sup> )	0.00	0.00	0.09 ± 0.05	0.16 ± 0.08	0.26 ± 0.03	0.32 ± 0.07	0.33 ± 0.09
n-Butyrate (g L <sup>-1</sup> )	0.00	0.00	0.02 ± 0.02	0.00	0.00	0.00	0.00
iso-Valerate (g L <sup>-1</sup> )	0.00	0.00	0.14 ± 0.06	0.25 ± 0.16	0.22 ± 0.03	0.35 ± 0.05	0.56 ± 0.06
n-Valerate (g L <sup>-1</sup> )	0.00	0.00	0.00	0.00	0.00	0.00	0.00
A/P ratio	0.00	0.00	0.98 ± 0.44	0.48 ± 0.27	0.19 ± 0.06	0.09 ± 0.10	0.22 ± 0.03
IA (g CaCO <sub>3</sub> L <sup>-1</sup> )	0.16 ± 0.00	0.42 ± 0.14	1.07 ± 0.18	1.23 ± 0.15	1.29 ± 0.12	1.06 ± 0.15	1.49 ± 0.15
IA/TA ratio	0.35 ± 0.04	0.24 ± 0.03	0.39 ± 0.08	0.35 ± 0.03	0.34 ± 0.03	0.32 ± 0.02	0.38 ± 0.01
IA/PA ratio	0.55 ± 0.11	0.32 ± 0.05	0.68 ± 0.25	0.54 ± 0.07	0.34 ± 0.03	0.46 ± 0.05	0.62 ± 0.03
pH	7.68	8.19 ± 0.15	8.13 ± 0.15	8.20 ± 0.09	8.27 ± 0.12	8.21 ± 0.14	8.18 ± 0.06
<b>Removal efficiency</b>							
TS removal (%)	34.21 ± 8.76	46.59 ± 0.00	27.00 ± 14.00	48.57 ± 18.66	29.32 ± 12.96	49.39 ± 5.05	22.30 ± 18.51
VS removal (%)	35.67 ± 6.79	54.28 ± 2.09	34.66 ± 7.78	55.41 ± 2.44	35.68 ± 4.46	49.38 ± 2.95	34.08 ± 4.06
<b>Biogas characteristics</b>							
Biogas prod. rate (m <sup>3</sup> m <sup>-3</sup> reactor d <sup>-1</sup> )	0.23	0.17 ± 0.02	0.13 ± 0.06	0.31 ± 0.09	0.27 ± 0.07	0.64 ± 0.08	0.62 ± 0.06
Specific biogas prod. (m <sup>3</sup> kgVS <sub>fed</sub> <sup>-1</sup> )	0.45	0.36 ± 0.04	0.27 ± 0.11	0.42 ± 0.16	0.40 ± 0.10	0.31 ± 0.03	0.21 ± 0.03
Biogas yield (m <sup>3</sup> kgVS <sub>removed</sub> <sup>-1</sup> )	1.46	0.50 ± 0.12	0.62 ± 0.34	0.60 ± 0.30	0.95 ± 0.45	0.73 ± 0.20	0.62 ± 0.03
Methane prod. rate (m <sup>3</sup> m <sup>-3</sup> reactor d <sup>-1</sup> )	0.13	0.12 ± 0.01	0.08 ± 0.05	0.18 ± 0.08	0.16 ± 0.07	0.40 ± 0.03	0.40 ± 0.05
Specific methane prod. (m <sup>3</sup> kgVS <sub>fed</sub> <sup>-1</sup> )	0.27	0.30 ± 0.00	0.16 ± 0.10	0.26 ± 0.15	0.24 ± 0.10	0.20 ± 0.02	0.13 ± 0.02
Methane yield (m <sup>3</sup> kgVS <sub>removed</sub> <sup>-1</sup> )	0.88	0.29 ± 0.00	0.42 ± 0.22	0.39 ± 0.20	0.66 ± 0.27	0.46 ± 0.12	0.40 ± 0.03
Methane content (%)	61.33 ± 1.13	68.56 ± 11.39	61.95 ± 5.37	65.03 ± 1.75	64.15 ± 2.81	61.90 ± 1.39	64.52 ± 3.10
<b>Stability period</b>							
Time (d)	1-22	44-59	78-130	145-203	319-369	442-465	522-553

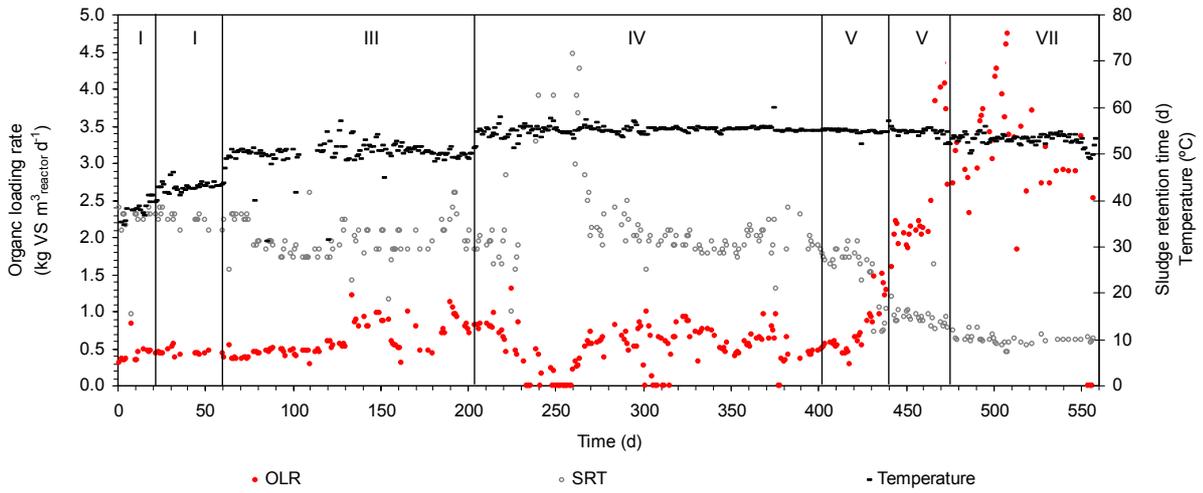


Figure 4.1. Organic loading rate (OLR), sludge retention time (SRT) and temperature in Reactor R1

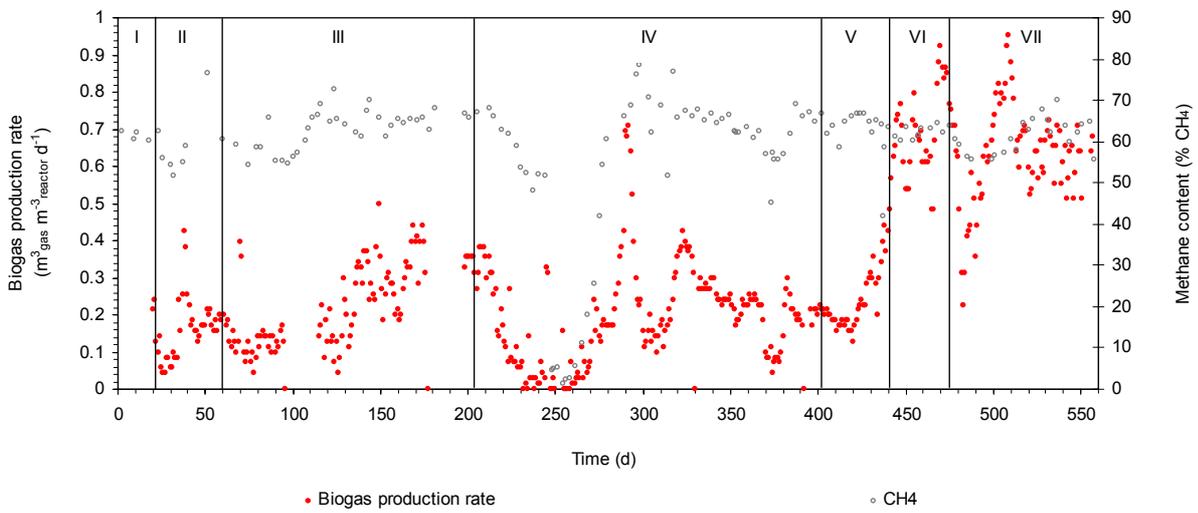


Figure 4.2. Biogas production rate and methane content in biogas in Reactor R1

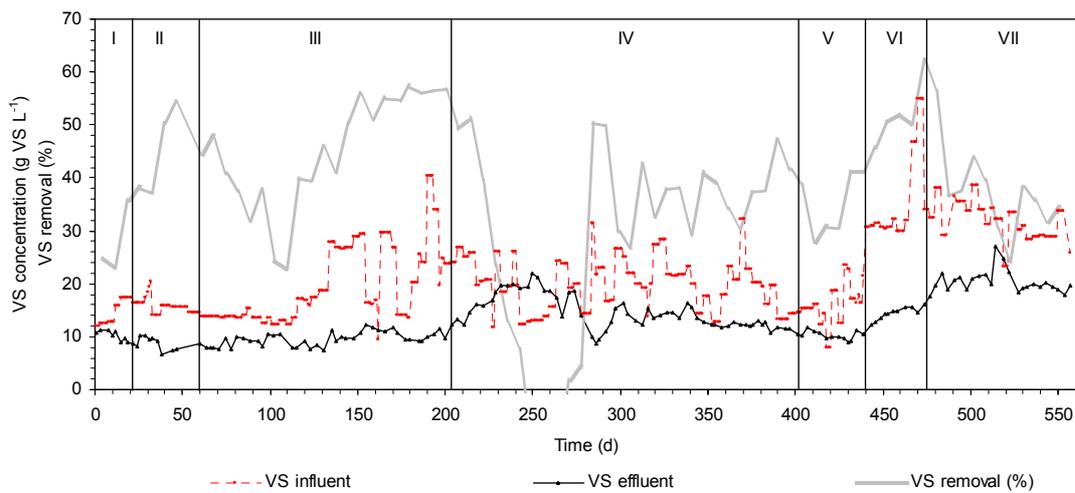
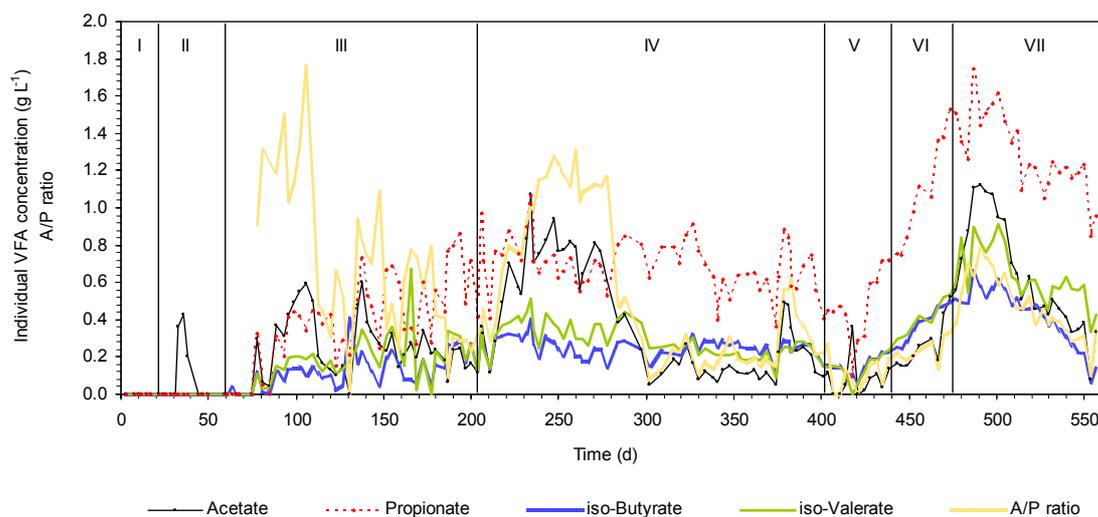
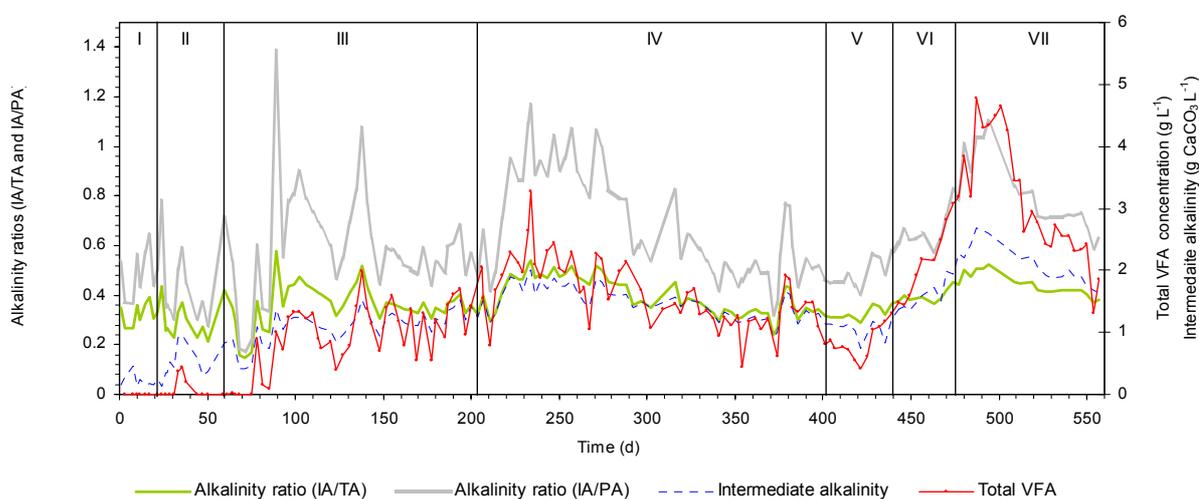


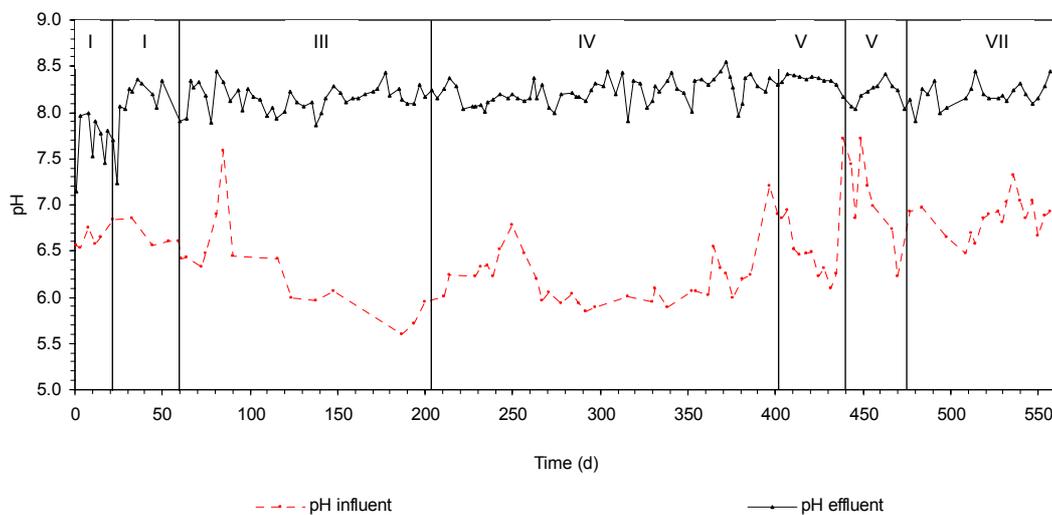
Figure 4.3. Influent and effluent sludge volatile solids (VS) concentration and VS removal in Reactor R1



**Figure 4.4.** Individual volatile fatty acids (VFA) and acetate to propionate ratio (A/P ratio) in the effluent of Reactor R1

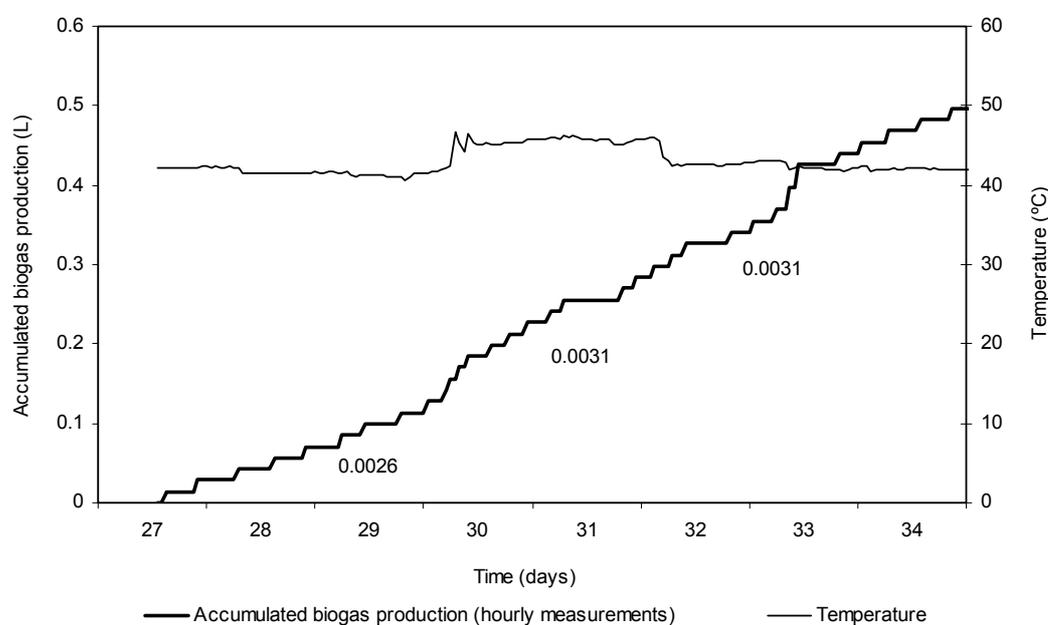


**Figure 4.5.** Intermediate alkalinity (IA); alkalinity ratios between IA and total alkalinity (IA/TA) and IA and partial alkalinity (IA/PA); and total volatile fatty acids (VFA) in the effluent of Reactor R1



**Figure 4.6.** Influent and effluent pH in Reactor R1

During these periods, influent sludge was quite diluted ( $TS < 23 \text{ g L}^{-1}$ ), which resulted in OLR as low as  $0.4\text{-}0.5 \text{ kg VS m}^{-3} \text{ reactor d}^{-1}$  and biogas production rates below  $0.3 \text{ m}^3 \text{ biogas m}^{-3} \text{ reactor d}^{-1}$  (Figures 4.1 and 4.2, periods I and II). VFA were generally below detection limits, except for a period following days 29-31 in which process temperature increased to  $46 \text{ }^\circ\text{C}$ , resulting in immediate VFA build-up (Figures 4.4 and 4.5, period II). The only VFA detected was acetic acid ( $< 0.5 \text{ g L}^{-1}$ ) over a period of one week (until day 38). However, the process did not seem to be affected by such temperature fluctuation in terms of biogas production, since biogas production rate remained fairly constant, as seen from the slope of the curve of accumulated biogas production before ( $0.0026$ ), during ( $0.0031$ ) and after ( $0.0031$ ) the temperature raise to  $46 \text{ }^\circ\text{C}$  (Figure 4.7). This suggests that VFA accumulation was not caused by a decrease in the methanogenic activity, but by an increase in the acetogenic activity.



**Figure 4.7.** Accumulated biogas production from hourly measurements during mesophilic digestion at  $43 \text{ }^\circ\text{C}$ . The values correspond to mean hourly biogas production rate (i.e. slope of the curve) before, during and after the temperature raise to  $46 \text{ }^\circ\text{C}$

#### *Period III: thermophilic anaerobic digestion at $50 \text{ }^\circ\text{C}$ , feeding low-solids sludge*

At day 60, process temperature was raised to  $50 \text{ }^\circ\text{C}$  and the system seemed to adapt well since no immediate effect was detected. Biogas production was maintained, although still at very low production rates ( $< 0.2 \text{ m}^3 \text{ biogas m}^{-3} \text{ reactor d}^{-1}$ ), methane content in biogas remained around 60 % and VFA under detection limits. However, at day 78 (after approximately half a SRT), a sudden peak VFA concentration of  $0.9 \text{ g L}^{-1}$  was detected. Although it disappeared within one week,

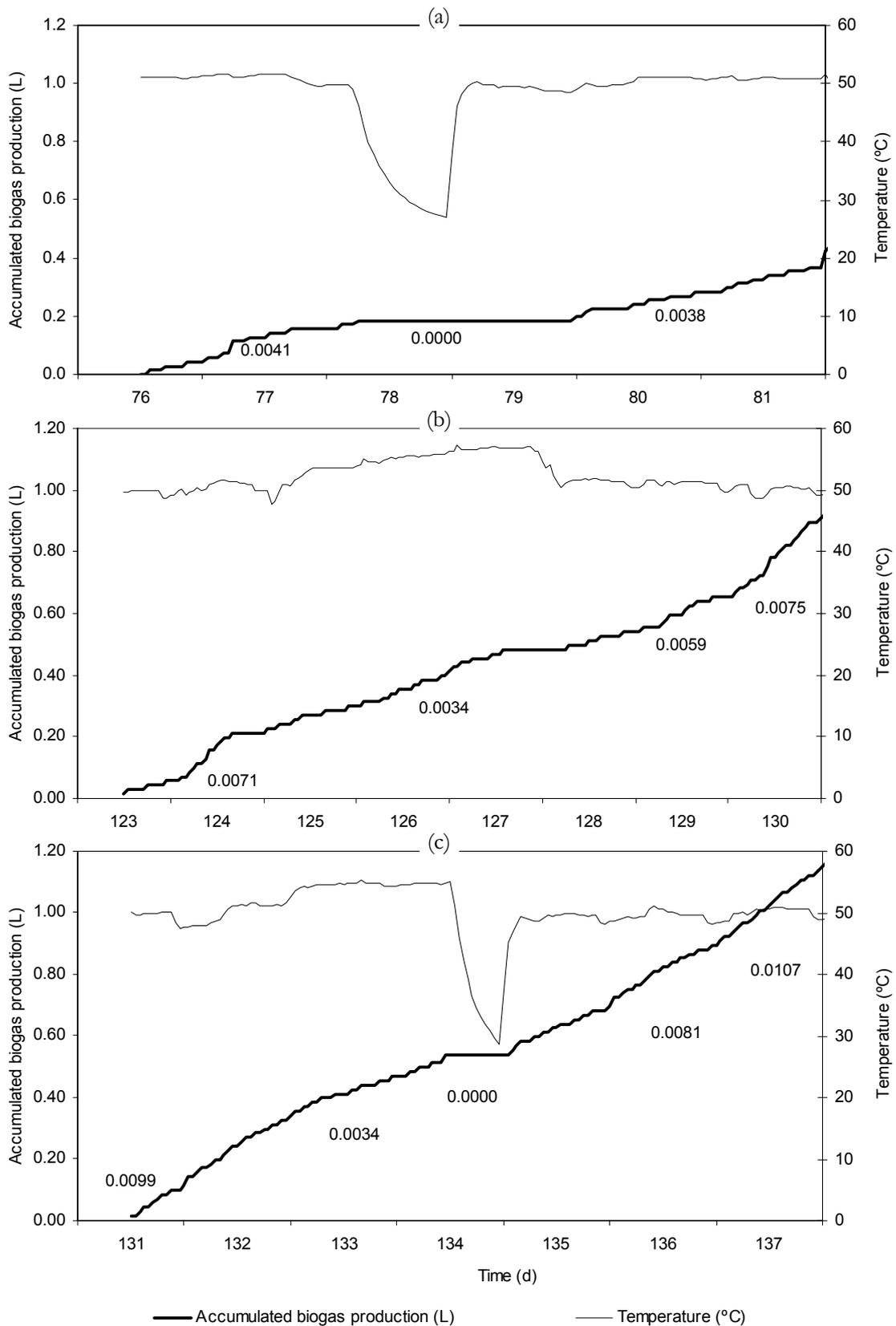
subsequent VFA build-up after day 89 resulted in persistent VFA in the effluent of the thermophilic digester in concentrations of at least  $0.38 \text{ g L}^{-1}$  (Figures 4.4 and 4.5, all periods).

During this period temperature fluctuations occurred frequently, due to operational problems with the thermostatic bath. Process temperature drop below  $47 \text{ }^{\circ}\text{C}$  on days 71-72, 77-78, 84, 88, 100, 119-120, 134 and 151; whereas it increased above  $53 \text{ }^{\circ}\text{C}$  on days 114-115, 118-120, 125-127, 132-133, 140-141, 146-148, 159-160, as can be seen in Figure 4.1 (period III). From the study of hourly biogas production rates it seemed that such temperature fluctuations did not have a severe persistent effect on the system. Apparently, biogas production ceased during low temperature intervals, but started as soon as process temperature was recovered, as deduced from Figure 4.8 (a). Similar patterns to that shown in Figure 4.8 (a) were observed for all temperature drop episodes. On the other hand, when process temperature raised above  $53 \text{ }^{\circ}\text{C}$ , it seemed that biogas production rate decreased only slightly, eventually ceasing if temperature raised above  $56 \text{ }^{\circ}\text{C}$  (Figure 4.8 (b)). Again, similar patterns to that shown in Figure 4.8 (b) were observed for all temperature raise episodes. Figure 4.8 (c) corresponds to a combination of both.

With regards to VFA, it might be speculated that the above mentioned temperature fluctuations, with subsequent cease in biogas production, would result in a certain accumulation of VFA. The profile of VFA concentration during days 78 to 120 might be explained like this, with peak concentrations on days 78, 89, 100 and 120; in which temperature drop episodes occurred (Figures 4.4 and 4.5, period III).

However, the latter also corresponds to peak concentrations of influent VS (Figure 4.3, period III) and, consequently, peak OLR (Figure 4.1, period III). Subsequent VFA peaks on days 138, 155, 165, 180 or 194 have its homologues on VS and OLR. Additionally, during the whole experimental period it was observed that the higher the solids concentration, the higher the VFA in the influent sludge. For example, on day 138, high influent VS resulted in high OLR, with high influent VFA ( $> 3 \text{ g L}^{-1}$ ) and effluent VFA (nearly  $2 \text{ g L}^{-1}$ ).

If we take into account that after day 151 no temperature drops were registered, process temperature being much steadier, peaks of VFA concentration are likely to be the result of higher influent VS, with higher VFA, thus higher OLR. Therefore, it seems that VFA accumulations resulted both from sudden organic overloading or from temperature drop episodes.



**Figure 4.8.** Accumulated biogas production from hourly measurements during thermophilic digestion at 50 °C. The values correspond to mean hourly biogas production rate (i.e. slope of the curve) before, during and after temperature fluctuation episodes: (a) temperature drop < 47 °C; (b) temperature raise > 53 °C; (c) temperature raise > 53 °C followed by a temperature drop < 47 °C

For the assessment of process performance during thermophilic digestion at 50 °C two periods ought to be distinguished. Initially, the composition of influent sludge was similar to that during mesophilic operation. Process efficiency at 50 °C and still at high SRT ( $\geq 30$  days) was quite similar to that at 43 °C (Table 4.3, period III (a)), although relatively poorer regarding solids removal (27 and 35 % for TS and VS, respectively), biogas production ( $\sim 0.13 \text{ m}^3_{\text{biogas}} \text{ m}^{-3}_{\text{reactor}} \text{ d}^{-1}$ ) and methane content in biogas (62 %).

After day 136, the solids concentration in the influent was some 50 % higher and so was the OLR, because the SRT was constant. Consequently, process performance rapidly improved (Table 4.3, period III (b)). Biogas production rate was doubled ( $\sim 0.3 \text{ m}^3_{\text{biogas}} \text{ m}^{-3}_{\text{reactor}} \text{ d}^{-1}$ ), with methane contents around 65 %. TS and VS removals were around 48.5 and 55.5 %, respectively; similar to those obtained at 43 °C. The major difference was VFA concentration, which was consistently higher, oscillating between 0.5 and 1.7 g L<sup>-1</sup>.

*Periods IV and V: thermophilic anaerobic digestion at 55 °C, feeding low-solids sludge*

At day 204 process temperature was raised to 55 °C. For the first 10 days the system did not seem to be affected, as all process parameters remained fairly constant. The solids content in the influent sludge was still quite high ( $> 30 \text{ g L}^{-1}$ ) and some operating problems with the peristaltic pumps began to occur. For this reason, on days 215 and 247 the reactor was opened, which obviously had a dramatic effect on process performance, especially regarding methanogenesis. Biogas production progressively decreased from days 216 to 231, until it almost ceased for about 40 days. Methane content in biogas initially fell to 50 % (between days 229-243) and then sharply to 5 % on day 247 (Figure 4.2, period IV). However, measurements of biogas composition from days 248 to 260 basically correspond to air which remained in the headspace of the reactor until it was displaced by the biogas produced. For this reason, between days 248 and 260 both CH<sub>4</sub> and CO<sub>2</sub> were extremely low ( $< 30 \%$ ), but the fact that CO<sub>2</sub> was higher indicates methanogenesis inhibition. After that, CH<sub>4</sub> content increased rapidly and accordingly to biogas production up to day 278 when the process seemed fully recovered.

At the same time, VFA increased up to a maximum concentration of over 3 g L<sup>-1</sup> at day 234 and then remained above 2 g L<sup>-1</sup> until day 257 (Figure 4.5, period IV); indicating that although methanogenesis was almost non-existent, hydrolysis and acidogenesis were not inhibited.

The pH never seemed to be affected (Figure 4.6, period IV), probably due to the high alkalinity of the system, which also remained fairly constant. On the contrary, intermediate alkalinity and alkalinity ratios increased in parallel with VFA (Figure 4.5, period IV), as they are actually indirect measurements of VFA (Ripley, 1986).

After digester failure the organic loading was stopped for a few days and then gradually increased at a very conservative SRT. As the system seemed to react well to organic loading, it was further increased up to initial values on day 270 when the system started to recover.

During the following stability period at 55 °C and 30 days SRT, process efficiency in terms of biogas production and yield was quite similar to that obtained under similar working conditions but at 50 °C (Table 4.3, periods III (b) and IV). In both cases, biogas production rate was around  $0.3 \text{ m}^3_{\text{biogas}} \text{ m}^{-3}_{\text{reactor}} \text{ d}^{-1}$  and biogas yield around  $0.4 \text{ m}^3_{\text{biogas}} \text{ kg VS}_{\text{fed}}^{-1}$ , with 64-65 %  $\text{CH}_4$ . Regarding the quality of the effluent sludge, VFA concentration was similar ( $1.1 - 1.3 \text{ g L}^{-1}$ ), but solids content was higher, which is also reflected by lower TS and VS destruction ( $\sim 29.5$  and  $35.5$  %, respectively).

*Periods VI and VII: thermophilic anaerobic digestion at 55 °C, feeding high-solids sludge*

After a transition period in which the SRT was gradually reduced from 30 to 15 days, influent low-solids sludge was replaced by high-solids sludge ( $> 40 \text{ g L}^{-1}$ ) on day 440. Consequently, the OLR increased to  $2 \text{ kg VS m}^{-3}_{\text{reactor}} \text{ d}^{-1}$  (Figure 4.1, periods VI and VII) and biogas production rate reached its maximum values around  $0.64 \text{ m}^3_{\text{biogas}} \text{ m}^{-3}_{\text{reactor}} \text{ d}^{-1}$  (Table 4.3, periods VI and VII). Therefore, biogas production rate was doubled with respect to that obtained at 30 days SRT. In terms of biogas yield, it was a bit lower ( $0.3 \text{ m}^3_{\text{biogas}} \text{ kg}^{-1} \text{ VS}_{\text{fed}}$ ), since more VS were fed to the digester but VS removal remained the same; thus specific methane production was also lower ( $0.73 \text{ m}^3_{\text{biogas}} \text{ kg}^{-1} \text{ VS}_{\text{removed}}$ ). Methane content in biogas was around 62 %. VFA were consistently higher (near  $2 \text{ g L}^{-1}$ ) but never exceeding influent VFA which, as earlier mentioned, increased with increasing influent VS.

The SRT was gradually reduced (between days 467 and 477) down to the minimum 10 days SRT of this work, which was maintained until the end of the study. Initially, biogas production rate followed a decreasing trend for 10 days (until day 488), increasing thereafter to reach values in the range of those obtained at 15 days SRT. This trend was somehow parallel to the OLR (see Figures 4.1 and 4.2, period VII). However, VFA rose to its highest concentration of nearly  $5 \text{ g L}^{-1}$

(Figure 4.5, period VII), being even slightly higher than influent VFA and indicating some VFA accumulation in the system. From Figure 4.3 (period VII) it can be seen that VS concentration in the effluent was also the highest ( $> 21 \text{ g L}^{-1}$ ).

On day 491 the presence of a surface scum layer became evident, its level increasing and tending to occupy all the headspace volume. Once a scum layer is formed, unless it is broken down, it accumulates and can markedly reduce the effective volume of the digester, impeding the release of gas from the liquor (Stafford, 1982). For this reason, on day 513 the reactor was opened to remove the scum. A second propeller was placed at surface level, in order to impede the formation of a new scum layer, or break it in case it was formed.

Surprisingly, methanogens were not inhibited during this process, since neither biogas production, nor methane content in biogas decreased. In fact, a fairly stable period followed, in which biogas production rate ( $0.62 \text{ m}^3_{\text{biogas}} \text{ m}^{-3}_{\text{reactor}} \text{ d}^{-1}$ ) approached that obtained at 15 days SRT, with 64.5 %  $\text{CH}_4$  in biogas. Again, biogas yield was lower ( $0.2 \text{ m}^3_{\text{biogas}} \text{ kg VS}_{\text{fed}}^{-1}$ ); since more VS were fed to the digester ( $\text{OLR} \sim 3 \text{ kg VS m}^{-3}_{\text{reactor}} \text{ d}^{-1}$ ) but its destruction was lower ( $\sim 35 \%$ ). The quality of the effluent was poorer, with TS and VS around 30 and  $20 \text{ g L}^{-1}$ , respectively; and VFA above  $2 \text{ g L}^{-1}$  (see Table 4.3, periods VI and VII)

Concerning VS destruction, it should be noted that the results obtained during all the experimental work clearly demonstrate that the calculated value of solids destruction is most affected by solids content in the influent sludge. As shown in Figure 4.3, within each experimental period effluent VS remained fairly constant regardless of influent VS. Thus, the calculated VS removal (Equation 3.3; Section 3.3.2) varies according to influent VS concentration; meaning that solids destruction is not as reliable for stability assessment as other parameters like effluent VS or effluent VFA.

#### 4.3.2.2. *Transition from mesophilic to thermophilic operation*

The transition of a thermophilic lab-scale anaerobic reactor treating thickened mixture of primary and waste activated sludge was carried out by changing from mesophilic (38-43 °C) to thermophilic (50 °C) temperature without causing apparent process disturbance. Since in mesophilic sludge the presence of thermophiles is reported to be as low as 10 % (Chen, 1983), a certain process instability might have been expected during the transition period in which mesophilic microorganisms were to be replaced by thermophilic homologues. Because

methanogens have the lowest growth rates, a certain accumulation of VFA and decrease in methane production ought to be expected. Such effect has been described after a single-step process temperature increase from 37 to 55 °C (Bousková *et al.*, 2005; Palatsi *et al.*, 2006). Furthermore, temperatures around 47 °C have been pointed out as the most critical during step-wise temperature increase, because they are in between optimal growth rate temperatures for mesophilic and thermophilic bacteria (De la Rubia, 2003; Bousková *et al.*, 2005; Palatsi *et al.*, 2006). However, according to other authors, a successful conversion of full-scale digesters from mesophilic to thermophilic conditions without disturbing process performance could be achieved by means of slow and gradual temperature increase (Zábranská *et al.*, 2000a).

To study the transition from mesophilic to thermophilic operation, process temperature was first increased from 38 to 43 °C, and then to 50 °C. In this way, it was changed from the upper limit for the growth of mesophiles to the lower limit for the growth of thermophiles (Tolner *et al.*, 1997). The reactor had been operating at the upper mesophilic limit for 35 days (one SRT). It could then be speculated that such high mesophilic temperatures (43 °C) may have favoured the development of thermophiles to a higher extent, compared to lower mesophilic temperatures (37-38 °C). Besides, two operating parameters may have contributed to the successful transition. Firstly, a high SRT of 35 days, similar to Zábranská *et al.* (2000a); and secondly, the low-solids sludge fed to the reactor. The combination of both resulted in lower OLR ( $< 0.5 \text{ kg VS m}^{-3}_{\text{reactor}} \text{ d}^{-1}$ ) than in previous studies ( $> 1.38 \text{ kg VS m}^{-3}_{\text{reactor}} \text{ d}^{-1}$ ) by Bousková *et al.* (2005) and Palatsi *et al.* (2006).

#### 4.3.2.3. Comparison of process efficiency at 43, 50 and 55 °C

The results obtained during stable periods at 43, 50 and 55 °C show that there are little differences in process performance working at high SRT ( $\geq 30$  days) in terms of gas production, provided that the OLR is the same. Gas production rate during mesophilic operation ( $\sim 0.2 \text{ m}^3_{\text{biogas}} \text{ m}^{-3}_{\text{reactor}} \text{ d}^{-1}$  and  $0.12 \text{ m}^3_{\text{CH}_4} \text{ m}^{-3}_{\text{reactor}} \text{ d}^{-1}$ ) was in the low range of the values reported in the literature (Speece, 1988), similar to that obtained with WAS as a sole substrate (Bolzonella *et al.* 2005; Mininni *et al.*, 2006). This might be explained by the fact that storm episodes are likely to affect the solids concentration of PS to a higher extent than WAS, the resulting diluted sludge having a higher proportion of WAS in the mixture. Operational problems in the WWTP (i.e. sludge thickeners) can also affect the proportion of PS and WAS in the mixture. At 50 and 55 °C, methane production rate was  $0.16\text{-}0.18 \text{ m}^3_{\text{CH}_4} \text{ m}^{-3}_{\text{reactor}} \text{ d}^{-1}$ . De la Rubia *et al.*, (2006) obtained  $0.17\text{-}$

$0.19 \text{ m}^3_{\text{CH}_4} \text{ m}^{-3}_{\text{reactor}} \text{ d}^{-1}$  working at  $55 \text{ }^\circ\text{C}$  with SRT of 40 and 27 days, respectively. In both studies, methane production rate was increased to  $0.4 \text{ m}^3_{\text{CH}_4} \text{ m}^{-3}_{\text{reactor}} \text{ d}^{-1}$  by reducing the SRT to 15 days.

Some authors suggest that the benefits of thermophilic digestion, in terms of solids destruction and methane production, only become evident at low SRT (Gavala *et al.*, 2003). The time needed for full conversion of solids depends of microbial growth rates and these are around 2-3 times higher for thermophilic methanogens compared to mesophilic methanogens (Van Lier *et al.*, 1993; Mladenovska and Ahring, 2000). Thus, the minimum design SRT of 15 days for mesophilic digesters (Metcalf and Eddy, 2003), could be reduced to 5-8 days for thermophilic digesters. Several references to thermophilic sludge digestion at SRT ranging from 15 to 6 days are found in the literature (Buhr and Andrews, 1977; Lafitte-Trouqué and Forster, 2002; Benabdallah *et al.*, 2006; Lu *et al.*, 2007). In the present study, during thermophilic operation at  $55 \text{ }^\circ\text{C}$  the SRT was gradually reduced to 25, 15 and 10 days, resulting in stable performance even at the lowest SRT, with gas production rates like those obtained at 15 days SRT ( $0.62\text{-}0.64 \text{ m}^3_{\text{biogas}} \text{ m}^{-3}_{\text{reactor}} \text{ d}^{-1}$  and  $0.4 \text{ m}^3_{\text{CH}_4} \text{ m}^{-3}_{\text{reactor}} \text{ d}^{-1}$ ).

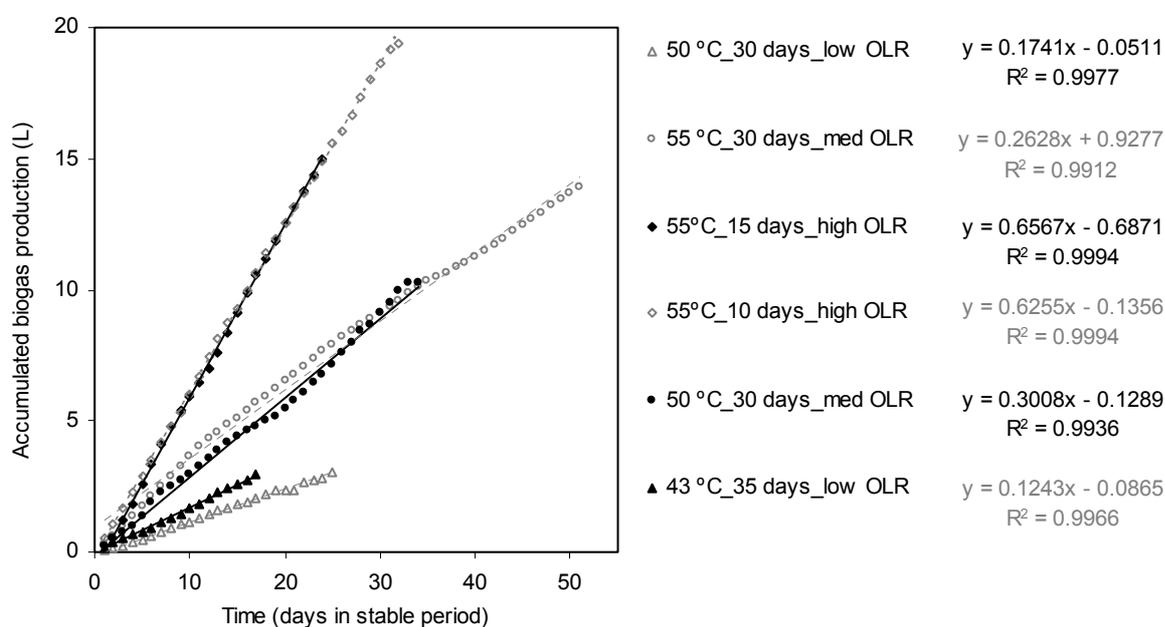
The efficiency of the process was not only increased by decreasing the SRT, but also as a result of increasing the influent sludge solids concentration (Bouallagui *et al.*, 2004). Two types of sludge were used, the limit between the so-called high-solids and low-solids sludge corresponding to a TS concentration of 4 % (Lay *et al.*, 1997). Additionally, eventual changes in the solids content resulted from typical seasonal changes. Consequently, the OLR was as low as  $0.4\text{-}0.5 \text{ kg VS m}^{-3} \text{ d}^{-1}$  during period II and the first phase of period III; around  $0.6\text{-}0.8 \text{ kg VS m}^{-3} \text{ d}^{-1}$  during the second phase of period III and period IV; and as high as  $2\text{-}3 \text{ kg VS m}^{-3} \text{ d}^{-1}$  during periods VI and VII.

Figure 4.9 shows accumulated biogas production corresponding to stable periods under each condition assayed. The slope of these curves (linear regressions) corresponds to biogas production rate (Table 4.3). The results suggest that biogas production rate was most affected by sludge solids content and OLR, which is explained as follows.

Working at high SRT ( $\geq 30$  days) and feeding low-solids sludge, when the OLR was the lowest ( $0.4\text{-}0.5 \text{ kg VS m}^{-3} \text{ d}^{-1}$ ), biogas production rate was also the lowest  $0.12\text{-}0.17 \text{ m}^3_{\text{biogas}} \text{ m}^{-3}_{\text{reactor}} \text{ d}^{-1}$  (at  $50$  and  $43 \text{ }^\circ\text{C}$ , respectively).

Still working at 50 °C, the solids concentration eventually increased, resulting in a medium OLR that enhanced biogas production ( $0.30 \text{ m}^3_{\text{biogas}} \text{ m}^{-3}_{\text{reactor}} \text{ d}^{-1}$ ). Therefore, process performance was improved by changing only the OLR. After the subsequent temperature increase to 55 °C, the OLR was still medium, although a bit lower ( $0.64$  vs.  $0.75 \text{ kg VS m}^{-3} \text{ d}^{-1}$ ), and so was biogas production rate ( $0.26$  vs.  $0.30 \text{ m}^3_{\text{biogas}} \text{ m}^{-3}_{\text{reactor}} \text{ d}^{-1}$ ).

Finally, feeding high-solids sludge, decreasing the SRT to 15 and 10 days (at 55 °C) resulted in the highest OLR of 3 and 2  $\text{kg VS m}^{-3} \text{ d}^{-1}$ , respectively; and the highest biogas production rate ( $0.62$ - $0.65 \text{ m}^3_{\text{biogas}} \text{ m}^{-3}_{\text{reactor}} \text{ d}^{-1}$ ). Therefore, biogas production rate increased with the sludge solids content and OLR, as will be thoroughly discussed in Section 4.3.3.



**Figure 4.9.** Accumulated biogas production and biogas production rates during stable periods under each operating condition assayed. Each treatment is identified in the legend by: process temperature (°C) \_ sludge retention time (d) \_ organic loading rate (low /medium/ high)

The main difference between mesophilic and thermophilic effluents referred to VFA: in general they were not detected during mesophilic operation, while they were always present during thermophilic operation (both at 50 and 55 °C); ranging from 0.5 to 2.5  $\text{g L}^{-1}$  during stability periods, and up to 4-5  $\text{g L}^{-1}$  during instability periods. In general, VFA are either not detected or found in very low concentrations ( $< 1 \text{ g L}^{-1}$ ) in mesophilic effluents (Speece, 1988; De la Rubia *et al.*, 2002; Gavala *et al.*, 2003; Bousková *et al.*, 2005). On the contrary, they are generally present in thermophilic effluents, in concentrations as high as 5  $\text{g L}^{-1}$  (De la Rubia *et al.*, 2006). Indeed, this

is a major disadvantage of thermophilic digesters, resulting in more highly loaded effluent supernatants compared to mesophilic ones.

If process efficiency at 50 and 55 °C are compared (Table 4.3), it seems that the process behaved similarly with regards to methane production ( $0.16\text{-}0.18 \text{ m}^3_{\text{CH}_4} \text{ m}^{-3}_{\text{reactor}} \text{ d}^{-1}$ ) and yield ( $0.24\text{-}0.26 \text{ m}^3_{\text{CH}_4} \text{ kg VS}^{-1}$ ), methane content in biogas (64-65 %  $\text{CH}_4$ ) and effluent VFA ( $\sim 1.2 \text{ g L}^{-1}$ ). This suggests that the process was most affected by sludge solids content (or OLR), regardless of process temperature. In fact, some authors have referred to quality and quantity of input material as the most influential factors of anaerobic digestion in full-scale digesters (Illmer and Gstraunthaler, 2008).

#### 4.3.2.4. Effluent hygienisation

Mesophilic and thermophilic effluents also differed in the concentration of pathogen indicator microorganisms (Table 4.4). Compared to the values obtained in influent sludge samples, a 3 log reduction of *E. coli* was achieved by mesophilic digestion at 43 °C, while complete destruction of such microorganisms was achieved under all thermophilic conditions assayed, even at 10 days SRT. *Salmonella* spp. was never detected.

**Table 4.4.** Microbiological analyses of influent and effluent sludge samples in R1

Pathogens	Influent (PS+WAS)	Effluent				
		43°C_37 d	50°C_30 d	55°C_30 d	55°C_15 d	55°C_10 d
<i>E.coli</i> (CFU mL <sup>-1</sup> )	$1.0 \times 10^6$	$1.7 \times 10^3$	Absence	Absence	Absence	Absence
<i>Salmonella</i> spp. (in 50 mL)	Absence	Absence	Absence	Absence	Absence	Absence

Thermophilic effluent hygienisation is widely reported in the literature (Zábranská *et al.*, 2000a; Lafitte-Trouqué and Forster, 2002; Skiadas *et al.*, 2004; Lu *et al.*, 2007), showing the potential of thermophilic treatment in preventing the spread of pathogens in the environment upon land application of digestates. After two-stage thermophilic digestion (55/52 °C), residual concentrations were  $10^4$  CFU g<sup>-1</sup> for coliforms, and  $10^3$  CFU g<sup>-1</sup> for faecal coliforms and enterococci; while these values were  $10^6$ ,  $10^5$  and  $10^4\text{-}10^5$  after two-stage mesophilic digestion (38/35 °C) (Zábranská *et al.*, 2000a). Similarly,  $10^3$  CFU mL<sup>-1</sup> of faecal streptococci remained after single-stage digestion at 55 °C, while a complete destruction was achieved after two-stage (70/55 °C) process (Lu *et al.*, 2007).

## 4.3.2.5. Sludge dewaterability

Sludge dewaterability, expressed as the Capillary Suction Time (CST) of influent and effluent sludge samples, is shown in Table 4.5. It can be observed that the results were similar for the influent and effluents at 50-55 °C and SRT of 30 days (430-440 s), and twice such values for the effluent at 55 °C and 10 days SRT (850 s). Since the value of CST measured depends on the solids concentration in the sample, the results differ expressed as CST per g TS or g VS: they were similar for all thermophilic effluents (~30 and 45 s per g TS and VS, respectively) and almost 2 fold influent values (18 and 27 s per g TS and VS, respectively). Therefore, thermophilic anaerobic digestion does not seem to enhance subsequent sludge dewatering step.

This is in accordance with previous works by Houghton *et al.* (2000) and Houghton and Stephenson (2002). These authors reported that mesophilic digested sludge was more difficult to dewater than raw sludge, and found a good correlation between changes in sludge dewaterability and changes in microbial extracellular polymer (ECP) composition occurring during anaerobic digestion of sludge. ECP, which is produced by bacteria and is found either associated with the bacterial cell wall or in suspension; is extremely hydrated in order to prevent desiccation of bacterial cells. Thus, changing the sludge structure through pre-treatment processes has been regarded as the only way of enhancing sludge dewaterability (Neyens and Baeyens, 2002).

**Table 4.5.** Sludge dewaterability measured as the Capillary Suction Time (CST)

Sludge dewaterability	Influent (PS+WAS)	Effluent		
		50°C_30 d	55°C_30 d	55°C_10 d
CST(s)	437	432	439	850
CST (s) / g TS L <sup>-1</sup>	18	30	29	29
CST (s) / g VS L <sup>-1</sup>	27	45	44	44

## 4.3.2.6. Temperature considerations regarding thermophilic operation

On the whole, it seems that similar process performance should be expected from anaerobic digesters operating at 50 °C and 55 °C, provided that all other operating parameters remain the same. Angelidaki and Ahring (1994) did not find any differences in biogas yields at temperatures in the range of 40-55 °C working at 15 days SRT, either. Some workers point out that the optimal temperature for thermophilic operation is 50 °C (Buhr and Andrews, 1977). El-Mashad *et al.* (2004) found higher methane production at 50 °C compared to 60 °C; whilst Ahring *et al.* (2001a)

found higher methane yield at 55 °C compared to 65 °C, which was attributed to reduced activity and amount of methanogens on the upper thermophilic range ( $\geq 60$  °C).

Besides, a reactor operating at 50 °C was more resistant to upward and downward temperature fluctuations of 10 °C compared to a reactor operating at 60 °C; upward temperature fluctuations having more severe effect on the specific methanogenic activity than downward temperature fluctuations (El-Mashad *et al.*, 2004). In the present work, temperature fluctuations were frequent during thermophilic operation at 50 °C. If the temperature dropped below 47 °C, biogas production immediately ceased. If the temperature increased above 53 °C, biogas production rate slightly decreased, eventually ceasing at temperatures higher than 56 °C. However, no lasting effect on the subsequent digestion was noted when the digester was returned to its original operating temperature, as also described in Buhr and Andrews (1977).

According to the results, if a similar performance of thermophilic reactors operating at 50 and 55 °C with long SRT is assumed, then a thermophilic process at 50 °C should be better and less costly in terms of energy consumption.

### 4.3.3. Thermophilic anaerobic sludge digestion at decreasing SRT (Reactor R2)

#### 4.3.3.1. Process performance

Process performance during the long term operation of Reactor R<sub>2</sub> (654 days) is illustrated in Figures 4.10 to 4.16. In such Figures, periods corresponding to the different operating conditions shown in Table 4.2 are separated by vertical lines. Mean values of operating and efficiency parameters during stable periods under each condition assayed are summarised in Table 4.6.

#### *Periods I and II: thermophilic anaerobic digestion at 30 days SRT, feeding low-solids sludge*

The process was start-up by seeding the digester with mesophilic sludge and rising process temperature from 38 to 55° C in a single-step. This resulted in an immediate VFA build-up (total VFA  $\geq 1$  g L<sup>-1</sup>) (Figures 4.13 and 4.14, period I), while methane content in biogas decreased to concentrations below 50 % for a few days (Figure 4.11, period I). The pH was not affected, being always above 8 (Figure 4.15, period I). Effluent VS concentration was fairly constant and remarkably low (10-12 g L<sup>-1</sup>), although low-solids sludge (VS < 18 g L<sup>-1</sup>), was fed during this period (Figure 4.12, period I).

**Table 4.6.** Average feed and digested sludge characteristics and operational parameters during semi-continuous anaerobic digestion of low- and high-solids sludge in Reactor R2

Parameter	Period					
	II (a)	II (b)	III	IV	V	VI
<b>Working conditions</b>						
T (°C)	55.26 ± 1.18	55.36 ± 1.26	55.40 ± 0.52	55.27 ± 0.16	54.72 ± 0.38	54.18 ± 1.68
SRT (d)	29.11 ± 1.48	30.33 ± 3.27	25.41 ± 4.44	20.43 ± 2.80	16.03 ± 1.70	10.39 ± 0.49
OLR (kg VS m <sup>-3</sup> reactor d <sup>-1</sup> )	0.47 ± 0.03	0.69 ± 0.14	0.97 ± 0.47	1.05 ± 0.23	1.38 ± 0.29	1.65 ± 0.34
<b>Feed composition</b>						
TS (g L <sup>-1</sup> )	19.63 ± 1.67	32.77 ± 8.04	31.48 ± 10.84	30.34 ± 7.38	28.86 ± 6.86	23.22 ± 5.17
VS (g L <sup>-1</sup> )	13.30 ± 0.85	22.16 ± 4.91	23.25 ± 7.70	21.34 ± 4.12	21.01 ± 5.14	17.93 ± 3.85
VS/TS	68.90 ± 4.67	68.21 ± 0.74	74.23 ± 1.79	70.59 ± 2.20	74.78 ± 1.80	77.52 ± 2.00
Total VFA (g L <sup>-1</sup> )	1.16 ± 0.19	2.85 ± 0.46	2.28 ± 0.29	1.84 ± 0.34	2.99 ± 0.50	2.43 ± 0.49
pH	6.97 ± 0.57	6.04 ± 0.11	5.75 ± 0.18	6.25 ± 0.12	5.92 ± 0.07	6.13 ± 0.29
<b>Effluent composition</b>						
TS (g L <sup>-1</sup> )	13.09 ± 1.74	17.60 ± 1.58	14.92 ± 1.15	20.11 ± 2.80	17.59 ± 0.94	18.90 ± 4.63
VS (g L <sup>-1</sup> )	7.90 ± 0.92	11.15 ± 1.18	9.55 ± 0.87	13.50 ± 0.78	11.62 ± 0.68	14.00 ± 2.31
VS/TS	61.76 ± 0.98	63.19 ± 1.68	63.94 ± 1.14	64.81 ± 1.27	66.39 ± 2.34	70.06 ± 0.86
Total VFA (g L <sup>-1</sup> )	0.60 ± 0.36	1.40 ± 0.34	1.01 ± 0.51	1.53 ± 0.29	1.56 ± 0.14	2.02 ± 0.39
Acetate (g L <sup>-1</sup> )	0.12 ± 0.17	0.31 ± 0.13	0.17 ± 0.15	0.17 ± 0.05	0.03 ± 0.04	0.22 ± 0.12
Propionate (g L <sup>-1</sup> )	0.29 ± 0.12	0.69 ± 0.13	0.51 ± 0.24	0.79 ± 0.15	0.92 ± 0.07	0.99 ± 0.10
iso-Butyrate (g L <sup>-1</sup> )	0.07 ± 0.05	0.19 ± 0.04	0.12 ± 0.09	0.24 ± 0.06	0.27 ± 0.02	0.29 ± 0.03
Butyrate (g L <sup>-1</sup> )	0.01 ± 0.02	0.00	0.00	0.00	0.34 ± 0.03	0.08 ± 0.10
iso-Valerate (g L <sup>-1</sup> )	0.12 ± 0.06	0.21 ± 0.08	0.20 ± 0.10	0.34 ± 0.07	0.34 ± 0.03	0.42 ± 0.07
Valerate (g L <sup>-1</sup> )	0.00	0.00	0.00	0.00	0.00	0.03 ± 0.04
A/P ratio	0.46 ± 0.39	0.44 ± 0.17	0.39 ± 0.33	0.21 ± 0.07	0.08 ± 0.03	0.22 ± 0.09
IA (g CaCO <sub>3</sub> L <sup>-1</sup> )	0.88 ± 0.08	1.26 ± 0.18	1.09 ± 0.23	1.32 ± 0.13	1.40 ± 0.12	1.47 ± 0.19
IA/TA ratio	0.31 ± 0.03	0.43 ± 0.04	0.37 ± 0.06	0.39 ± 0.04	0.41 ± 0.02	0.46 ± 0.03
IA/PA ratio	0.45 ± 0.07	0.75 ± 0.12	0.59 ± 0.16	0.65 ± 0.09	0.71 ± 0.07	0.86 ± 0.12
pH	8.18 ± 0.11	8.03 ± 0.09	8.15 ± 0.17	8.08 ± 0.11	7.86 ± 0.12	7.91 ± 0.09
<b>Removal efficiency</b>						
TS removal (%)	30.71 ± 10.97	39.66 ± 15.87	50.13 ± 14.22	36.10 ± 17.11	34.98 ± 17.67	27.50 ± 20.95
VS removal (%)	42.18 ± 5.95	44.06 ± 5.89	53.44 ± 2.99	40.46 ± 9.14	43.19 ± 4.97	22.70 ± 4.46
<b>Biogas characteristics</b>						
Biogas prod. rate (m <sup>3</sup> m <sup>-3</sup> reactor d <sup>-1</sup> )	0.18 ± 0.06	0.28 ± 0.07	0.35 ± 0.12	0.41 ± 0.14	0.36 ± 0.11	0.56 ± 0.14
Specific biogas prod. (m <sup>3</sup> kg VS <sub>fed</sub> <sup>-1</sup> )	0.37 ± 0.11	0.36 ± 0.07	0.42 ± 0.12	0.43 ± 0.08	0.29 ± 0.10	0.37 ± 0.10
Biogas yield (m <sup>3</sup> kg VS <sub>removed</sub> <sup>-1</sup> )	0.63 ± 0.09	0.70 ± 0.10	0.90 ± 0.43	0.99 ± 0.47	0.81 ± 0.68	1.15 ± 0.20
Methane prod. rate (m <sup>3</sup> m <sup>-3</sup> reactor d <sup>-1</sup> )	0.08 ± 0.02	0.22 ± 0.04	0.20 ± 0.04	0.30 ± 0.07	0.24 ± 0.03	0.36 ± 0.11
Specific methane prod. (m <sup>3</sup> kg VS <sub>fed</sub> <sup>-1</sup> )	0.17 ± 0.03	0.26 ± 0.03	0.28 ± 0.08	0.29 ± 0.08	0.19 ± 0.04	0.23 ± 0.06
Methane yield (m <sup>3</sup> kg VS <sub>removed</sub> <sup>-1</sup> )	0.40 ± 0.05	0.47 ± 0.05	0.61 ± 0.29	0.70 ± 0.31	0.59 ± 0.43	0.71 ± 0.13
Methane content (%)	63.64 ± 3.03	64.57 ± 4.86	65.07 ± 2.58	66.21 ± 1.20	64.02 ± 1.37	61.78 ± 1.49
<b>Stability period</b>						
Time (d)	78-115	131-161	162-203	204-250	274-304	337-366

**Table 4.6 (cont.).** Average feed and digested sludge characteristics and operational parameters during semi-continuous anaerobic digestion of low- and high-solids sludge in Reactor R2

Parameter	Period		
	IX	XI	XII
<b>Working conditions</b>			
T (°C)	53.24 ± 0.30	53.62 ± 1.10	52.28 ± 1.54
SRT (d)	9.41 ± 0.81	6.23 ± 1.30	10.12 ± 1.10
OLR (kg VS m <sup>-3</sup> reactor d <sup>-1</sup> )	3.71 ± 0.40	5.24 ± 0.52	2.40 ± 0.33
<b>Feed composition</b>			
TS (g L <sup>-1</sup> )	45.39 ± 3.52	54.61 ± 7.65	40.60 ± 10.93
VS (g L <sup>-1</sup> )	34.86 ± 2.34	31.21 ± 3.60	24.23 ± 2.70
VS/TS	75.71 ± 0.59	58.08 ± 10.29	62.02 ± 9.11
Total VFA (g L <sup>-1</sup> )	3.46 ± 0.49	1.92 ± 0.60	1.03 ± 0.14
pH	6.61 ± 0.12	6.81 ± 0.31	7.05 ± 0.25
<b>Effluent composition</b>			
TS (g L <sup>-1</sup> )	21.91 ± 2.34	37.97 ± 9.69	24.33 ± 6.40
VS (g L <sup>-1</sup> )	14.94 ± 1.72	18.49 ± 4.02	14.39 ± 2.76
VS/TS	68.08 ± 0.79	49.07 ± 2.82	60.18 ± 4.78
Total VFA (g L <sup>-1</sup> )	3.40 ± 0.47	1.65 ± 0.58	2.28 ± 0.44
Acetate (g L <sup>-1</sup> )	0.58 ± 0.18	0.18 ± 0.27	0.52 ± 0.20
Propionate (g L <sup>-1</sup> )	1.43 ± 0.07	1.03 ± 0.12	1.17 ± 0.15
iso-Butyrate (g L <sup>-1</sup> )	0.52 ± 0.03	0.10 ± 0.09	0.18 ± 0.10
Butyrate (g L <sup>-1</sup> )	0.06 ± 0.08	0.01 ± 0.03	0.01 ± 0.01
iso-Valerate (g L <sup>-1</sup> )	0.78 ± 0.10	0.33 ± 0.16	0.40 ± 0.13
Valerate (g L <sup>-1</sup> )	0.02 ± 0.03	0.00	0.00
A/P ratio	0.40 ± 0.11	0.16 ± 0.22	0.45 ± 0.19
IA (g CaCO <sub>3</sub> L <sup>-1</sup> )	2.09 ± 0.17	1.64 ± 0.30	2.18 ± 0.13
IA/TA ratio	0.44 ± 0.03	0.39 ± 0.03	0.40 ± 0.02
IA/PA ratio	0.79 ± 0.11	0.63 ± 0.07	0.66 ± 0.07
pH	8.03 ± 0.11	8.13 ± 0.04	8.18 ± 0.07
<b>Removal efficiency</b>			
TS removal (%)	50.17 ± 7.36	39.83 ± 11.08	37.21 ± 19.01
VS removal (%)	57.32 ± 4.18	40.60 ± 10.06	38.59 ± 10.63
<b>Biogas characteristics</b>			
Biogas prod. rate (m <sup>3</sup> m <sup>-3</sup> reactor d <sup>-1</sup> )	1.07 ± 0.15	1.46 ± 0.14	0.61 ± 0.14
Biogas yield (m <sup>3</sup> kg VS <sub>fed</sub> <sup>-1</sup> )	0.30 ± 0.03	0.28 ± 0.03	0.27 ± 0.04
Specific biogas prod. (m <sup>3</sup> kg VS <sub>removed</sub> <sup>-1</sup> )	0.51 ± 0.20	0.71 ± 0.21	0.59 ± 0.14
Methane prod. rate (m <sup>3</sup> m <sup>-3</sup> reactor d <sup>-1</sup> )	0.62 ± 0.13	0.86 ± 0.12	0.38 ± 0.08
Methane yield (m <sup>3</sup> kg VS <sub>fed</sub> <sup>-1</sup> )	0.18 ± 0.04	0.17 ± 0.03	0.16 ± 0.03
Specific methane prod. (m <sup>3</sup> kg VS <sub>removed</sub> <sup>-1</sup> )	0.35 ± 0.11	0.43 ± 0.11	0.38 ± 0.09
Methane content (%)	62.13 ± 3.46	64.33 ± 7.50	63.81 ± 3.75
<b>Stability period</b>			
Time (days)	493-512	569-599	631-651

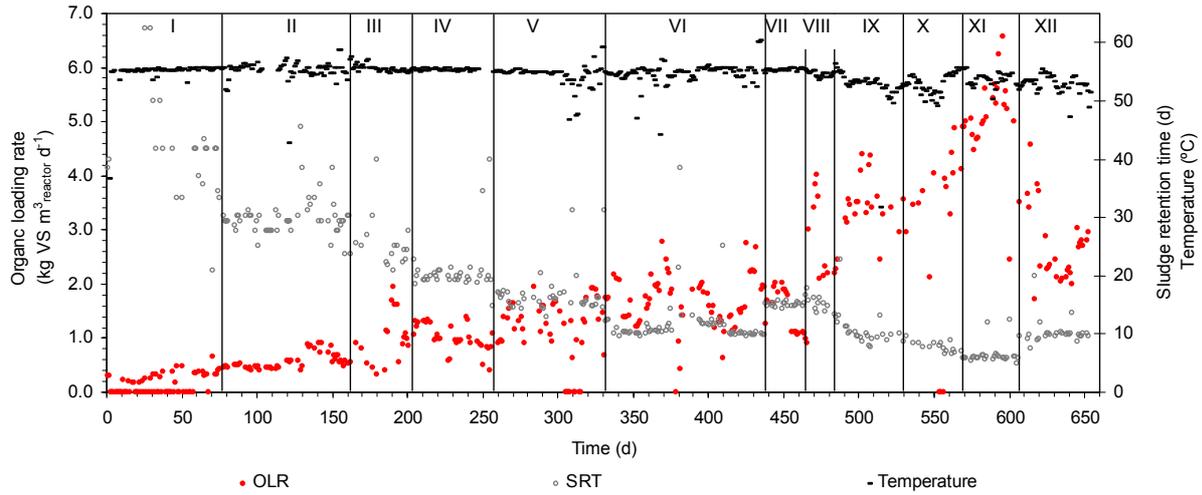


Figure 4.10. Organic loading rate (OLR), sludge retention time (SRT) and temperature in Reactor R2

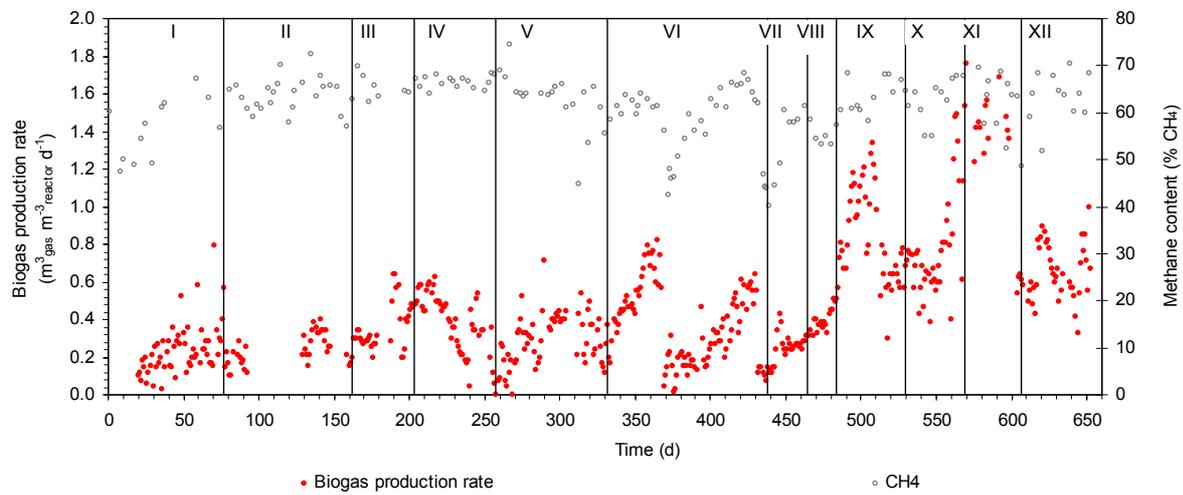


Figure 4.11. Biogas production rate and methane content in biogas in Reactor R2

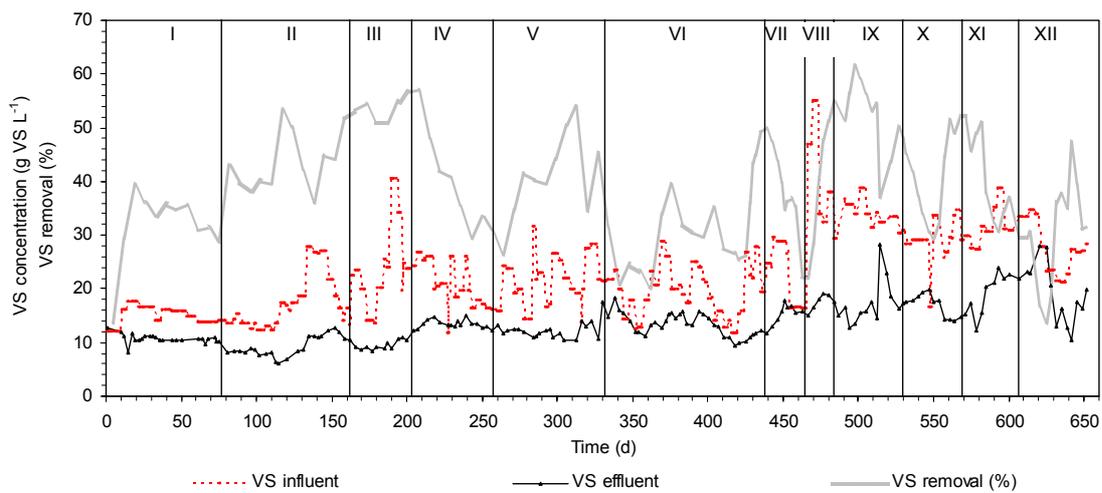
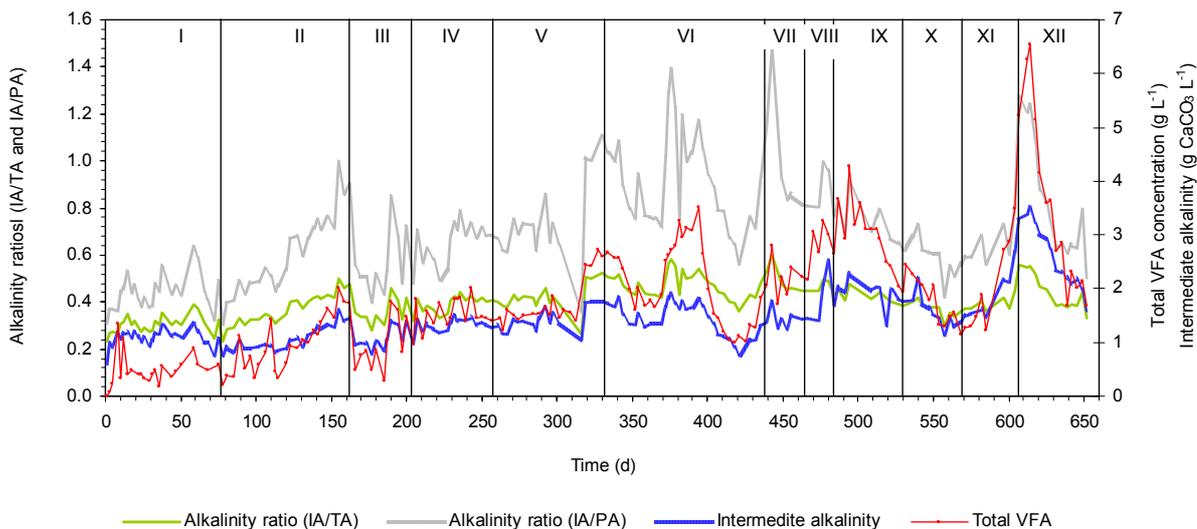
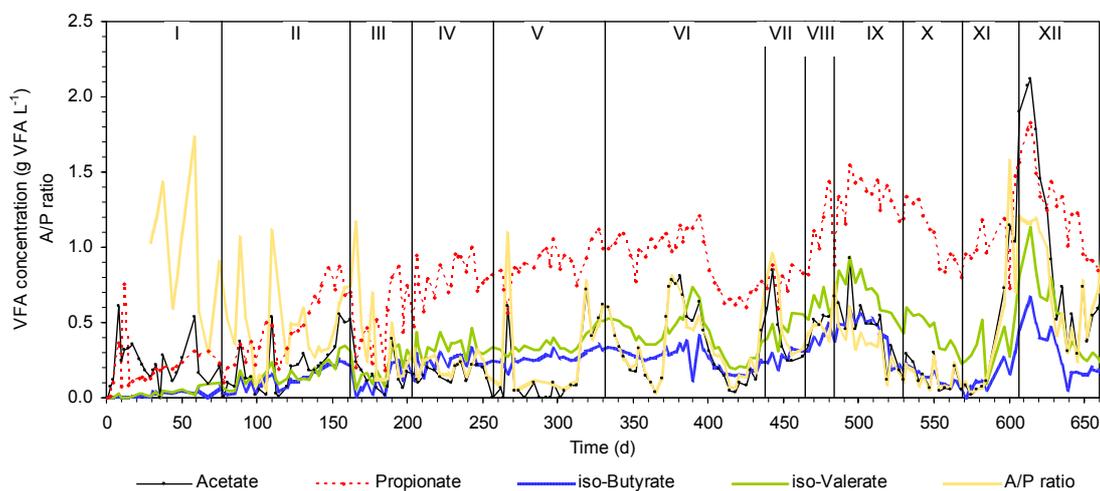


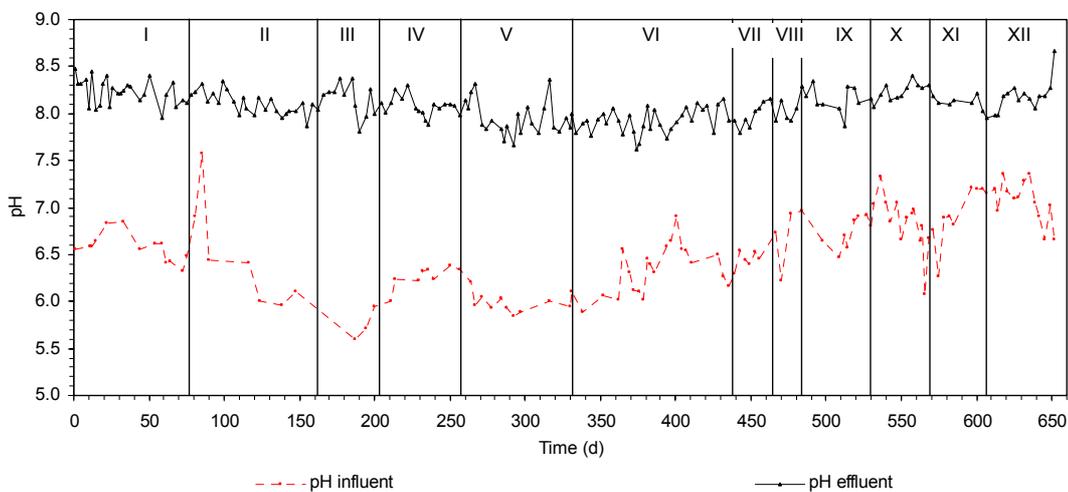
Figure 4.12. Influent and effluent sludge volatile solids (VS) concentration and VS removal in Reactor R2



**Figure 4.13.** Individual volatile fatty acids (VFA) and acetate to propionate ratio (A/P ratio) in the effluent of Reactor R2



**Figure 4.14.** In intermediate alkalinity (IA); alkalinity ratios between IA and total alkalinity (IA/TA) and IA and partial alkalinity (IA/PA); and total volatile fatty acids (VFA) in the effluent of Reactor R2



**Figure 4.15.** Influent and effluent pH in Reactor R2

The SRT was set to 30 days for 3 SRT. Initially, the OLR was as low as  $0.47 \text{ kg VS m}^{-3}_{\text{reactor}} \text{ d}^{-1}$ , but it eventually increased to  $0.69 \text{ kg VS m}^{-3}_{\text{reactor}} \text{ d}^{-1}$  due to increased solids concentration in the feed sludge ( $\text{VS} > 17 \text{ g L}^{-1}$ ) (Figure 4.12, period II). As a result, process efficiency improved, with methane production rates around  $0.22 \text{ m}^3_{\text{CH}_4} \text{ m}^{-3}_{\text{reactor}} \text{ d}^{-1}$  and 40-50 % VS destruction (Table 4.6, period II). Such values are in the range of those reported in the literature for thermophilic digestion of sewage sludge at high SRT; for example De la Rubia *et al.* (2006) obtained around  $0.19 \text{ m}^3_{\text{CH}_4} \text{ m}^{-3}_{\text{reactor}} \text{ d}^{-1}$  and 53 % VS removal working at 27 days SRT. Higher effluent VFA concentration ( $1\text{-}2 \text{ g L}^{-1}$ ) might be a consequence of the sudden increase in influent VFA, resulting from increased influent VS. In general, it was observed that the higher the influent VS, the higher the influent VFA, and usually the higher the effluent VFA (see peak concentrations of VS and VFA around days 140, 190, 130, 140, 285, 300, 390 in Figures 4.12 - 4.14).

*Periods III and IV: thermophilic anaerobic digestion at 25 and 20 days SRT, feeding low-solids sludge*

The SRT was subsequently reduced to 25 and 20 days, on days 162 and 204, respectively. During these periods, the OLR was very similar ( $\sim 1 \text{ kg VS m}^{-3}_{\text{reactor}} \text{ d}^{-1}$ ) and so was process performance (Table 4.6, periods III and IV), with biogas production rates between  $0.3$  and  $0.5 \text{ m}^3_{\text{biogas}} \text{ m}^{-3}_{\text{reactor}} \text{ d}^{-1}$  and 62-68 %  $\text{CH}_4$  in biogas. However, VS destruction was lower at 20 days SRT (40 % vs. 53 %), which is possibly related to fluctuations in influent VS concentration (Figure 4.12, periods III and IV). De la Rubia *et al.* (2002) and Gavala *et al.* (2003) obtained similar results with thermophilic digestion of PS and WAS at 20 days SRT ( $\sim 0.4 \text{ m}^3_{\text{biogas}} \text{ m}^{-3}_{\text{reactor}} \text{ d}^{-1}$ , 60-65 %  $\text{CH}_4$  in biogas,  $\sim 53$  % VS destruction).

*Periods III and IV: thermophilic anaerobic digestion at 15 and 10 days SRT, feeding low-solids sludge*

On days 257 and 332 the SRT was further decreased to 15 and 10 days, and the OLR consequently increased to  $1\text{-}1.6$  and  $1.5\text{-}2 \text{ kg VS m}^{-3}_{\text{reactor}} \text{ d}^{-1}$ , respectively. Working at 10 days SRT substantially increased biogas and methane production rates, up to  $0.56$  and  $0.36 \text{ m}^3 \text{ m}^{-3} \text{ d}^{-1}$ , respectively (Table 4.6, periods V and VI); reaching the highest values of all the experimental period feeding low-solids sludge. Interestingly, gas production rates obtained by Benabdallah *et al.* (2006) at 15 days SRT ( $\sim 0.58 \text{ m}^3_{\text{biogas}} \text{ m}^{-3}_{\text{reactor}} \text{ d}^{-1}$  and  $0.39 \text{ m}^3_{\text{CH}_4} \text{ m}^{-3}_{\text{reactor}} \text{ d}^{-1}$ ) are equivalent to those obtained at 10 days SRT, rather than 15 days SRT, in the present study.

*Periods VII, VIII and IX: transition from low- to high-solids sludge, operating at 15-10 days SRT*

From day 465 onwards, high-solids sludge with solids concentration in the range of 40-60 g TS L<sup>-1</sup> and 30-35 g VS L<sup>-1</sup> was fed (Figure 4.12, periods VIII-XII). To sustain this increase, the SRT had been set back to 15 days at day 438, and it was maintained until day 484, when it was gradually reduced to 12.5 days and then to 10 days. The OLR increased sharply to values between 3-4 kg VS m<sup>-3</sup><sub>reactor</sub> d<sup>-1</sup>. The result of doubling the solids content in the feed sludge, hence the OLR was that biogas production rate was doubled from around 0.5 to 1 m<sup>3</sup><sub>biogas</sub> m<sup>-3</sup><sub>reactor</sub> d<sup>-1</sup> feeding low- and high-solids sludge, respectively, while operating at the same SRT of 10 days (Table 4.6, periods VI and IX). Methane content in biogas remained above 60 %. The solids concentration in the effluent was fairly similar (19 vs. 22 g L<sup>-1</sup>), which is the reason why the calculated VS removal increased up to 57 %. Effluent VFA reached concentrations higher than 3 g L<sup>-1</sup> (Figure 4.14), but always within the range of influent VFA.

*Periods X, XI and XII: thermophilic anaerobic digestion at 8-6 days SRT, feeding high-solids sludge*

Next, on day 530 the SRT was gradually decreased to 8 and 7 days, and finally to 6 days at day 569. During this last period, the OLR ranged between 4.5 and 6.5 kg VS m<sup>-3</sup><sub>reactor</sub> d<sup>-1</sup>. Such a high OLR resulted from both a relatively high solids concentration in the feed sludge (~ 54 g TS L<sup>-1</sup> and 31 g VS L<sup>-1</sup>) and a relatively low SRT for a single-stage digester, which are amongst the highest OLR and lowest SRT values found in the literature (Buhr and Andrews, 1977; Speece, 1988; De la Rubia *et al.*, 2006; Benabdallah, 2006). Biogas production reached its highest rates around 1.5 m<sup>3</sup><sub>biogas</sub> m<sup>-3</sup><sub>reactor</sub> d<sup>-1</sup>, still maintaining 58-69 % CH<sub>4</sub> in biogas. VS destruction was also high (40 %), even though effluent VS were slightly higher than in previous periods (> 20 g L<sup>-1</sup>), especially after day 585 (Figure 4.12, period XI).

In fact, until day 583 the OLR was already high but always below 5 kg VS m<sup>-3</sup><sub>reactor</sub> d<sup>-1</sup>; whereas from day 583 onwards it was consistently above 5 kg VS m<sup>-3</sup><sub>reactor</sub> d<sup>-1</sup>, and even higher than 6 kg VS m<sup>-3</sup><sub>reactor</sub> d<sup>-1</sup> (Figure 4.10, period XI). Effluent VFA, which had remained below 2 g L<sup>-1</sup> increased sharply, up to its highest value of 6.5 g L<sup>-1</sup> (Figure 4.14, periods XI and XII). Methane content in biogas drop below 50 % (Figure 4.11, period XI) and alkalinity started a relentless increase (Figure 4.14, period XI). With some delay, the VS removal dropped to 13 % (Figure 4.12, periods XI and XII). Since these symptoms suggested an eventual digester failure, the SRT was set back from 6 to 10 days to avoid such a failure. Methane content in biogas rose up to 60 % within the first week and accumulated VFA were removed within 2 weeks (Figures 4.11 and 4.13-4.14, period XII). Compared to previous periods working at 10 days SRT with low- and

high-solids sludge, the OLR was now intermediate, and so were in general most process parameters (Table 4.6, period XII).

#### 4.3.3.2. Process stability

During almost two years of experimental work with reactor R2, process performance was immediately altered whenever the OLR increased, either as a result of decreasing the SRT or due to fluctuations in the solids content of the feed sludge. Additionally, alterations were detected whenever temperature fluctuations occurred, and especially when they happened together with organic overloading.

In Reactor R2, between days 290 and 513, some problems with the temperature control system caused occasional temperature drops to 45-50 °C, and some temperature rises to 56-59 °C, as can be seen in Figure 4.10. The immediate response of the system was a decrease in methane content in biogas from around 60 % to below 50 % and VFA accumulation (Figures 4.11 and 4.13-4.14) as a result of decreased methanogenic activity.

#### *Volatile fatty acids*

Although the concentration of all the VFA increased, the rise in acetate concentration was perhaps the most accentuated. Throughout the whole experimental period, acetate fluctuated within a wider range of concentrations, compared to other major VFA like propionate, iso-butyrate and iso-valerate. From Figure 4.13 it is evident that those three VFA followed parallel trends, propionate concentration always being the highest. On the other hand, acetate concentration ranged from almost 0 to nearly 1 g L<sup>-1</sup>. This clearly indicates that temperature fluctuations and organic overloading affected methanogens to a higher extent than acidogens, with subsequent accumulations of acetate in the liquor. Since changes in propionate concentrations were less pronounced, the profile of acetate concentration was very similar to that of the ratio between acetate and propionate (A/P ratio), as can be seen from Figure 4.13.

As well as individual and total VFA, some authors have proposed acetate concentration and A/P ratio as valuable indicators to predict process failure (Marchaim and Krause, 1993; Pind *et al.*, 2002). For manure, an acetic acid concentration of 0.8 g L<sup>-1</sup> and an A/P ratio of 1.4 have been proposed as limit values (Hill *et al.*, 1987; cited in Marchaim and Krause, 1993). To our knowledge, such limit values for thermophilic sewage sludge digestion have not yet been

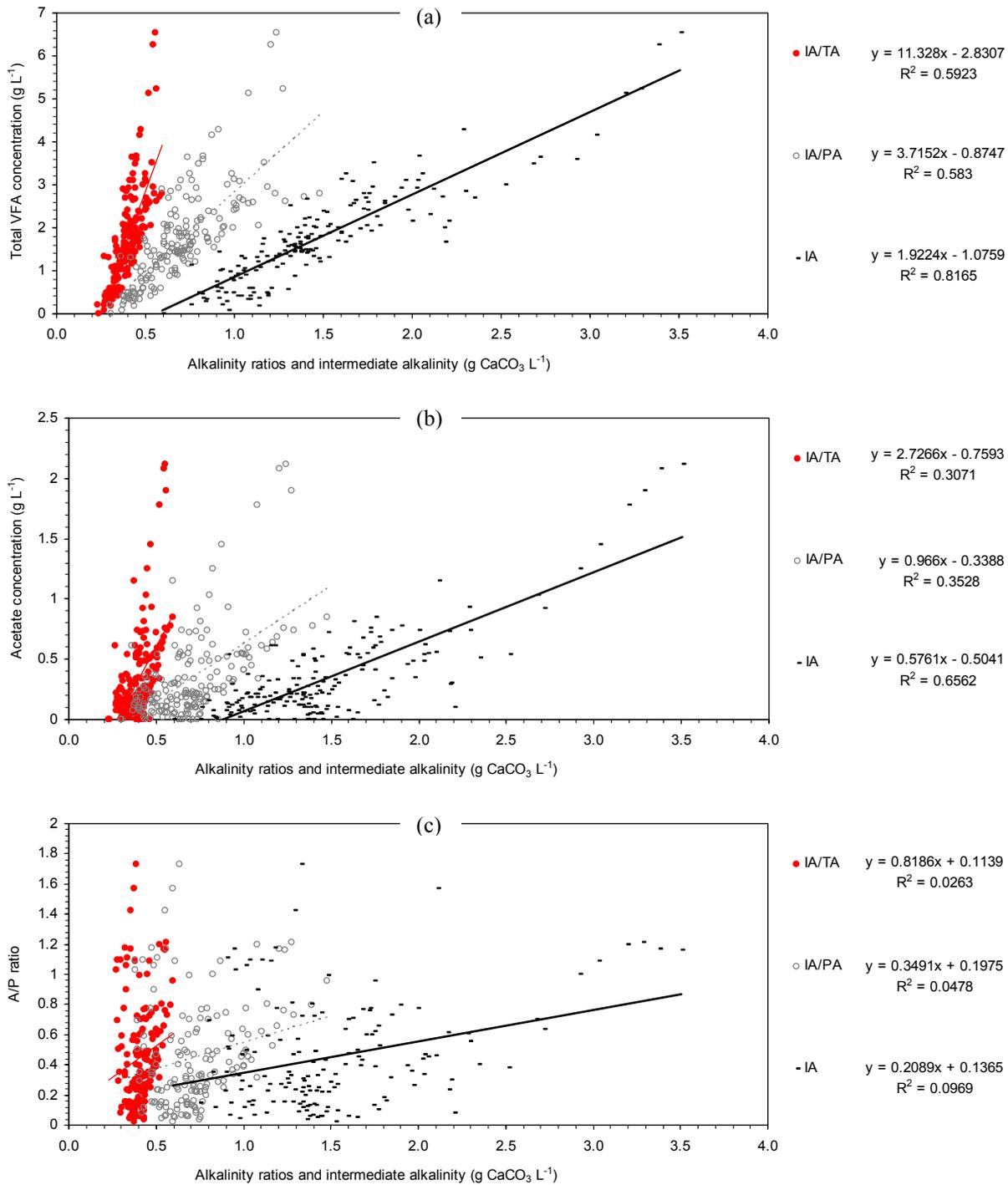
proposed. In the present study, acetate concentration was usually below  $0.5 \text{ g L}^{-1}$  (Tables 4.3 and 4.6, all periods) and only in cases of organic overloading or temperature fluctuations did this value rise above  $0.5 \text{ g L}^{-1}$  and up to  $2 \text{ g L}^{-1}$ , as seen in Figures 4.4 and 4.13, respectively. Furthermore, concentrations above  $1 \text{ g L}^{-1}$  were only reached when the SRT was reduced to 6 days, with OLR greater than  $5 \text{ kg VS m}^{-3}_{\text{reactor}} \text{ d}^{-1}$ , as shown in Figure 4.13 (period XII). Therefore, a limit concentration of  $0.5 \text{ g L}^{-1}$  of acetic acid would seem more appropriate to predict digester failure during thermophilic sewage sludge digestion. Similarly, during stability periods the A/P ratio was below 0.5 (Tables 4.3 and 4.6, all periods); hence the limit A/P ratio to predict digester failure ought to be reduced to around 0.5. In this way, both an acetate concentration higher than half the propionate concentration; or higher than  $0.5 \text{ g L}^{-1}$  might suggest process unbalance, anticipating an eventual digester failure. The total VFA concentration corresponding to such values may approximate  $2.5 \text{ g L}^{-1}$ .

### *Alkalinity*

An indirect measurement of VFA is the intermediate alkalinity (IA), and the alkalinity ratios between intermediate and total (IA/TA) or partial (IA/PA) alkalinities are alternative process indicators (Ripley *et al.*, 1986). In the present study, the profile of the IA/PA ratio was indeed very similar to that of total VFA, acetate concentration and A/P ratio in Figures 4.4-4.5 and 4.13-4.14; while variations in the IA/TA ratio were less pronounced. Since total alkalinity was fairly constant, the higher the intermediate alkalinity, the lower the partial alkalinity. Consequently, the increase in the alkalinity ratio was higher for the IA/PA ratio than for the IA/TA ratio, meaning that the IA/PA ratio was more sensible to variations in the VFA concentration.

Figure 4.16 shows total VFA concentration (a), acetate concentration (b) and A/P ratio (c); as a function of the alkalinity ratios and intermediate alkalinity. Obviously, the best correlated parameter is intermediate alkalinity, followed by IA/PA ratio and ultimately IA/TA ratio. Although all the correlation coefficients were low, the best correlations were obtained with respect to total VFA concentration ( $R^2 \leq 0.82$ ), while the correlations with acetate concentration were very poor ( $R^2 \leq 0.65$ ) and no correlations were found with the A/P ratio ( $R^2 \sim 0$ ).

If threshold values were to be set in order to predict process failure based on the measurement of alkalinity; the values corresponding to the aforementioned VFA limit concentration of  $2.5 \text{ g L}^{-1}$  would be: an IA/PA ratio around 0.9, an IA/TA ratio around 0.5 and an intermediate alkalinity around  $1.8 \text{ g CaCO}_3 \text{ L}^{-1}$ .



**Figure 4.16.** Correlation between the intermediate alkalinity (IA), IA to total alkalinity (IA/TA) ratio or IA to partial alkalinity (IA/PA) ratio and: (a) total volatile fatty acids (VFA) concentration, (b) acetate concentration and (c) acetate to propionate (A/P) ratio, during thermophilic sludge digestion

*Methane content in biogas*

With regards to the methane content in biogas, during stable periods this value always ranged between 60-70 % (Tables 4.3 and 4.6, all periods), which is typically reported in the literature for

thermophilic sludge digestion (Krugel *et al.* 1998; Záborská *et al.* 2000a; Lafitte-Trouqué and Forster, 2002; Gavala *et al.*, 2003; Bousková *et al.*, 2005; De la Rubia *et al.* 2006; Benabdallah *et al.*, 2006; Pavan *et al.* 2006; Palatsi *et al.*, 2006; Lu *et al.*, 2007). It only fell below 55 % in cases of organic overloading or temperature fluctuation, which suggests an alert concentration of 55 % for thermophilic sludge digestion. It should be noticed that such value would be within the normal range reported for other processes, for instance in digesters treating the organic fraction of municipal solid wastes methane content in biogas ranges from 50-60 % (Mata-Álvarez, 2002).

### *pH*

In terms of pH, this parameter was fairly constant and remarkably high (around 8). Even in the above mentioned episodes of digester instability, it only decreased to 7.6-7.8. Working at 6 days SRT and with the highest OLR ( $> 5 \text{ kg VS m}^{-3} \text{ reactor d}^{-1}$ ), when all other indicator parameters were above the limit values proposed, the pH was still 8. The reason for this is that the alkalinity of the system was also the highest; hence the buffer capacity of the system prevented from an eventual pH drop caused by VFA accumulation. In sewage sludge digesters, sufficient alkalinity is generally found ( $3\text{-}5 \text{ g CaCO}_3 \text{ L}^{-1}$ ) to prevent the pH from failing below the limit for methanogenesis inhibition (Metcalf and Eddy, 2003). Studies with high-solids sludge (4-10 % TS) have shown that the optimum pH range for high rate digestion is 6.6-7.8, while the acceptable pH range is 6.1-8.3; meaning that below 6.1 the process may fail due to an excessively low methanogenesis rate compared to acidogenesis rate, while above 8.3 the process might be inhibited by free ammonia (Lay *et al.*, 1997).

The limit concentration proposed to detect and prevent digester failure during thermophilic sewage sludge digestion, based on the results obtained in this study, are summarised in Table 4.7.

**Table 4.7.** Limit values proposed to prevent thermophilic anaerobic sludge digestion failure

Parameter	Limit value for thermophilic anaerobic sludge digestion
Acetate concentration ( $\text{g L}^{-1}$ )	0.5
A/P ratio	0.5
VFA concentration ( $\text{g L}^{-1}$ )	2.5
Intermediate alkalinity ( $\text{g CaCO}_3 \text{ L}^{-1}$ )	1.8
IA/PA ratio	0.9
IA/TA ratio	0.5
Methane content in biogas (% $\text{CH}_4$ )	55

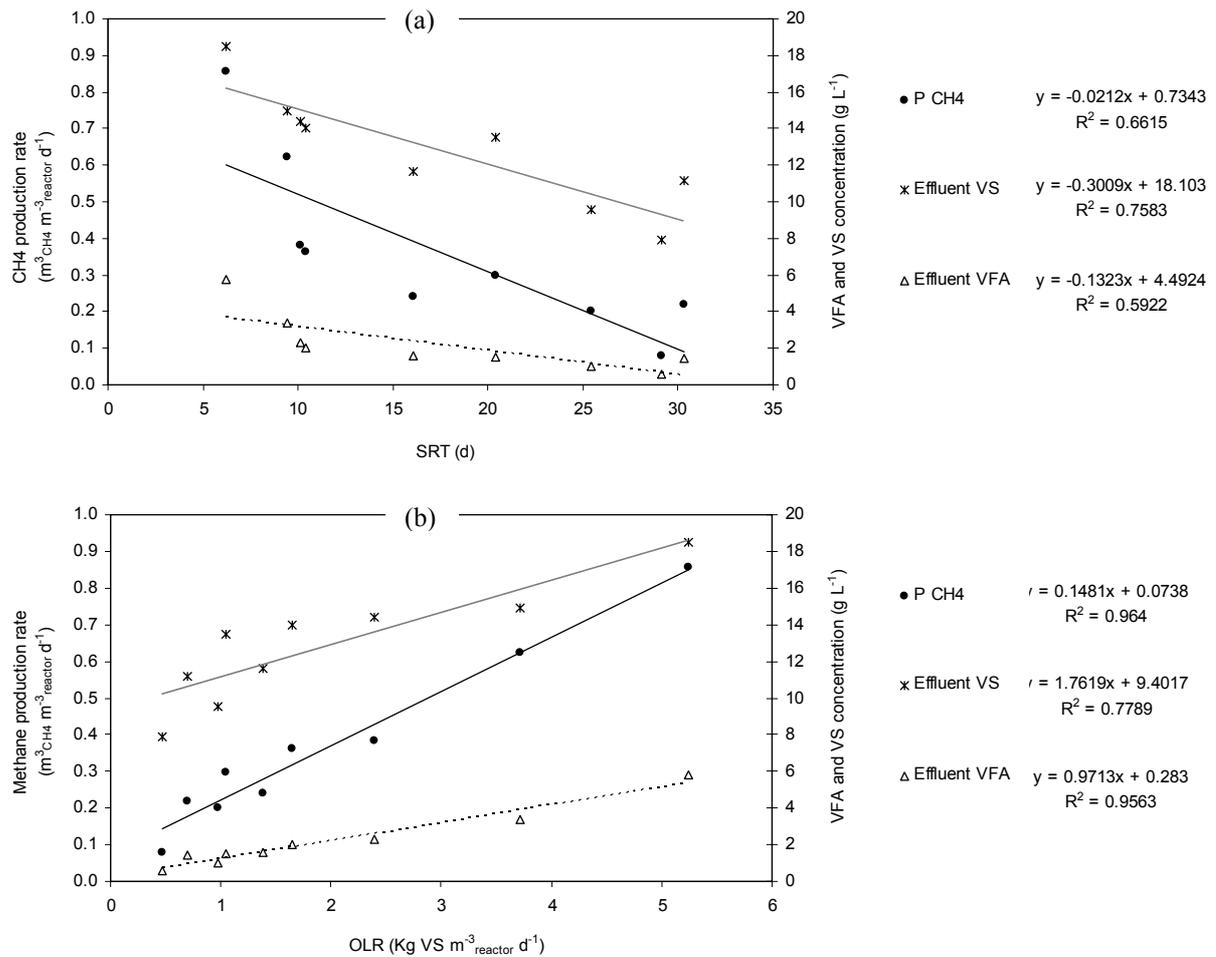
#### 4.3.3.3. Effect of SRT and OLR on process efficiency and stability

The main objective of decreasing the SRT was to determine the minimum SRT allowing a stable anaerobic process performance at 55 °C. Bearing in mind that the minimum design SRT is around 15 days at 35 °C (Metcalf and Eddy, 2003), and that the growth rates of thermophilic methanogens are 2-3 times higher than those of mesophilic homologues, (Van Lier *et al.*, 1993; Mladenovska and Ahring, 2000), the theoretical SRT may be reduced to 5-8 days at 55 °C. However, such a reduction is likely to deteriorate process efficiency, especially regarding the quality of the effluent which is generally poorer in thermophilic digesters (Buhr and Andrews, 1977). Digested sludge dewaterability might consequently be degraded. At the same time, the destruction of pathogenic microorganisms might also be affected.

For the purposes of this study, the SRT was gradually reduced from 30 to 6 days. However, because the feeding sludge was collected weekly from the WWTP, seasonal variations affected its composition and organic content. Furthermore, low-solids and high-solids sludge were used. Whilst operating under a fixed SRT, the OLR was affected by the sludge organic content; thus it was also necessary to assess the effect of OLR on the thermophilic sludge digestion.

Figure 4.17 shows methane production rate, effluent VFA and effluent VS as a function of SRT (a) and OLR (b). In general, correlations were higher with OLR than with SRT, especially for methane production rate vs. OLR ( $R^2=0.96$ ) and VFA vs. OLR ( $R^2=0.93$ ), while they were poorer for VS ( $R^2=0.77$ ). This means that daily methane production, hence methanogenic activity, was very much dependant on the OLR; regardless of SRT, at least for SRT above 6 days. Similarly, acidogenesis increased with the OLR (Figure 4.17), but short SRT were not enough to convert all VFA to methane, which means that a portion of hydrolysed organic compounds did not end up yielding methane. As a result, the concentration of VS was also higher at shorter SRT.

Notice that the value of effluent VFA ( $5.79 \text{ g L}^{-1}$ ) corresponding to a SRT of 6 days and an OLR of  $5.24 \text{ kg VS m}^{-3} \text{ d}^{-1}$  (Figure 4.17), is the mean value measured during the days following the SRT increase from 6 to 10 days (Figure 4.14, period XII), undertaken to avoid an eventual digester failure. Although this value was measured from the effluent of the reactor working at 10 days SRT, it might be speculated that it was a consequence of the previous period of operation at 6 days SRT.



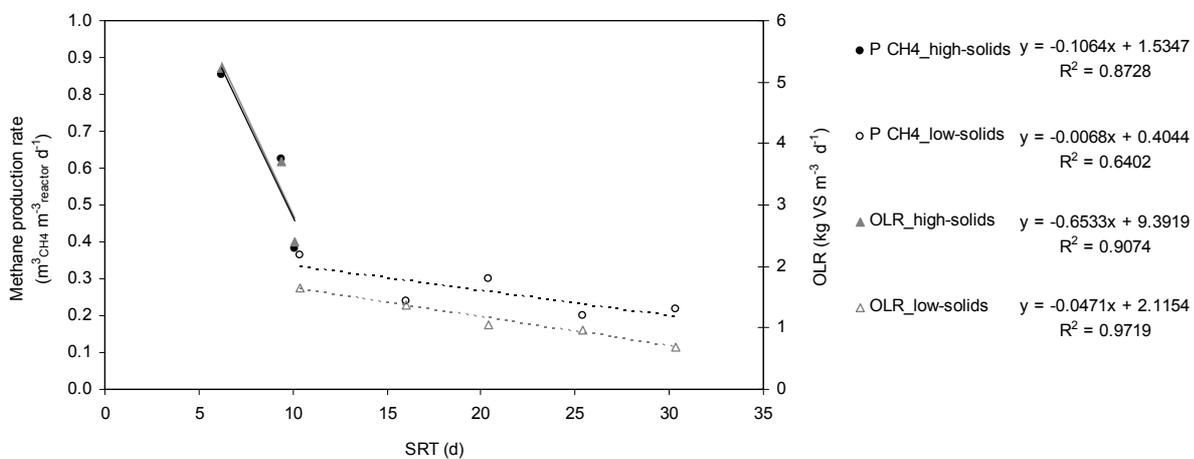
**Figure 4.17.** Methane production rate ( $P_{CH_4}$ ), effluent volatile fatty acids (VFA) and volatile solids (VS) as a function of: (a) sludge retention time (SRT) and (b) organic loading rate (OLR), during thermophilic sludge digestion. Note: The value of effluent VFA ( $5.79 \text{ g L}^{-1}$ ) corresponding to a SRT of 6 days and an OLR of  $5.24 \text{ kg VS m}^{-3} \text{ d}^{-1}$ , is the mean value measured during the days following the SRT increase from 6 to 10 days (period XII, Figure 4.14)

In Figure 4.18, methane production rate versus SRT, and OLR versus SRT, are plotted separately for low- and high-solids sludge. It is evident that both parameters followed parallel trends when low- and high-solids sludge were fed. It is also clear that they increased with decreasing SRT, especially in the case of high-solids sludge. Since both decreasing the SRT and feeding more concentrated sludge resulted in increased OLR, daily methane production improved in either case. As expected, the OLR and methane production were more sensitive to the solids concentration in the sludge at shorter SRT, while they were less sensitive at higher SRT.

De la Rubia *et al.* (2006) found a similar dependence of methane production rate on OLR and SRT over the range of 15-75 days during thermophilic anaerobic digestion of PS and WAS. COD

mass balances indicated that the amount of COD used for methane generation increased at decreasing SRT or increasing OLR. The results obtained by these authors suggest that higher OLR ( $> 2.2 \text{ kg VS m}^{-3} \text{ d}^{-1}$ ) or lower SRT ( $< 15$  days) might have resulted in further methane production improvement ( $> 0.4 \text{ m}^3_{\text{CH}_4} \text{ m}^{-3} \text{ d}^{-1}$ ).

Miron *et al.* (2000) report that, during psychrophilic digestion of PS, SRT of 10 days were enough to obtain methanogenic conditions in the reactor, while lower SRT (8 days) resulted in acidogenic conditions. Taking into account that reaction rates are higher under thermophilic conditions, it might be speculated that the homologous SRT for a thermophilic process would be lower.



**Figure 4.18.** Methane production rate ( $P_{\text{CH}_4}$ ) and organic loading rate (OLR) as a function of sludge retention time (SRT), during thermophilic anaerobic digestion of low- and high-solids sludge

As far as methanogenesis is concerned, Lin *et al.* (1985) found that it occurred normally at SRT as low as 4.43 days with OLR of  $70 \text{ g COD L}^{-1}$ ; and at 2.91 days with OLR of  $20 \text{ g COD L}^{-1}$ , the calculated minimum SRT for microbial populations being 2.42 days. In the study by Zhang and Noike (1994), even at SRT of 1.5 h methane was produced by  $\text{H}_2$ -utilizing methanogens; although SRT above 12 h were required to avoid the washout of acetate-utilizing methanogens. Since methanogenesis is the rate limiting step for the anaerobic degradation of soluble substrates, such low SRT might have been sufficient for the whole conversion of the substrate into methane in the above mentioned studies. However, when it comes to particulate substrates, like sewage sludge, hydrolysis tends to be slow and rate limiting. Therefore, longer SRT are required.

In the present study, the minimum SRT assayed was 6 days, but the minimum SRT ensuring a stable performance was 10 days. Methane production under thermophilic conditions was

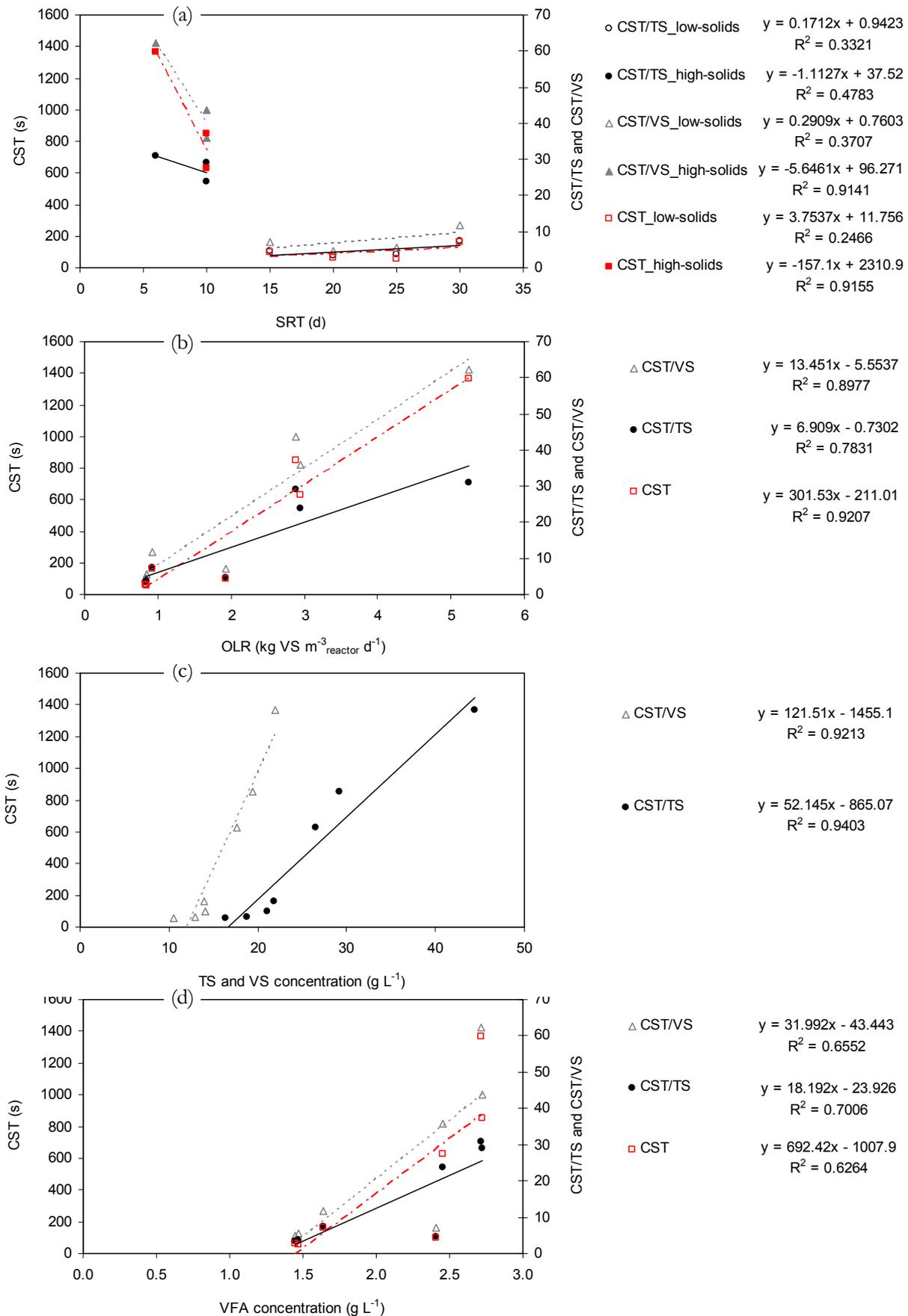
improved by decreasing the SRT from 30 to 10 days. It was further enhanced at 6 days SRT with an OLR higher than  $5 \text{ kg VS m}^{-3} \text{ d}^{-1}$ , feeding high-solids sludge. However, when the OLR eventually increased ( $> 6 \text{ kg VS m}^{-3} \text{ d}^{-1}$ ) as a result of fluctuations in the solids content of the feed sludge, methanogenic activity was severely affected; as indicated by decreased biogas production, with methane content below 50 %, and a sudden accumulation of VFA, with a total concentration higher than  $6 \text{ g L}^{-1}$ . Furthermore, the quality of the effluent in terms of VS content was worsened.

On the other hand, working at SRT of 10 days still with high OLR ( $3\text{-}4 \text{ kg VS m}^{-3} \text{ d}^{-1}$ ), the process was more stable. Biogas and methane production rates ( $0.55\text{-}0.6$  and  $0.35\text{-}0.4 \text{ m}^3 \text{ m}^{-3} \text{ reactor} \text{ d}^{-1}$ ) were increased by 50 % compared previous results at higher SRT. Gas production at 10 days SRT was within the range obtained by other authors at 15 days SRT (De la Rubia *et al.*, 2006; Benabdallah *et al.*, 2006); but clearly higher than that obtained at 20 days SRT (De la Rubia *et al.*, 2002; Gavala *et al.*, 2003). In practise, this means that the sludge daily flow rate could be doubled or the digester volume reduced, while producing the same amount of methane, thus of energy. However, higher effluent VS and especially higher VFA, ought to be expected at this reduced SRT; which might deteriorate subsequent sludge dewatering.

#### 4.3.3.4. Sludge dewaterability

Sludge dewaterability was measured by determining the capillary suction time (CST) of digested sludge samples obtained during each stability period. In Figure 4.19, the values of CST, as well as CST per g TS and CST per g VS in the sludge sample are represented versus the SRT (a) and OLR (b). In Figure 4.19 (a) such values are plotted separately for low- and high-solids sludge. Effluents from digesters treating low-solids sludge at high SRT ( $\geq 15$  days) were all similar, while CST increased at decreasing SRT feeding high-solids sludge. Indeed, CST increased proportionally to the OLR, as indicated by a high correlation coefficient ( $R^2=0.92$ ) in Figure 4.19 (b). The trends are similar when the CST is expressed per g TS or g VS.

A clear dependence of CST on the solids concentration in the sludge sample is shown in Figure 4.19 (c): the higher the solids concentration, the higher the CST. Hence, it may be speculated that any increase in effluent VS and TS resulting from changing the OLR and/or SRT may ultimately affect digested sludge dewaterability. From the results of this study, it seems that digested sludge dewaterability was deteriorated with TS higher than  $26 \text{ g L}^{-1}$  and VS higher than  $17 \text{ g L}^{-1}$ ; which corresponded to OLR above  $3 \text{ g VS m}^{-3} \text{ d}^{-1}$  and SRT below 10 days.



**Figure 4.19.** Capillary suction time (CST) of thermophilic digested sludge: (a) CST, CST per total solids (CST/TS) and CST per volatile solids (CST/VS) vs. sludge retention time (SRT); (b) CST, CST/TS and CST/VS vs. organic loading rate (OLR); (c) CST vs. TS and VS; (d) CST, CST/TS and CST/VS vs. OLR

According to the work by Miron *et al.* (2000), the dewaterability of PS worsened under acidogenic conditions ( $SRT \leq 8$  days), while it improved under methanogenic conditions ( $SRT \geq 10$  days). This was related to a decrease in the mean particle size, thus an increase in the total surface area, under acidogenic conditions. Moreover, only at high SRT of 15 days was digested sludge dewaterability improved compared to that of influent sludge. The results of the present study are quite consistent with those findings, since only at SRT above 15 days was the CST value (60-160 s) below that of influent sludge (437 s). Sludge dewaterability was worsened (CST  $\sim$  630-1370 s) at shorter SRT (10-6 days), which were typically associated to higher effluent VFA, thus higher soluble VS. Indeed, an increasing trend was followed by CST with respect to effluent VFA (Figure 4.19 (d)).

In the literature some controversy exists regarding the effect of anaerobic digestion on sludge dewaterability, and it is still not clear whether mesophilic and thermophilic digestion has any effect in sludge dewaterability. It has been shown that sludge dewaterability, as well as the amount of chemicals required for sludge conditioning, are directly dependant on the concentration of biopolymer in the solution (Novak *et al.*, 2003). Houghton *et al.* (2000) and Houghton and Stephenson (2002) reported that the composition of microbial extracellular polymer (ECP) varied after sludge digestion and was also affected by the feed composition; attributing excess ECP production to acidogenic bacteria. This might also explain higher CST values obtained in the present study in samples with higher VFA concentration, in which the presence of acidogenic bacteria should be higher.

#### 4.3.3.5. Effluent hygienisation

Sludge hygienisation was assessed by quantifying pathogen indicator *Escherichia coli* and *Salmonella* spp. from digested sludge samples obtained during each stability period, and comparing them to the values obtained in influent sludge samples. While *Salmonella* spp. was never detected; the concentration of the *E. coli* in the influent sludge was in the range of  $10^6$  CFU mL<sup>-1</sup>. A complete destruction of *E. coli* was achieved at SRT higher than 20 days, but concentrations in the range of  $10^1$  and  $10^2$  CFU mL<sup>-1</sup> were found at SRT of 10-15 days and 6 days, respectively (Table 4.8). Apparently, the concentration of *E. coli* in the effluent was influenced by the OLR, suggesting a certain effect of the initial *E. coli* concentration in the influent sludge.

**Table 4.8.** Microbiological analyses of influent and effluent sludge samples in R2

Pathogens	Influent (PS+WAS)	Effluent					
		30 d	25 d	20 d	15 d	10 d	6 d
<i>E. coli</i> (CFU mL <sup>-1</sup> )	1.0 × 10 <sup>6</sup>	Absence	Absence	Absence	1.0 × 10 <sup>1</sup>	1.0 × 10 <sup>1</sup>	1.1 × 10 <sup>2</sup>
<i>Salmonella</i> spp. (in 50 mL)	Absence	Absence	Absence	Absence	Absence	Absence	Absence

The rate of die-off of bacteria as a result of thermal stress follows first order kinetics (Krugel *et al.*, 1998). According to this simple model, it basically depends on process temperature, time and the initial concentration of bacteria. Laboratory studies conducted by Lang and Smith (2007) revealed that the death of enteric bacteria was instantaneous ( $\leq 40$  s) at 70 °C; it took place within 1 h at 55 °C; and was only marginal at 35 °C. These authors concluded that pathogen removal rates during high-temperature sludge treatment depended largely upon time-temperature decay kinetics, but that mesophilic temperatures did not exert a specific thermal stress on the decay of *E. coli* and *Salmonella* spp. In full-scale reactors, such decay is also influenced by operational factors and sludge characteristics, especially those affecting the heat transfer; and therefore longer times are required.

Hygienisation of thermophilic effluent sludge in laboratory and full-scale reactors working at a range of SRT is reported in the literature (Zábranská *et al.*, 2000a; Laffite-Trouqué and Forster, 2002; Skiadas *et al.*, 2004; Lu *et al.*, 2007). It is in fact a major advantage of thermophilic anaerobic digestion, compared to mesophilic operation. In this study, *E. coli* and *Salmonella* spp. concentrations in all effluent samples were below the limits proposed in the 3<sup>rd</sup> Draft EU Working Document on Sludge (Environment DG, EU, 2000) for land application of digested sludge; which suggests that a minimum SRT of 6 days at 55 °C might be sufficient to prevent the spread of pathogens in the environment upon land application of digestates.

However, Higgins *et al.* (2007) point out that high concentrations of indicator bacteria such as faecal coliforms have been measured in anaerobically digested sludges immediately after dewatering; even though low concentrations had been measured prior to dewatering. Since the reasons for this are not yet clear, measures like continued storage of the cake may provide a simple solution to achieve the desired hygienisation effect.

#### 4.4. CONCLUSIONS

Two lab-scale digesters were operated for 18 and 21 months, respectively, treating low-solids and high-solids mixture of PS and WAS. The effect of process temperature (43, 50 and 55 °C), sludge retention time (30-6 days) and organic loading rate on methane production and on the quality of the effluent sludge were evaluated. Process stability versus temperature fluctuations and organic overloading was also studied. From this work the following conclusions can be drawn:

- (1) The transition from a mesophilic (43 °C) to a thermophilic (50 °C) operation was carried out without causing any apparent process disturbance (as indicated by unaltered biogas production rate, methane content in biogas and pH), working at high SRT ( $\geq 30$  days) while feeding low-solids sludge ( $OLR \leq 0.5 \text{ kg VS m}^{-3}_{\text{reactor}} \text{ d}^{-1}$ ).
- (2) Working at long SRT ( $\geq 30$  days), the main difference between mesophilic (43 °C) and thermophilic (50- 55 °C) process performance referred to VFA and effluent hygienisation. During stable mesophilic operation, VFA were always under detection limits; whereas a certain accumulation (0.5-2.5 g VFA L<sup>-1</sup>) was always detected during thermophilic operation. A 3 log reduction in *E. coli* was achieved by mesophilic digestion; with concentrations around 10<sup>3</sup> CFU mL<sup>-1</sup> in the mesophilic sludge. On the other hand, thermophilic digestion ensured either complete destruction or residual concentrations of 10<sup>1</sup>-10<sup>2</sup> CFU mL<sup>-1</sup> in the thermophilic sludge. *Samonella* spp. was never detected.
- (3) Thermophilic sludge digestion at 50 °C and 55 °C behaved very similarly in terms of biogas production and effluent stabilisation, hygienisation and dewaterability; provided that other process parameters were the same. In general, the process was more efficient at higher OLR, resulting from higher solids content in the feed sludge. Within the studied range, the higher the sludge solids content, the higher the biogas production both at 50 °C and 55 °C.
- (4) A linear correlation was found between methane production rate and OLR, as well as between effluent characteristics (VS concentration, VFA concentration and sludge dewaterability) and OLR, during thermophilic operation at 55 °C. No such correlations were found with respect to the SRT, due to fluctuations in the solids content of the influent sludge, affecting the OLR.

- (5) Methane production rate at 55 °C was increased (from 0.2 to 0.4-0.6  $\text{m}^3_{\text{CH}_4} \text{m}^3_{\text{reactor}} \text{d}^{-1}$ ) by decreasing the SRT from 30 to 10, while increasing the OLR from 0.5 to 2.5-3.5  $\text{kg VS m}^3_{\text{reactor}} \text{d}^{-1}$ . Although it was further improved at the lowest SRT of 6 days, with an OLR higher than 5  $\text{kg VS m}^3_{\text{reactor}} \text{d}^{-1}$ , progressive VFA accumulation ( $> 5 \text{ g L}^{-1}$ ) and reduced methane content in biogas ( $< 50 \%$ ) suggested poor methanogenic activity and process unbalance.
- (6) Temperature fluctuations during thermophilic operation at 50 °C did not show a severe effect on the system. Biogas production ceased during temperature drops ( $< 47 \text{ °C}$ ) or increases ( $> 56 \text{ °C}$ ), but no lasting effect on the subsequent digestion was noted when the digester was returned to its original operating temperature. Peak VFA concentrations were detected in close relation with both temperature fluctuations and organic overloading, resulting from sudden increases in the influent VS, thus in the influent VFA.
- (7) Exposing the digester to aerobic conditions (as a result of opening the reactor to solve operating problems), had a severe effect when the sludge had been adapted for a relatively short period to thermophilic temperatures; whereas no such effect was detected after long term thermophilic operation.
- (8) According to the values of indicator parameters during stability and instability periods, the following concentrations might be useful to detect and prevent an eventual digester failure during thermophilic sludge digestion: total VFA ( $2.5 \text{ g L}^{-1}$ ), acetate ( $0.5 \text{ g L}^{-1}$ ), A/P ratio (0.5), intermediate alkalinity ( $1.8 \text{ g CaCO}_3 \text{ L}^{-1}$ ), intermediate alkalinity/partial alkalinity ratio (0.9), intermediate alkalinity/total alkalinity ratio (0.5), methane content in biogas (55 %).

**Chapter 5. Study of the effect of low temperature  
pre-treatment on the thermophilic  
anaerobic digestion of sewage sludge**

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## Chapter 5. STUDY OF THE EFFECT OF LOW TEMPERATURE PRE-TREATMENT ON THE THERMOPHILIC ANAEROBIC DIGESTION OF SEWAGE SLUDGE

### Abstract

Thermophilic anaerobic digestion is more efficient than mesophilic and psychrophilic anaerobic digestion, in terms of biogas and methane production, volatile solids removal and pathogens destruction. The process might be further accelerated by sludge pre-treatment, promoting sludge solubilization and hydrolysis.

The objective of this Chapter was to investigate the effect of low temperature pre-treatment (70 °C) on the efficiency of thermophilic anaerobic digestion of primary and waste activated sludge. Firstly, the effect of sludge pre-treatment time (9, 24, 48 and 72 h) was evaluated by measuring the increase in volatile dissolved solids (VDS), volatile fatty acids (VFA) and biogas production in thermophilic batch tests. Secondly, semi-continuous process performance was studied in a lab-scale reactor (5 L) working at 55 °C with a sludge retention time of 10 days.

The 70 °C pre-treatment showed an initial solubilization effect (increasing VDS by almost 10 times after 9 h), followed by a progressive generation of VFA (from 0 to nearly 5 g L<sup>-1</sup> after 72 h). Biogas production increased up to 30 % both in batch tests and in semi-continuous experiments. Methane content in biogas also increased, from 64 to 69 % CH<sub>4</sub> with raw and pre-treated sludge, respectively. These results suggest that a short period (9 h) low temperature pre-treatment should be enough to enhance methane production through thermophilic anaerobic digestion of sludge.

*Based on:*

*Ferrer I., Ponsá S., Vázquez F. and Font, X. (2008). Increasing biogas production by thermal (70 °C) sludge pre-treatment prior to thermophilic anaerobic digestion. Biochemical Engineering Journal, 42(2), 186-192.*

## 5.1. INTRODUCTION AND OBJECTIVES

### 5.1.1. Introduction

Anaerobic digestion is a treatment process used in many municipal wastewater treatment plants (WWTP) for sludge stabilization. Mass reduction, methane production and improved dewatering properties of the treated sludge are the main features of the process. Slow degradation of sewage sludge is a disadvantage of anaerobic digestion, leading to high sludge retention times (SRT) of 20-30 days in conventional mesophilic digesters. This fact implies significant space requirements due to large digesters. Anaerobic digestion may be carried out under psychrophilic (< 25 °C), mesophilic (35-40 °C) and thermophilic conditions (50-55 °C). In general, mesophilic anaerobic digestion of sewage sludge is more widely used compared to thermophilic digestion, mainly because of the lower energy requirements and higher stability of the process. Thermophilic digestion, however, is more efficient in terms of organic matter removal and methane production (Buhr and Andrews, 1977; Ahring *et al.*, 2001b). Moreover, it enhances the destruction of pathogens, weed seeds and insect eggs; thus enabling effluent hygienisation (Zábranská *et al.*, 2000a), which might be required in the short term for land application, as suggested in the 3<sup>rd</sup> Draft EU Working Document on Sludge (Environment DG, EU, 2000). Increased energy requirements may be met by implementing a system allowing heat recovery from the effluent and cogeneration with biogas (Zupančič and Roš, 2003).

#### 5.1.1.1. Sludge pre-treatment

Hydrolysis is the rate limiting step of anaerobic digestion of semi-solid wastes. In this step both solubilization of particulate matter and biological decomposition of organic polymers to monomers or dimers take place. Thermal, chemical, biological and mechanical processes, as well as combinations of these, have been studied as possible pre-treatments to accelerate sludge hydrolysis. These pre-treatments cause the lysis or disintegration of sludge cells permitting the release of intracellular matter that becomes more accessible to anaerobic microorganisms. This fact improves the overall digestion process velocity and the degree of sludge degradation, thus reducing digester retention time and increasing methane production rates (Müller, 2000).

Mechanical sludge disintegration methods are generally based on the disruption of microbial cell walls by shear stress. Stirred ball mills, high pressure homogenisers and mechanical jet smash techniques have been used for mechanical pre-treatment application although the most used technique is sludge sonication (Weemaes and Verstraete, 1998; Müller, 2000; Bourgrier *et al.*,

2006; Benabdallah El-Hadj *et al.*, 2006; Climent *et al.*, 2007). Microwaves have also been used for cell lysis. However, they have been scarcely used for sludge disintegration (Banik *et al.*, 2003; Park *et al.*; 2004; Eskicioglu *et al.*, 2006; Eskicioglu *et al.*, 2007; Climent *et al.*, 2007). The use of heat has been widely reported for the disintegration of sludge (Stuckey and McCarty, 1984; Müller, 2000; Valo *et al.*, 2004; Záborská *et al.*, 2000b; Záborská *et al.*, 2006; Bourgrier *et al.*, 2007; Climent *et al.*, 2007). A wide range of temperatures has been studied, ranging from 60 to 270 °C, although the most common pre-treatment temperatures are between 60 and 180 °C, since temperatures above 200 °C have been found responsible for refractory compound formation (Stuckey and McCarty, 1984). Pre-treatments applied at temperatures below 100 °C are considered as low temperature thermal pre-treatments. Such pre-treatments have been pointed out as effective in increasing biogas production from both primary and secondary sludge (Gavala *et al.*, 2003; Climent *et al.*, 2007).

Similarly, two-stage systems coupling a hyperthermophilic digester (68-70 °C) and a thermophilic digester (55 °C) have been found to be more efficient in terms of methane production compared to single stage thermophilic digesters treating primary and secondary sludge (Skiadas *et al.*, 2004; Lu *et al.*, 2007) and cattle manure (Nielsen *et al.* 2004). In these studies, it is suggested that thermal pre-treatment applied at temperatures around 70 °C enhances biological activity of some thermophilic bacteria population with optimum activity temperatures in the high values of the thermophilic range. Thus, low temperature pre-treatments may be considered a predigestion step.

In general, the efficiency of pre-treatments has been assessed by the increase of soluble organic matter (i.e. volatile dissolved solids (VDS), soluble chemical oxygen demand or soluble proteins). Some studies also focus on anaerobic biodegradability and biogas production, mainly in mesophilic batch assays (Valo *et al.*, 2004; Bourgrier *et al.*, 2006; Eskicioglu *et al.*, 2006; Eskicioglu *et al.*, 2007). But little work has been done on the effect of sludge pre-treatment on thermophilic anaerobic digestion (Gavala *et al.*, 2003, Climent *et al.*, 2007), especially in continuous digesters (Laffite-Trouqué and Forster, 2002; Benabdallah El-Hadj *et al.*, 2006; Záborská *et al.*, 2006). To our knowledge, no such work exists for low temperature pre-treatment of the mixture of primary and waste activated sludge prior to continuous thermophilic anaerobic digestion.

### 5.1.2. Objectives

The objective of this Chapter was to address the enhancement of thermophilic anaerobic digestion of the mixture of thickened primary sludge (PS) and waste activated sludge (WAS), by

means of low temperature (70 °C) pre-treatment. Firstly by studying the effect of pre-treatment time on organic matter solubilization, volatile fatty acids (VFA) generation and biogas production in thermophilic batch tests; and secondly by evaluating process efficiency in a semi-continuous lab-scale reactor at 55 °C and 10 days SRT. The effect on the hygienisation of sludge was also studied.

## **5.2. METHODOLOGY**

### **5.2.1. Low temperature (70 °C) sludge pre-treatment**

The low temperature sludge pre-treatment was carried out at 70 °C in order to enhance thermal solubilization of particulate material, as well as enzymatic hydrolysis. Bearing in mind that the effect of thermal pre-treatments depends both on treatment temperature and time (Li and Noike, 1992), in the present study the effect of pre-treatment duration was evaluated by taking samples at different pre-treatment times (9, 24, 48 and 72 h) in order to study the combined effect. Sludge samples were pre-treated following the procedure explained in Chapter 3 (Section 3.5). Sewage sludge was obtained from the municipal WWTP described in Chapter 3 (Section 3.1).

### **5.2.2. Anaerobic batch tests**

Biogas production of raw and pre-treated sludge samples (at 70 °C for 9, 24, 48 and 72 h) was initially determined by means of batch tests at 55 °C. The objective was to study the effect of the duration of 70 °C pre-treatment, in terms of anaerobic biodegradability and biogas production under thermophilic conditions. Anaerobic batch tests were carried out as indicated in Chapter 3 (Section 3.4). Biogas production was measured manometrically, with a device designed for the purposes of this study (Ferrer, 2003; Fornés, 2004; Ferrer *et al.*, 2004b).

### **5.2.3. Lab-scale thermophilic anaerobic digestion**

The effect of 70 °C pre-treatment on semi-continuous process performance was studied in the experimental set-up described in Chapter 3 (Section 3.2).

Prior to the experiments with pre-treated sludge, Reactor R1 had been working at 55 °C for one year, at decreasing SRT from 30 to 10 days (Chapter 4), at which it was maintained under stable conditions for 2 months. This is the control treatment to which experiments with pre-treated sludge were compared. Keeping the same SRT of 10 days, the digester was subsequently fed with

pre-treated sludge (at 70 °C, for 9, 24 and 48 h), with a total experimental duration of 6 months. Experimental procedures and analytical methods are described in Chapter 3 (Sections 3.3 and 3.6).

### 5.3. RESULTS AND DISCUSSION

#### 5.3.1. Sludge composition

General characteristics of the feeding sludge, mixture of thickened PS and WAS, are summarised in Table 5.1. TS content was around 39 g L<sup>-1</sup> (3.9 %) and total VS around 29 g L<sup>-1</sup> (2.9 %), with a VS/TS ratio of 0.74 (74 %), a high organic content typical from fresh non-stabilized materials. Furthermore, only a small proportion of this organic material was soluble, as shown by the low volatile dissolved solids to total volatile solids ratio (0.05 VDS/VS), which may be indicating that little hydrolysis had occurred. This matches with the almost absence of volatile fatty acids (VFA), meaning very scarce fermentative activity. The only VFA detected were acetate and propionate.

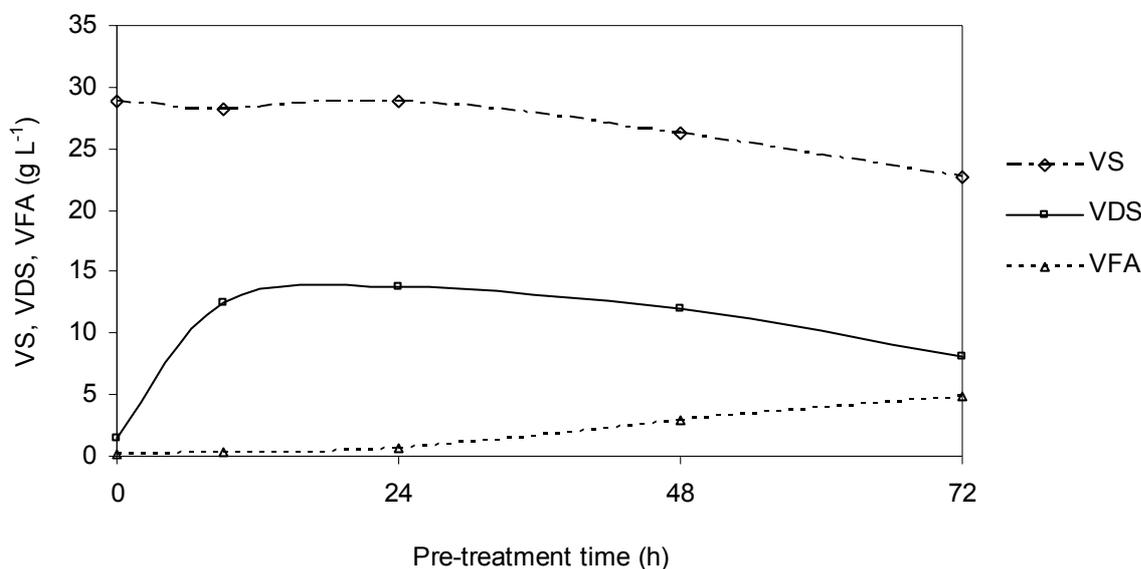
**Table 5.1.** Sewage sludge composition

Parameter	Value
TS (g L <sup>-1</sup> )	38.97
VS (g L <sup>-1</sup> )	28.87
VS/TS	0.74
TDS (g L <sup>-1</sup> )	2.54
VDS (g L <sup>-1</sup> )	1.51
VDS/TDS	0.59
VDS/VS	0.05
Total VFA (g L <sup>-1</sup> )	0.11
Acetate (g L <sup>-1</sup> )	0.06
Propionate (g L <sup>-1</sup> )	0.05
iso-Butyrate (g L <sup>-1</sup> )	0.00
n-Butyrate (g L <sup>-1</sup> )	0.00
iso-Valerate (g L <sup>-1</sup> )	0.00
n-Valerate (g L <sup>-1</sup> )	0.00
pH	7.96

### 5.3.2. Effect of the 70 °C pre-treatment on VDS and VFA

The expected effect after thermal pre-treatment of sludge was an increase in soluble materials, with interest focused on soluble organic solids (i.e. VDS), thus enhancing hydrolysis. Since the feeding sludge was a mixture of thickened PS and WAS, and WAS consists of a complex activated sludge floc structure, the disruption of this structure may release biopolymers such as proteins or sugars from the floc into the soluble phase (Eskicioglu *et al.*, 2006). At the same time, disruption of microbial cells from WAS should lead to their solubilization into carbohydrates, proteins, lipids and even lower molecular weight products like VFA (Li and Noike, 1992).

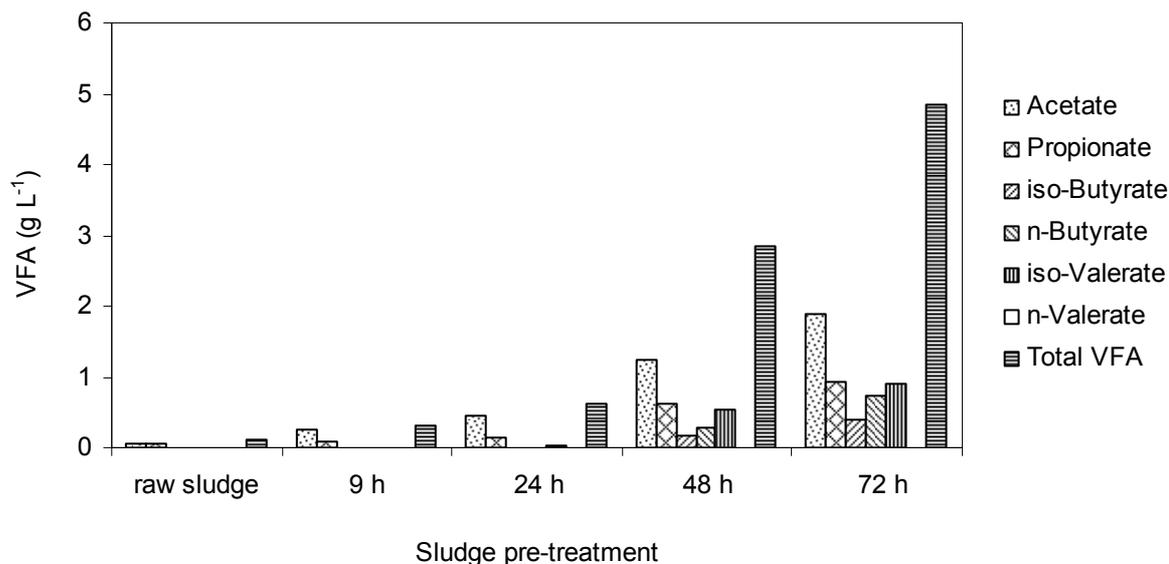
As expected, TDS and VDS concentrations increased after thermal pre-treatment at 70 °C. An increase from around 1.5 g L<sup>-1</sup>VDS in the raw sludge to 11.9-13.9 g L<sup>-1</sup> VDS after 9, 24 and 48 h thermal pre-treatment was detected (Figure 5.1), resulting in an increase in VDS/VS ratio from 0.05 to 0.44-0.48. This means that the proportion of soluble to total organic matter increased by almost 10 times, from 5 % to almost 50 % after 70 °C pre-treatment. Regarding VFA concentration, it increased along pre-treatment time, from about 0 in the raw sludge to nearly 5 g L<sup>-1</sup> after 72 h thermal pre-treatment. After 24 h acetic and propionic acids were the main VFA generated, whereas butyric and valeric acids were mostly detected after 48 h (Figure 5.2).



**Figure 5.1.** Evolution of volatile solids (VS), volatile dissolved solids (VDS) and volatile fatty acids (VFA) along 70 °C pre-treatment time (9, 24, 48 and 72 h)

Comparing the evolution of VDS and VFA (Figure 5.1), it is clear that there was a sharp increase in VDS, which was followed by a progressive generation of VFA after 24 h. According to this, sludge solubilization due to 70 °C pre-treatment would occur rapidly, reaching a maximum

concentration of VDS within 9-24 h. Other studies indicate that even shorter periods (30-60 min) are needed for WAS solubilization at 60-80 °C (Li and Noike, 1992; Wang *et al.* 1997). On the other hand, longer pre-treatments at 70 °C may favour the activity of thermophilic or hyperthermophilic bacteria, promoting enzymatic hydrolysis and resulting in a predigestion step (Skiadas *et al.*, 2004; Nielsen *et al.*, 2004; Lu *et al.*, 2007). The relentless increase in VFA after 9 h, and especially after 24 h, might result from the aforementioned process.

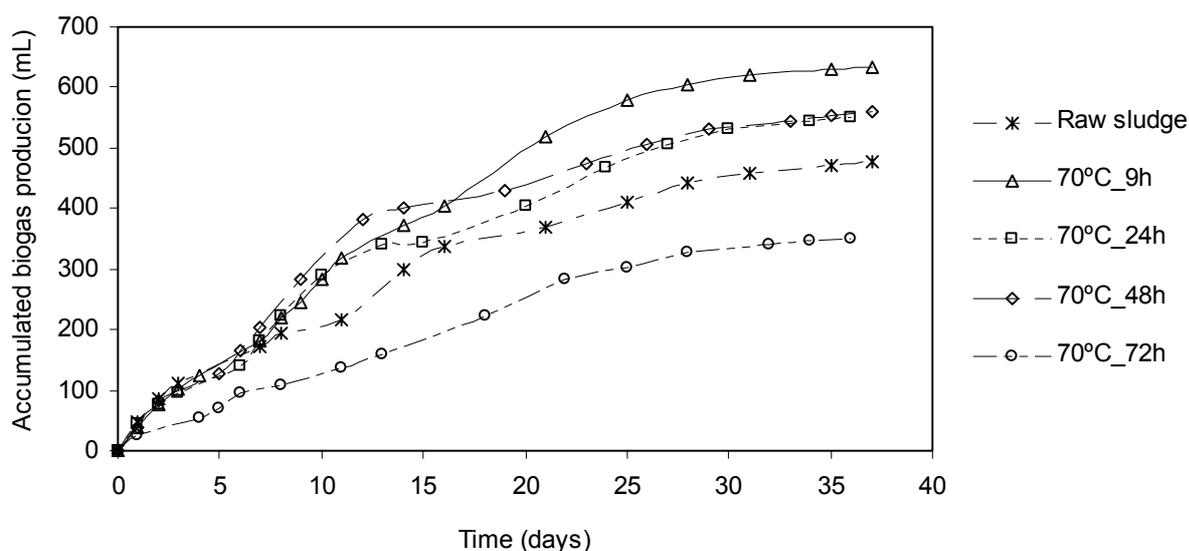


**Figure 5.2.** Generation of individual and total volatile fatty acids (VFA) along 70 °C pre-treatment time (9, 24, 48 and 72 h)

### 5.3.3. Effect of the 70 °C pre-treatment on biogas production in batch tests

Biogas production under thermophilic conditions was initially assessed by means of anaerobic batch tests using raw and pre-treated sludge samples. Figure 5.3 shows the evolution of net accumulated biogas production during the 37 days of assay. Initial biogas production rate (indicated by the slope of the curve) up to day 7 was similar in all cases, except for the 72 h pre-treated sludge. However, at day 10 (which corresponds to the SRT assayed in the continuous process) accumulated production was nearly 300 mL for 9, 24, and 48 h pre-treated samples, whereas for the control treatment it was around 200 mL, representing an almost 50 % volume increase. Final values were somewhat higher for the 9 h treatment (30 % increase) followed by the 24 and 48 h treatments (15 % increase). Gavala *et al.* (2003) found increased thermophilic methane potential after 70 °C pre-treatment, but only for primary sludge samples, whereas production rate was increased both with primary and secondary sludge samples.

Lower values for 72 h treated sludge could be related to process inhibition caused by initial accumulation of VFA. The concentration of VFA in the sludge after 72 h of thermal pre-treatment was remarkably high ( $4.86 \text{ g L}^{-1}$ ), even higher than in the thermophilic inoculum used for the tests ( $2.12 \text{ g L}^{-1}$ ). This initial accumulation was not observed after shorter pre-treatments (9-48 h) in which final VFA concentration were much lower ( $0.32$ - $2.86 \text{ g L}^{-1}$ ). In addition, partial biodegradation of organic compounds during pre-treatment itself might be responsible for lower final biogas volume; as suggested by lower VS and VDS in Figure 5.1.



**Figure 5.3.** Biogas production in thermophilic batch tests with raw and 70 °C pre-treated sludge (9, 24, 48 and 72 h)

#### 5.3.4. Performance of thermophilic anaerobic digestion

Table 5.2 shows characteristics and operational parameters during semi-continuous thermophilic anaerobic digestion of raw sludge and 70 °C pre-treated mixture of primary and secondary waste sludge.

##### 5.3.4.1. Thermophilic anaerobic digestion of raw sludge at 10 days SRT

Thermophilic digestion of raw sludge after 1 year of operation at decreasing SRT from 30 to 10 days (Chapter 4, Section 4.3.2), and more than 2 months at the lowest SRT of only 10 days, proved to be very stable. Average efficiencies were around 27 % and 33 % for TS and VS removal, respectively; biogas production rate around  $0.63 \text{ m}^3_{\text{biogas}} \text{ m}^{-3}_{\text{reactor}} \text{ d}^{-1}$  and methane content in biogas around 64 % (Table 5.2).

**Table 5.2.** Average feed and digested sludge characteristics and operational parameters during semi-continuous thermophilic anaerobic digestion with raw and 70 °C pre-treated sewage sludge

Parameter	70 °C pre-treatment time (h)			
	0	9	24	48
<b><i>Working conditions</i></b>				
Temperature (°C)	55.09 ± 0.63			
SRT (d)	9.97 ± 0.58			
OLR (kg VS m <sup>-3</sup> reactor d <sup>-1</sup> )	3.03 ± 0.33	2.93 ± 0.76	2.40 ± 0.83	2.94 ± 0.29
<b><i>Feed composition</i></b>				
TS (g L <sup>-1</sup> )	38.53 ± 6.26	55.47 ± 11.75	38.33 ± 9.90	54.43 ± 4.43
VS (g L <sup>-1</sup> )	30.08 ± 2.89	30.45 ± 3.59	26.59 ± 6.63	27.88 ± 2.12
VS/TS	0.78	0.55	0.69	0.51
pH	6.92 ± 0.18	6.67 ± 0.46	7.28 ± 0.29	7.15 ± 0.18
<b><i>Effluent composition</i></b>				
TS (g L <sup>-1</sup> )	31.17 ± 4.93	34.87 ± 5.92	33.95 ± 5.43	36.88 ± 5.64
VS (g L <sup>-1</sup> )	19.93 ± 1.88	18.95 ± 2.29	19.64 ± 3.52	18.56 ± 1.69
VS/TS	0.64	0.54	0.58	0.50
Total VFA (g L <sup>-1</sup> )	2.40 ± 0.42	1.27 ± 0.38	2.07 ± 0.45	1.42 ± 0.34
Acetate (g L <sup>-1</sup> )	0.32 ± 0.13	0.15 ± 0.10	0.67 ± 0.23	0.40 ± 0.29
Propionate (g L <sup>-1</sup> )	1.14 ± 0.12	0.88 ± 0.09	1.11 ± 0.17	0.86 ± 0.10
iso-Butyrate (g L <sup>-1</sup> )	0.30 ± 0.13	0.05 ± 0.08	0.09 ± 0.04	0.07 ± 0.04
n-Butyrate (g L <sup>-1</sup> )	0.00 ± 0.00	0.00 ± 0.00	0.01 ± 0.01	0.00 ± 0.00
iso-Valerate (g L <sup>-1</sup> )	0.53 ± 0.09	0.18 ± 0.13	0.19 ± 0.14	0.11 ± 0.02
n-Valerate (g L <sup>-1</sup> )	0.00 ± 0.00	0.00 ± 0.00	0.00 ± 0.00	0.00 ± 0.00
pH	8.22 ± 0.10	8.27 ± 0.10	8.32 ± 0.13	8.25 ± 0.12
<b><i>Removal efficiency</i></b>				
TS removal (%)	26.89 ± 6.07	31.16 ± 15.44	28.35 ± 15.38	30.66 ± 8.70
VS removal (%)	33.23 ± 5.49	36.55 ± 5.72	24.64 ± 9.09	32.61 ± 4.27
<b><i>Biogas characteristics</i></b>				
Biogas prod. rate (m <sup>3</sup> biogas m <sup>-3</sup> reactor d <sup>-1</sup> )	0.63 ± 0.06	0.87 ± 0.17	0.69 ± 0.18	0.81 ± 0.15
Biogas yield (m <sup>3</sup> biogas kg VS <sub>fed</sub> <sup>-1</sup> )	0.22 ± 0.04	0.30 ± 0.04	0.28 ± 0.05	0.29 ± 0.05
Specific biogas prod. (m <sup>3</sup> biogas kg VS <sub>removal</sub> <sup>-1</sup> )	0.61 ± 0.16	0.82 ± 0.17	0.81 ± 0.13	0.94 ± 0.14
Methane prod. rate (m <sup>3</sup> CH <sub>4</sub> m <sup>-3</sup> reactor d <sup>-1</sup> )	0.40 ± 0.04	0.56 ± 0.22	0.48 ± 0.14	0.59 ± 0.05
Methane yield (m <sup>3</sup> CH <sub>4</sub> kg VS <sub>fed</sub> <sup>-1</sup> )	0.15 ± 0.05	0.18 ± 0.08	0.18 ± 0.02	0.12 ± 0.10
Specific methane prod. (m <sup>3</sup> CH <sub>4</sub> kg VS <sub>removal</sub> <sup>-1</sup> )	0.44 ± 0.11	0.49 ± 0.23	0.41 ± 0.26	0.40 ± 0.35
Methane content (%)	63.73 ± 3.52	69.77 ± 3.36	68.73 ± 5.48	67.84 ± 5.13
<b><i>Stability period</i></b>				
Time (d)	522-553	558-598	599-648	649-680

Our results are quite consistent with those obtained under similar conditions, treating WAS at 8-12 days SRT (Laffite-Trouqué and Forster, 2002), or the mixture of PS and WAS at 15 days SRT (Benabdallah El-Hadj *et al.*, 2006) and 20 days SRT (Gavala *et al.*, 2003). However, from the comparison of these results it is clear that VS removal is lower at 10 days SRT (33 % vs. 46 and 52 % at 15 and 20 days SRT, respectively). On the other hand, biogas production rate is considerably higher (0.63 vs. 0.58 and 0.43 L L<sup>-1</sup> day<sup>-1</sup> at 15 and 20 days, respectively). This suggests that lower SRT are more efficient in terms of energy production, but less efficient in terms of effluent stabilization; as predicted by kinetic models when hydrolysis is the rate-limiting step of anaerobic digestion (Vavilin *et al.*, 2007). Hence, depending on sludge final disposal (i.e. land application) a stabilisation post-treatment such as composting may be appropriate to further stabilise the effluent.

Higher VS concentration in the effluent should possibly be related to a certain accumulation of VFA in the effluent, especially propionate, which degradation tends to be slower than the rest (Pind *et al.*, 2002). Apparently, though, this did not affect process stability. In fact, despite being high compared to mesophilic sludge (in which VFA concentration is typically low or even not detected); VFA concentration was still low compared to other thermophilic digesters with stable operation at SRT between 15 and 75 days (De la Rubia, 2006). Stable operation in spite of relatively high VFA concentration might be attributed to high buffer capacity in the system (i.e. alkalinity) and to the fact that anaerobes were already adapted to high OLR (~ 3 g VS L<sup>-1</sup> day<sup>-1</sup>) working at 10 days SRT.

Regarding effluent hygienisation, pathogens concentration was reduced from >10<sup>6</sup> CFU to absence per mL for *E. coli*; whereas *Salmonella* spp. was always absence per 50 mL (both in raw and digested sludge samples), which was also found by Záborská *et al.* (2000a). From a sanitary point of view, this effluent would fulfil the requirements for land application proposed in the 3<sup>rd</sup> Draft EU Working Document on Sludge (Environment DG, EU, 2000). Destruction of pathogens from primary or secondary waste sludge through one and two-stage thermophilic digestion has also been reported by other authors (Laffite-Trouqué and Forster, 2002; Skiadas *et al.*, 2004; Lu *et al.*, 2007).

#### 5.3.4.2. Thermophilic anaerobic digestion of 70 °C pre-treated sludge at 10 days SRT

The results with pre-treated sludge (Table 5.2) clearly show that the process was more efficient in terms of biogas production and yield in all cases, with increases in the range of 30-40 %,

following the tendency observed in the batch tests. Lower increase with the 24 h pre-treatment (10%) may be attributed to lower VS content in the influent sludge obtained from the WWTP during this experimental period. Notice that, in spite of the variability of solids concentration in the influent sludge, solids concentration in the effluent is fairly similar for all treatments. Apparently, the higher the VS fed, the higher the VS removed, and the higher the biogas production. According to this, under the conditions assayed, increasing solids concentration in the influent sludge up to of 55 g TS L<sup>-1</sup> and 30 g VS L<sup>-1</sup>, allows to increase biogas production (i.e. energy production) maintaining the quality of the effluent. Biogas yield (i.e. biogas produced per VS fed) was also enhanced in all cases, being some 30 % higher with pre-treated sludge (0.28-0.30 L gVS<sub>fed</sub><sup>-1</sup>) than with raw sludge (0.22 L gVS<sub>fed</sub><sup>-1</sup>). The same pattern described for biogas production applies to methane production. Moreover, methane content in biogas was also always higher after sludge pre-treatment, around 69 % vs. 64 % with raw sludge.

According to our results, it seems that 70 °C sludge pre-treatment has a similar effect in subsequent thermophilic digestion regardless of pre-treatment time. If no additional benefits are obtained, the shorter the pre-treatment time, the lower the costs related to energy consumption and reactor volume. Therefore, 9 h pre-treatment should be enough to enhance thermophilic digestion of sludge at 10 days SRT. Two-stage systems coupling a hyperthermophilic digester (68-70 °C, 2-3 days SRT) and a thermophilic digester (55 °C, 12-13 days SRT) have also been found to be more efficient in terms of methane production than single stage thermophilic digesters (55 °C, 15 days SRT) treating primary and secondary sludge (Skiadas *et al.*, 2004; Lu *et al.*, 2007) and cattle manure (Nielsen *et al.* 2004). In such studies it is suggested that positive effect of low temperature pre-treatments upon thermophilic digestion are related to the fact that they accelerate hydrolysis-acidogenesis by promoting the activity of thermophilic bacteria, resulting in the so-called predigestion step. Our study shows that 70 °C pre-treatment time as well as the overall SRT of thermophilic anaerobic digestion can be further reduced, maintaining the efficiency in terms of biogas and methane production. Other pre-treatments such as ultrasounds are more effective at enhancing mesophilic than thermophilic sludge digestion (Benabdallah El-Hadj *et al.*, 2006), which has been attributed to higher hydrolysis rate under thermophilic conditions, thus reducing the benefits from sludge solubilization prior to digestion process.

From an energetic point of view, full-scale application of low temperature sludge pre-treatment is amongst the less energy demanding pre-treatments, since influent sludge might be heated up to 70 °C by means of a heat-exchanger, using the waste heat from a conventional heat and power

generation unit fuelled with biogas. According to theoretical energy balances, the extra energy requirements would be fully covered by the energy generated from the extra methane production (Lu *et al.*, 2007).

#### 5.4. CONCLUSIONS

A thermophilic lab-scale digester was operated for over 6 months treating raw and pre-treated (70 °C) mixture of PS and WAS. From this period of study the following conclusions can be drawn:

- (1) Sludge solubilization due to low temperature (70 °C) pre-treatment can increase VDS concentration as much as 10 times (from  $\sim 1.5$  g VDS L<sup>-1</sup> in raw sludge to  $\sim 12.73$  g VDS L<sup>-1</sup> in pre-treated samples), representing an increase from around 5 % to 50 % in the VDS/VS ratio. This effect occurred already after the shorter pre-treatment times assayed (9 and 24 h). However, VFA generation was only enhanced after 24 h, which might be the threshold for the so-called predigestion step. From this moment, VFA concentration increased along pre-treatment time, up to a maximum concentration of nearly 5 g VFA L<sup>-1</sup> after 72 h.
- (2) Thermophilic batch tests showed that initial biogas production rate was similar for raw and for 9, 24 and 48 h pre-treated sludge samples. However, at day 10 accumulated biogas productions were 50 % higher for 9, 24, and 48 h pre-treatments, and final values were 30 % higher for 9 h pre-treatment, and 15 % for 24 and 48 h pre-treatments. Lower production in the 72 h pre-treatment could be related to initial inhibition caused by VFA accumulation, and to partial biodegradation of solubilized compounds during thermal pre-treatment.
- (3) Sludge pre-treatment at 70 °C enhanced biogas and methane productions in lab-scale digesters working at 55 °C with a SRT of 10 days. Biogas yield was some 30 % higher with pre-treated sludge ( $0.28\text{-}0.30$  L gVS<sub>fed</sub><sup>-1</sup>) than with raw sludge ( $0.22$  L gVS<sub>fed</sub><sup>-1</sup>). Methane content in biogas was also higher after sludge pre-treatment, around 69 % vs. 64 % with raw sludge.
- (4) The comparison of thermophilic anaerobic digestion of raw sludge at 10 days SRT with other studies at 15 and 20 days SRT shows that lower SRT are more efficient in terms of energy production, but less efficient in terms of effluent stabilization. This suggests that, depending on sludge final disposal, a stabilisation post-treatment such as composting may be appropriate to further stabilise the effluent.

- (5) Regarding effluent hygienisation, the thermophilic digester treating raw sludge at 10 days SRT was capable of reducing *E. coli* from over  $10^6$  CFU in the raw sludge to absence per mL in the digested effluent, whereas *Salmonella* spp. was never detected.
  
- (6) The results suggest that a short period (9 h) low temperature (70 °C) pre-treatment should be enough to enhance biogas and methane production through thermophilic anaerobic sludge digestion. The assessment of even shorter pre-treatment times should be considered in future research studies.



**Chapter 6. Assessment of sewage sludge anaerobic  
digestion from an energy perspective**

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## Chapter 6. ASSESSMENT OF SEWAGE SLUDGE ANAEROBIC DIGESTION FROM AN ENERGY PERSPECTIVE

### Abstract

Energy consumption accounts for some 30 % of the total operating costs of intensive sewage treatment systems. In conventional wastewater treatment plants employing an activated sludge process, around 15-20 % of this energy is used in the sludge treatment line, including sludge pumping, thickening, stabilisation and dewatering. Therefore, optimisation of sludge management can substantially contribute in the reduction of wastewater treatment costs.

The objective of this Chapter was to assess, from an energy perspective, alternatives for the enhancement of anaerobic sludge digestion. First of all, data from laboratory-, pilot- and full-scale digesters were used to compare energy production and consumption (i.e. energy balance) under hypothetic operating conditions of full-scale digesters. Secondly, a first order kinetic model was used to evaluate the efficiency of alternative sludge treatment systems.

According to the results, thermophilic anaerobic sludge digestion would result in net energy production, during cold and warm seasons, only in digesters with wall insulation and with energy recovery from both the biogas produced and the effluent sludge. In such a case, the energetic efficiency would be similar for thermophilic digesters working at half the sludge retention time (SRT) (10-15 days) of mesophilic digesters (20-30 days), meaning that the sludge daily flow rate could be doubled, or the reactor volume reduced, with subsequent savings in terms of sludge treatment costs. Additionally, two-stage systems (70/55 °C) may result in higher net energy production compared to single-stage systems (55 °C). However, the amount of surplus energy generated increases with digester volume. In spite of the decrease in methane production rate at increasing SRT, energy production is still higher than energy consumption, and therefore the bigger the amount of sludge in the digester, the higher the energy production.

*Partly based on:*

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## 6.1. INTRODUCTION AND OBJECTIVES

### 6.1.1. Introduction

Energy consumption accounts for some 30 % of the total operating costs of intensive sewage treatment systems. In conventional wastewater treatment plants (WWTP) employing an activated sludge process, around 15-20 % of this energy is used in the sludge treatment line, including sludge pumping, thickening, stabilisation and dewatering (Metcalf and Eddy, 2003). Furthermore, the total costs of sludge management for its treatment, transport and final disposal may represent some 50 % of the total facility costs of operation and maintenance (Mujeriego and Carbó, 1994). Therefore, optimisation of sludge management can substantially contribute in the reduction of wastewater treatment costs.

Anaerobic digestion allows simultaneous sludge stabilisation and energy recovery from the biogas produced, in such a way that anaerobic digesters can potentially be “energy-sufficient”. Sludge heating accounts for the major energy requirements, although electricity is also needed, for sludge pumping and mixing. Energy production depends on methane production rate, hence on organic solids removal, which in turn depends on the substrate composition (i.e. biodegradable fraction) and process operation (i.e. temperature, sludge retention time (SRT), organic loading rate (OLR), etc.).

#### *6.1.1.1. Review on thermophilic and mesophilic anaerobic sludge digestion*

Some figures on the efficiency of laboratory-, pilot- and full-scale reactors treating sewage sludge, obtained by means of an extensive literature review, are shown in Tables 6.1 through 6.4. Such Tables summarise information on: the type of sludge treated (primary sludge (PS), waste activated sludge (WAS) or the mixture of PS and WAS) and its solids content; the reactor design and volume; process temperature, SRT and OLR; biogas and methane production; effluent solids concentration and volatile solids (VS) removal. To ease comprehension, data on mesophilic and thermophilic processes has been separated into Tables 6.1-6.2 and 6.3-6.4, respectively. Table 6.4 summarises experimental data from the present work, corresponding to Chapters 4 and 5.

It should be noticed that, due to the variability between operating parameters, the comparison of data from different studies is not straightforward. However, if we look at the results obtained in studies comparing mesophilic and thermophilic performance of reactors operating under the same conditions, it seems that the efficiency of the process is similar regardless of the

temperature for SRT over 20 days; with gas production rates around  $0.3\text{-}0.4 \text{ m}^3_{\text{biogas}} \text{ m}^{-3} \text{ d}^{-1}$  and VS removals of 53 % (Gavala *et al.*, 2003; De la Rubia *et al.*, 2002). On the other hand, reducing the SRT to 15 days at 55 °C increased gas production rate by 60 % (from 0.36 to  $\sim 0.6 \text{ m}^3_{\text{biogas}} \text{ m}^{-3} \text{ d}^{-1}$ ) and VS removal by 12 % (from 41.6 to 46.3 %) compared to the mesophilic process at 20 days SRT (De la Rubia *et al.*, 2006; Benabdallah El-Hadj *et al.*, 2006). Similarly, gas production rate during thermophilic operation was 100 % and 200 % higher at low SRT of 10 and 8 days, respectively, compared to mesophilic operation at the same SRT (Laffité-Trouqué and Forster, 2002). Therefore, by operating within the thermophilic range of temperatures, it seems feasible to reduce the SRT, while increasing methane production, thus energy production.

In general, methane content in biogas ranges between 60-70 % and, in most cases, VS removal ranges between 30-60 %. Values below 30 % correspond to digesters treating WAS, in which gas production rate is also the lowest, below  $0.2 \text{ m}^3_{\text{biogas}} \text{ m}^{-3} \text{ d}^{-1}$  (Laffité-Trouqué and Forster, 2002; Bolzonella *et al.*, 2005).

Even though methane yield should be constant for a given waste, according to the results reported in the literature it clearly ranges from 0.1 to  $0.8 \text{ m}^3_{\text{CH}_4} \text{ kg VS}_{\text{removed}}^{-1}$ . This should be explained by sludge heterogeneity resulting from factors like the proportion of PS and WAS in the mixture and, in the case of WAS, the SRT in the activated sludge process (Bolzonella *et al.*, 2005), amongst others. The data on WAS indicates variability within the range of  $0.17\text{-}0.43 \text{ m}^3_{\text{CH}_4} \text{ kg VS}_{\text{removed}}^{-1}$ ; which is lower than the theoretical value calculated for biomass ( $0.5 \text{ m}^3_{\text{CH}_4} \text{ kg VS}_{\text{removed}}^{-1}$ ). On the other hand, according to the data on PS, methane yields are higher ( $0.4\text{-}0.8 \text{ m}^3_{\text{CH}_4} \text{ kg VS}_{\text{removed}}^{-1}$ ) and apparently equal to or higher than those of equivalent processes treating mixture of PS and WAS, both in the mesophilic range, 0.8 vs.  $0.3\text{-}0.5 \text{ m}^3_{\text{CH}_4} \text{ kg VS}_{\text{removed}}^{-1}$  (Krugel *et al.*, 1998; Gavala *et al.*, 2003; Benabdallah El-Hadj *et al.*, 2006) and in the thermophilic range,  $0.4\text{-}0.6 \text{ m}^3_{\text{CH}_4} \text{ kg VS}_{\text{removed}}^{-1}$  (De la Rubia *et al.*, 2006; Benabdallah El-Hadj *et al.*, 2006; Lu *et al.*, 2007).

In the work by Lu *et al.*, (2007), VS removal increased by 20 % (from 50 to 60 %), and biogas and methane production and yield by 40-60 %, as a result of implementing a hyperthermophilic step (70 °C), which is the only reference found in the literature regarding 70 °C sludge pre-treatment by means of a two-stage process. Záborská *et al.* (2000a) report an improvement by 27 % on gas production rate from two-stage thermophilic systems (55/52 °C) compared to two-stage mesophilic (38/35 °C) systems.

### 6.1.1.2. Energy considerations

On the whole, it seems that thermophilic anaerobic sludge digestion, in one or two-stage systems, and the use of low temperature pre-treatments are successful approaches to upgrade conventional mesophilic digestion. A major drawback of these alternatives is increased energy consumption. According to the study by Zupančič and Roš (2003), heat requirements in thermophilic sludge digestion are about twice those of mesophilic digestion, but they may be covered with a combined heat and power (CHP) unit fuelled with biogas, together with heat regeneration from the effluent sludge. Záborská *et al.* (2000a) report that heat requirements for two-stage thermophilic digesters are fully covered by increased biogas production; and that additionally surplus electric energy is yielded. Similarly, extra energy requirements for the operation of a thermal pre-treatment step might not only be covered but result in net energy production (Bourgrier *et al.*, 2007; Lu *et al.*, 2007).

Besides temperature considerations, some authors point out the importance of solids concentration in the feed sludge, since dilute sludges (total solids < 4.7 %) result in poorer biogas production and increased heating requirements (Speece, 1988). In such a case, digesters may not be able to self-sustain even mesophilic operation (Bolzonella *et al.*, 2005).

### 6.1.1.3. Prediction of energy production using mathematical models

The theoretical energy production of an anaerobic digester may be calculated by predicting methane production under certain operating conditions, using mathematical models. A number of complex mathematical models have been proposed during the last decades for modelling anaerobic digestion processes. Siegrist *et al.* (2002) developed a specific model for mesophilic and thermophilic anaerobic sewage sludge digestion. The Anaerobic Digestion Model No 1 (ADM1) (IWA 2002) may also be useful for predicting the behaviour of sewage sludge treatment, but the substrate has to be well characterised in terms of organic contents and biodegradability (Parker, 2005).

Hydrolysis of organic matter has generally been described by first order kinetics. Although more complex models have also been used, they are only slightly better than first order models, and therefore its use has been recommended by default by the IWA Task Group for Mathematical Modelling and Anaerobic Digestion Processes, especially when the amount of biomass in the reactor is not rate-limiting (IWA 2002).

**Table 6.1.** Operational characteristics of full-, pilot- and laboratory-scale mesophilic anaerobic digesters treating sewage sludge.

Sludge	Process	Volume m <sup>3</sup>	T °C	SRT days	OLR kg VS m <sup>-3</sup> d <sup>-1</sup>	Gas rate m <sup>3</sup> m <sup>-3</sup> d <sup>-1</sup>	CH <sub>4</sub> rate m <sup>3</sup> m <sup>-3</sup> d <sup>-1</sup>	CH <sub>4</sub> yield m <sup>3</sup> kg VS <sup>-1</sup> d <sup>-1</sup>	CH <sub>4</sub> yield m <sup>3</sup> kg VS <sub>rem</sub> <sup>-1</sup>	CH <sub>4</sub> %	TS <sub>e</sub> %	VS <sub>e</sub> %	TS <sub>i</sub> %	VS <sub>i</sub> %	VS rem %	Adapted from
PS	1 stage (CSTR)	4,800	39	19				0.775	63.0						47.00	Krugel <i>et al.</i> (1998)
PS+WAS	2 stage (CSTR+not)	4,823 × 2	38+35	12+11		0.482	0.320		66.3	2.69	54.52					Zábranská <i>et al.</i> (2000a)
	1 stage (CSTR)	3,367	37.6	21	1.000	0.180				3.8	57.90			22		
	1 stage (CSTR)		38.5	20	0.800	0.070				2.6	69.23			17		
WAS (no PT)	1 stage (CSTR)	2,200	35	33	0.800	0.080				3.5	62.86			27		Bolzonella <i>et al.</i> (2005)
	1 stage (CSTR)	2,200	34	40	0.700	0.040				3.9	64.10			13		
	1 stage (CSTR)	1,500-3,500	36	22	1.000	0.150				2.7	66.67			15		
WAS	1 stage (CSTR)	1,600	34.5	37.2	0.530	0.100	0.068	0.088	68.0	2.58	54.00	3.60	62.00			Mininni <i>et al.</i> (2006)
	1 stage (CSTR)	4,400	M	40		0.261										
PS+WAS	1 stage (CSTR)	1,800	M	35												Zábranská <i>et al.</i> (2006)
	1 stage (CSTR)	3,500-6,500	38	19	1.130	0.368										
	2 stage (CSTR+not)	4,823 × 2	38+35	6.7+18.6		0.331										
PS+WAS	1 stage (CSTR)	5,000	39	22.5	1.124	0.380	0.166	0.223	66.0	4.20	60.00	50.00				This work
PS+WAS	1 stage (CSTR)	0.15	35	27	1.300	0.360	0.250	0.192	0.363	69.4	5.50	70.90	53.00			De la Rubia <i>et al.</i> (2002)
WAS	1 stage (CSTR)	0.0005	35	10		0.084		0.041	0.153					27.00		Laffite-Trouqué and Forster (2002)
				12		0.088		0.051	0.283					18.00		
								0.048	0.252					19.00		
PS+WAS	1 stage (CSTR)	0.001	37	20	1.433	0.406	0.250	0.175	0.324	61.6	2.40	55.10	4.29	66.80	53.82	Gavala <i>et al.</i> (2003)
PS+WAS (40/60%)	1 stage (CSTR)	0.0026	37	20	1.380	0.551	0.385	0.279	0.593	69.5	3.04	47.04	4.60	58.70	47.04	Bousková <i>et al.</i> (2005)
PS+WAS (75/25%)	1 stage (CSTR)	0.005	35	20	1.380	0.515	0.355	0.257	0.560	68.8	3.25	44.92	4.60	58.70	45.93	
										63.6	2.30	57.77	2.98	76.55	41.60	Benabdallah <i>et al.</i> (2006)

PS: primary sludge; WAS: waste activated sludge; CSTR: continuous stirred tank reactor; CSTR+not: continuous stirred tank reactor and not stirred tank reactor; Gas rate: biogas production rate; CH<sub>4</sub> rate: methane production rate; TS: total solids; VS: volatile solids; VS rem: VS removal. Note: full-scale: > 1,000 m<sup>3</sup>; pilot-scale: 0.15-0.2 m<sup>3</sup>; laboratory-scale < 0.02 m<sup>3</sup>

**Table 6.2.** Operational characteristics of full-scale mesophilic and psychrophilic anaerobic digesters treating sewage sludge.

Sludge Process	Volume m <sup>3</sup>	T °C	SRT days	OLR kg VS m <sup>-3</sup> d <sup>-1</sup>	Gas rate m <sup>3</sup> m <sup>-3</sup> d <sup>-1</sup>	CH <sub>4</sub> rate m <sup>3</sup> m <sup>-3</sup> d <sup>-1</sup>	CH <sub>4</sub> yield m <sup>3</sup> kg VS <sub>fed</sub> <sup>-1</sup>	CH <sub>4</sub> yield m <sup>3</sup> kg VS <sub>rem</sub> <sup>-1</sup>	CH <sub>4</sub> %	TS <sub>e</sub> %	VS <sub>e</sub> % TS	TS <sub>i</sub> %	VS <sub>i</sub> % TS	VS rem %	Adapted from
	37,500		147	0.218								5.90	70.00	73.00	
	6,750	35.0	23	1.276	0.545							5.40	70.00	45.00	
	13,125	38.9	50	0.404	0.345							3.00	78.00	70.00	
	8,625	35.0	10	3.014	2.067							4.50	76.00		
	51,750		26	0.819	0.339							4.40	66.00	41.00	
	3,375	28.9	166	0.059	0.671							3.20	50.00		
	112,500	35.0	17	1.377	0.755							5.20	64.00	27.00	
	86,250	16.7	50	0.331	0.085							7.30	56.00		
	5,625	26.7	8	1.792								3.20	63.00		
	30,000	28.3	80	0.506								7.60	69.00	50.00	
	25,875	33.3	42	0.510	0.328							4.40	66.00	35.00	
	12,750	35.6	42	0.647	0.444							4.50	75.00	71.00	
	168,750	35.6	35	0.724	0.537							4.00	75.00	60.00	
	213,750	34.4	17	1.362	0.887							4.20	71.00	46.00	
	9,375	36.7	42	0.950	0.845							5.50	81.00	58.00	Speece (1988)
	15,375	35.0	16	0.989	0.736							24.00	76.00	53.00	
	3,750	37.2	111	0.160	0.190							3.00	75.00	82.00	
	60,000	30.0	8	2.409	0.255							4.20	66.00	71.00	
	55,500	37.8	44	0.302	0.342							5.50	67.00	58.00	
	55,500	34.4	44	0.243	0.342							3.30	75.00	58.00	
	10,500	35.0	133	0.526	0.135							7.90	89.00	80.00	
	48,750	25.6		0.538								3.20	69.00	23.00	
	40,500		28	1.073	0.678							5.40	79.00	58.00	
	108,750	35.6	24	1.063	0.677							5.00	68.00	50.00	
	24,750	35.6	18	1.386	1.715							4.50	70.00	56.00	
	9,750		17	1.138	0.464							3.30	74.00	29.00	
	3,750	28.9		4.181								6.60	56.00	27.00	
	30,000	35.0	11	4.700	0.849							8.00	75.00	72.00	
	22,500	21.1	17	0.844								5.00	50.00	56.00	

Volume: total volume of all digesters; Gas rate: biogas production rate; CH<sub>4</sub> rate: methane production rate; TS: total solids; VS: volatile solids; VS rem: VS removal

**Table 6.3.** Operational characteristics of full-, pilot- and laboratory-scale thermophilic anaerobic digesters treating sewage sludge.

Sludge	Process	Volume m <sup>3</sup>	T °C	SRT days	OLR kg VS m <sup>-3</sup> d <sup>-1</sup>	Gas rate m <sup>3</sup> m <sup>-3</sup> d <sup>-1</sup>	CH <sub>4</sub> rate m <sup>3</sup> m <sup>-3</sup> d <sup>-1</sup>	CH <sub>4</sub> yield m <sup>3</sup> kgVS <sub>fed</sub> <sup>-1</sup>	CH <sub>4</sub> yield m <sup>3</sup> kgVS <sub>rem</sub> <sup>-1</sup>	CH <sub>4</sub> %	TS <sub>e</sub> %	VS <sub>e</sub> %	TS <sub>i</sub> %	VS <sub>i</sub> %	VS rem %	Adapted from
PS+WAS	1 stage (CSTR)	12,000	54.6	21				0.626		62.6					60.00	Krugel <i>et al.</i> (1998)
	2 stage (CSTR)	3,100 × 2	55	22+22				0.420		64.6					80.00	
PS+WAS	2 stage (CSTR+not)	4,823 × 2	55+52	12+11		0.613	0.407			66.3	2.03	53.51				Zábranská <i>et al.</i> (2000a)
PS+WAS	1 stage (CSTR)	0.15	55	20	1.870	0.367	0.220	0.118	0.310	60	5.50	70.90	5.50	63.64	38.00	De la Rubia <i>et al.</i> (2002)
PS+WAS	1 stage (CSTR)	0.15	55	75	0.420	0.033	0.020	0.048	0.065	60	5.50	63.64	5.50	63.64	73.00	De la Rubia <i>et al.</i> (2006)
				40	0.800	0.283	0.170	0.213	0.313	60	5.50	63.64	5.50	63.64	68.00	
				27	1.480	0.310	0.190	0.128	0.242	65	5.50	63.64	5.50	63.64	53.00	
				20	1.870	0.400	0.260	0.139	0.65	65	5.50	63.64	5.50	63.64	49.00	
				15	2.190	0.615	0.400	0.183	0.373	65	5.50	63.64	5.50	63.64	49.00	
WAS	1 stage (CSTR)	0.2	55	18	2.190	0.120	0.080	0.107	0.174	67	2.10	58.22	3.00	62.70	61.77	Pavan <i>et al.</i> (2006)
WAS	1 stage (CSTR)	0.0005	55	8		0.246		0.093	0.321						29.00	Laffie-Trouqué and Forster (2002)
				10		0.172		0.169	0.412							
Diluted sludge	1 stage (CSTR)	0.001	55	20	1.433	0.426	0.256	0.179	0.338	60.2	2.43	55.63	4.29	66.80	52.92	Gavala <i>et al.</i> (2003)
				1	7.000	3.000	1.500	0.030	0.120	50.0	1.00	70.00	25.00			
				2	3.500	11.250	6.750	0.135	0.422	60.0	1.00	70.00	32.00			
				3	2.333	20.000	13.000	0.260	0.765	65.0	1.00	70.00	34.00			
				4	1.750	21.250	14.875	0.298	0.804	70.0	1.00	70.00	37.00			
				5	1.400	23.000	16.100	0.322	0.805	70.0	1.00	70.00	40.00			
				6	1.167	24.000	16.800	0.336	0.800	70.0	1.00	70.00	42.00			
				7	1.000	25.000	17.500	0.350	0.814	70.0	1.00	70.00	43.00			
				8	0.875	25.500	17.850	0.357	0.811	70.0	1.00	70.00	44.00			
				9	0.778	27.000	18.900	0.378	0.804	70.0	1.00	70.00	47.00			
				10	0.700	28.500	19.950	0.399	0.814	70.0	1.00	70.00	49.00			
				PS+WAS (40/60%)	1 stage (CSTR)	0.0026	55	20	1.380	0.677	0.417	0.302	0.702	66.4	3.52	45.17
20	1.380	0.688	0.451					0.327	0.742	66.0	3.55	45.35	4.60	58.70	44.00	
PS+WAS (50/50%)	1 stage (CSTR)	0.005	55	15	1.519	0.578	0.391	0.257	0.555	67.6	2.27	53.90	2.98	76.55	46.34	Benabdallah <i>et al.</i> (2006)
				21.4	1.730	0.616	0.371	0.617	60.10							
				23.4	1.560	0.613	0.399	0.689	57.90							
PS	2 stage (2 CSTR)	0.0005+0.003	70+55	2+13	0.693	0.350	0.242	0.349	0.571	69.0	0.92	43.97	1.65	63.16	61.02	Lu <i>et al.</i> (2007)
				15	0.693	0.232	0.146	0.211	0.415	63.0	1.24	41.18	1.65	63.16	50.82	

PS: primary sludge; WAS: waste activated sludge; CSTR: continuous stirred tank reactor; CSTR+not: continuous stirred tank reactor and not stirred tank reactor; Gas rate: biogas production rate; CH<sub>4</sub> rate: methane production rate; TS: total solids; VS: volatile solids; VS rem: VS removal. Note: full-scale: > 1,000 m<sup>3</sup>; pilot-scale: 0.15-0.2 m<sup>3</sup>; laboratory-scale < 0.02 m<sup>3</sup>

**Table 6.4.** Operational characteristics of laboratory-scale anaerobic digesters treating sewage sludge (data from Chapters 4 and 5).

Sludge	Process	Volume m <sup>3</sup>	T °C	SRT days	OLR kg VS m <sup>-3</sup> d <sup>-1</sup>	Gas rate m <sup>3</sup> m <sup>-3</sup> d <sup>-1</sup>	CH <sub>4</sub> rate m <sup>3</sup> m <sup>-3</sup> d <sup>-1</sup>	CH <sub>4</sub> yield m <sup>3</sup> kg VS <sup>-1</sup> fed <sup>-1</sup>	CH <sub>4</sub> yield m <sup>3</sup> kg VS <sup>rem</sup> <sup>-1</sup>	CH <sub>4</sub> %	TS <sub>c</sub>		VS <sub>c</sub>		TS <sub>i</sub>		VS <sub>i</sub>		Adapted from
											%	%	%	%	%	%	%	%	
PS+WAS (50/50%)	1 stage (CSTR)	0.005	43	36	0.442	0.173	0.121	0.297	0.289	68.6	1.21	61.91	2.08	70.70	54.28	This work (Chapter 4)			
PS+WAS (75/25%)	1 stage (CSTR)	0.005	50	30	0.479	0.134	0.082	0.160	0.419	62.0	1.44	62.35	2.16	68.65	34.66				
			50	32	0.749	0.310	0.179	0.264	0.392	65.0	1.60	65.35	3.25	72.29	55.41	This work (Chapter 4)			
			55	31	0.643	0.265	0.161	0.244	0.658	64.1	2.03	66.17	2.72	77.03	35.68				
PS+WAS (75/25%)	1 stage (CSTR)	0.005	55	15	2.063	0.645	0.404	0.196	0.459	61.9	2.10	66.51	4.14	74.72	49.38				
			55	10	3.034	0.621	0.400	0.132	0.403	64.5	2.94	66.72	3.92	73.57	34.08				
			29	30	0.465	0.180	0.080	0.168	0.398	63.6	1.31	61.76	1.96	68.90	42.18				
PS+WAS (75/25%)	1 stage (CSTR)	0.005	55	9	3.710	1.073	0.624	0.185	0.345	62.1	2.19	68.08	4.54	75.71	57.32				
			6	10	5.238	1.464	0.855	0.172	0.430	64.3	3.80	49.07	5.46	58.08	40.60				
			10	10	2.398	0.607	0.383	0.160	0.379	63.8	2.43	60.18	4.06	62.02	38.59				
PS+WAS (75/25%)	1 stage (CSTR) pret (9h 70°C) + 1 CSTR pret (24h 70°C) + 1 CSTR pret (48h 70°C) + 1 CSTR	0.005	55	10	3.008	0.630	0.400	0.150	0.440	63.7	3.12	63.94	3.85	78.07	33.23	This work (Chapter 5); Ferrer <i>et al.</i> (2008)			
			70+55	10	3.045	0.870	0.560	0.180	0.490	69.8	3.49	54.34	5.55	54.89	36.55				
			10	10	2.659	0.690	0.480	0.180	0.410	68.7	3.40	57.85	3.83	69.37	24.64				
				10	2.788	0.810	0.590	0.120	0.400	67.8	3.69	50.57	5.44	51.22	32.61				

PS: primary sludge; WAS: waste activated sludge; CSTR: continuous stirred tank reactor; Gas rate: biogas production rate; CH<sub>4</sub> rate: methane production rate; TS: total solids; VS: volatile solids; VS rem: VS removal

Furthermore, first order kinetics can also be used to predict methane production when hydrolysis is slower than acidogenesis, acetogenesis and methanogenesis; thus the rate-limiting step of the overall anaerobic digestion process (Vavilin *et al.* 2008). For a given substrate, once the first order kinetic constant is adjusted, methane production depends on the SRT. Therefore, theoretical energy production from the methane can be predicted and compared to theoretical energy consumption of the system.

### **6.1.2. Objectives**

The objective of this study was to assess, from an energy perspective, alternatives for the enhancement of anaerobic sludge digestion.

First of all, data from laboratory-, pilot- and full-scale sludge digesters were used to compare energy production and consumption (i.e. energy balance) under hypothetical operating conditions of full-scale digesters.

Secondly, a first order kinetic model was adjusted using the above mentioned data, in order to predict energy production and consumption (i.e. energy balance) in alternative scenarios and evaluate the efficiency of alternative sludge treatment systems.

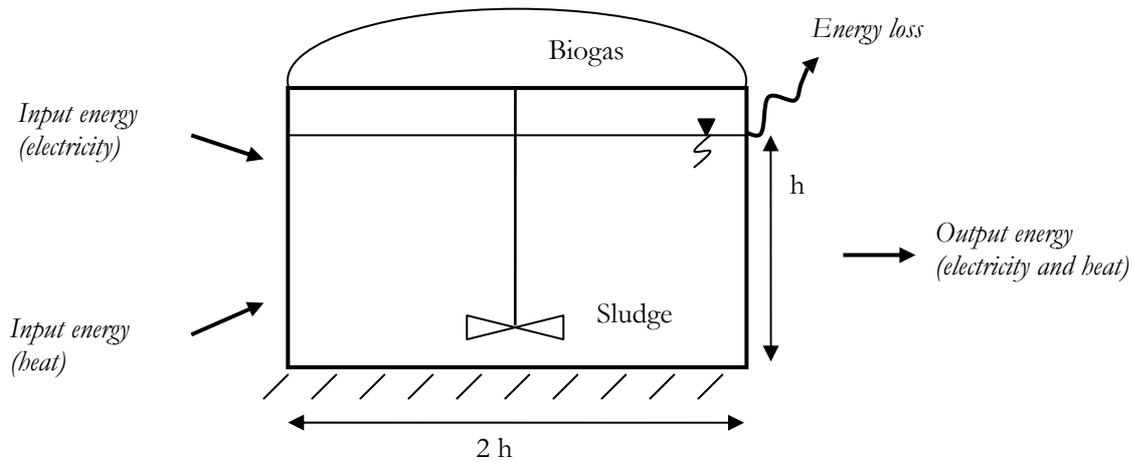
## **6.2. METHODOLOGY**

### **6.2.1. Fundamentals of the energy balance**

In anaerobic digesters, organic matter is converted into primary fuel source (biogas). This fuel source can be converted into usable energy through different processes, for example it can be burnt directly to provide heat; or it can be provided to a combined heat and power unit to produce electricity and heat. In the present study, the second alternative is considered, resulting in two forms of output energy: output electricity and output heat.

The anaerobic digesters considered are completely stirred tank reactors (CSTR), which means that input electricity is needed for sludge mixing and pumping. It is assumed that sludge digesters operate either in the mesophilic or in the thermophilic range of temperatures, thus input heat is needed to raise sludge temperature from ambient to process temperature; and to compensate for the heat loss through the walls of the digester and piping.

A schematic diagram of the energy balance and the anaerobic digester considered is shown in Figure 6.1.



**Figure 6.1.** Schematic diagram of the energy balance in the anaerobic digesters considered

## 6.2.2. Description of the systems

### 6.2.2.1. Anaerobic digesters

The digesters were designed as cylindrical tanks (Figure 6.1) with a width to eighth ratio of 2:1 (Metcalf and Eddy, 2003). The sludge volume in the digesters, or working volume ( $V$ ), was supposed to be 80 % of the total volume; leaving the remaining 20 % for gas collection under the cover of the digester. It was assumed that digestion tanks were made of concrete, wall insulation reducing the heat transfer coefficient from 5 to 1  $\text{W m}^{-2} \text{°C}^{-1}$  (Metcalf and Eddy, 2003).

All digesters were assumed to be CSTR operated in continuous mode. Thus, the calculated working volume was a function of the sludge daily flow rate ( $Q$ ) and SRT.

### 6.2.2.2. System configuration

Two system configurations were considered, namely single-stage and two-stage digestion. Furthermore, systems including a low temperature sludge pre-treatment step were also evaluated. The pre-treatment step was conceptually defined as the first digester of a two-stage process, and not as a batch pre-treatment followed by a single-stage digester.

Process temperature for the single-stage system was either mesophilic (30-40 °C) or thermophilic (50-55 °C); while two-stage systems combined a mesophilic, thermophilic or hyperthermophilic (70 °C) first step, with a mesophilic or thermophilic second step.

#### 6.2.2.3. Energy recovery

Two alternatives were assessed in terms of energy recovery: a system with energy recovery from the biogas produced and a system with energy recovery from the biogas produced and from the effluent sludge.

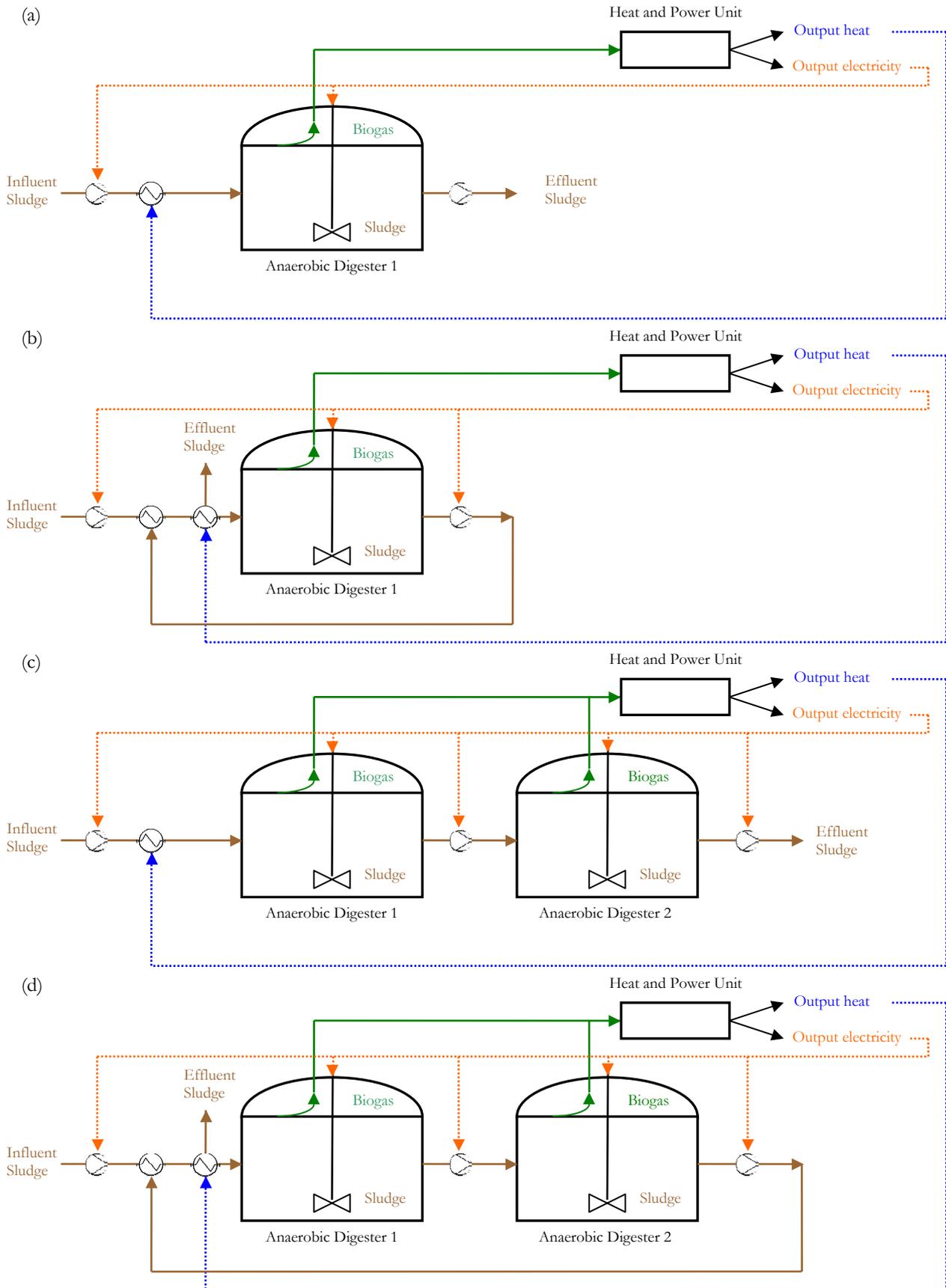
In all cases it was assumed that biogas was fuelled to a cogeneration or CHP unit, generating electricity and heat. Output electricity would cover electricity requirements for sludge pumping and mixing, whereas output heat would be used to heat influent sludge by means of a sludge-to-water heat exchanger.

In the system with heat recovery from the effluent sludge, recovered heat would be used to rise temperature up of influent sludge by means of an additional heat exchanger (i.e. a sludge-to-sludge heat exchanger); while cooling the digested sludge prior to dewatering (Krugel *et al.*, 1998).

#### 6.2.2.4. Scenarios

Based on system configuration and energy recovery, four scenarios are considered in this study. A schematic diagram of each scenario is shown in Figure 6.2:

- Scenario (a): single-stage (mesophilic or thermophilic) sludge digestion with energy recovery from the biogas produced.
- Scenario (b): single-stage (mesophilic or thermophilic) sludge digestion with energy recovery from the biogas produced and from the effluent sludge.
- Scenario (c): Two-stage (mesophilic or thermophilic) sludge digestion with energy recovery from the biogas produced.
- Scenario (d): Two-stage (mesophilic or thermophilic) sludge digestion with energy recovery from the biogas produced and from the effluent sludge.



**Figure 6.2.** Anaerobic systems considered: (a) Single-stage with energy recovery from biogas; (b) Single-stage with energy recovery from the biogas and effluent sludge; (c) Two-stage with energy recovery from biogas; (d) Two-stage with energy recovery from biogas and effluent sludge

### 6.2.3. Energy balance

#### 6.2.3.1. Input electricity

Electricity requirements were calculated according to Equation 6.1. Input electricity for the pumping of influent and effluent sludge was estimated as  $1.8 \times 10^3 \text{ kJ m}^{-3}_{\text{sludge}}$ , while that for the stirring of the digester was estimated as  $3 \times 10^2 \text{ kJ m}^{-3}_{\text{reactor}} \text{ d}^{-1}$  (Lu *et al.*, 2007).

$$E (\text{input, electricity}) = Q \theta + V \omega \quad \text{Eq. 6.1.}$$

where:  $E(\text{input, electricity})$  is the total electricity requirement ( $\text{kJ d}^{-1}$ )  
 $Q$  is the sludge daily flow rate ( $\text{m}^3_{\text{sludge}} \text{ d}^{-1}$ )  
 $V$  is the volume of sludge in the reactor, or working volume ( $\text{m}^3_{\text{reactor}}$ )  
 $\theta$  is the electrical energy consumption for pumping ( $\text{kJ m}^{-3}_{\text{sludge}}$ )  
 $\omega$  is the electrical energy consumption rate for stirring ( $\text{kJ m}^{-3}_{\text{reactor}} \text{ d}^{-1}$ )

#### 6.2.3.2. Input heat

Heat requirements were calculated using Equation 6.2; which includes the amount of heat needed to raise the influent sludge temperature to process temperature; and to compensate for heat losses through the walls of the digester and piping (Salter and Banks, 2008).

Heat requirements to raise the influent sludge temperature can be calculated assuming that sludge specific density and specific heat are essentially the same as those of water, thus  $10^3 \text{ kg m}^{-3}$  and  $4.18 \text{ kJ kg}^{-1} \text{ }^\circ\text{C}^{-1}$ , respectively (Metcalf and Eddy, 2003).

For two-stage systems, it was considered that process temperature in the second reactor was always lower than process temperature in the first reactor, which means that the second stage does not need extra heat. For example, a two-stage mesophilic process may operate at 38 /35  $^\circ\text{C}$ ; and two-stage thermophilic processes at 55/52  $^\circ\text{C}$  or 70/55  $^\circ\text{C}$ . On the contrary, in some cases the effluent of the first reactor would have to be cooled down. This energy could be recovered by means of a heat exchanger, with efficiency for heat recovery up to 85 % (Lu *et al.* 2007).

Heat losses depend on the surface area of the reactor, the heat transfer coefficient and environmental conditions. For the purposes of this study, only the heat losses through the walls of the digester were calculated, since they account for the major energy loss of the system

(Martin, 1998). Furthermore, it has been shown that heat losses of the digester represent only 2-8 % of the total heat requirements (Zupančič and Roš, 2003). The surface area of the reactor wall ( $A$ ) was then calculated for the reactor working volume (i.e. 80 % of the total volume).

$$E (\text{input, heat}) = Q \rho \gamma (T_r - T_{\text{sludge}}) (1 - \lambda) + k A (T_r - T_{\text{amb}}) 86.4 \quad \text{Eq. 6.2.}$$

where:

- $E(\text{input, heat})$  is the total heat requirement ( $\text{kJ d}^{-1}$ )
- $Q$  is the sludge daily flow rate ( $\text{m}^3_{\text{sludge}} \text{d}^{-1}$ )
- $\rho$  is the specific density of sludge ( $\text{kg m}^{-3}_{\text{sludge}}$ )
- $\gamma$  is the specific heat of sludge ( $\text{kJ kg}^{-1} \text{ } ^\circ\text{C}^{-1}$ )
- $T_r$  is process temperature ( $^\circ\text{C}$ )
- $T_{\text{sludge}}$  is influent sludge temperature ( $^\circ\text{C}$ )
- $\lambda$  is the percentage of heat recovered from effluent sludge (%)
- $T_{\text{amb}}$  is ambient temperature ( $^\circ\text{C}$ )
- $k$  is the heat transfer coefficient ( $\text{W m}^{-2} \text{ } ^\circ\text{C}^{-1}$ )
- $A$  is the surface area of the reactor wall ( $\text{m}^2$ )
- 86.4 is the conversion coefficient of W into  $\text{kJ d}^{-1}$ .

### 6.2.3.3. Output electricity and heat

Energy output depends on methane production rate, hence on organic solids removal, which in turn depends on the substrate composition (i.e. biodegradable fraction) and process operation (i.e. temperature, SRT, organic loading rate (OLR), etc.). The concentrations of organic solids in the feed sludge, together with the SRT, determine the OLR. The energy content of methane is  $35,800 \text{ kJ m}^{-3}$  (Metcalf and Eddy, 2003).

Output energy is calculated with Equation 6.3; and output electricity and heat according to Equations 6.4 and 6.5, respectively. In this study we hypothesised that all biogas produced was fuelled to a CHP unit, with conversion efficiencies of 35 % and 55 % for electricity and heat, respectively; energy loss accounting for the remaining 10 % (Zupančič and Roš, 2003).

$$E (\text{output}) = P_{\text{CH}_4} V \xi \quad \text{Eq. 6.3.}$$

$$E (\text{output, electricity}) = P_{\text{CH}_4} V \xi \eta \quad \text{Eq. 6.4.}$$

$$E (\text{output, heat}) = P_{\text{CH}_4} V \xi \psi \quad \text{Eq. 6.5.}$$

where:  $E(\text{output})$  is the total energy produced ( $\text{kJ d}^{-1}$ )  
 $P_{\text{CH}_4}$  is the methane production rate ( $\text{m}^3_{\text{CH}_4} \text{m}^{-3}_{\text{reactor}} \text{d}^{-1}$ )  
 $V$  is the volume of sludge in the reactor, or working volume ( $\text{m}^3_{\text{reactor}}$ )  
 $\xi$  is the lower heating value of methane ( $\text{kJ m}^{-3}_{\text{CH}_4}$ )  
 $E(\text{output, electricity})$  is the total electricity produced ( $\text{kJ d}^{-1}$ )  
 $\eta$  is the efficiency coefficient of the CHP unit for electricity generation (%)  
 $E(\text{output})$  is the total heat produced ( $\text{kJ d}^{-1}$ )  
 $\Psi$  is the efficiency coefficient of the CHP unit for heat generation (%)

#### 6.2.3.4. Energy balance and energy ratio

The term energy balance is used to express the difference between the energy output and input of the process, which is calculated by Equation 6.6. If we look at the energy balance in terms of electricity or heat separately, then they are calculated according to Equations 6.7 and 6.8, respectively. The results may be expressed as daily energy production and consumption ( $\text{kJ d}^{-1}$ ;  $\text{GJ d}^{-1}$ ) or as daily energy production and consumption per unit of reactor volume ( $\text{MJ d}^{-1} \text{m}^{-3}_{\text{reactor}}$ ).

$$\Delta E (\text{global}) = E (\text{output}) - E (\text{input, electricity}) - E (\text{input, heat}) \quad \text{Eq. 6.6.}$$

$$\Delta E (\text{electricity}) = E (\text{output, electricity}) - E (\text{input, electricity}) \quad \text{Eq. 6.7}$$

$$\Delta E (\text{heat}) = E (\text{output, heat}) - E (\text{input, heat}) \quad \text{Eq. 6.8.}$$

The energy ratio between output and input total energy, electricity or heat, is calculated according to Equations 6.9-6.11, respectively. This value enables to compare the efficiency of different reactors and processes (Pavan *et al.*, 2008; Salter and Banks, 2008).

$$\text{Energy ratio} = \frac{E (\text{output})}{E (\text{input, electricity}) + E (\text{input, heat})} \quad \text{Eq. 6.9.}$$

$$\text{Electricity ratio} = \frac{E(\text{output, electricity})}{E(\text{input, electricity})} \quad \text{Eq. 6.10}$$

$$\text{Heat ratio} = \frac{E(\text{output, heat})}{E(\text{input, heat})} \quad \text{Eq. 6.11.}$$

### 6.2.3.5. Summary of parameters and input data

The parameters used to calculate theoretical energy balances for full-scale digesters by extrapolating data from laboratory-, pilot- and full-scale experiences are summarised in Table 6.5. Process temperature ranged from 30 to 70 °C, while SRT ranged from 8 to 133 days; according to data reported in the literature (Tables 6-1-6.4).

The sludge daily flow rate treated was  $100 \text{ m}^3_{\text{sludge}} \text{ d}^{-1}$ . The reactor volume was not fixed, since it depended on the sludge daily flow rate and SRT. Two ambient temperatures were considered, corresponding to warm seasons (20 °C) and extreme cold seasons (0 °C) in a Mediterranean location like Barcelona Metropolitan Area. The minimum sludge temperature was assumed to be 10 °C when ambient temperature was 0 °C (Metcalf and Eddy, 2003; Zupančič and Roš, 2003).

**Table 6.5.** Parameters used for the calculation of energy balances from real data (Tables 6.1-6.4)

Parameter	Symbol	Value	Source
Process temperature (°C)	$T_r$	30-70	Literature (Tables 6.1-6.4)
SRT (d)	SRT	8-133	Literature (Tables 6.1-6.4)
Sludge daily flow rate ( $\text{m}^3_{\text{sludge}} \text{ d}^{-1}$ )	Q	100	Defined for calculation
Ambient temperature (°C)	$T_r$	0, 20	Defined for calculation
Heat transfer coefficient, insulated ( $\text{W m}^{-2} \text{ °C}^{-1}$ )	k	1	Metcalf and Eddy (2003)
Heat transfer coefficient, not insulated ( $\text{W m}^{-2} \text{ °C}^{-1}$ )	k	5	Metcalf and Eddy (2003)
Energy consumption for pumping ( $\text{kJ m}^{-3}_{\text{sludge}}$ )	$\theta$	$1.8 \times 10^3$	Lu <i>et al.</i> (2007)
Energy consumption rate for stirring ( $\text{kJ m}^{-3}_{\text{reactor}} \text{ d}^{-1}$ )	$\omega$	$3 \times 10^2$	Lu <i>et al.</i> (2007)
Specific density of sludge ( $\text{kg m}^{-3}_{\text{sludge}}$ )	$\rho$	$10^3$	Metcalf and Eddy (2003)
Specific heat of sludge ( $\text{kJ kg}^{-1} \text{ °C}^{-1}$ )	$\gamma$	4.18	Metcalf and Eddy (2003)
Lower heating value of methane ( $\text{kJ m}^{-3}$ )	$\xi$	35,800	Metcalf and Eddy (2003)
Efficiency of the CHP unit for electricity generation (%)	$\eta$	35	Zupančič and Roš (2003)
Efficiency of the CHP unit for heat generation (%)	$\psi$	55	Zupančič and Roš (2003)
Efficiency of heat recovery from effluent sludge (%)	$\lambda$	85	Lu <i>et al.</i> (2007)

## 6.2.4. First order kinetic model

### 6.2.4.1. Fitting of the first order kinetic model

The first order kinetic model for a CSTR operating under steady state conditions may be expressed by the following equations for substrate and specific methane production, when hydrolysis is the rate-limiting step (Vavilin *et al.*, 2008):

$$S_e = \frac{S_i}{1 + k_b SRT} \quad \text{Eq. 6.12.}$$

$$SP_{CH_4} = SP_o \frac{(S_i - S_e)}{S_i} = SP_o \frac{k_b SRT}{1 + k_b SRT} \quad \text{Eq. 6.13.}$$

where:

- $S_e$  is the effluent VS concentration (%)
- $S_i$  is the influent VS concentration (%)
- $k_b$  is the first order rate coefficient (d<sup>-1</sup>)
- $SRT$  is the sludge retention time (d)
- $SP_{CH_4}$  is the specific methane production (m<sup>3</sup><sub>CH<sub>4</sub></sub> kg VS<sup>-1</sup><sub>fed</sub>)
- $SP_o$  is the maximum specific methane production (m<sup>3</sup><sub>CH<sub>4</sub></sub> kg VS<sup>-1</sup><sub>fed</sub>)

The theoretical conversion coefficient of VS to methane or specific methane production ( $SP_o$ ) can be calculated from the steady state mass balance for chemical oxygen demand (COD), transforming the result into VS. From such balance, the methane produced as a result of COD conversion is 0.35 m<sup>3</sup><sub>CH<sub>4</sub></sub> kg COD<sub>removed</sub><sup>-1</sup> (at standard conditions). Since the COD of sewage sludge depends on its organic composition, a theoretical value of 1.425 kg COD kg VS<sup>-1</sup> may be used. This value is calculated using the formula C<sub>18</sub>H<sub>19</sub>NO<sub>9</sub>, which approximates the composition of organic solids in the sludge. According to this, the methane produced as a result of VS conversion is approximately 0.5 m<sup>3</sup><sub>CH<sub>4</sub></sub> kg VS<sub>removed</sub><sup>-1</sup> (at standard conditions). Alternatively, the specific methane production may be calculated using experimental data on methane production and VS removal. For sewage sludge, an average value around 0.4 m<sup>3</sup><sub>CH<sub>4</sub></sub> kg VS<sub>removed</sub><sup>-1</sup> was found.

The first order rate coefficient ( $k_b$ ) may be obtained by adjusting experimental data to Equations 6.12 and/or 6.13. In this study Equation 6.12 was used, because the amount of data available was higher for VS than for methane production, especially regarding full-scale experiences (Tables 6.1-6.4). Therefore, the  $k_b$  value for mesophilic and thermophilic sludge digestion was obtained

by adjusting Equation 6.12 with data from single-stage mesophilic and thermophilic digesters in Tables 6-1-6.4.

The quality of the fitting may be illustrated comparing the VS removal obtained from experimental data (Equation 3.3) with the VS removal predicted by the model (Equation 6.14). Equation 6.14 is obtained combining Equations 3.3 and 6.12.

$$VS_{\text{removal}}(\%) = \frac{S_i - S_e}{S_i} 100 = \frac{S_i - \frac{S_i}{1 + k_b SRT}}{S_i} 100 = 1 - \frac{1}{1 + k_b SRT} 100 \quad \text{Eq. 6.14.}$$

where:  $VS_{\text{removal}}$  is the amount of VS removed with respect to influent VS (%)  
 $S_i$  is the influent VS concentration (%)  
 $S_e$  is the effluent VS concentration (%)  
 $k_b$  is the first order rate coefficient ( $d^{-1}$ )  
 $SRT$  is the sludge retention time (d)

#### 6.2.4.2. Prediction of methane and energy production

Total energy production from methane can be calculated using Equations 6.15 and 6.16. Equation 6.15 is obtained by introducing the OLR into Equation 6.13. Equation 6.16 is obtained by combining Equations 6.15 and 6.3. Similarly, by combining Equation 6.16 with Equations 6.4 and 6.5, output electricity and output heat can be obtained.

$$P_{CH_4} = SP_{CH_4} OLR = SP_o \frac{k_b SRT}{1 + k_b SRT} OLR \quad \text{Eq. 6.15.}$$

$$E(\text{output}) = P_{CH_4} V \xi = SP_{CH_4} OLR V \xi = SP_o \frac{k_b SRT}{1 + k_b SRT} OLR V \xi \quad \text{Eq. 6.16.}$$

where:  $P_{CH_4}$  is the methane production rate ( $m^3_{CH_4} m^{-3}_{\text{reactor}} d^{-1}$ )  
 $E(\text{output})$  is the total energy produced ( $kJ d^{-1}$ )  
 $SP_{CH_4}$  is the specific methane production ( $m^3_{CH_4} kg VS^{-1}$ )  
 $OLR$  is the organic loading rate ( $kg VS^{-1} m^3_{\text{reactor}} d^{-1}$ )  
 $V$  is the volume of sludge in the reactor, or working volume ( $m^3_{\text{reactor}}$ )  
 $\xi$  is the lower heating value of methane ( $kJ m^{-3}_{CH_4}$ )

$SP_o$  is the maximum specific methane production ( $\text{m}^3_{\text{CH}_4} \text{kg VS}^{-1}_{\text{fed}}$ )

$k_b$  is the first order rate coefficient ( $\text{d}^{-1}$ )

SRT is the sludge retention time (d)

In Equation 6.16, energy production is expressed as a function of the SRT and OLR; the reactor volume depending on the sludge daily flow rate and the SRT. The anaerobic biodegradability of the substrate is reflected by the conversion coefficient of VS to product or specific methane production. In this way, once  $k_b$  is determined, energy production at different SRT and OLR can be predicted. Energy requirements are calculated as explained in Sections 6.2.3.1 and 6.2.3.2; and energy balances as explained in Section 6.2.3.4.

#### 6.2.4.3. Summary of parameters and input data

The parameters used to calculate theoretical energy balances for full-scale digesters using the predictions of the first order kinetic model are summarised in Table 6.6.

**Table 6.6.** Parameters used for the calculation of energy balances using the first order kinetic model

Parameter	Symbol	Value	Source
Process temperature ( $^{\circ}\text{C}$ )	$T_r$	35, 55	Defined for calculation
SRT (d)	SRT	5, 10, 15, 20, 25, 30	Defined for calculation
Concentration of VS in the feed sludge ( $\text{kg m}^{-3}_{\text{sludge}}$ )	Q	10, 20, 30,40	Defined for calculation
Sludge daily flow rate ( $\text{m}^3_{\text{sludge}} \text{d}^{-1}$ )	$T_r$	100	Defined for calculation
Ambient temperature ( $^{\circ}\text{C}$ )	k	0, 20	Defined for calculation
Heat transfer coefficient, insulated ( $\text{W m}^{-2} \text{ }^{\circ}\text{C}^{-1}$ )	k	1	Metcalf and Eddy (2003)
Heat transfer coefficient, not insulated ( $\text{W m}^{-2} \text{ }^{\circ}\text{C}^{-1}$ )	$\theta$	5	Metcalf and Eddy (2003)
Energy consumption for pumping ( $\text{kJ m}^{-3}_{\text{sludge}}$ )	$\varpi$	$1.8 \times 10^3$	Lu <i>et al.</i> (2007)
Energy consumption for stirring ( $\text{kJ m}^{-3}_{\text{reactor}} \text{d}^{-1}$ )	$\rho$	$3 \times 10^2$	Lu <i>et al.</i> (2007)
Specific density of sludge ( $\text{kg m}^{-3}_{\text{sludge}}$ )	$\gamma$	$10^3$	Metcalf and Eddy (2003)
Specific heat of sludge ( $\text{kJ kg}^{-1} \text{ }^{\circ}\text{C}^{-1}$ )	$\xi$	4.18	Metcalf and Eddy (2003)
Lower heating value of methane ( $\text{kJ m}^{-3}$ )	$\eta$	35,800	Metcalf and Eddy (2003)
Efficiency of the CHP unit for electricity generation (%)	$\psi$	35	Zupančič and Roš (2003)
Efficiency of the CHP unit for heat generation (%)	$\lambda$	55	Zupančič and Roš (2003)
Efficiency of heat recovery from effluent sludge (%)		85	Lu <i>et al.</i> (2007)

In this case, only single-stage processes were considered. Process temperature was assumed to be  $35^{\circ}\text{C}$  in mesophilic digesters and  $55^{\circ}\text{C}$  in thermophilic digesters. SRT in the range of 10-30 days were evaluated. The OLR depended on the concentration of organic solids in the feed sludge (10-

40 kg VS m<sup>-3</sup><sub>sludge</sub>) and the SRT; the resulting value ranging from 0.1-8 kg VS m<sup>-3</sup><sub>reactor</sub> d<sup>-1</sup>. The VS concentrations in the feed sludge were defined according to the values obtained during the previous experimental work (Chapters 4 and 5) and in the literature review (Tables 6.1-6.4).

The same as before, the sludge daily flow rate treated was 100 m<sup>3</sup><sub>sludge</sub> d<sup>-1</sup>; the reactor volume depending on the sludge daily flow rate and the SRT. Again, two ambient temperatures were considered, corresponding to warm seasons (20 °C) and extreme cold seasons (0 °C); and the minimum sludge temperature was assumed to be 10 °C (Metcalf and Eddy, 2003; Zupančič and Roš, 2003).

## 6.3. RESULTS

### 6.3.1. Assessment of anaerobic digestion systems from an energy perspective

In this Section, the results of theoretical electricity, heat and total energy balances; as well as energy ratios of single-stage and two-stage mesophilic and thermophilic sludge digesters are presented. With data from Chapters 4 and 5 (Table 6.4), theoretical performance of full-scale digesters treating the mixture of thickened PS and WAS was assessed. Data from other studies (Tables 6.1-6.2) were also used to calculate theoretical energy balances; in order to compare the performance of sludge digesters under different operating conditions (i.e. process temperature, SRT, etc.) from an energy perspective. The calculated theoretical energy inputs and outputs, energy balances and energy ratios; are summarised in the Appendix (Tables 1-24).

#### 6.3.1.1. Single-stage anaerobic digestion (Scenarios a and b)

Figures 6.3 and 6.4 show theoretical energy balances calculated with experimental results from Chapter 4, for single-stage thermophilic digesters with energy recovery from the biogas produced (Figure 6.3) and from the biogas and effluent sludge (Figure 6.4). In both Figures, graphs (a) and (b) correspond to digesters with wall insulation at ambient temperatures of 20 and 0 °C, respectively; while graphs (c) and (d) correspond to digesters without wall insulation at ambient temperatures of 20 and 0 °C, respectively.

According to theoretical calculations, sludge digestion results in surplus electricity generation. Output electricity obtained by cogeneration from the biogas produced is much higher than electricity consumption for sludge pumping and mixing; thus electricity balances are always positive and electricity ratios above 1 (Tables 6.7-6.8). Since electricity inputs and outputs do not

depend on ambient temperature or digester insulation, electricity balances and ratios are equal for each operating condition (i.e. each row in Tables 6.7-6.8).

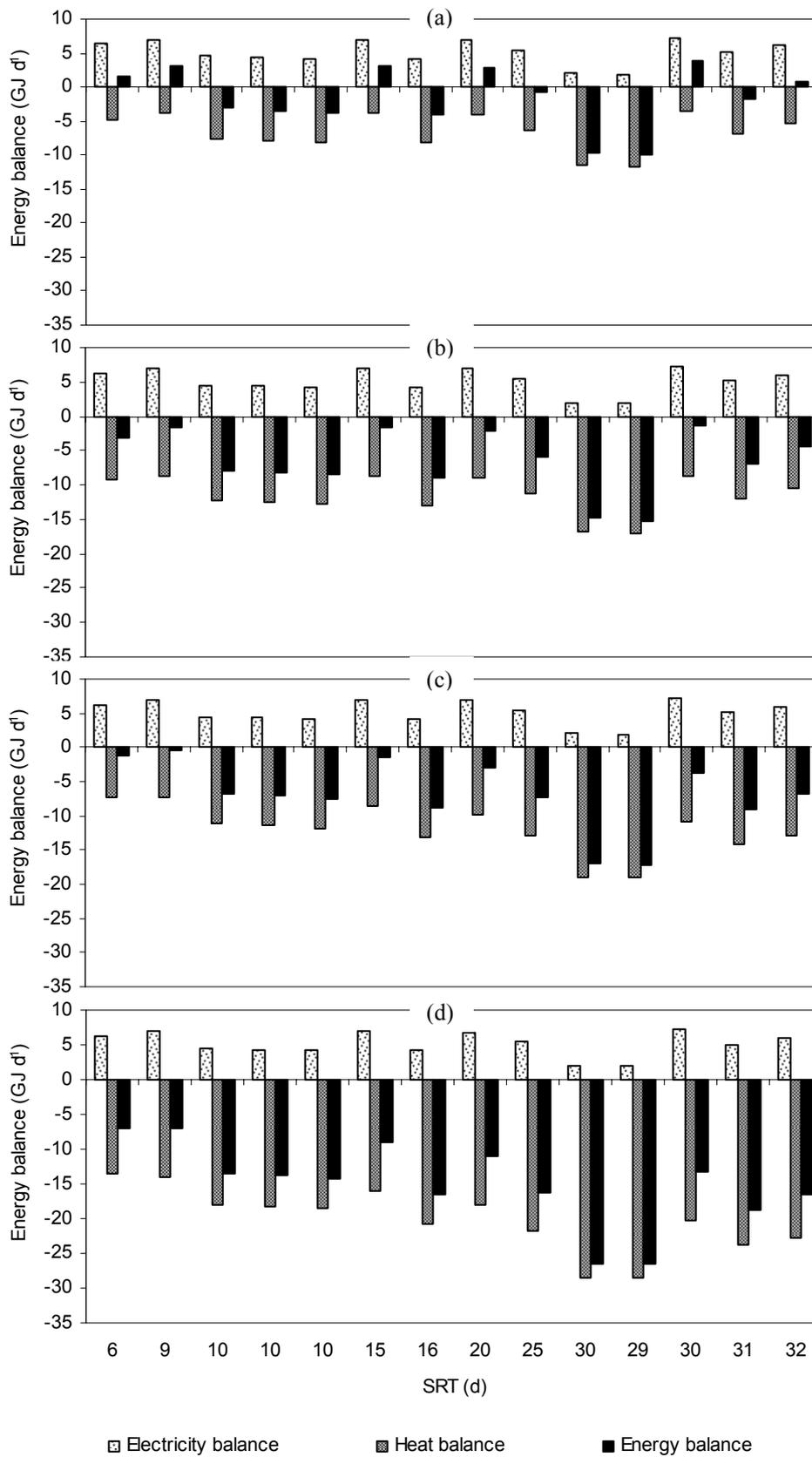
Electricity ratios are directly dependant on the methane production rate, thus they depend on the SRT and OLR. The highest electricity ratios in Tables 6.7 and 6.8 are obtained with the lowest SRT and highest OLR (i.e. SRT of 6-9 days and OLR around  $3\text{-}5 \text{ kg VS m}^{-3} \text{ reactor d}^{-1}$ ).

Contrary to electricity balances and ratios, heat balances are much affected by ambient temperature and tank insulation. As expected, the amount of input heat is directly dependant on the difference between influent sludge and process temperature; while the heat loss through the walls of the tank depends on the difference between process and ambient temperature, and tank insulation.

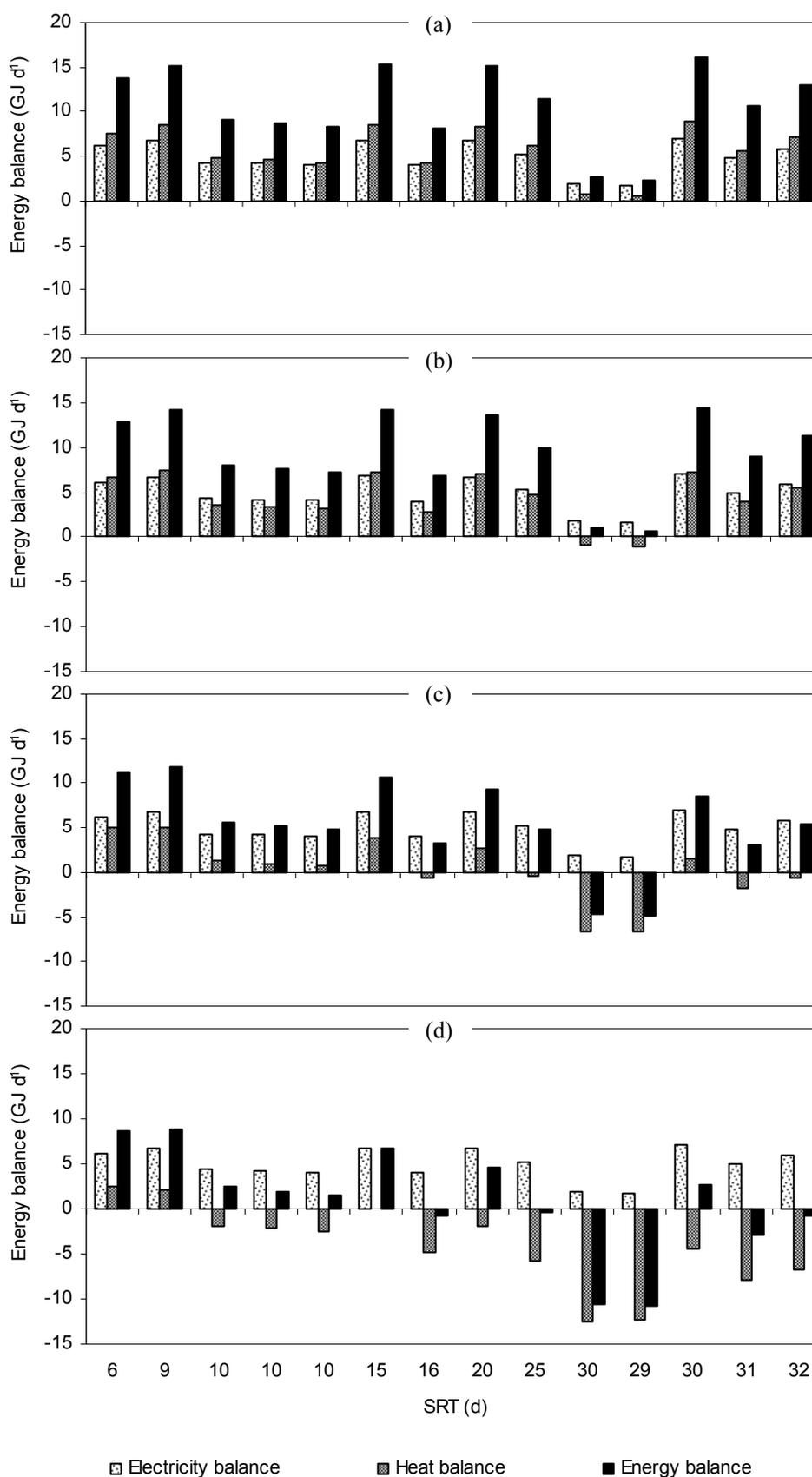
Figure 6.3 shows that all the heat balances are negative in digesters with only energy recovery from biogas. A tendency to become more negative at increasing SRT may be speculated, especially in digesters without wall insulation and at an ambient temperature of  $0 \text{ }^{\circ}\text{C}$ . As deduced from Table 6.7, given a daily flow rate ( $100 \text{ m}^3_{\text{sludge}} \text{ d}^{-1}$ ), the reactor volume increases with the SRT; resulting in higher surface area and heat loss through the walls of the digester. The results are worsened at lower OLR ( $< 0.5 \text{ kg VS m}^{-3} \text{ reactor d}^{-1}$ ), resulting in poor methane production rates ( $< 0.1 \text{ m}^3_{\text{CH}_4} \text{ m}^{-3} \text{ reactor d}^{-1}$ ), and output heat from cogeneration with biogas. Thus, heat ratios are below 1 in all cases.

The effect of wall insulation can be deduced from the comparison of graphs (a) and (c), or (b) and (d); while the effect of ambient temperature can be deduced from the comparison of graphs (a) and (b), or (c) and (d). Apparently, the heat deficit is almost doubled in reactors without insulation; and similarly occurs when ambient temperatures are  $0 \text{ }^{\circ}\text{C}$  compared to  $20 \text{ }^{\circ}\text{C}$ . The final result is that only with insulated digesters, and during warm seasons ( $20 \text{ }^{\circ}\text{C}$ ), would the thermophilic digesters studied result in net energy production when only energy recovery from biogas is considered.

Therefore, successful thermophilic sludge digestion requires energy recovery from the effluent, as suggested by Zupančič and Roš (2003). For this reason, sludge-to-sludge heat exchangers are used in full-scale plants (Krugel *et al.*, 1998).



**Figure 6.3.** Electricity, heat and total energy balance for single-stage thermophilic anaerobic digesters treating  $100 \text{ m}^3_{\text{sludge}} \text{ d}^{-1}$  at different sludge retention time (SRT) (data from Table 6.4); and with energy recovery from biogas: Digesters with wall insulation at an ambient temperature of (a)  $20 \text{ }^\circ\text{C}$  or (b)  $0 \text{ }^\circ\text{C}$ , digesters without wall insulation at an ambient temperature of (c)  $20 \text{ }^\circ\text{C}$  or (d)  $0 \text{ }^\circ\text{C}$ .



**Figure 6.4.** Electricity, heat and total energy balance for single-stage thermophilic anaerobic digesters treating  $100 \text{ m}^3_{\text{sludge}} \text{ d}^{-1}$  at different sludge retention time (SRT) (data from Table 6.4); and with energy recovery from biogas and the effluent sludge: Digesters with wall insulation at an ambient temperature of (a)  $20 \text{ }^\circ\text{C}$  or (b)  $0 \text{ }^\circ\text{C}$ , digesters without wall insulation at an ambient temperature of (c)  $20 \text{ }^\circ\text{C}$  or (d)  $0 \text{ }^\circ\text{C}$ .

If we look at Figure 6.4 corresponding to digesters with energy recovery from biogas and from the effluent sludge, almost all heat balances become positive, resulting in net energy production. This is true for all insulated digesters, both at 20 and 0 °C. For non-insulated digesters at 20 °C, the only negative values correspond to the lowest OLR; whereas at 0 °C most heat balances are negative, but the overall energy balance is neutral or positive, except for the cases of low OLR mentioned above. This is also deduced from the heat and energy ratios in Table 6.8.

### 6.3.1.2. Two-stage anaerobic digestion

Figure 6.5 shows the electricity, heat and total energy balances of the two-stage system corresponding to Chapter 5. It consists of a first 70 °C step, with a SRT of 9, 24 or 48h; and a second step at 55 °C with a SRT of 10 days. Graph (a) corresponds to the system with energy recovery from the biogas and graph (b) to energy recovery from the biogas and from the effluent sludge. Within each graph, the balances for ambient temperatures of 20 and 0 °C, for digesters with and without wall insulation are shown.

Again, electricity balances are always positive. In spite of higher electricity consumption for sludge pumping and mixing in a two-stage system, output electricity is still much higher than input electricity. It should be noticed that, as earlier mentioned, electricity balances are equal for each operating condition.

Also, all heat balances and overall energy balances are negative when only energy from biogas is recovered (Figure 6.5 (a)). Although results are similar for all 70 °C SRT, they are slightly poorer for the 24 h step, which is in accordance with lower methane production rate ( $0.56-0.59$  vs.  $0.48$   $\text{m}^3_{\text{CH}_4} \text{m}^{-3}_{\text{reactor}} \text{d}^{-1}$ ) in Table 6.4.

Nevertheless, when energy recovery from the effluent sludge is also accounted for (Figure 6.5 (b)), all balances become positive; except for the non-insulated reactor at 0 °C, which has a negative heat balance but positive overall balance, due to surplus electricity generation. At 20 °C without digester insulation, energy production is almost half of that with insulated digesters, corroborating the necessity of digester insulation. Provided that digesters are insulated, ambient temperature (0-20 °C) has little effect on net energy production. In this case, the major part of heat requirements would be for sludge heating. By heat recovery from the effluent, external energy requirements are reduced, hence net energy production results from the stabilisation of sludge in such system.

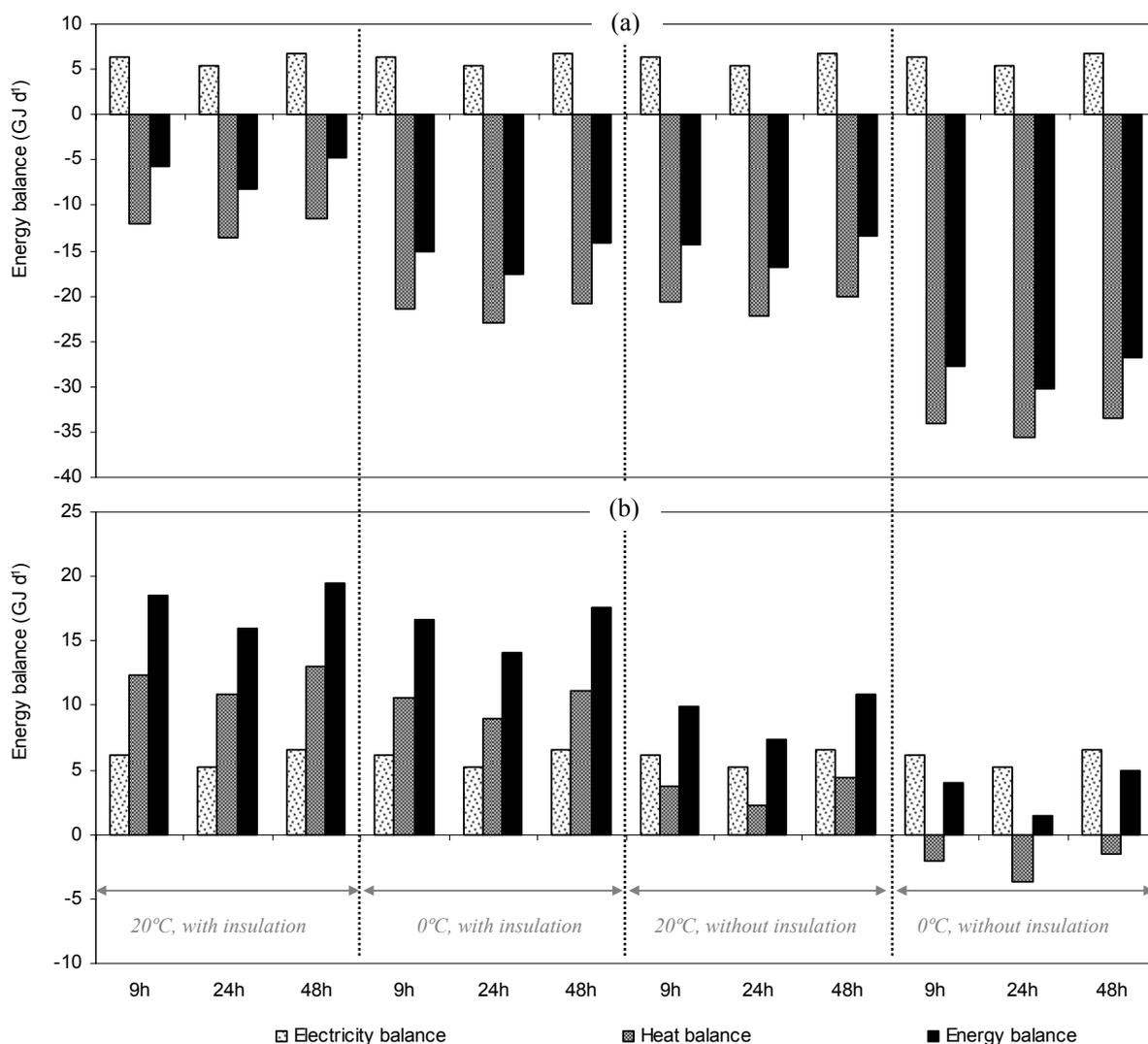
**Table 6.7.** Energy ratios calculated for single-stage thermophilic digesters with energy recovery from biogas (data from Table 6.4, corresponding to Chapter 4).

N <sup>o</sup>	Experimental conditions and results				100 m <sup>3</sup> sludge d <sup>-1</sup>			Insulated; T <sub>ext</sub> = 20 °C			Insulated; T <sub>ext</sub> = 0 °C			Non-insulated; T <sub>ext</sub> = 20 °C			Non-insulated; T <sub>ext</sub> = 0 °C			
	Sludge	T (°C)	SRT (d)	OLR (kg VS m <sup>-3</sup> d <sup>-1</sup> )	CH <sub>4</sub> rate (m <sup>3</sup> CH <sub>4</sub> m <sup>-3</sup> d <sup>-1</sup> )	V (m <sup>3</sup> )	Surface area (m <sup>2</sup> )	Energy ratio	Electricity ratio	Heat ratio	Energy ratio	Electricity ratio	Heat ratio	Energy ratio	Electricity ratio	Heat ratio	Energy ratio	Electricity ratio	Heat ratio	Reference
1	PS+WAS	55	6	5.238	0.855	623	214	1.22	18.19	0.69	0.94	18.19	0.53	1.05	18.19	0.59	0.79	18.19	0.44	
2	PS+WAS	55	9	3.710	0.624	941	281	1.32	15.91	0.75	1.02	15.91	0.57	1.09	15.91	0.61	0.81	15.91	0.45	
3	PS+WAS	55	10	3.034	0.400	997	292	0.89	10.43	0.51	0.69	10.43	0.39	0.73	10.43	0.41	0.54	10.43	0.30	
4	PS+WAS	55	10	2.398	0.383	1,012	295	0.87	10.04	0.49	0.67	10.04	0.38	0.71	10.04	0.40	0.53	10.04	0.30	
5	PS+WAS	55	10	1.650	0.363	1,039	301	0.84	9.61	0.48	0.65	9.61	0.37	0.69	9.61	0.39	0.51	9.61	0.29	
6	PS+WAS	55	15	2.063	0.404	1,504	384	1.32	12.06	0.76	1.02	12.06	0.58	1.03	12.06	0.59	0.76	12.06	0.43	
7	PS+WAS	55	16	1.381	0.241	1,603	401	0.84	7.31	0.48	0.65	7.31	0.37	0.65	7.31	0.37	0.48	7.31	0.27	
8	PS+WAS	55	20	1.045	0.299	2,043	472	1.30	9.65	0.75	1.00	9.65	0.57	0.97	9.65	0.55	0.71	9.65	0.40	
9	PS+WAS	55	25	0.972	0.201	2,541	545	1.06	6.78	0.62	0.82	6.78	0.47	0.77	6.78	0.44	0.56	6.78	0.32	
10	PS+WAS	55	30	0.479	0.082	3,029	613	0.51	2.86	0.30	0.39	2.86	0.23	0.36	2.86	0.20	0.26	2.86	0.15	
11	PS+WAS	55	29	0.465	0.080	2,911	597	0.48	2.79	0.28	0.37	2.79	0.21	0.34	2.79	0.19	0.25	2.79	0.14	
12	PS+WAS	55	30	0.694	0.218	3,033	614	1.35	7.62	0.79	1.04	7.62	0.60	0.95	7.62	0.55	0.69	7.62	0.39	
13	PS+WAS	55	31	0.643	0.161	3,087	621	1.01	5.61	0.59	0.78	5.61	0.45	0.71	5.61	0.41	0.51	5.61	0.29	
14	PS+WAS	55	32	0.749	0.179	3,208	637	1.16	6.30	0.68	0.89	6.30	0.52	0.81	6.30	0.47	0.59	6.30	0.33	This work

**Table 6.8.** Energy ratios calculated for single-stage thermophilic digesters with energy recovery from biogas and from the effluent sludge (data from Table 6.4, corresponding to Chapter 4).

N <sup>o</sup>	Experimental conditions and results				100 m <sup>3</sup> sludge d <sup>-1</sup>		Insulated; T <sub>ext</sub> = 20 °C		Insulated; T <sub>ext</sub> = 0 °C		Non-insulated; T <sub>ext</sub> = 20 °C		Non-insulated; T <sub>ext</sub> = 0 °C		Reference				
	Sludge	T (°C)	SRT (d)	OLR (kg VS m <sup>-3</sup> d <sup>-1</sup> )	CH <sub>4</sub> rate (m <sup>3</sup> CH <sub>4</sub> m <sup>-3</sup> d <sup>-1</sup> )	V (m <sup>3</sup> )	Surface area (m <sup>2</sup> )	Energy ratio	Electricity ratio	Heat ratio	Energy ratio	Electricity ratio	Heat ratio	Energy ratio		Electricity ratio	Heat ratio		
1	PS+WAS	55	6	5.238	0.855	623	214	5.63	12.21	3.69	4.35	12.21	2.73	3.19	12.21	1.93	2.26	12.21	1.33
2	PS+WAS	55	9	3.710	0.624	941	281	5.70	11.45	3.80	4.38	11.45	2.78	2.96	11.45	1.79	2.07	11.45	1.22
3	PS+WAS	55	10	3.034	0.400	997	292	3.82	7.58	2.55	2.93	7.58	1.86	1.96	7.58	1.19	1.37	7.58	0.80
4	PS+WAS	55	10	2.398	0.383	1,012	295	3.70	7.32	2.47	2.84	7.32	1.81	1.89	7.32	1.15	1.32	7.32	0.78
5	PS+WAS	55	10	1.650	0.363	1,039	301	3.58	7.04	2.39	2.74	7.04	1.75	1.82	7.04	1.10	1.27	7.04	0.75
6	PS+WAS	55	15	2.063	0.404	1,504	384	5.22	9.39	3.56	3.98	9.39	2.57	2.47	9.39	1.49	1.70	9.39	1.00
7	PS+WAS	55	16	1.381	0.241	1,603	401	3.25	5.75	2.23	2.48	5.75	1.61	1.52	5.75	0.92	1.05	5.75	0.61
8	PS+WAS	55	20	1.045	0.299	2,043	472	4.76	7.87	3.32	3.62	7.87	2.38	2.12	7.87	1.29	1.46	7.87	0.86
9	PS+WAS	55	25	0.972	0.201	2,541	545	3.68	5.70	2.61	2.79	5.70	1.86	1.58	5.70	0.96	1.08	5.70	0.64
10	PS+WAS	55	30	0.479	0.082	3,029	613	1.67	2.45	1.21	1.27	2.45	0.85	0.70	2.45	0.43	0.48	2.45	0.28
11	PS+WAS	55	29	0.465	0.080	2,911	597	1.60	2.38	1.15	1.22	2.38	0.81	0.67	2.38	0.41	0.46	2.38	0.27
12	PS+WAS	55	30	0.694	0.218	3,033	614	4.46	6.54	3.22	3.38	6.54	2.27	1.86	6.54	1.14	1.27	6.54	0.75
13	PS+WAS	55	31	0.643	0.161	3,087	621	3.31	4.83	2.40	2.51	4.83	1.69	1.38	4.83	0.84	0.94	4.83	0.56
14	PS+WAS	55	32	0.749	0.179	3,208	637	3.78	5.44	2.74	2.87	5.44	1.93	1.56	5.44	0.96	1.07	5.44	0.63

This work



**Figure 6.5.** Electricity, heat and total energy balance versus 70 °C pre-treatment time, in two-stage anaerobic digesters (70 °C at 9, 24 or 48 h of SRT/55 °C at 10 days of SRT) treating 100 m<sup>3</sup>sludge d<sup>-1</sup>; with energy recoveries from: (a) biogas and (b) biogas and the effluent sludge.

### 6.3.1.3. Comparison of the process under different operating conditions

So far, the results suggest that thermophilic sludge digestion in insulated digesters and with energy recovery from both the biogas produced and the effluent sludge, results in net energy production regardless of ambient temperature (0-20 °C).

If we compare the two-stage (70/55 °C) system (Figure 6.5) with single-stage (55 °C) sludge digestion at SRT around 10 days (Figures 6.3 and 6.4), it seems that higher energy would be obtained with the former, which is in accordance with higher methane production rates (Table 6.4). Other authors have suggested surplus energy production through a hyperthermophilic/

thermophilic two step process treating primary sludge, at SRT of 2 and 13 days, suggesting that even lower SRT be used for the methanogenic digester (Lu *et al.*, 2007).

If we compare the results with data of mesophilic and thermophilic two-stage systems in Tables 6.1-6.4, the highest net energy production (almost double) is obtained with the results of the present work, which correspond to the lowest SRT for the first and second stage reactors. This should be taken into account, since lowering the SRT enables to reduce the reactor volume, hence its capital cost, and consequently the costs of sludge management and wastewater treatment. Throughout this work it has been shown that thermophilic sludge treatment at SRT as low as 10 days results in stable and efficient performance.

If we look at the energy ratios calculated for single-stage digesters using data from Tables 6.1 and 6.2, which are shown in Tables 6.9 and 6.10 for systems with energy recovery from biogas and from the effluent sludge, respectively; it seems that similar energy production would be expected from thermophilic digesters operating at SRT of 10-20 days and mesophilic digesters operating at SRT of 20 days. Again, this means that through thermophilic operation, either smaller reactors can be used, or higher sludge flow rates treated, whilst maintaining the energetic efficiency of the process.

Regarding the type of sludge, energy ratios in Tables 6.9 and 6.10 are consistently higher for digesters treating the mixture of PS and WAS, compared to digesters treating only WAS, both under mesophilic and thermophilic conditions. See for example the value for thermophilic digestion of WAS at 18 days SRT (N° 25), versus PS and WAS at 15-20 days SRT (N° 35-41).

Since anaerobic biodegradability and methane production rate are higher for PS compared to WAS, the proportion between them in the sludge mixture is expected to affect its maximum biodegradability and reaction rate (Gavala *et al.*, 2003). This may account for some differences between reactors operating at the same temperature, SRT and OLR; but different sludge composition. Furthermore, Bolzonella *et al.* (2005) found a relationship between the SRT in the activated sludge process and the specific gas production during mesophilic anaerobic digestion of WAS, showing that the higher the activated sludge SRT, the lower the specific gas production. As a result, digesters treating WAS as a sole substrate, with low VS content and low specific methane production, might not be able to self-sustain process temperature, even in the mesophilic range, especially during cold seasons.

**Table 6.9.** Energy ratios calculated for single-stage digesters with energy recovery from biogas (data from Tables 6.1 and 6.2).

N°	Experimental conditions and results					Insulated; T <sub>ext</sub> =20 °C			Insulated; T <sub>amb</sub> =0 °C			Non-insulated; T <sub>amb</sub> =20 °C			Non-insulated; T <sub>amb</sub> =0 °C			Reference			
	Sludge	T (°C)	SRT (d)	OLR (kg VS m <sup>-3</sup> d <sup>-1</sup> )	CH <sub>4</sub> rate (m <sup>3</sup> CH <sub>4</sub> m <sup>-3</sup> d <sup>-1</sup> )	V (m <sup>3</sup> )	Surface area (m <sup>2</sup> )	100 m <sup>3</sup> sludge d <sup>-1</sup>	Energy ratio	Electricity ratio	Heat ratio	Energy ratio	Electricity ratio	Heat ratio	Energy ratio	Electricity ratio	Heat ratio				
15	WAS	35	8	1.000	0.055	800	252	800	0.22	1.30	0.13	0.13	1.30	0.08	0.19	1.30	0.11	0.11	1.30	0.06	Laffite-Trouqué and Forster (2002)
16	WAS	35	10	1.000	0.057	1,000	293	1,000	0.29	1.49	0.17	0.17	1.49	0.10	0.24	1.49	0.14	0.14	1.49	0.08	
17	WAS	37.6	21	1.000	0.117	2,100	480	2,100	0.99	3.80	0.60	0.63	3.80	0.37	0.74	3.80	0.44	0.44	3.80	0.25	
18	WAS	38.5	20	0.800	0.046	2,000	465	2,000	0.35	1.46	0.21	0.23	1.46	0.13	0.27	1.46	0.16	0.16	1.46	0.09	Bolzonella <i>et al.</i> (2005)
19	WAS	36	22	0.800	0.098	2,200	495	2,200	0.93	3.20	0.57	0.58	3.20	0.34	0.70	3.20	0.42	0.42	3.20	0.23	
20	WAS	35	33	0.700	0.052	3,300	649	3,300	0.74	1.84	0.48	0.45	1.84	0.27	0.53	1.84	0.32	0.32	1.84	0.17	
21	WAS	34.5	37.2	0.530	0.068	3,720	703	3,720	1.10	2.45	0.72	0.66	2.45	0.40	0.77	2.45	0.48	0.48	2.45	0.24	Mininni <i>et al.</i> (2006)
22	WAS	34	40	1.000	0.026	4,000	738	4,000	0.46	0.94	0.30	0.27	0.94	0.17	0.32	0.94	0.20	0.20	0.94	0.10	Bolzonella <i>et al.</i> (2005)
23	WAS	55	8	1.000	0.160	800	252	800	0.29	3.82	0.16	0.22	3.82	0.13	0.24	3.82	0.14	0.14	3.82	0.10	Laffite-Trouqué and Forster (2002)
24	WAS	55	10	1.000	0.112	1,000	293	1,000	0.25	2.92	0.14	0.19	2.92	0.11	0.20	2.92	0.12	0.12	2.92	0.09	
25	WAS	55	18	1.000	0.080	1,800	433	1,800	0.31	2.51	0.18	0.24	2.51	0.14	0.24	2.51	0.13	0.13	2.51	0.10	Pavan <i>et al.</i> (2006)
26	PS	55	15	0.693	0.146	1,500	384	1,500	0.48	4.36	0.27	0.37	4.36	0.21	0.37	4.36	0.21	0.21	4.36	0.15	Lu <i>et al.</i> (2007)
27	n.d.	38	19	1.130	0.241	1,900	449	1,900	1.82	7.63	1.09	1.17	7.63	0.68	1.39	7.63	0.82	0.82	7.63	0.47	Zábranská <i>et al.</i> (2006)
28	PS+WAS	35	20	1.140	0.229	2,000	465	2,000	2.14	7.36	1.31	1.30	7.36	0.76	1.63	7.36	0.97	0.97	7.36	0.52	Benabdallah <i>et al.</i> (2006)
28	PS+WAS	37	20	1.433	0.250	2,000	465	2,000	2.09	8.04	1.26	1.32	8.04	0.77	1.58	8.04	0.94	0.94	8.04	0.53	Gavala <i>et al.</i> (2003)
30	PS+WAS	37	20	1.380	0.355	2,000	465	2,000	2.96	11.39	1.79	1.87	11.39	1.09	2.25	11.39	1.33	1.33	11.39	0.75	Bousková <i>et al.</i> (2005)
31	PS+WAS	37	20	1.380	0.385	2,000	465	2,000	3.21	12.36	1.94	2.03	12.36	1.19	2.44	12.36	1.44	1.44	12.36	0.81	
32	PS+WAS	39	22.5	1.124	0.166	2,250	503	2,250	1.39	5.46	0.84	0.91	5.46	0.53	1.03	5.46	0.61	0.61	5.46	0.36	This work
33	PS+WAS	35	27	1.300	0.250	2,700	568	2,700	3.02	8.54	1.90	1.84	8.54	1.09	2.21	8.54	1.34	1.34	8.54	0.70	De la Rubia <i>et al.</i> (2002)
34	PS+WAS	43	36	0.442	0.121	3,600	688	3,600	1.27	4.33	0.78	0.89	4.33	0.52	0.88	4.33	0.52	0.52	4.33	0.32	Ferrer <i>et al.</i> (2008)
35	PS+WAS	55	15	1.519	0.391	1,500	384	1,500	1.28	11.66	0.73	0.99	11.66	0.56	1.00	11.66	0.56	0.56	11.66	0.41	Benabdallah <i>et al.</i> (2006)
36	PS+WAS	55	15	2.190	0.400	1,500	384	1,500	1.31	11.93	0.75	1.01	11.93	0.57	1.02	11.93	0.58	0.58	11.93	0.42	De la Rubia <i>et al.</i> (2006)
37	PS+WAS	55	20	1.870	0.220	2,000	465	2,000	0.94	7.07	0.54	0.72	7.07	0.41	0.70	7.07	0.40	0.40	7.07	0.29	De la Rubia <i>et al.</i> (2002)
38	PS+WAS	55	20	1.433	0.257	2,000	465	2,000	1.09	8.24	0.63	0.84	8.24	0.48	0.82	8.24	0.47	0.47	8.24	0.34	Gavala <i>et al.</i> (2003)
39	PS+WAS	55	20	1.870	0.260	2,000	465	2,000	1.11	8.35	0.64	0.85	8.35	0.49	0.83	8.35	0.47	0.47	8.35	0.34	De la Rubia <i>et al.</i> (2006)
40	PS+WAS	55	20	1.380	0.417	2,000	465	2,000	1.77	13.38	1.02	1.37	13.38	0.78	1.33	13.38	0.76	0.76	13.38	0.55	
41	PS+WAS	55	20	1.380	0.451	2,000	465	2,000	1.92	14.48	1.11	1.48	14.48	0.84	1.44	14.48	0.82	0.82	14.48	0.59	Bousková <i>et al.</i> (2005)
42	PS+WAS	55	27	1.480	0.190	2,700	568	2,700	1.06	6.49	0.62	0.82	6.49	0.47	0.76	6.49	0.44	0.44	6.49	0.31	
43	PS+WAS	55	40	0.800	0.170	4,000	738	4,000	1.33	6.17	0.79	1.03	6.17	0.60	0.90	6.17	0.52	0.52	6.17	0.37	De la Rubia <i>et al.</i> (2006)
44	PS+WAS	55	75	0.420	0.020	7,500	1,122	7,500	0.26	0.77	0.16	0.20	0.77	0.12	0.16	0.77	0.09	0.09	0.77	0.06	

**Table 6.10.** Energy ratios calculated for single-stage digesters with energy recovery from biogas and from the effluent sludge (data from Tables 6.1 and 6.2).

N <sup>o</sup>	Experimental conditions and results				100 m <sup>3</sup> sludge d <sup>-1</sup>		Insulated; T <sub>amb</sub> =0 °C		Non-insulated; T <sub>amb</sub> =20 °C		Non-insulated; T <sub>amb</sub> =0 °C		Reference						
	Sludge	T (°C)	SRT (d)	OLR (kg VS m <sup>-3</sup> d <sup>-1</sup> )	CH <sub>4</sub> rate (m <sup>3</sup> CH <sub>4</sub> m <sup>-3</sup> d <sup>-1</sup> )	V (m <sup>3</sup> )	Surface area (m <sup>2</sup> )	Energy ratio	Electricity ratio	Heat ratio	Energy ratio	Electricity ratio		Heat ratio					
15	WAS	35	8		0.055	800	252	0.84	0.91	0.68	0.53	0.91	0.37	0.49	0.91	0.33	0.26	0.91	0.16
16	WAS	35	10		0.057	1,000	293	1.03	1.09	0.85	0.66	1.09	0.46	0.59	1.09	0.40	0.31	1.09	0.19
17	WAS	37.6	21	1.000	0.117	2,100	480	3.11	3.11	2.64	2.05	3.11	1.47	1.53	3.11	1.02	0.84	3.11	0.51
18	WAS	38.5	20	0.800	0.046	2,000	465	1.14	1.19	0.94	0.76	1.19	0.54	0.56	1.19	0.37	0.31	1.19	0.19
19	WAS	36	22	0.800	0.098	2,200	495	2.84	2.63	2.50	1.83	2.63	1.33	1.41	2.63	0.95	0.74	2.63	0.45
20	WAS	35	33	0.700	0.052	3,300	649	1.96	1.59	1.90	1.26	1.59	0.96	0.95	1.59	0.66	0.48	1.59	0.30
21	WAS	34.5	37.2	0.530	0.068	3,720	703	2.77	2.15	2.78	1.77	2.15	1.37	1.33	2.15	0.94	0.67	2.15	0.41
22	WAS	34	40	1.000	0.026	4,000	738	1.12	0.84	1.16	0.71	0.84	0.56	0.54	0.84	0.38	0.27	0.84	0.17
23	WAS	55	8		0.160	800	252	1.29	2.67	0.85	0.99	2.67	0.63	0.69	2.67	0.42	0.49	2.67	0.29
24	WAS	55	10		0.112	1,000	293	1.07	2.12	0.71	0.82	2.12	0.52	0.55	2.12	0.33	0.38	2.12	0.23
25	WAS	55	18		0.080	1,800	433	1.17	2.00	0.81	0.89	2.00	0.58	0.53	2.00	0.32	0.37	2.00	0.22
26	PS	55	15	0.693	0.146	1,500	384	1.88	3.39	1.29	1.44	3.39	0.93	0.89	3.39	0.54	0.62	3.39	0.36
27	n.d.	38	19	1.130	0.241	1,900	449	5.93	6.16	4.92	3.93	6.16	2.78	2.95	6.16	1.95	1.63	6.16	0.99
28	PS+WAS	35	20	1.140	0.229	2,000	465	6.55	5.98	5.84	4.17	5.98	3.03	3.34	5.98	2.28	1.72	5.98	1.05
28	PS+WAS	37	20	1.433	0.250	2,000	465	6.61	6.53	5.63	4.33	6.53	3.10	3.29	6.53	2.20	1.78	6.53	1.08
30	PS+WAS	37	20	1.380	0.355	2,000	465	9.37	9.26	7.99	6.13	9.26	4.39	4.67	9.26	3.12	2.52	9.26	1.53
31	PS+WAS	37	20	1.380	0.385	2,000	465	10.17	10.04	8.66	6.65	10.04	4.76	5.06	10.04	3.38	2.73	10.04	1.66
32	PS+WAS	39	22.5	1.124	0.166	2,250	503	4.37	4.51	3.64	2.93	4.51	2.09	2.10	4.51	1.38	1.18	4.51	0.71
33	PS+WAS	35	27	1.300	0.250	2,700	568	8.49	7.23	7.93	5.42	7.23	4.05	4.17	7.23	2.88	2.13	7.23	1.31
34	PS+WAS	43	36	0.442	0.121	3,600	688	3.67	3.79	3.05	2.57	3.79	1.85	1.60	3.79	1.04	0.96	3.79	0.58
35	PS+WAS	55	15	1.519	0.391	1,500	384	5.04	9.07	3.44	3.85	9.07	2.48	2.38	9.07	1.44	1.65	9.07	0.97
36	PS+WAS	55	15	2.190	0.400	1,500	384	5.16	9.28	3.52	3.94	9.28	2.54	2.44	9.28	1.48	1.68	9.28	0.99
37	PS+WAS	55	20	1.870	0.220	2,000	465	3.45	5.74	2.41	2.63	5.74	1.72	1.55	5.74	0.94	1.06	5.74	0.62
38	PS+WAS	55	20	1.433	0.257	2,000	465	4.03	6.70	2.81	3.07	6.70	2.01	1.80	6.70	1.09	1.24	6.70	0.73
39	PS+WAS	55	20	1.870	0.260	2,000	465	4.08	6.79	2.84	3.11	6.79	2.04	1.83	6.79	1.11	1.26	6.79	0.74
40	PS+WAS	55	20	1.380	0.417	2,000	465	6.54	10.87	4.56	4.98	10.87	3.26	2.93	10.87	1.78	2.01	10.87	1.18
41	PS+WAS	55	20	1.380	0.451	2,000	465	7.08	11.77	4.93	5.39	11.77	3.53	3.17	11.77	1.92	2.18	11.77	1.28
42	PS+WAS	55	27	1.480	0.190	2,700	568	3.61	5.49	2.58	2.75	5.49	1.83	1.54	5.49	0.94	1.05	5.49	0.62
43	PS+WAS	55	40	0.800	0.170	4,000	738	4.07	5.46	3.02	3.09	5.46	2.12	1.63	5.46	1.00	1.11	5.46	0.66
44	PS+WAS	55	75	0.420	0.020	7,500	1,122	0.66	0.72	0.53	0.50	0.72	0.36	0.25	0.72	0.15	0.17	0.72	0.10

Process enhancement by feeding concentrated sludge is not only a matter of OLR, since equal OLR can be obtained with more or less diluted sludges depending on the daily flow rate. Up to a limit, concentrated sludges result in higher solids destruction and increased methane production rate, while consuming the same input energy for an equal SRT. Indeed, in the survey carried out by Speece (1988), diluted sludges were identified as a major root cause of several negative impacts on digester operation, including reduced SRT, reduced VS destruction, reduced methane generation, reduced alkalinity, increased volumes of digested sludge, increased costs for digested sludge post-treatment and disposal, and increased heating requirements.

### 6.3.2. First order kinetic model

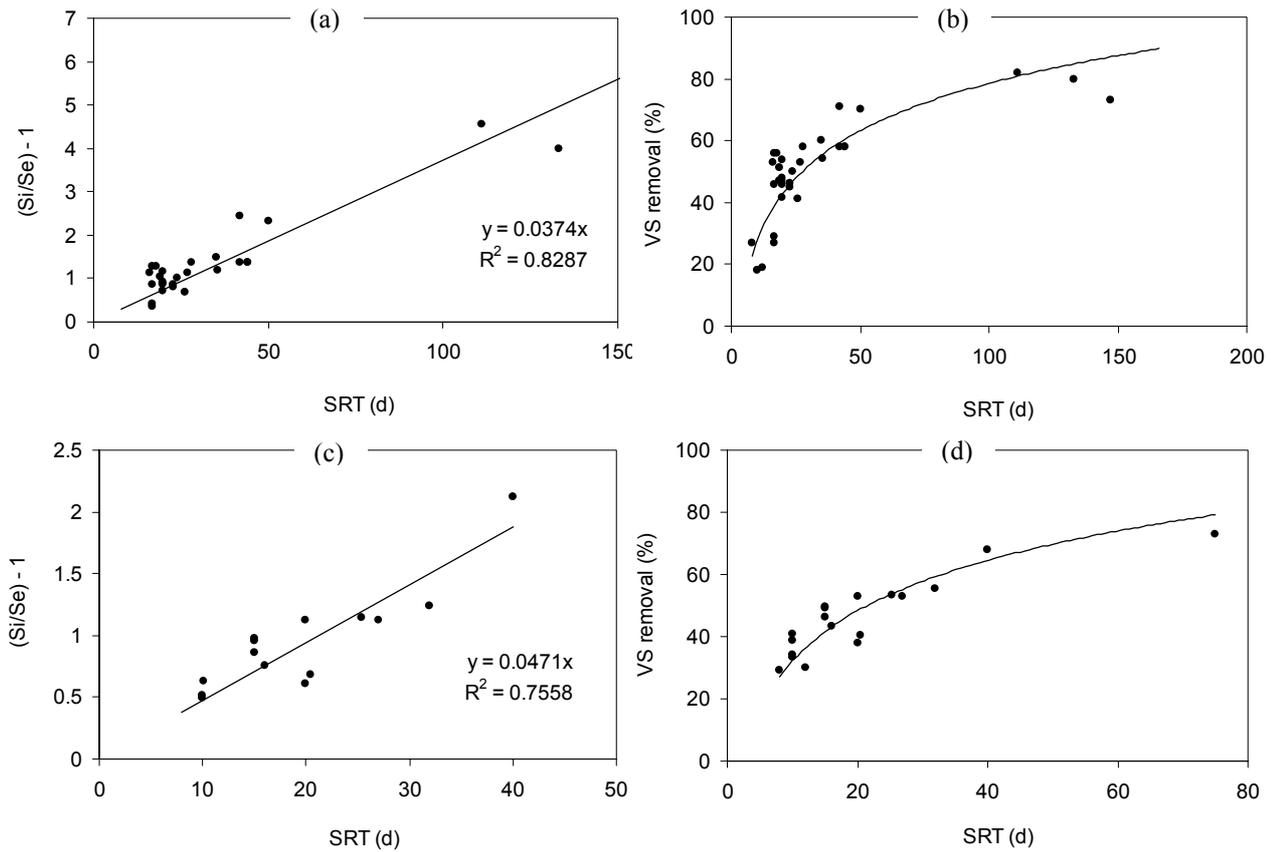
#### 6.3.2.1. Fitting of the first order kinetic model

The first order rate coefficients ( $k_b$ ) for single-stage mesophilic and thermophilic anaerobic sludge digestion were determined using experimental data from Tables 6.1-6.4. By linearising Equation 6.12 and plotting it versus the SRT, the slope of the curve corresponds to  $k_b$ , as shown in Figure 6.6 (a) and (c) for mesophilic and thermophilic digestion, respectively. Graphs (b) and (d) show VS removal versus SRT, obtained from experimental data on mesophilic and thermophilic sludge digestion (Tables 6.1-6.4). The curve represents VS removal predicted by the model, calculated according to Equation 6.11.

According to Figure 6.6 (a) and (c), the values of  $k_b$  were approximately  $0.037 \text{ d}^{-1}$  and  $0.047 \text{ d}^{-1}$  for mesophilic and thermophilic digestion of PS and WAS mixture, respectively. It should be mentioned here that data corresponding to digestion of diluted sludge, and PS or WAS as solely substrates, was not used; in order to reduce the variability of substrate composition. Nevertheless, when these data were also included, it was observed that  $k_b$  values ranged between  $0.035\text{-}0.04 \text{ d}^{-1}$  for the mesophilic process and between  $0.045\text{-}0.05 \text{ d}^{-1}$  for the thermophilic process; depending on the data set used. Thus, the aforementioned  $0.037 \text{ d}^{-1}$  and  $0.047 \text{ d}^{-1}$  were representative of single-stage mesophilic and thermophilic processes, respectively.

#### 6.3.2.2. Prediction of methane production

Methane production as a function of SRT was predicted by substituting the  $k_b$  values determined into Equation 6.15, for VS concentrations in the feed sludge in the range of  $10\text{-}40 \text{ kg VS m}^{-3} \text{ sludge}$ . The results are shown in Figure 6.7.

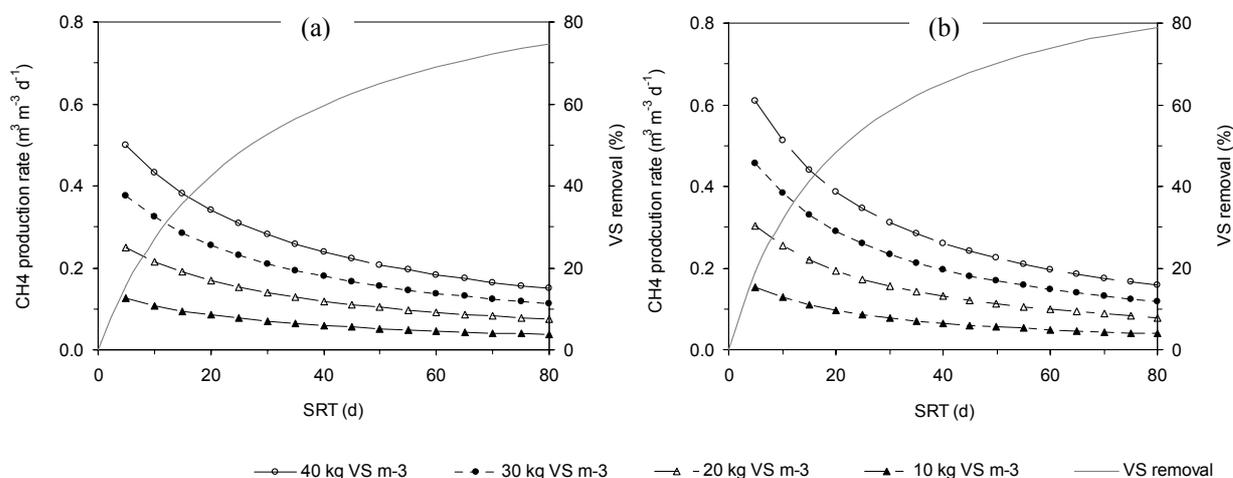


**Figure 6.6.** Fitting of the first order kinetic model with experimental data from single-stage anaerobic digestion of PS and WAS: (a) Linearised Equation 6.9, the slope of the curve represents the mesophilic first order rate coefficient; (b) Experimental and predicted values of VS removal vs. SRT during mesophilic digestion; (c) Linearised Equation 6.9, the slope of the curve represents the thermophilic first order rate coefficient; (d) Experimental and predicted values of VS removal vs. SRT during thermophilic digestion

The conversion coefficient of VS to methane calculated theoretically was  $0.5 \text{ m}^3_{\text{CH}_4} \text{ kg VS}^{-1}$ . However, such value was calculated assuming that all organic solids in the sludge were cells, with a theoretical composition approximated by the formula  $\text{C}_{18}\text{H}_{19}\text{NO}_9$  and a theoretical COD of  $1.425 \text{ kg COD kg VS}^{-1}$ . The conversion coefficient calculated from experimental results may differ from the theoretical value, especially when the proportion of PS in the sludge mixture is high. Indeed, the average conversion coefficient calculated from Tables 6.1-6.4 was around  $0.4 \text{ m}^3_{\text{CH}_4} \text{ kg VS}^{-1}$ , which corresponds to 80 % of the theoretical value. Thus, the empirical value of  $0.4 \text{ m}^3_{\text{CH}_4} \text{ kg VS}^{-1}$  replaced the theoretical value of  $0.5 \text{ m}^3_{\text{CH}_4} \text{ kg VS}^{-1}$  in this model.

According to this model, methane production rate decreases exponentially with SRT. Therefore, it might be speculated that, as long as SRT above the minimum or washout SRT are considered (i.e.  $\text{SRT} > 5$  days); the lower the SRT, the higher the daily methane production. For a given SRT;

the higher the VS concentration in the feed sludge, the higher the OLR and daily methane production; the upper limit for the OLR depending on the operating conditions.



**Figure 6.7.** Predicted methane production rate and volatile solids (VS) removal, using first order kinetics with first order rate coefficients ( $k_d$ ) of 0.037 d<sup>-1</sup> for mesophilic (a) and 0.047 d<sup>-1</sup> for thermophilic (b) anaerobic digestion of PS and WAS; influent VS: 10-40 kg m<sup>-3</sup>; conversion coefficient: 0.4 m<sup>3</sup><sub>CH<sub>4</sub></sub> kg VS<sup>-1</sup>

Since methane production rate increases proportionally to the OLR (Equation 6.15), which in turn increases proportionally to the influent VS concentration and SRT, the effect of changing the influent VS concentration is more pronounced at short SRT. For example, in thermophilic digesters, reducing the influent VS from 40 to 10 kg VS m<sup>-3</sup><sub>sludge</sub> would decrease methane production rate from 0.51 to 0.13 m<sup>3</sup><sub>CH<sub>4</sub></sub> m<sup>-3</sup><sub>reactor</sub> d<sup>-1</sup> working at 10 days SRT; and from 0.39 to 0.10 m<sup>3</sup><sub>CH<sub>4</sub></sub> m<sup>-3</sup><sub>reactor</sub> d<sup>-1</sup> working at 20 days SRT. Although the percentage of reduction is the same, the absolute value of methane, thus energy production, would be reduced to a higher extent at lower SRT.

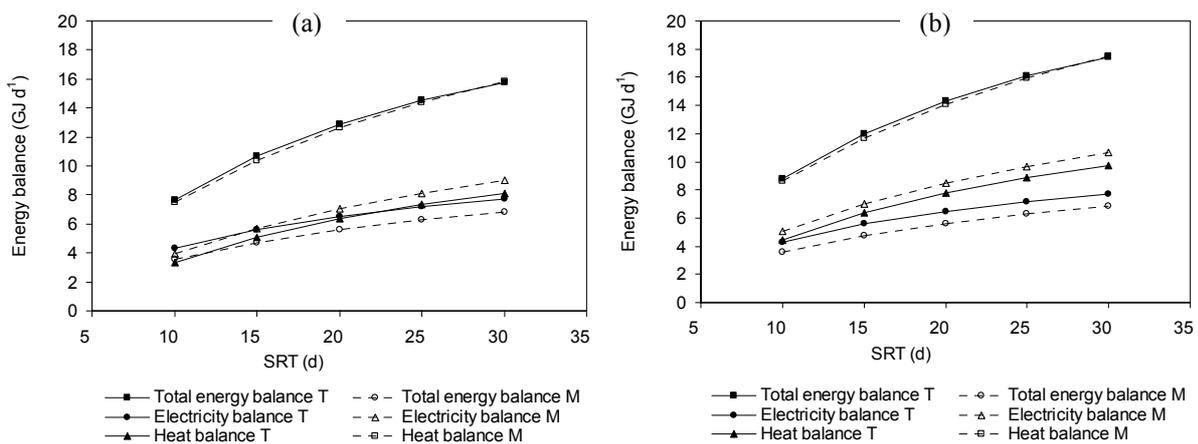
Similarly, VS removal (%) does not depend on the OLR (Equation 6.14), although the amount of VS removed (g VS<sub>removed</sub>) does. Therefore, the curve shown in Figure 6.7 (a) and (b) applies to any influent VS concentration for mesophilic and thermophilic processes, respectively. According to this model, the SRT required for 50 % VS removal would be 27 and 21 days under mesophilic and thermophilic conditions, respectively.

From Figure 6.7 it might be speculated that similar results (i.e. methane production rate and VS removal) would be obtained at thermophilic temperatures with SRT some 5-6 days lower, compared to mesophilic temperatures.

6.3.2.3. Prediction of energy production and energy balance

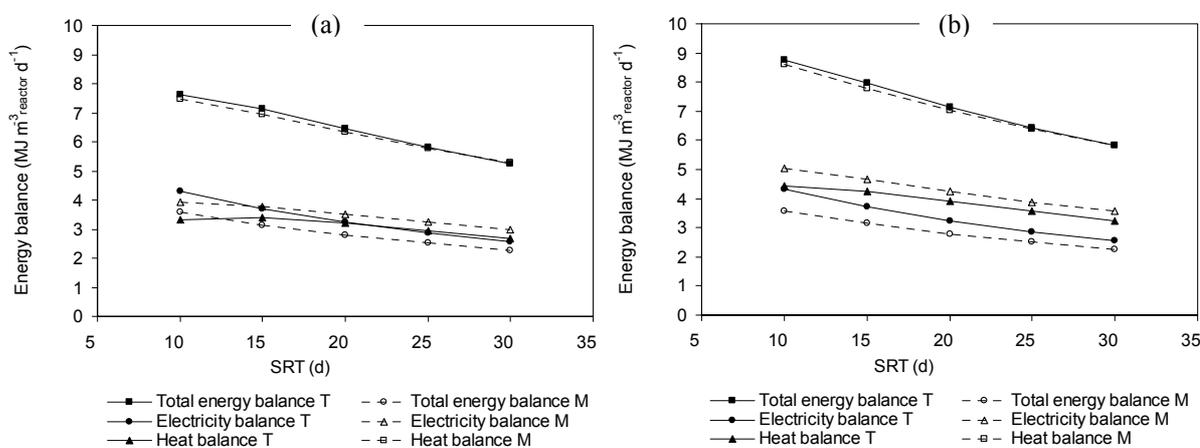
Finally, theoretical energy output was calculated with Equation 6.16, and theoretical energy balances and ratios as explained in Section 6.2.3; using the predicted methane production rate for SRT of 10-30 days and a sludge daily flow rate of  $100 \text{ m}^3_{\text{sludge}} \text{ d}^{-1}$ .

Figure 6.8 shows the expected energy balances for single-stage thermophilic and mesophilic digesters treating  $100 \text{ m}^3 \text{ d}^{-1}$  of thickened PS and WAS, with an organic solids concentration of  $30 \text{ g VS kg}^{-1}$ . It is assumed that the digesters are insulated and energy is recovered from both the biogas produced and the effluent sludge. Ambient temperatures represent cold seasons (a) and warm seasons (b). From Figure 6.8 it is clear that net energy production increases with the SRT and thus the digester volume. In spite of the decrease in methane production rate at increasing SRT (Figure 6.7), energy production is still higher than energy consumption, and therefore the bigger the amount of sludge in the digester, the higher the energy production. However, if we look at energy production per unit of digester volume (Figure 6.9), it is evident that the energetic efficiency decreases with SRT.



**Figure 6.8.** Electricity, heat and total energy balance of single-stage thermophilic (T) and mesophilic (M) anaerobic digesters treating  $100 \text{ m}^3_{\text{sludge}} \text{ d}^{-1}$  at different SRT; and with energy recoveries from biogas and effluent sludge. The digesters are insulated and ambient temperature is: (a)  $0^\circ \text{C}$  and (b)  $20^\circ \text{C}$

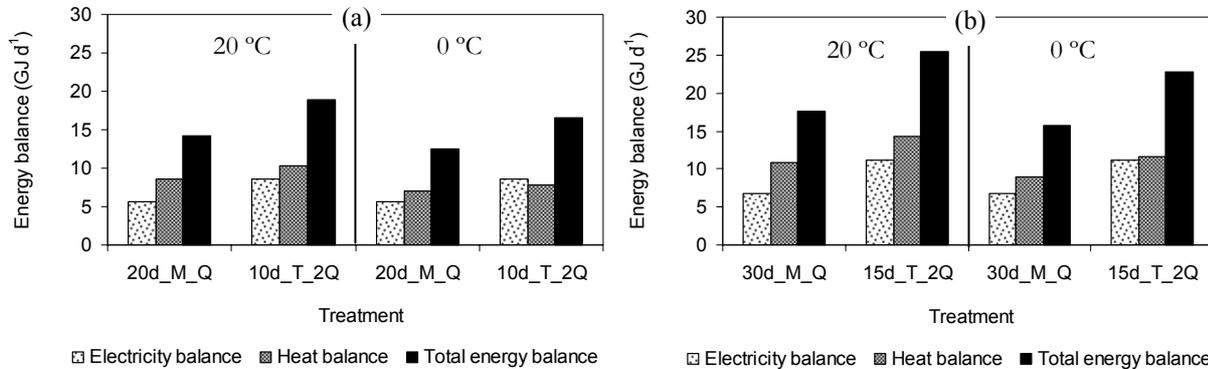
This means that, for a given sludge daily flow rate requiring stabilisation, provided that digesters are insulated and that energy is recovered from both the biogas and the effluent sludge, the higher the SRT and reactor volume, the higher the net energy production, but also the capital cost. On the other hand, shorter SRT and smaller reactors are more efficient and less costly. Theoretically, little differences exist between mesophilic and thermophilic systems from an energetic point of view. In practise, SRT as low as 10 days may not be feasible at mesophilic temperatures, since the growth rates of mesophilic methanogens require a minimum SRT around 15 days (Metcalf and Eddy, 2003). On the contrary, thermophilic systems would be feasible at this low SRT, enabling to maximise energy production per unit of reactor volume and cost.



**Figure 6.9.** Electricity, heat and total energy balance per unit of working volume in single-stage thermophilic (T) and mesophilic (M) anaerobic digesters treating 100 m<sup>3</sup> sludge d<sup>-1</sup> at different SRT; and with energy recoveries from biogas and effluent sludge. The digesters are insulated and ambient temperature is: (a) 0° C and (b) 20 °C

An example for the comparison of mesophilic and thermophilic digesters with the same working volume is given in Figure 6.10. In such Figure, the energy balance of a mesophilic reactor treating a sludge flow rate  $Q$  (100 m<sup>3</sup> d<sup>-1</sup>) at 20 days SRT, is plot beside the energy balance of a thermophilic reactor treating a sludge flow rate  $2Q$  (200 m<sup>3</sup> d<sup>-1</sup>) at 10 SRT (Figure 6.10 (a)). Similarly, the energy balance of a mesophilic reactor treating a sludge flow rate  $Q$  (100 m<sup>3</sup> d<sup>-1</sup>) at 30 days SRT, is plot beside the energy balance of a thermophilic reactor treating a sludge flow rate  $2Q$  (200 m<sup>3</sup> d<sup>-1</sup>) at 15 SRT (Figure 6.10 (b)). This enables the comparison between digesters with the same working volume: thermophilic at 10 days SRT vs. mesophilic at 20 days SRT; and thermophilic at 15 days SRT vs. mesophilic at 30 days SRT.

From Figure 6.10, it seems that thermophilic reactors treating twice the sludge daily flow rate (2Q) of mesophilic reactors (Q) with the same working volume would be similarly efficient in terms of surplus energy production.



**Figure 6.10.** Electricity, heat and total energy balance in single-stage anaerobic digesters treating 100 m<sup>3</sup>sludge d<sup>-1</sup> (Q) at SRT of 20 or 30 days under mesophilic conditions; and treating 200 m<sup>3</sup>sludge d<sup>-1</sup> (2Q) at SRT of 10 or 15 days under thermophilic conditions. Energy is recovered from both the biogas and effluent sludge. The digesters are insulated and ambient temperature is 0° C or 20 °C

## 6.4. CONCLUSIONS

Anaerobic digestion of sewage sludge was evaluated from an energy perspective. The performance of single-stage and two-stage mesophilic and thermophilic digesters working at a range of SRT and VS concentrations in the feed sludge, with and without wall insulation, with energy recovery from the biogas produced (through cogeneration) and from the effluent sludge, was assessed. This study highlights the following conclusions:

- (1) Thermophilic anaerobic sludge digestion would result in net energy production, during cold and warm seasons, provided that digesters with wall insulation and with energy recovery from both the biogas produced and the effluent sludge are used.
- (2) In this case, the energetic efficiency would be similar for thermophilic digesters working at half the SRT (10-15 days) of mesophilic digesters (20-30 days), meaning that the sludge daily flow rate could be doubled, or the reactor volume reduced, with subsequent savings in terms of sludge and wastewater treatment costs.

- (3) Two-stage systems (70/55 °C) may result in higher net energy production compared to single-stage systems (55 °C). Since the 70 °C step increases methane production in the 55 °C step, energy output is also increased, while energy requirements are similar, provided that the digesters are insulated and that energy is recovered from both the biogas produced and the effluent sludge.
- (4) The amount of surplus energy generated increases with digester volume. In spite of the decrease in methane production rate at increasing SRT, energy production is still higher than energy consumption, and therefore the bigger the amount of sludge in the digester, the higher the energy production.
- (5) The efficiency tends to increase in proportion with the VS concentration in the feed sludge. Therefore, feeding highly concentrated sludges is a way of increasing net energy production, as long as the equipment for pumping, mixing, etc, withstands this increase. At the same time, the digestion of the mixture of PS and WAS is more efficient compared to the digestion of solely WAS, which would hardly result in net energy production during cold seasons.



## **Chapter 7. Conclusions and future work**

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## Chapter 7. CONCLUSIONS AND FUTURE WORK

The experimental results presented in this PhD thesis were obtained by operating two lab-scale reactors for almost two years. Previous work included the design and implementation of the experimental set-up (MSc Thesis). Additionally, batch anaerobic tests were carried out with a device designed specifically for the study.

During this period, the effect of process temperature, sludge retention time (SRT), organic loading rate (OLR) and 70 °C sludge pre-treatment on the anaerobic digestion of sewage sludge was studied. The process was evaluated in terms of energy production (i.e. biogas and methane production) and the quality of the effluent sludge (i.e. volatile solids (VS) and volatile fatty acids (VFA) content, sludge dewaterability and hygienisation). Focus was put on the stability of the process at decreasing SRT and increasing OLR. Process efficiency during stable performance under each operating condition assayed was compared. Finally, the results were assessed from an energy perspective, by means of theoretical energy balances and ratios; and compared to the results obtained with experimental data from other studies. A first order kinetic model was also used.

The conclusions that can be drawn from the different issues dealt in this work are:

- (1) During anaerobic sludge digestion, the transition from a mesophilic (43 °C) to a thermophilic operation (50 °C) may be carried out without disturbing the process, by operating the reactors at high SRT ( $SRT \geq 30$  days) and low OLR ( $OLR \leq 0.5 \text{ kg VS m}^{-3}_{\text{reactor}} \text{ d}^{-1}$ ). Under such conditions, some VFA accumulation ( $0.5\text{-}2.5 \text{ g L}^{-1}$ ) and enhanced pathogen destruction (residual *E. coli*  $\leq 10^2 \text{ CFU mL}^{-1}$ ) would be the main differences of thermophilic (50-55 °C) compared to mesophilic (38-43 °C) reactors. Thermophilic sludge digestion at 50 °C and 55 °C should be similar in terms of biogas production and effluent stabilisation, hygienisation and dewaterability; provided that other process parameters are the same.
- (2) Methane production rate tends to increase proportionally to the OLR, thus to the SRT and VS concentration in the feed sludge. Similarly, the quality of the effluent sludge (VS content, VFA content, and sludge dewaterability) is also affected by the OLR. According to the results obtained at 55 °C, methane production rate increased by 2-3 times (from 0.2 to 0.4-

$0.6 \text{ m}^3_{\text{CH}_4} \text{ m}^3_{\text{reactor}} \text{ d}^{-1}$ ) by decreasing the SRT from 30 to 15-10 days, while increasing the OLR from 0.5 to 2.5-3.5  $\text{kg VS m}^3_{\text{reactor}} \text{ d}^{-1}$ . However, process unbalance resulted from subsequent SRT reduction to 6 days, with OLR above 5  $\text{kg VS m}^3_{\text{reactor}} \text{ d}^{-1}$ . The following concentrations might be useful to detect and prevent digester failure during thermophilic sludge digestion: total VFA ( $2.5 \text{ g L}^{-1}$ ), acetate ( $0.5 \text{ g L}^{-1}$ ), acetate/propionate ratio (0.5), intermediate alkalinity ( $1.8 \text{ g CaCO}_3 \text{ L}^{-1}$ ), intermediate alkalinity/partial alkalinity ratio (0.9), intermediate alkalinity/total alkalinity ratio (0.5), methane content in biogas (55 %).

- (3) The low temperature ( $70 \text{ }^\circ\text{C}$ ) sludge pre-treatment may initially promote sludge solubilization, increasing the concentration of soluble to total organic matter from 5 % to 50 % within 9-24 h; which is followed by a progressive VFA generation after 24 h. Subsequent anaerobic digestion of pre-treated sludge samples (9-48 h) should increase biogas production by 30-40 %, working at  $55 \text{ }^\circ\text{C}$  with a SRT of 10 days. Biogas yield is some 30 % higher with pre-treated sludge ( $0.28\text{-}0.30$  vs.  $0.22 \text{ L gVS}_{\text{fed}}^{-1}$ ) and methane content in biogas is also higher with pre-treated sludge (69 % vs. 64 %).
- (4) Thermophilic anaerobic sludge digestion would result in net energy production, during cold and warm seasons, provided that digesters with wall insulation and with energy recovery from both the biogas produced and the effluent sludge are used. In this case, the energetic efficiency would be similar for thermophilic digesters working at half the SRT (10-15 days) of mesophilic digesters (20-30 days), meaning that the sludge daily flow rate could be doubled, or the reactor volume reduced, with subsequent savings in terms of sludge treatment costs. Furthermore, two-stage systems ( $70/55 \text{ }^\circ\text{C}$ ) may result in higher net energy production compared to single-stage ( $55 \text{ }^\circ\text{C}$ ) systems at 10 days SRT. However, the amount of surplus energy generated increases with digester volume. In spite of the decrease in methane production rate at increasing SRT, energy production is still higher than energy consumption, and therefore the bigger the amount of sludge in the digester, the higher the energy production.

The overall conclusions and suggested future work can be summarised as follows:

In practise, there are little differences in terms of output energy production between mesophilic and thermophilic reactors treating sewage sludge under the same conditions (i.e. sludge daily flow rate, OLR, SRT, etc.).

However, the SRT can only be reduced to 10-15 days in thermophilic reactors, while in mesophilic reactors the minimum SRT is around 15-20 days. Therefore, the only way of working at the minimum stable SRT is by operating under thermophilic conditions. This allows for reactor volume reduction (i.e. capital cost), or sludge daily flow rate increase, at expenses of reducing the quality of the effluent sludge. Therefore, additional post-treatment may be required prior to sludge final disposal.

On the other hand, the higher the SRT and reactor volume, the higher the surplus energy production. Since sludge stabilisation is also higher at long SRT, it seems that working at high SRT and bigger reactor volumes, is the best way of optimising sewage sludge digestion.

In addition, effluent sludge hygienisation is only fulfilled in thermophilic reactors.

In this context, thermophilic anaerobic digestion would be of interest in the following situations:

- (1) To reduce the capital cost of the digester by reducing the reactor volume.
- (2) To increase the sludge daily flow rate of an existing reactor.
- (3) To ensure sludge hygienisation.

If there are no economical and spatial constraints, long SRT and reactors should be more efficient in terms of energy production; while operating under thermophilic conditions would be desirable for pathogen destruction.

An integrated approach suggests the use of the Life Cycle Assessment (LCA) methodology to compare and select the most appropriate solution for each particular case. Future studies will be focused on this topic.

The energetic assessment should be improved by using data from full-scale digesters in wastewater treatment plants. In spite of the challenges involved in accessing accurate and reliable data, this approach would provide more realistic results. Finally, an economic assessment ought to be performed.



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## Appendix

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**Table 1.** Calculations for the results from Chapter 4 (Table 6.4), in an insulated digester with energy recovery from biogas, at 20 °C ( $Q = 100 \text{ m}^3 \text{ sludge d}^{-1}$ ;  $T_{\text{amb}} = 20 \text{ °C}$ ; Heat transfer coefficient = 1)

N <sup>o</sup>	Sludge	T	SRT	OLR	CH <sub>4</sub> rate	V	Surface area	Input electricity	Input heat	Heat loss	Output energy	Output electricity	Output heat	Energy balance	Electricity balance	Heat balance	Energy ratio	Electricity ratio	Heat ratio	Reference
		°C	d	kg VS m <sup>-3</sup> d <sup>-1</sup>	m <sup>3</sup> CH <sub>4</sub> m <sup>-3</sup> d <sup>-1</sup>	m <sup>3</sup>	m <sup>2</sup>	MJ d <sup>-1</sup> m <sup>-3</sup>												
1	PS+WAS	55	6	5.238	0.855	623	214	0.59	23.49	1.04	30.62	10.72	16.84	2.44	10.13	-7.69	1.22	18.19	0.69	
2	PS+WAS	55	9	3.710	0.624	941	281	0.49	15.54	0.90	22.33	7.82	12.28	3.16	7.32	-4.16	1.32	15.91	0.75	
3	PS+WAS	55	10	3.034	0.400	997	292	0.48	14.68	0.89	14.32	5.01	7.88	-3.16	4.53	-7.69	0.89	10.43	0.51	
4	PS+WAS	55	10	2.398	0.383	1,012	295	0.48	14.46	0.88	13.71	4.80	7.54	-3.48	4.32	-7.80	0.87	10.04	0.49	
5	PS+WAS	55	10	1.650	0.363	1,039	301	0.47	14.08	0.87	13.00	4.55	7.15	-3.73	4.08	-7.81	0.84	9.61	0.48	
6	PS+WAS	55	15	2.063	0.404	1,504	384	0.42	9.73	0.77	14.47	5.06	7.96	2.10	4.64	-2.55	1.32	12.06	0.76	
7	PS+WAS	55	16	1.381	0.241	1,603	401	0.41	9.12	0.76	8.61	3.01	4.74	-2.54	2.60	-5.15	0.84	7.31	0.48	This work
8	PS+WAS	55	20	1.045	0.299	2,043	472	0.39	7.16	0.70	10.71	3.75	5.89	1.39	3.36	-1.97	1.30	9.65	0.75	
9	PS+WAS	55	25	0.972	0.201	2,541	545	0.37	5.76	0.65	7.19	2.52	3.95	-0.31	2.14	-2.45	1.06	6.78	0.62	
10	PS+WAS	55	30	0.479	0.082	3,029	613	0.36	4.83	0.61	2.94	1.03	1.62	-3.16	0.67	-3.83	0.51	2.86	0.30	
11	PS+WAS	55	29	0.465	0.080	2,911	597	0.36	5.03	0.62	2.88	1.01	1.58	-3.42	0.65	-4.06	0.48	2.79	0.28	
12	PS+WAS	55	30	0.694	0.218	3,033	614	0.36	4.82	0.61	7.82	2.74	4.30	1.24	2.38	-1.14	1.35	7.62	0.79	
13	PS+WAS	55	31	0.643	0.161	3,087	621	0.36	4.74	0.61	5.75	2.01	3.16	-0.53	1.65	-2.19	1.01	5.61	0.59	
14	PS+WAS	55	32	0.749	0.179	3,208	637	0.36	4.56	0.60	6.41	2.24	3.53	0.25	1.89	-1.64	1.16	6.30	0.68	

**Table 2.** Calculations for the results from Chapter 4 (Table 6.4), in an insulated digester with energy recovery from biogas, at 0 °C ( $Q = 100 \text{ m}^3_{\text{sludge}} \text{ d}^{-1}$ ;  $T_{\text{amb}} = 0 \text{ °C}$ ; Heat transfer coefficient = 1)

N°	Sludge	T	SRT	OLR	CH4 rate	V	Surface area	Input electricity	Input heat	Heat loss	Output energy	Output electricity	Output heat	Energy balance	Electricity balance	Heat balance	Energy ratio	Electricity ratio	Heat ratio	Reference
		°C	d	kg VS $\text{m}^{-3} \text{d}^{-1}$	$\text{m}^3 \text{CH}_4 \text{m}^{-3} \text{d}^{-1}$	$\text{m}^3$	$\text{m}^2$	$\text{MJ d}^{-1} \text{m}^{-3}$												
1	PS+WAS	55	6	5.238	0.855	623	214	0.59	30.20	1.63	30.62	10.72	16.84	-4.86	10.13	-14.99	0.94	18.19	0.53	
2	PS+WAS	55	9	3.710	0.624	941	281	0.49	19.98	1.42	22.33	7.82	12.28	-1.79	7.32	-9.12	1.02	15.91	0.57	
3	PS+WAS	55	10	3.034	0.400	997	292	0.48	18.87	1.39	14.32	5.01	7.88	-7.86	4.53	-12.39	0.69	10.43	0.39	
4	PS+WAS	55	10	2.398	0.383	1,012	295	0.48	18.59	1.39	13.71	4.80	7.54	-8.12	4.32	-12.44	0.67	10.04	0.38	
5	PS+WAS	55	10	1.650	0.363	1,039	301	0.47	18.10	1.37	13.00	4.55	7.15	-8.25	4.08	-12.33	0.65	9.61	0.37	
6	PS+WAS	55	15	2.063	0.404	1,504	384	0.42	12.51	1.21	14.47	5.06	7.96	-1.12	4.64	-5.77	1.02	12.06	0.58	
7	PS+WAS	55	16	1.381	0.241	1,603	401	0.41	11.73	1.19	8.61	3.01	4.74	-5.58	2.60	-8.18	0.65	7.31	0.37	
8	PS+WAS	55	20	1.045	0.299	2,043	472	0.39	9.21	1.10	10.71	3.75	5.89	-1.06	3.36	-4.42	1.00	9.65	0.57	
9	PS+WAS	55	25	0.972	0.201	2,541	545	0.37	7.40	1.02	7.19	2.52	3.95	-2.33	2.14	-4.47	0.82	6.78	0.47	This work
10	PS+WAS	55	30	0.479	0.082	3,029	613	0.36	6.21	0.96	2.94	1.03	1.62	-4.89	0.67	-5.56	0.39	2.86	0.23	
11	PS+WAS	55	29	0.465	0.080	2,911	597	0.36	6.46	0.97	2.88	1.01	1.58	-5.21	0.65	-5.85	0.37	2.79	0.21	
12	PS+WAS	55	30	0.694	0.218	3,033	614	0.36	6.20	0.96	7.82	2.74	4.30	-0.49	2.38	-2.86	1.04	7.62	0.60	
13	PS+WAS	55	31	0.643	0.161	3,087	621	0.36	6.09	0.96	5.75	2.01	3.16	-2.23	1.65	-3.89	0.78	5.61	0.45	
14	PS+WAS	55	32	0.749	0.179	3,208	637	0.36	5.86	0.94	6.41	2.24	3.53	-1.39	1.89	-3.28	0.89	6.30	0.52	

**Table 3.** Calculations for the results from Chapter 4 (Table 6.4), in a non-insulated digester with energy recovery from biogas, at 20 °C ( $Q = 100 \text{ m}^3 \text{ stage d}^{-1}$ ;  $T_{\text{amb}} = 20 \text{ °C}$ ; Heat transfer coefficient = 5)

N <sup>o</sup>	Sludge	T	SRT	OLR	CH <sub>4</sub> rate	V	Surface area	Input electricity	Input heat	Heat loss	Output energy	Output electricity	Output heat	Energy balance	Electricity balance	Heat balance	Energy ratio	Electricity ratio	Heat ratio	Reference
		°C	d	kg VS m <sup>-3</sup> d <sup>-1</sup>	m <sup>3</sup> CH <sub>4</sub> m <sup>-3</sup> d <sup>-1</sup>	m <sup>3</sup>	m <sup>2</sup>	MJ d <sup>-1</sup> m <sup>-3</sup>												
1	PS+WAS	55	6	5.238	0.855	623	214	0.59	23.49	5.19	30.62	10.72	16.84	-1.71	10.13	-11.84	1.05	18.19	0.59	
2	PS+WAS	55	9	3.710	0.624	941	281	0.49	15.54	4.52	22.33	7.82	12.28	-0.45	7.32	-7.78	1.09	15.91	0.61	
3	PS+WAS	55	10	3.034	0.400	997	292	0.48	14.68	4.43	14.32	5.01	7.88	-6.70	4.53	-11.23	0.73	10.43	0.41	
4	PS+WAS	55	10	2.398	0.383	1,012	295	0.48	14.46	4.41	13.71	4.80	7.54	-7.01	4.32	-11.33	0.71	10.04	0.40	
5	PS+WAS	55	10	1.650	0.363	1,039	301	0.47	14.08	4.37	13.00	4.55	7.15	-7.23	4.08	-11.30	0.69	9.61	0.39	
6	PS+WAS	55	15	2.063	0.404	1,504	384	0.42	9.73	3.87	14.47	5.06	7.96	-1.00	4.64	-5.64	1.03	12.06	0.59	
7	PS+WAS	55	16	1.381	0.241	1,603	401	0.41	9.12	3.78	8.61	3.01	4.74	-5.57	2.60	-8.17	0.65	7.31	0.37	This work
8	PS+WAS	55	20	1.045	0.299	2,043	472	0.39	7.16	3.49	10.71	3.75	5.89	-1.40	3.36	-4.76	0.97	9.65	0.55	
9	PS+WAS	55	25	0.972	0.201	2,541	545	0.37	5.76	3.25	7.19	2.52	3.95	-2.91	2.14	-5.05	0.77	6.78	0.44	
10	PS+WAS	55	30	0.479	0.082	3,029	613	0.36	4.83	3.06	2.94	1.03	1.62	-5.61	0.67	-6.28	0.36	2.86	0.20	
11	PS+WAS	55	29	0.465	0.080	2,911	597	0.36	5.03	3.10	2.88	1.01	1.58	-5.90	0.65	-6.54	0.34	2.79	0.19	
12	PS+WAS	55	30	0.694	0.218	3,033	614	0.36	4.82	3.06	7.82	2.74	4.30	-1.21	2.38	-3.58	0.95	7.62	0.55	
13	PS+WAS	55	31	0.643	0.161	3,087	621	0.36	4.74	3.04	5.75	2.01	3.16	-2.97	1.65	-4.62	0.71	5.61	0.41	
14	PS+WAS	55	32	0.749	0.179	3,208	637	0.36	4.56	3.00	6.41	2.24	3.53	-2.15	1.89	-4.04	0.81	6.30	0.47	

**Table 4.** Calculations for the results from Chapter 4 (Table 6.4), in a non-insulated digester with energy recovery from biogas, at 0 °C ( $Q = 100 \text{ m}^3 \text{ sludge d}^{-1}$ ;  $T_{\text{amb}} = 0 \text{ °C}$ ; Heat transfer coefficient = 5)

N°	Sludge	T	SRT	OLR	CH4 rate	V	Surface area	Input electricity	Input heat	Heat loss	Output energy	Output electricity	Output heat	Energy balance	Electricity balance	Heat balance	Energy ratio	Electricity ratio	Heat ratio	Reference
		°C	d	kg VS $\text{m}^{-3} \text{d}^{-1}$	$\text{m}^3 \text{CH}_4 \text{m}^{-3} \text{d}^{-1}$	$\text{m}^3$	$\text{m}^2$	$\text{MJ d}^{-1} \text{m}^{-3}$												
1	PS+WAS	55	6	5.238	0.855	623	214	0.59	30.20	8.15	30.62	10.72	16.84	-11.38	10.13	-21.51	0.79	18.19	0.44	
2	PS+WAS	55	9	3.710	0.624	941	281	0.49	19.98	7.10	22.33	7.82	12.28	-7.47	7.32	-14.80	0.81	15.91	0.45	
3	PS+WAS	55	10	3.034	0.400	997	292	0.48	18.87	6.97	14.32	5.01	7.88	-13.43	4.53	-17.96	0.54	10.43	0.30	
4	PS+WAS	55	10	2.398	0.383	1,012	295	0.48	18.59	6.93	13.71	4.80	7.54	-13.66	4.32	-17.98	0.53	10.04	0.30	
5	PS+WAS	55	10	1.650	0.363	1,039	301	0.47	18.10	6.87	13.00	4.55	7.15	-13.75	4.08	-17.82	0.51	9.61	0.29	
6	PS+WAS	55	15	2.063	0.404	1,504	384	0.42	12.51	6.07	14.47	5.06	7.96	-5.98	4.64	-10.63	0.76	12.06	0.43	
7	PS+WAS	55	16	1.381	0.241	1,603	401	0.41	11.73	5.95	8.61	3.01	4.74	-10.34	2.60	-12.94	0.48	7.31	0.27	This work
8	PS+WAS	55	20	1.045	0.299	2,043	472	0.39	9.21	5.49	10.71	3.75	5.89	-5.45	3.36	-8.80	0.71	9.65	0.40	
9	PS+WAS	55	25	0.972	0.201	2,541	545	0.37	7.40	5.10	7.19	2.52	3.95	-6.41	2.14	-8.55	0.56	6.78	0.32	
10	PS+WAS	55	30	0.479	0.082	3,029	613	0.36	6.21	4.81	2.94	1.03	1.62	-8.74	0.67	-9.41	0.26	2.86	0.15	
11	PS+WAS	55	29	0.465	0.080	2,911	597	0.36	6.46	4.87	2.88	1.01	1.58	-9.11	0.65	-9.75	0.25	2.79	0.14	
12	PS+WAS	55	30	0.694	0.218	3,033	614	0.36	6.20	4.81	7.82	2.74	4.30	-4.33	2.38	-6.71	0.69	7.62	0.39	
13	PS+WAS	55	31	0.643	0.161	3,087	621	0.36	6.09	4.78	5.75	2.01	3.16	-6.06	1.65	-7.71	0.51	5.61	0.29	
14	PS+WAS	55	32	0.749	0.179	3,208	637	0.36	5.86	4.72	6.41	2.24	3.53	-5.17	1.89	-7.06	0.59	6.30	0.33	

**Table 5.** Calculations for the results from Chapter 4 (Table 6.4), in an insulated digester with energy recovery from biogas and effluent sludge, at 20 °C ( $Q = 100 \text{ m}^3 \text{ sludge} \cdot \text{d}^{-1}$ ;  $T_{\text{amb}} = 20 \text{ °C}$ ; Heat transfer coefficient = 1)

N°	Sludge	T	SRT	OLR	CH4 rate	V	Surface area	Input electricity	Input heat	Heat loss	Output energy	Output electricity	Output heat	Energy balance	Electricity balance	Heat balance	Energy ratio	Electricity ratio	Heat ratio	Reference
		°C	d	$\frac{\text{kg VS}}{\text{m}^3 \cdot \text{d}}$	$\frac{\text{m}^3 \text{CH}_4}{\text{m}^3 \cdot \text{d}}$	$\text{m}^3$	$\text{m}^2$	$\text{MJ d}^{-1} \cdot \text{m}^{-3}$												
1	PS+WAS	55	6	5.238	0.855	623	214	0.88	3.52	1.04	30.62	10.72	16.84	22.12	9.84	12.28	5.63	12.21	3.69	
2	PS+WAS	55	9	3.710	0.624	941	281	0.68	2.33	0.90	22.33	7.82	12.28	16.18	7.13	9.05	5.70	11.45	3.80	
3	PS+WAS	55	10	3.034	0.400	997	292	0.66	2.20	0.89	14.32	5.01	7.88	9.14	4.35	4.79	3.82	7.58	2.55	
4	PS+WAS	55	10	2.398	0.383	1,012	295	0.66	2.17	0.88	13.71	4.80	7.54	8.63	4.14	4.49	3.70	7.32	2.47	
5	PS+WAS	55	10	1.650	0.363	1,039	301	0.65	2.11	0.87	13.00	4.55	7.15	8.06	3.90	4.16	3.58	7.04	2.39	
6	PS+WAS	55	15	2.063	0.404	1,504	384	0.54	1.46	0.77	14.47	5.06	7.96	10.25	4.52	5.72	5.22	9.39	3.56	
7	PS+WAS	55	16	1.381	0.241	1,603	401	0.52	1.37	0.76	8.61	3.01	4.74	5.10	2.49	2.61	3.25	5.75	2.23	This work
8	PS+WAS	55	20	1.045	0.299	2,043	472	0.48	1.07	0.70	10.71	3.75	5.89	7.39	3.27	4.12	4.76	7.87	3.32	
9	PS+WAS	55	25	0.972	0.201	2,541	545	0.44	0.86	0.65	7.19	2.52	3.95	4.51	2.07	2.44	3.68	5.70	2.61	
10	PS+WAS	55	30	0.479	0.082	3,029	613	0.42	0.72	0.61	2.94	1.03	1.62	0.89	0.61	0.28	1.67	2.45	1.21	
11	PS+WAS	55	29	0.465	0.080	2,911	597	0.42	0.75	0.62	2.88	1.01	1.58	0.79	0.58	0.21	1.60	2.38	1.15	
12	PS+WAS	55	30	0.694	0.218	3,033	614	0.42	0.72	0.61	7.82	2.74	4.30	5.28	2.32	2.96	4.46	6.54	3.22	
13	PS+WAS	55	31	0.643	0.161	3,087	621	0.42	0.71	0.61	5.75	2.01	3.16	3.44	1.59	1.84	3.31	4.83	2.40	
14	PS+WAS	55	32	0.749	0.179	3,208	637	0.41	0.68	0.60	6.41	2.24	3.53	4.07	1.83	2.24	3.78	5.44	2.74	

**Table 6.** Calculations for the results from Chapter 4 (Table 6.4), in an insulated digester with energy recovery from biogas and effluent sludge, at 0 °C ( $Q = 100 \text{ m}^3 \text{ sludge d}^{-1}$ ;  $T_{\text{amb}} = 0 \text{ °C}$ ; Heat transfer coefficient = 1)

N°	Sludge	T	SRT	OLR	CH4 rate	V	Surface area	Input electricity	Input heat	Heat loss	Output energy	Output electricity	Output heat	Energy balance	Electricity balance	Heat balance	Energy ratio	Electricity ratio	Heat ratio	Reference
		°C	d	$\frac{\text{kg VS}}{\text{m}^3 \cdot \text{d}}$	$\frac{\text{m}^3 \text{CH}_4}{\text{m}^3 \cdot \text{d}}$	$\text{m}^3$	$\text{m}^2$	$\text{MJ d}^{-1} \cdot \text{m}^{-3}$												
1	PS+WAS	55	6	5.238	0.855	623	214	0.88	4.53	1.63	30.62	10.72	16.84	20.52	9.84	10.68	4.35	12.21	2.73	
2	PS+WAS	55	9	3.710	0.624	941	281	0.68	3.00	1.42	22.33	7.82	12.28	15.00	7.13	7.87	4.38	11.45	2.78	
3	PS+WAS	55	10	3.034	0.400	997	292	0.66	2.83	1.39	14.32	5.01	7.88	8.00	4.35	3.65	2.93	7.58	1.86	
4	PS+WAS	55	10	2.398	0.383	1,012	295	0.66	2.79	1.39	13.71	4.80	7.54	7.51	4.14	3.37	2.84	7.32	1.81	
5	PS+WAS	55	10	1.650	0.363	1,039	301	0.65	2.72	1.37	13.00	4.55	7.15	6.96	3.90	3.06	2.74	7.04	1.75	
6	PS+WAS	55	15	2.063	0.404	1,504	384	0.54	1.88	1.21	14.47	5.06	7.96	9.39	4.52	4.87	3.98	9.39	2.57	
7	PS+WAS	55	16	1.381	0.241	1,603	401	0.52	1.76	1.19	8.61	3.01	4.74	4.28	2.49	1.79	2.48	5.75	1.61	This work
8	PS+WAS	55	20	1.045	0.299	2,043	472	0.48	1.38	1.10	10.71	3.75	5.89	6.68	3.27	3.41	3.62	7.87	2.38	
9	PS+WAS	55	25	0.972	0.201	2,541	545	0.44	1.11	1.02	7.19	2.52	3.95	3.90	2.07	1.82	2.79	5.70	1.86	
10	PS+WAS	55	30	0.479	0.082	3,029	613	0.42	0.93	0.96	2.94	1.03	1.62	0.33	0.61	-0.28	1.27	2.45	0.85	
11	PS+WAS	55	29	0.465	0.080	2,911	597	0.42	0.97	0.97	2.88	1.01	1.58	0.22	0.58	-0.36	1.22	2.38	0.81	
12	PS+WAS	55	30	0.694	0.218	3,033	614	0.42	0.93	0.96	7.82	2.74	4.30	4.73	2.32	2.41	3.38	6.54	2.27	
13	PS+WAS	55	31	0.643	0.161	3,087	621	0.42	0.91	0.96	5.75	2.01	3.16	2.89	1.59	1.29	2.51	4.83	1.69	
14	PS+WAS	55	32	0.749	0.179	3,208	637	0.41	0.88	0.94	6.41	2.24	3.53	3.53	1.83	1.70	2.87	5.44	1.93	

**Table 7.** Calculations for the results from Chapter 4 (Table 6.4), in a non-insulated digester with energy recovery from biogas and effluent sludge, at 20 °C ( $Q = 100 \text{ m}^3 \text{ sludge} \cdot \text{d}^{-1}$ ;  $T_{\text{amb}} = 20 \text{ }^\circ\text{C}$ ; Heat transfer coefficient = 5)

N°	Sludge	T	SRT	OLR	CH4 rate	V	Surface area	Input electricity	Input heat	Heat loss	Output energy	Output electricity	Output heat	Energy balance	Electricity balance	Heat balance	Energy ratio	Electricity ratio	Heat ratio	Reference
		°C	d	$\frac{\text{kg VS}}{\text{m}^3 \cdot \text{d}}$	$\frac{\text{m}^3 \text{CH}_4}{\text{m}^3 \cdot \text{d}}$	$\text{m}^3$	$\text{m}^2$	$\text{MJ d}^{-1} \cdot \text{m}^{-3}$												
1	PS+WAS	55	6	5.238	0.855	623	214	0.88	3.52	5.19	30.62	10.72	16.84	17.97	9.84	8.13	3.19	12.21	1.93	
2	PS+WAS	55	9	3.710	0.624	941	281	0.68	2.33	4.52	22.33	7.82	12.28	12.57	7.13	5.43	2.96	11.45	1.79	
3	PS+WAS	55	10	3.034	0.400	997	292	0.66	2.20	4.43	14.32	5.01	7.88	5.59	4.35	1.24	1.96	7.58	1.19	
4	PS+WAS	55	10	2.398	0.383	1,012	295	0.66	2.17	4.41	13.71	4.80	7.54	5.10	4.14	0.96	1.89	7.32	1.15	
5	PS+WAS	55	10	1.650	0.363	1,039	301	0.65	2.11	4.37	13.00	4.55	7.15	4.56	3.90	0.66	1.82	7.04	1.10	
6	PS+WAS	55	15	2.063	0.404	1,504	384	0.54	1.46	3.87	14.47	5.06	7.96	7.16	4.52	2.63	2.47	9.39	1.49	
7	PS+WAS	55	16	1.381	0.241	1,603	401	0.52	1.37	3.78	8.61	3.01	4.74	2.07	2.49	-0.42	1.52	5.75	0.92	This work
8	PS+WAS	55	20	1.045	0.299	2,043	472	0.48	1.07	3.49	10.71	3.75	5.89	4.59	3.27	1.32	2.12	7.87	1.29	
9	PS+WAS	55	25	0.972	0.201	2,541	545	0.44	0.86	3.25	7.19	2.52	3.95	1.92	2.07	-0.16	1.58	5.70	0.96	
10	PS+WAS	55	30	0.479	0.082	3,029	613	0.42	0.72	3.06	2.94	1.03	1.62	-1.56	0.61	-2.17	0.70	2.45	0.43	
11	PS+WAS	55	29	0.465	0.080	2,911	597	0.42	0.75	3.10	2.88	1.01	1.58	-1.69	0.58	-2.27	0.67	2.38	0.41	
12	PS+WAS	55	30	0.694	0.218	3,033	614	0.42	0.72	3.06	7.82	2.74	4.30	2.83	2.32	0.52	1.86	6.54	1.14	
13	PS+WAS	55	31	0.643	0.161	3,087	621	0.42	0.71	3.04	5.75	2.01	3.16	1.00	1.59	-0.59	1.38	4.83	0.84	
14	PS+WAS	55	32	0.749	0.179	3,208	637	0.41	0.68	3.00	6.41	2.24	3.53	1.67	1.83	-0.16	1.56	5.44	0.96	

**Table 8.** Calculations for the results from Chapter 4 (Table 6.4), in a non-insulated digester with energy recovery from biogas and effluent sludge, at 0 °C ( $Q = 100 \text{ m}^3 \text{ sludge d}^{-1}$ ;  $T_{\text{amb}} = 0 \text{ °C}$ ; Heat transfer coefficient = 5)

N°	Sludge	T	SRT	OLR	CH4 rate	V	Surface area	Input electricity	Input heat	Heat loss	Output energy	Output electricity	Output heat	Energy balance	Electricity balance	Heat balance	Energy ratio	Electricity ratio	Heat ratio	Reference
		°C	d	$\frac{\text{kg VS}}{\text{m}^3 \text{d}^{-1}}$	$\frac{\text{m}^3 \text{CH}_4}{\text{m}^3 \text{d}^{-1}}$	$\text{m}^3$	$\text{m}^2$	$\text{MJ d}^{-1} \text{m}^{-3}$												
1	PS+WAS	55	6	5.238	0.855	623	214	0.88	4.53	8.15	30.62	10.72	16.84	14.00	9.84	4.16	2.26	12.21	1.33	
2	PS+WAS	55	9	3.710	0.624	941	281	0.68	3.00	7.10	22.33	7.82	12.28	9.32	7.13	2.18	2.07	11.45	1.22	
3	PS+WAS	55	10	3.034	0.400	997	292	0.66	2.83	6.97	14.32	5.01	7.88	2.43	4.35	-1.92	1.37	7.58	0.80	
4	PS+WAS	55	10	2.398	0.383	1,012	295	0.66	2.79	6.93	13.71	4.80	7.54	1.96	4.14	-2.18	1.32	7.32	0.78	
5	PS+WAS	55	10	1.650	0.363	1,039	301	0.65	2.72	6.87	13.00	4.55	7.15	1.46	3.90	-2.44	1.27	7.04	0.75	
6	PS+WAS	55	15	2.063	0.404	1,504	384	0.54	1.88	6.07	14.47	5.06	7.96	4.53	4.52	0.01	1.70	9.39	1.00	
7	PS+WAS	55	16	1.381	0.241	1,603	401	0.52	1.76	5.95	8.61	3.01	4.74	-0.48	2.49	-2.97	1.05	5.75	0.61	This work
8	PS+WAS	55	20	1.045	0.299	2,043	472	0.48	1.38	5.49	10.71	3.75	5.89	2.29	3.27	-0.98	1.46	7.87	0.86	
9	PS+WAS	55	25	0.972	0.201	2,541	545	0.44	1.11	5.10	7.19	2.52	3.95	-0.18	2.07	-2.26	1.08	5.70	0.64	
10	PS+WAS	55	30	0.479	0.082	3,029	613	0.42	0.93	4.81	2.94	1.03	1.62	-3.52	0.61	-4.13	0.48	2.45	0.28	
11	PS+WAS	55	29	0.465	0.080	2,911	597	0.42	0.97	4.87	2.88	1.01	1.58	-3.68	0.58	-4.26	0.46	2.38	0.27	
12	PS+WAS	55	30	0.694	0.218	3,033	614	0.42	0.93	4.81	7.82	2.74	4.30	0.88	2.32	-1.44	1.27	6.54	0.75	
13	PS+WAS	55	31	0.643	0.161	3,087	621	0.42	0.91	4.78	5.75	2.01	3.16	-0.94	1.59	-2.53	0.94	4.83	0.56	
14	PS+WAS	55	32	0.749	0.179	3,208	637	0.41	0.88	4.72	6.41	2.24	3.53	-0.24	1.83	-2.07	1.07	5.44	0.63	

**Table 9.** Calculations for the results from the literature review (Tables 6.1-6.2), in an insulated digester with energy recovery from biogas, at 20 °C ( $Q = 100 \text{ m}^3 \text{ sludge d}^{-1}$ ;  $T_{\text{amb}} = 20 \text{ °C}$ ; Heat transfer coefficient = 1)

N°	Sludge	T	SRT	OLR	CH4 rate	V	Surface area	Input electricity	Input heat	Heat loss	Output energy	Output electricity	Output heat	Energy balance	Electricity balance	Heat balance	Energy ratio	Electricity ratio	Heat ratio	Reference
		°C	d	$\frac{\text{kg VS}}{\text{m}^3 \text{ d}^1}$	$\frac{\text{m}^3 \text{ CH}_4}{\text{m}^3 \text{ d}^1}$	$\text{m}^3$	$\text{m}^2$	$\text{MJ d}^{-1} \text{ m}^{-3}$												
15	WAS	35	8		0.055	800	252	0.53	7.84	0.41	1.95	0.68	1.08	-7.01	0.16	-7.17	0.22	1.30	0.13	Lafitte-Trouqué and Forster (2002)
16	WAS	35	10		0.057	1,000	293	0.48	6.27	0.38	2.05	0.72	1.13	-5.29	0.24	-5.52	0.29	1.49	0.17	
17	WAS	37.6	21		1.000	2,100	480	0.39	3.50	0.35	4.19	1.47	2.30	-0.47	1.08	-1.55	0.99	3.80	0.60	
18	WAS	38.5	20		0.800	2,000	465	0.39	3.87	0.37	1.63	0.57	0.90	-3.16	0.18	-3.34	0.35	1.46	0.21	Bolzonella <i>et al.</i> (2005)
19	WAS	36	22		0.800	2,200	495	0.38	3.04	0.31	3.49	1.22	1.92	-0.59	0.84	-1.43	0.93	3.20	0.57	
20	WAS	35	33		0.700	3,300	649	0.35	1.90	0.25	1.86	0.65	1.02	-0.83	0.30	-1.13	0.74	1.84	0.48	
21	WAS	34.5	37.2		0.530	3,720	703	0.35	1.63	0.24	2.43	0.85	1.34	-0.02	0.50	-0.53	1.10	2.45	0.72	Mininni <i>et al.</i> (2006)
22	WAS	34	40		1.000	4,000	738	0.35	1.46	0.22	0.93	0.33	0.51	-1.19	-0.02	-1.17	0.46	0.94	0.30	Bolzonella <i>et al.</i> (2005)
23	WAS	55	8		0.160	800	252	0.53	18.29	0.95	5.72	2.00	3.15	-14.61	1.48	-16.09	0.29	3.82	0.16	Lafitte-Trouqué and Forster (2002)
24	WAS	55	10		0.112	1,000	293	0.48	14.63	0.89	4.00	1.40	2.20	-12.39	0.92	-13.31	0.25	2.92	0.14	
25	WAS	55	18		0.080	1,800	433	0.40	8.13	0.73	2.86	1.00	1.58	-6.68	0.60	-7.28	0.31	2.51	0.18	Pavan <i>et al.</i> (2006)
26	PS	55	15		0.693	1,500	384	0.42	9.75	0.77	5.23	1.83	2.88	-6.24	1.41	-7.65	0.48	4.36	0.27	Lu <i>et al.</i> (2007)
27	n.d.	38	19		1.130	1,900	449	0.39	3.96	0.37	8.61	3.01	4.74	3.03	2.62	0.41	1.82	7.63	1.09	Zábranská <i>et al.</i> (2006)
28	PS+WAS	35	20		1.140	2,000	465	0.39	3.14	0.30	8.20	2.87	4.51	3.55	2.48	1.07	2.14	7.36	1.31	Benabdallah <i>et al.</i> (2006)
28	PS+WAS	37	20		1.433	2,000	465	0.39	3.55	0.34	8.95	3.13	4.92	3.77	2.74	1.03	2.09	8.04	1.26	Gavala <i>et al.</i> (2003)
30	PS+WAS	37	20		1.380	2,000	465	0.39	3.55	0.34	12.70	4.44	6.98	7.14	4.05	3.09	2.96	11.39	1.79	Bousková <i>et al.</i> (2005)
31	PS+WAS	37	20		1.380	2,000	465	0.39	3.55	0.34	13.77	4.82	7.57	8.11	4.43	3.68	3.21	12.36	1.94	
32	PS+WAS	35	27		1.300	2,700	568	0.37	2.32	0.27	8.95	3.13	4.92	5.09	2.77	2.33	3.02	8.54	1.90	De la Rubia <i>et al.</i> (2002)
33	PS+WAS	43	36		0.442	3,600	688	0.35	2.67	0.38	4.33	1.52	2.38	0.50	1.17	-0.67	1.27	4.33	0.78	This work
34	PS+WAS	55	10		3.003	1,000	293	0.48	14.63	0.89	14.32	5.01	7.88	-3.11	4.53	-7.64	0.90	10.44	0.51	Ferrer <i>et al.</i> (2008)
35	PS+WAS	55	15		1.519	1,500	384	0.42	9.75	0.77	13.99	4.90	7.69	1.64	4.48	-2.83	1.28	11.66	0.73	Benabdallah <i>et al.</i> (2006)
36	PS+WAS	55	15		2.190	1,500	384	0.42	9.75	0.77	14.32	5.01	7.88	1.94	4.59	-2.65	1.31	11.93	0.75	De la Rubia <i>et al.</i> (2006)
37	PS+WAS	55	20		1.870	2,000	465	0.39	7.32	0.70	7.88	2.76	4.33	-1.32	2.37	-3.69	0.94	7.07	0.54	De la Rubia <i>et al.</i> (2002)
38	PS+WAS	55	20		1.433	2,000	465	0.39	7.32	0.70	9.18	3.21	5.05	-0.14	2.82	-2.97	1.09	8.24	0.63	Gavala <i>et al.</i> (2003)
39	PS+WAS	55	20		1.870	2,000	465	0.39	7.32	0.70	9.31	3.26	5.12	-0.03	2.87	-2.90	1.11	8.35	0.64	De la Rubia <i>et al.</i> (2006)
40	PS+WAS	55	20		1.380	2,000	465	0.39	7.32	0.70	14.91	5.22	8.20	5.01	4.83	0.18	1.77	13.38	1.02	
41	PS+WAS	55	20		1.380	2,000	465	0.39	7.32	0.70	16.14	5.65	8.88	6.12	5.26	0.86	1.92	14.48	1.11	Bousková <i>et al.</i> (2005)
42	PS+WAS	55	27		1.480	2,700	568	0.37	5.42	0.64	6.80	2.38	3.74	-0.30	2.01	-2.31	1.06	6.49	0.62	De la Rubia <i>et al.</i> (2006)
43	PS+WAS	55	40		0.800	4,000	738	0.35	3.66	0.56	6.09	2.13	3.35	0.92	1.79	-0.87	1.33	6.17	0.79	
44	PS+WAS	55	75		0.420	7,500	1,122	0.32	1.95	0.45	0.72	0.25	0.39	-2.08	-0.07	-2.01	0.26	0.77	0.16	

**Table 10.** Calculations for the results from the literature review (Tables 6.1-6.2), in an insulated digester with energy recovery from biogas, at 0 °C ( $Q = 100 \text{ m}^3 \text{ sludge d}^{-1}$ ;  $T_{\text{amb}} = 0 \text{ °C}$ ; Heat transfer coefficient = 1)

N°	Sludge	T °C	SRT d	OLR $\frac{\text{kg VS}}{\text{m}^3 \text{ d}^{-1}}$	CH4 rate $\frac{\text{m}^3 \text{ CH}_4}{\text{m}^3 \text{ d}^{-1}}$	V $\text{m}^3$	Surface area $\text{m}^2$	Input electricity $\text{MJ d}^{-1} \text{ m}^{-3}$	Input heat $\text{MJ d}^{-1} \text{ m}^{-3}$	Heat loss $\text{MJ d}^{-1} \text{ m}^{-3}$	Output energy $\text{MJ d}^{-1} \text{ m}^{-3}$	Output electricity $\text{MJ d}^{-1} \text{ m}^{-3}$	Output heat $\text{MJ d}^{-1} \text{ m}^{-3}$	Energy balance $\text{MJ d}^{-1} \text{ m}^{-3}$	Electricity balance $\text{MJ d}^{-1} \text{ m}^{-3}$	Heat balance $\text{MJ d}^{-1} \text{ m}^{-3}$	Energy ratio	Electricity ratio	Heat ratio	Reference
15	WAS	35	8	n.d.	0.055	800	252	0.53	13.06	0.95	1.95	0.68	1.08	-12.78	0.16	-12.94	0.13	1.30	0.08	Lafitte-Trouqué and Forster (2002)
16	WAS	35	10	n.d.	0.057	1,000	293	0.48	10.45	0.89	2.05	0.72	1.13	-9.97	0.24	-10.21	0.17	1.49	0.10	
17	WAS	37.6	21	1.000	0.117	2,100	480	0.39	5.49	0.74	4.19	1.47	2.30	-2.85	1.08	-3.93	0.63	3.80	0.37	
18	WAS	38.5	20	0.800	0.046	2,000	465	0.39	5.96	0.77	1.63	0.57	0.90	-5.65	0.18	-5.83	0.23	1.46	0.13	Bolzonella <i>et al.</i> (2005)
19	WAS	36	22	0.800	0.098	2,200	495	0.38	4.94	0.70	3.49	1.22	1.92	-2.88	0.84	-3.72	0.58	3.20	0.34	
20	WAS	35	33	0.700	0.052	3,300	649	0.35	3.17	0.59	1.86	0.65	1.02	-2.44	0.30	-2.74	0.45	1.84	0.27	
21	WAS	34.5	37.2	0.530	0.068	3,720	703	0.35	2.75	0.56	2.43	0.85	1.34	-1.47	0.50	-1.98	0.66	2.45	0.40	Mininni <i>et al.</i> (2006)
22	WAS	34	40	1.000	0.026	4,000	738	0.35	2.51	0.54	0.93	0.33	0.51	-2.56	-0.02	-2.54	0.27	0.94	0.17	Bolzonella <i>et al.</i> (2005)
23	WAS	55	8	n.d.	0.160	800	252	0.53	23.51	1.50	5.72	2.00	3.15	-20.38	1.48	-21.86	0.22	3.82	0.13	Lafitte-Trouqué and Forster (2002)
24	WAS	55	10	n.d.	0.112	1,000	293	0.48	18.81	1.39	4.00	1.40	2.20	-17.08	0.92	-18.00	0.19	2.92	0.11	
25	WAS	55	18	n.d.	0.080	1,800	433	0.40	10.45	1.14	2.86	1.00	1.58	-9.42	0.60	-10.02	0.24	2.51	0.14	Pavan <i>et al.</i> (2006)
26	PS	55	15	0.693	0.146	1,500	384	0.42	12.54	1.22	5.23	1.83	2.88	-9.47	1.41	-10.88	0.37	4.36	0.21	Lu <i>et al.</i> (2007)
27	n.d.	38	19	1.130	0.241	1,900	449	0.39	6.16	0.78	8.61	3.01	4.74	0.42	2.62	-2.20	1.17	7.63	0.68	Zábranská <i>et al.</i> (2006)
28	PS+WAS	35	20	1.140	0.229	2,000	465	0.39	5.23	0.70	8.20	2.87	4.51	1.06	2.48	-1.42	1.30	7.36	0.76	Benabdallah <i>et al.</i> (2006)
28	PS+WAS	37	20	1.433	0.250	2,000	465	0.39	5.64	0.74	8.95	3.13	4.92	1.28	2.74	-1.46	1.32	8.04	0.77	Gavala <i>et al.</i> (2003)
30	PS+WAS	37	20	1.380	0.355	2,000	465	0.39	5.64	0.74	12.70	4.44	6.98	4.65	4.05	0.60	1.87	11.39	1.09	Bousková <i>et al.</i> (2005)
31	PS+WAS	37	20	1.380	0.385	2,000	465	0.39	5.64	0.74	13.77	4.82	7.57	5.62	4.43	1.19	2.03	12.36	1.19	
32	PS+WAS	35	27	1.300	0.250	2,700	568	0.37	3.87	0.64	8.95	3.13	4.92	3.18	2.77	0.42	1.84	8.54	1.09	De la Rubia <i>et al.</i> (2002)
33	PS+WAS	43	36	0.442	0.121	3,600	688	0.35	3.83	0.71	4.33	1.52	2.38	-0.99	1.17	-2.16	0.89	4.33	0.52	This work
34	PS+WAS	55	10	3.003	0.400	1,000	293	0.48	18.81	1.39	14.32	5.01	7.88	-7.79	4.53	-12.33	0.69	10.44	0.39	Ferrer <i>et al.</i> (2008)
35	PS+WAS	55	15	1.519	0.391	1,500	384	0.42	12.54	1.22	13.99	4.90	7.69	-1.59	4.48	-6.06	0.99	11.66	0.56	Benabdallah <i>et al.</i> (2006)
36	PS+WAS	55	15	2.190	0.400	1,500	384	0.42	12.54	1.22	14.32	5.01	7.88	-1.29	4.59	-5.88	1.01	11.93	0.57	De la Rubia <i>et al.</i> (2006)
37	PS+WAS	55	20	1.870	0.220	2,000	465	0.39	9.41	1.10	7.88	2.76	4.33	-3.81	2.37	-6.18	0.72	7.07	0.41	De la Rubia <i>et al.</i> (2002)
38	PS+WAS	55	20	1.433	0.257	2,000	465	0.39	9.41	1.10	9.18	3.21	5.05	-2.64	2.82	-5.46	0.84	8.24	0.48	Gavala <i>et al.</i> (2003)
39	PS+WAS	55	20	1.870	0.260	2,000	465	0.39	9.41	1.10	9.31	3.26	5.12	-2.52	2.87	-5.39	0.85	8.35	0.49	De la Rubia <i>et al.</i> (2006)
40	PS+WAS	55	20	1.380	0.417	2,000	465	0.39	9.41	1.10	14.91	5.22	8.20	2.52	4.83	-2.31	1.37	13.38	0.78	Bousková <i>et al.</i> (2005)
41	PS+WAS	55	20	1.380	0.451	2,000	465	0.39	9.41	1.10	16.14	5.65	8.88	3.62	5.26	-1.63	1.48	14.48	0.84	
42	PS+WAS	55	27	1.480	0.190	2,700	568	0.37	6.97	1.00	6.80	2.38	3.74	-2.21	2.01	-4.23	0.82	6.49	0.47	
43	PS+WAS	55	40	0.800	0.170	4,000	738	0.35	4.70	0.88	6.09	2.13	3.35	-0.45	1.79	-2.23	1.03	6.17	0.60	De la Rubia <i>et al.</i> (2006)
44	PS+WAS	55	75	0.420	0.020	7,500	1,122	0.32	2.51	0.71	0.72	0.25	0.39	-2.90	-0.07	-2.83	0.20	0.77	0.12	

**Table 11.** Calculations for the results from the literature review (Tables 6.1- 6.2), in a non-insulated digester with energy recovery from biogas, at 20 °C ( $Q = 100 \text{ m}^3 \text{ sludge d}^{-1}$ ;  $T_{\text{amb}} = 20 \text{ °C}$ ; Heat transfer coefficient = 5)

N°	Sludge	T	SRT	OLR	CH4 rate	V	Surface area	Input electricity	Input heat	Heat loss	Output energy	Output electricity	Output heat	Energy balance	Electricity balance	Heat balance	Energy ratio	Electricity ratio	Heat ratio	Reference
		°C	d	$\frac{\text{kg VS}}{\text{m}^3 \text{ d}^1}$	$\frac{\text{m}^3 \text{ CH}_4}{\text{m}^3 \text{ d}^1}$	$\text{m}^3$	$\text{m}^2$	$\text{MJ d}^{-1} \text{ m}^{-3}$												
15	WAS	35	8	n.d.	0.055	800	252	0.53	7.84	2.04	1.95	0.68	1.08	-8.65	0.16	-8.81	0.19	1.30	0.11	Lafitte-Trouqué and Forster (2002)
16	WAS	35	10	n.d.	0.057	1,000	293	0.48	6.27	1.90	2.05	0.72	1.13	-6.81	0.24	-7.04	0.24	1.49	0.14	
17	WAS	37.6	21	1.000	0.117	2,100	480	0.39	3.50	1.74	4.19	1.47	2.30	-1.86	1.08	-2.94	0.74	3.80	0.44	
18	WAS	38.5	20	0.800	0.046	2,000	465	0.39	3.87	1.86	1.63	0.57	0.90	-4.65	0.18	-4.83	0.27	1.46	0.16	Bolzonella <i>et al.</i> (2005)
19	WAS	36	22	0.800	0.098	2,200	495	0.38	3.04	1.56	3.49	1.22	1.92	-1.84	0.84	-2.68	0.70	3.20	0.42	
20	WAS	35	33	0.700	0.052	3,300	649	0.35	1.90	1.27	1.86	0.65	1.02	-1.85	0.30	-2.15	0.53	1.84	0.32	
21	WAS	34.5	37.2	0.530	0.068	3,720	703	0.35	1.63	1.18	2.43	0.85	1.34	-0.97	0.50	-1.47	0.77	2.45	0.48	Mininni <i>et al.</i> (2006)
22	WAS	34	40	1.000	0.026	4,000	738	0.35	1.46	1.12	0.93	0.33	0.51	-2.09	-0.02	-2.07	0.32	0.94	0.20	Bolzonella <i>et al.</i> (2005)
23	WAS	55	8	n.d.	0.160	800	252	0.53	18.29	4.77	5.72	2.00	3.15	-18.43	1.48	-19.91	0.24	3.82	0.14	Lafitte-Trouqué and Forster (2002)
24	WAS	55	10	n.d.	0.112	1,000	293	0.48	14.63	4.43	4.00	1.40	2.20	-15.94	0.92	-16.86	0.20	2.92	0.12	
25	WAS	55	18	n.d.	0.080	1,800	433	0.40	8.13	3.64	2.86	1.00	1.58	-9.59	0.60	-10.19	0.24	2.51	0.13	Pavan <i>et al.</i> (2006)
26	PS	55	15	0.693	0.146	1,500	384	0.42	9.75	3.87	5.23	1.83	2.88	-9.33	1.41	-10.74	0.37	4.36	0.21	Lu <i>et al.</i> (2007)
27	n.d.	38	19	1.130	0.241	1,900	449	0.39	3.96	1.84	8.61	3.01	4.74	1.56	2.62	-1.06	1.39	7.63	0.82	Zábranská <i>et al.</i> (2006)
28	PS+WAS	35	20	1.140	0.229	2,000	465	0.39	3.14	1.51	8.20	2.87	4.51	2.35	2.48	-0.13	1.63	7.36	0.97	Benabdallah <i>et al.</i> (2006)
28	PS+WAS	37	20	1.433	0.250	2,000	465	0.39	3.55	1.71	8.95	3.13	4.92	2.41	2.74	-0.34	1.58	8.04	0.94	Gavala <i>et al.</i> (2003)
30	PS+WAS	37	20	1.380	0.355	2,000	465	0.39	3.55	1.71	12.70	4.44	6.98	5.78	4.05	1.72	2.25	11.39	1.33	Bousková <i>et al.</i> (2005)
31	PS+WAS	37	20	1.380	0.385	2,000	465	0.39	3.55	1.71	13.77	4.82	7.57	6.74	4.43	2.31	2.44	12.36	1.44	
32	PS+WAS	35	27	1.300	0.250	2,700	568	0.37	2.32	1.36	8.95	3.13	4.92	4.00	2.77	1.24	2.21	8.54	1.34	De la Rubia <i>et al.</i> (2002)
33	PS+WAS	43	36	0.442	0.121	3,600	688	0.35	2.67	1.90	4.33	1.52	2.38	-1.02	1.17	-2.19	0.88	4.33	0.52	This work
34	PS+WAS	55	10	3.003	0.400	1,000	293	0.48	14.63	4.43	14.32	5.01	7.88	-6.65	4.53	-11.18	0.73	10.44	0.41	Ferrer <i>et al.</i> (2008)
35	PS+WAS	55	15	1.519	0.391	1,500	384	0.42	9.75	3.87	13.99	4.90	7.69	-1.45	4.48	-5.93	1.00	11.66	0.56	Benabdallah <i>et al.</i> (2006)
36	PS+WAS	55	15	2.190	0.400	1,500	384	0.42	9.75	3.87	14.32	5.01	7.88	-1.15	4.59	-5.75	1.02	11.93	0.58	De la Rubia <i>et al.</i> (2006)
37	PS+WAS	55	20	1.870	0.220	2,000	465	0.39	7.32	3.52	7.88	2.76	4.33	-4.13	2.37	-6.50	0.70	7.07	0.40	De la Rubia <i>et al.</i> (2002)
38	PS+WAS	55	20	1.433	0.257	2,000	465	0.39	7.32	3.52	9.18	3.21	5.05	-2.96	2.82	-5.78	0.82	8.24	0.47	Gavala <i>et al.</i> (2003)
39	PS+WAS	55	20	1.870	0.260	2,000	465	0.39	7.32	3.52	9.31	3.26	5.12	-2.84	2.87	-5.71	0.83	8.35	0.47	De la Rubia <i>et al.</i> (2006)
40	PS+WAS	55	20	1.380	0.417	2,000	465	0.39	7.32	3.52	14.91	5.22	8.20	2.20	4.83	-2.63	1.33	13.38	0.76	Bousková <i>et al.</i> (2005)
41	PS+WAS	55	20	1.380	0.451	2,000	465	0.39	7.32	3.52	16.14	5.65	8.88	3.30	5.26	-1.95	1.44	14.48	0.82	
42	PS+WAS	55	27	1.480	0.190	2,700	568	0.37	5.42	3.18	6.80	2.38	3.74	-2.84	2.01	-4.86	0.76	6.49	0.44	De la Rubia <i>et al.</i> (2006)
43	PS+WAS	55	40	0.800	0.170	4,000	738	0.35	3.66	2.79	6.09	2.13	3.35	-1.32	1.79	-3.10	0.90	6.17	0.52	
44	PS+WAS	55	75	0.420	0.020	7,500	1,122	0.32	1.95	2.26	0.72	0.25	0.39	-3.89	-0.07	-3.82	0.16	0.77	0.09	

**Table 12.** Calculations for the results from the literature review (Tables 6.1-6.2), in a non-insulated digester with energy recovery from biogas, at 0 °C ( $Q = 100 \text{ m}^3 \text{ sludge d}^{-1}$ ;  $T_{\text{amb}} = 0 \text{ °C}$ ; Heat transfer coefficient = 5)

N°	Sludge	T	SRT	OLR	CH4 rate	V	Surface area	Input electricity	Input heat	Heat loss	Output energy	Output electricity	Output heat	Energy balance	Electricity balance	Heat balance	Energy ratio	Electricity ratio	Heat ratio	Reference
		°C	d	$\frac{\text{kg VS}}{\text{m}^3 \text{ d}^1}$	$\frac{\text{m}^3 \text{ CH}_4}{\text{m}^3 \text{ d}^1}$	$\text{m}^3$	$\text{m}^2$	$\text{MJ d}^{-1} \text{ m}^{-3}$												
15	WAS	35	8	n.d.	0.055	800	252	0.53	13.06	4.77	1.95	0.68	1.08	-16.60	0.16	-16.76	0.11	1.30	0.06	Lafitte-Trouqué and Forster (2002)
16	WAS	35	10	n.d.	0.057	1,000	293	0.48	10.45	4.43	2.05	0.72	1.13	-13.52	0.24	-13.75	0.13	1.49	0.08	
17	WAS	37.6	21	1.000	0.117	2,100	480	0.39	5.49	3.72	4.19	1.47	2.30	-5.83	1.08	-6.91	0.44	3.80	0.25	
18	WAS	38.5	20	0.800	0.046	2,000	465	0.39	5.96	3.87	1.63	0.57	0.90	-8.75	0.18	-8.93	0.16	1.46	0.09	Bolzonella <i>et al.</i> (2005)
19	WAS	36	22	0.800	0.098	2,200	495	0.38	4.94	3.50	3.49	1.22	1.92	-5.68	0.84	-6.52	0.40	3.20	0.23	
20	WAS	35	33	0.700	0.052	3,300	649	0.35	3.17	2.97	1.86	0.65	1.02	-4.82	0.30	-5.12	0.29	1.84	0.17	
21	WAS	34.5	37.2	0.530	0.068	3,720	703	0.35	2.75	2.82	2.43	0.85	1.34	-3.73	0.50	-4.23	0.41	2.45	0.24	Mininni <i>et al.</i> (2006)
22	WAS	34	40	1.000	0.026	4,000	738	0.35	2.51	2.71	0.93	0.33	0.51	-4.73	-0.02	-4.71	0.17	0.94	0.10	Bolzonella <i>et al.</i> (2005)
23	WAS	55	8	n.d.	0.160	800	252	0.53	23.51	7.50	5.72	2.00	3.15	-26.38	1.48	-27.86	0.18	3.82	0.10	Lafitte-Trouqué and Forster (2002)
24	WAS	55	10	n.d.	0.112	1,000	293	0.48	18.81	6.96	4.00	1.40	2.20	-22.65	0.92	-23.57	0.15	2.92	0.09	
25	WAS	55	18	n.d.	0.080	1,800	433	0.40	10.45	5.72	2.86	1.00	1.58	-13.99	0.60	-14.60	0.17	2.51	0.10	Pavan <i>et al.</i> (2006)
26	PS	55	15	0.693	0.146	1,500	384	0.42	12.54	6.08	5.23	1.83	2.88	-14.33	1.41	-15.74	0.27	4.36	0.15	Lu <i>et al.</i> (2007)
27	n.d.	38	19	1.130	0.241	1,900	449	0.39	6.16	3.88	8.61	3.01	4.74	-2.69	2.62	-5.31	0.82	7.63	0.47	Zábranská <i>et al.</i> (2006)
28	PS+WAS	35	20	1.140	0.229	2,000	465	0.39	5.23	3.52	8.20	2.87	4.51	-1.75	2.48	-4.23	0.90	7.36	0.52	Benabdallah <i>et al.</i> (2006)
28	PS+WAS	37	20	1.433	0.250	2,000	465	0.39	5.64	3.72	8.95	3.13	4.92	-1.69	2.74	-4.43	0.92	8.04	0.53	Gavala <i>et al.</i> (2003)
30	PS+WAS	37	20	1.380	0.355	2,000	465	0.39	5.64	3.72	12.70	4.44	6.98	1.68	4.05	-2.38	1.30	11.39	0.75	Bousková <i>et al.</i> (2005)
31	PS+WAS	37	20	1.380	0.385	2,000	465	0.39	5.64	3.72	13.77	4.82	7.57	2.64	4.43	-1.79	1.41	12.36	0.81	
32	PS+WAS	35	27	1.300	0.250	2,700	568	0.37	3.87	3.18	8.95	3.13	4.92	0.64	2.77	-2.13	1.21	8.54	0.70	De la Rubia <i>et al.</i> (2002)
33	PS+WAS	43	36	0.442	0.121	3,600	688	0.35	3.83	3.55	4.33	1.52	2.38	-3.83	1.17	-5.00	0.56	4.33	0.32	This work
34	PS+WAS	55	10	3.003	0.400	1,000	293	0.48	18.81	6.96	14.32	5.01	7.88	-13.36	4.53	-17.89	0.55	10.44	0.31	Ferrer <i>et al.</i> (2008)
35	PS+WAS	55	15	1.519	0.391	1,500	384	0.42	12.54	6.08	13.99	4.90	7.69	-6.45	4.48	-10.93	0.73	11.66	0.41	Benabdallah <i>et al.</i> (2006)
36	PS+WAS	55	15	2.190	0.400	1,500	384	0.42	12.54	6.08	14.32	5.01	7.88	-6.15	4.59	-10.74	0.75	11.93	0.42	De la Rubia <i>et al.</i> (2006)
37	PS+WAS	55	20	1.870	0.220	2,000	465	0.39	9.41	5.52	7.88	2.76	4.33	-8.23	2.37	-10.60	0.51	7.07	0.29	De la Rubia <i>et al.</i> (2002)
38	PS+WAS	55	20	1.433	0.257	2,000	465	0.39	9.41	5.52	9.18	3.21	5.05	-7.05	2.82	-9.88	0.60	8.24	0.34	Gavala <i>et al.</i> (2003)
39	PS+WAS	55	20	1.870	0.260	2,000	465	0.39	9.41	5.52	9.31	3.26	5.12	-6.94	2.87	-9.81	0.61	8.35	0.34	De la Rubia <i>et al.</i> (2006)
40	PS+WAS	55	20	1.380	0.417	2,000	465	0.39	9.41	5.52	14.91	5.22	8.20	-1.90	4.83	-6.73	0.97	13.38	0.55	Bousková <i>et al.</i> (2005)
41	PS+WAS	55	20	1.380	0.451	2,000	465	0.39	9.41	5.52	16.14	5.65	8.88	-0.80	5.26	-6.05	1.05	14.48	0.59	
42	PS+WAS	55	27	1.480	0.190	2,700	568	0.37	6.97	5.00	6.80	2.38	3.74	-6.21	2.01	-8.22	0.55	6.49	0.31	De la Rubia <i>et al.</i> (2006)
43	PS+WAS	55	40	0.800	0.170	4,000	738	0.35	4.70	4.38	6.09	2.13	3.35	-3.95	1.79	-5.74	0.65	6.17	0.37	
44	PS+WAS	55	75	0.420	0.020	7,500	1,122	0.32	2.51	3.56	0.72	0.25	0.39	-5.74	-0.07	-5.67	0.11	0.77	0.06	

**Table 13.** Calculations for the results from the literature review (Tables 6.1 -6.2), in an insulated digester with energy recovery from biogas and effluent sludge, at 20 °C ( $Q=100\text{ m}^3\text{ sludge d}^{-1}$ ;  $T_{\text{amb}}=20\text{ °C}$ ; Heat transfer coef=1)

N°	Sludge	T °C	SRT d	OLR $\frac{\text{kg VS}}{\text{m}^3\text{ d}^1}$	CH4 rate $\frac{\text{m}^3\text{ CH}_4}{\text{m}^3\text{ d}^1}$	V $\text{m}^3$	Surface area $\text{m}^2$	Input electricity $\text{MJ d}^1\text{ m}^3$	Input heat $\text{MJ d}^1\text{ m}^3$	Heat loss $\text{MJ d}^1\text{ m}^3$	Output energy $\text{MJ d}^1\text{ m}^3$	Output electricity $\text{MJ d}^1\text{ m}^3$	Output heat $\text{MJ d}^1\text{ m}^3$	Energy balance $\text{MJ d}^1\text{ m}^3$	Electricity balance $\text{MJ d}^1\text{ m}^3$	Heat balance $\text{MJ d}^1\text{ m}^3$	Energy ratio	Electricity ratio	Heat ratio	Reference
15	WAS	35	8	n.d.	0.055	800	252	0.75	1.18	0.41	1.95	0.68	1.08	-0.58	-0.07	-0.51	0.84	0.91	0.68	Lafitte-Trouqué and Forster (2002)
16	WAS	35	10	n.d.	0.057	1,000	293	0.66	0.94	0.38	2.05	0.72	1.13	-0.14	0.06	-0.19	1.03	1.09	0.85	
17	WAS	37.6	21	1.000	0.117	2,100	480	0.47	0.53	0.35	4.19	1.47	2.30	2.42	0.99	1.43	3.11	3.11	2.64	Bolzonella <i>et al.</i> (2005)
18	WAS	38.5	20	0.800	0.046	2,000	465	0.48	0.58	0.37	1.63	0.57	0.90	0.03	0.09	-0.06	1.14	1.19	0.94	
19	WAS	36	22	0.800	0.098	2,200	495	0.46	0.46	0.31	3.49	1.22	1.92	1.91	0.76	1.15	2.84	2.63	2.50	
20	WAS	35	33	0.700	0.052	3,300	649	0.41	0.29	0.25	1.86	0.65	1.02	0.73	0.24	0.48	1.96	1.59	1.90	
21	WAS	34.5	37.2	0.530	0.068	3,720	703	0.40	0.24	0.24	2.43	0.85	1.34	1.31	0.46	0.86	2.77	2.15	2.78	Mininni <i>et al.</i> (2006)
22	WAS	34	40	1.000	0.026	4,000	738	0.39	0.22	0.22	0.93	0.33	0.51	0.01	-0.06	0.07	1.12	0.84	1.16	Bolzonella <i>et al.</i> (2005)
23	WAS	55	8	n.d.	0.160	800	252	0.75	2.74	0.95	5.72	2.00	3.15	0.70	1.25	-0.55	1.29	2.67	0.85	Lafitte-Trouqué and Forster (2002)
24	WAS	55	10	n.d.	0.112	1,000	293	0.66	2.19	0.89	4.00	1.40	2.20	-0.14	0.74	-0.88	1.07	2.12	0.71	
25	WAS	55	18	n.d.	0.080	1,800	433	0.50	1.22	0.73	2.86	1.00	1.58	0.13	0.50	-0.37	1.17	2.00	0.81	Pavan <i>et al.</i> (2006)
26	PS	55	15	0.693	0.146	1,500	384	0.54	1.46	0.77	5.23	1.83	2.88	1.93	1.29	0.64	1.88	3.39	1.29	Lu <i>et al.</i> (2007)
27	n.d.	38	19	1.130	0.241	1,900	449	0.49	0.59	0.37	8.61	3.01	4.74	6.30	2.52	3.77	5.93	6.16	4.92	Zábranská <i>et al.</i> (2006)
28	PS+WAS	35	20	1.140	0.229	2,000	465	0.48	0.47	0.30	8.20	2.87	4.51	6.13	2.39	3.74	6.55	5.98	5.84	Benabdallah <i>et al.</i> (2006)
28	PS+WAS	37	20	1.433	0.250	2,000	465	0.48	0.53	0.34	8.95	3.13	4.92	6.70	2.65	4.05	6.61	6.53	5.63	Gavala <i>et al.</i> (2003)
30	PS+WAS	37	20	1.380	0.355	2,000	465	0.48	0.53	0.34	12.70	4.44	6.98	10.07	3.96	6.11	9.37	9.26	7.99	Bousková <i>et al.</i> (2005)
31	PS+WAS	37	20	1.380	0.385	2,000	465	0.48	0.53	0.34	13.77	4.82	7.57	11.04	4.34	6.70	10.17	10.04	8.66	
32	PS+WAS	35	27	1.300	0.250	2,700	568	0.43	0.35	0.27	8.95	3.13	4.92	7.00	2.70	4.30	8.49	7.23	7.93	De la Rubia <i>et al.</i> (2002)
33	PS+WAS	43	36	0.442	0.121	3,600	688	0.40	0.40	0.38	4.33	1.52	2.38	2.72	1.12	1.60	3.67	3.79	3.05	This work
34	PS+WAS	55	10	3.003	0.400	1,000	293	0.66	2.19	0.89	14.32	5.01	7.88	9.15	4.35	4.80	3.83	7.59	2.56	Ferrer <i>et al.</i> (2008)
35	PS+WAS	55	15	1.519	0.391	1,500	384	0.54	1.46	0.77	13.99	4.90	7.69	9.81	4.36	5.46	5.04	9.07	3.44	Benabdallah <i>et al.</i> (2006)
36	PS+WAS	55	15	2.190	0.400	1,500	384	0.54	1.46	0.77	14.32	5.01	7.88	10.11	4.47	5.64	5.16	9.28	3.52	De la Rubia <i>et al.</i> (2006)
37	PS+WAS	55	20	1.870	0.220	2,000	465	0.48	1.10	0.70	7.88	2.76	4.33	4.81	2.28	2.53	3.45	5.74	2.41	De la Rubia <i>et al.</i> (2002)
38	PS+WAS	55	20	1.433	0.257	2,000	465	0.48	1.10	0.70	9.18	3.21	5.05	5.98	2.73	3.25	4.03	6.70	2.81	Gavala <i>et al.</i> (2003)
39	PS+WAS	55	20	1.870	0.260	2,000	465	0.48	1.10	0.70	9.31	3.26	5.12	6.10	2.78	3.32	4.08	6.79	2.84	De la Rubia <i>et al.</i> (2006)
40	PS+WAS	55	20	1.380	0.417	2,000	465	0.48	1.10	0.70	14.91	5.22	8.20	11.14	4.74	6.40	6.54	10.87	4.56	Bousková <i>et al.</i> (2005)
41	PS+WAS	55	20	1.380	0.451	2,000	465	0.48	1.10	0.70	16.14	5.65	8.88	12.24	5.17	7.08	7.08	11.77	4.93	
42	PS+WAS	55	27	1.480	0.190	2,700	568	0.43	0.81	0.64	6.80	2.38	3.74	4.24	1.95	2.29	3.61	5.49	2.58	De la Rubia <i>et al.</i> (2006)
43	PS+WAS	55	40	0.800	0.170	4,000	738	0.39	0.55	0.56	6.09	2.13	3.35	3.98	1.74	2.24	4.07	5.46	3.02	
44	PS+WAS	55	75	0.420	0.020	7,500	1,122	0.35	0.29	0.45	0.72	0.25	0.39	-0.45	-0.10	-0.35	0.66	0.72	0.53	

**Table 14.** Calculations for the results from the literature review (Tables 6.1 -6.2), in an insulated digester with energy recovery from biogas and effluent sludge, at 0 °C. (Q=100 m<sup>3</sup> sludge d<sup>-1</sup>; T<sub>amb</sub>=0 °C; Heat transfer coef=1)

N°	Sludge	T °C	SRT d	OLR kg VS m <sup>-3</sup> d <sup>-1</sup>	CH4 rate m <sup>3</sup> CH <sub>4</sub> m <sup>-3</sup> d <sup>-1</sup>	V m <sup>3</sup>	Surface area m <sup>2</sup>	Input electricity MJ d <sup>-1</sup> m <sup>-3</sup>	Input heat MJ d <sup>-1</sup> m <sup>-3</sup>	Heat loss MJ d <sup>-1</sup> m <sup>-3</sup>	Output energy MJ d <sup>-1</sup> m <sup>-3</sup>	Output electricity MJ d <sup>-1</sup> m <sup>-3</sup>	Output heat MJ d <sup>-1</sup> m <sup>-3</sup>	Energy balance MJ d <sup>-1</sup> m <sup>-3</sup>	Electricity balance MJ d <sup>-1</sup> m <sup>-3</sup>	Heat balance MJ d <sup>-1</sup> m <sup>-3</sup>	Energy ratio	Electricity ratio	Heat ratio	Reference
15	WAS	35	8	n.d.	0.055	800	252	0.75	1.96	0.95	1.95	0.68	1.08	-1.90	-0.07	-1.84	0.53	0.91	0.37	Lafitte-Trouqué and Forster (2002)
16	WAS	35	10	n.d.	0.057	1,000	293	0.66	1.57	0.89	2.05	0.72	1.13	-1.27	0.06	-1.33	0.66	1.09	0.46	
17	WAS	37.6	21	1,000	0.117	2,100	480	0.47	0.82	0.74	4.19	1.47	2.30	1.73	0.99	0.74	2.05	3.11	1.47	
18	WAS	38.5	20	0.800	0.046	2,000	465	0.48	0.89	0.77	1.63	0.57	0.90	-0.68	0.09	-0.77	0.76	1.19	0.54	Bolzonella <i>et al.</i> (2005)
19	WAS	36	22	0.800	0.098	2,200	495	0.46	0.74	0.70	3.49	1.22	1.92	1.24	0.76	0.48	1.83	2.63	1.33	
20	WAS	35	33	0.700	0.052	3,300	649	0.41	0.48	0.59	1.86	0.65	1.02	0.20	0.24	-0.05	1.26	1.59	0.96	
21	WAS	34.5	37.2	0.530	0.068	3,720	703	0.40	0.41	0.56	2.43	0.85	1.34	0.82	0.46	0.36	1.77	2.15	1.37	Mininni <i>et al.</i> (2006)
22	WAS	34	40	1,000	0.026	4,000	738	0.39	0.38	0.54	0.93	0.33	0.51	-0.47	-0.06	-0.41	0.71	0.84	0.56	Bolzonella <i>et al.</i> (2005)
23	WAS	55	8	n.d.	0.160	800	252	0.75	3.53	1.50	5.72	2.00	3.15	-0.62	1.25	-1.88	0.99	2.67	0.63	Lafitte-Trouqué and Forster (2002)
24	WAS	55	10	n.d.	0.112	1,000	293	0.66	2.82	1.39	4.00	1.40	2.20	-1.27	0.74	-2.01	0.82	2.12	0.52	
25	WAS	55	18	n.d.	0.080	1,800	433	0.50	1.57	1.14	2.86	1.00	1.58	-0.63	0.50	-1.14	0.89	2.00	0.58	Pavan <i>et al.</i> (2006)
26	PS	55	15	0.693	0.146	1,500	384	0.54	1.88	1.22	5.23	1.83	2.88	1.07	1.29	-0.22	1.44	3.39	0.93	Lu <i>et al.</i> (2007)
27	n.d.	38	19	1.130	0.241	1,900	449	0.49	0.92	0.78	8.61	3.01	4.74	5.56	2.52	3.03	3.93	6.16	2.78	Zábranská <i>et al.</i> (2006)
28	PS+WAS	35	20	1.140	0.229	2,000	465	0.48	0.78	0.70	8.20	2.87	4.51	5.41	2.39	3.02	4.17	5.98	3.03	Benabdallah <i>et al.</i> (2006)
28	PS+WAS	37	20	1.433	0.250	2,000	465	0.48	0.85	0.74	8.95	3.13	4.92	5.99	2.65	3.33	4.33	6.53	3.10	Gavala <i>et al.</i> (2003)
30	PS+WAS	37	20	1.380	0.355	2,000	465	0.48	0.85	0.74	12.70	4.44	6.98	9.36	3.96	5.39	6.13	9.26	4.39	Bousková <i>et al.</i> (2005)
31	PS+WAS	37	20	1.380	0.385	2,000	465	0.48	0.85	0.74	13.77	4.82	7.57	10.32	4.34	5.98	6.65	10.04	4.76	
32	PS+WAS	35	27	1.300	0.250	2,700	568	0.43	0.58	0.64	8.95	3.13	4.92	6.40	2.70	3.71	5.42	7.23	4.05	De la Rubia <i>et al.</i> (2002)
33	PS+WAS	43	36	0.442	0.121	3,600	688	0.40	0.57	0.71	4.33	1.52	2.38	2.21	1.12	1.10	2.57	3.79	1.85	This work
34	PS+WAS	55	10	3.003	0.400	1,000	293	0.66	2.82	1.39	14.32	5.01	7.88	8.01	4.35	3.66	2.94	7.59	1.87	Ferrer <i>et al.</i> (2008)
35	PS+WAS	55	15	1.519	0.391	1,500	384	0.54	1.88	1.22	13.99	4.90	7.69	8.95	4.36	4.60	3.85	9.07	2.48	Benabdallah <i>et al.</i> (2006)
36	PS+WAS	55	15	2.190	0.400	1,500	384	0.54	1.88	1.22	14.32	5.01	7.88	9.25	4.47	4.78	3.94	9.28	2.54	De la Rubia <i>et al.</i> (2006)
37	PS+WAS	55	20	1.870	0.220	2,000	465	0.48	1.41	1.10	7.88	2.76	4.33	4.09	2.28	1.82	2.63	5.74	1.72	De la Rubia <i>et al.</i> (2002)
38	PS+WAS	55	20	1.433	0.257	2,000	465	0.48	1.41	1.10	9.18	3.21	5.05	5.27	2.73	2.53	3.07	6.70	2.01	Gavala <i>et al.</i> (2003)
39	PS+WAS	55	20	1.870	0.260	2,000	465	0.48	1.41	1.10	9.31	3.26	5.12	5.38	2.78	2.60	3.11	6.79	2.04	De la Rubia <i>et al.</i> (2006)
40	PS+WAS	55	20	1.380	0.417	2,000	465	0.48	1.41	1.10	14.91	5.22	8.20	10.43	4.74	5.69	4.98	10.87	3.26	Bousková <i>et al.</i> (2005)
41	PS+WAS	55	20	1.380	0.451	2,000	465	0.48	1.41	1.10	16.14	5.65	8.88	11.53	5.17	6.36	5.39	11.77	3.53	
42	PS+WAS	55	27	1.480	0.190	2,700	568	0.43	1.05	1.00	6.80	2.38	3.74	3.64	1.95	1.70	2.75	5.49	1.83	De la Rubia <i>et al.</i> (2006)
43	PS+WAS	55	40	0.800	0.170	4,000	738	0.39	0.71	0.88	6.09	2.13	3.35	3.51	1.74	1.77	3.09	5.46	2.12	
44	PS+WAS	55	75	0.420	0.020	7,500	1,122	0.35	0.38	0.71	0.72	0.25	0.39	-0.79	-0.10	-0.69	0.50	0.72	0.36	

**Table 15.** Calculations for the results from the literature review (Tables 6.1 -6.2), in a non-insulated digester with energy recovery from biogas and effluent sludge, at 20 °C ( $Q=100\text{ m}^3\text{ sludge d}^{-1}$ ;  $T_{\text{amb}}=20\text{ °C}$ ; Heat transf coef=5)

N°	Sludge	T °C	SRT d	OLR $\frac{\text{kg VS}}{\text{m}^3\text{ d}^1}$	CH4 rate $\frac{\text{m}^3\text{ CH}_4}{\text{m}^3\text{ d}^1}$	V $\text{m}^3$	Surface area $\text{m}^2$	Input electricity $\text{MJ d}^{-1}\text{ m}^{-3}$	Input heat $\text{MJ d}^{-1}\text{ m}^{-3}$	Heat loss $\text{MJ d}^{-1}\text{ m}^{-3}$	Output energy $\text{MJ d}^{-1}\text{ m}^{-3}$	Output electricity $\text{MJ d}^{-1}\text{ m}^{-3}$	Output heat $\text{MJ d}^{-1}\text{ m}^{-3}$	Energy balance $\text{MJ d}^{-1}\text{ m}^{-3}$	Electricity balance $\text{MJ d}^{-1}\text{ m}^{-3}$	Heat balance $\text{MJ d}^{-1}\text{ m}^{-3}$	Energy ratio	Electricity ratio	Heat ratio	Reference
15	WAS	35	8	n.d.	0.055	800	252	0.75	1.18	2.04	1.95	0.68	1.08	-2.21	-0.07	-2.15	0.49	0.91	0.33	Lafitte-Trouqué and Forster (2002)
16	WAS	35	10	n.d.	0.057	1,000	293	0.66	0.94	1.90	2.05	0.72	1.13	-1.66	0.06	-1.71	0.59	1.09	0.40	
17	WAS	37.6	21	1.000	0.117	2,100	480	0.47	0.53	1.74	4.19	1.47	2.30	1.03	0.99	0.04	1.53	3.11	1.02	Bolzonnella <i>et al.</i> (2005)
18	WAS	38.5	20	0.800	0.046	2,000	465	0.48	0.58	1.86	1.63	0.57	0.90	-1.45	0.09	-1.54	0.56	1.19	0.37	
19	WAS	36	22	0.800	0.098	2,200	495	0.46	0.46	1.56	3.49	1.22	1.92	0.67	0.76	-0.09	1.41	2.63	0.95	
20	WAS	35	33	0.700	0.052	3,300	649	0.41	0.29	1.27	1.86	0.65	1.02	-0.29	0.24	-0.54	0.95	1.59	0.66	
21	WAS	34.5	37.2	0.530	0.068	3,720	703	0.40	0.24	1.18	2.43	0.85	1.34	0.37	0.46	-0.09	1.33	2.15	0.94	Mininni <i>et al.</i> (2006)
22	WAS	34	40	1.000	0.026	4,000	738	0.39	0.22	1.12	0.93	0.33	0.51	-0.89	-0.06	-0.82	0.54	0.84	0.38	Bolzonnella <i>et al.</i> (2005)
23	WAS	55	8	n.d.	0.160	800	252	0.75	2.74	4.77	5.72	2.00	3.15	-3.11	1.25	-4.37	0.69	2.67	0.42	Lafitte-Trouqué and Forster (2002)
24	WAS	55	10	n.d.	0.112	1,000	293	0.66	2.19	4.43	4.00	1.40	2.20	-3.68	0.74	-4.42	0.55	2.12	0.33	
25	WAS	55	18	n.d.	0.080	1,800	433	0.50	1.22	3.64	2.86	1.00	1.58	-2.78	0.50	-3.28	0.53	2.00	0.32	Pavan <i>et al.</i> (2006)
26	PS	55	15	0.693	0.146	1,500	384	0.54	1.46	3.87	5.23	1.83	2.88	-1.16	1.29	-2.45	0.89	3.39	0.54	Lu <i>et al.</i> (2007)
27	n.d.	38	19	1.130	0.241	1,900	449	0.49	0.59	1.84	8.61	3.01	4.74	4.83	2.52	2.30	2.95	6.16	1.95	Zábranská <i>et al.</i> (2006)
28	PS+WAS	35	20	1.140	0.229	2,000	465	0.48	0.47	1.51	8.20	2.87	4.51	4.92	2.39	2.53	3.34	5.98	2.28	Benabdallah <i>et al.</i> (2006)
28	PS+WAS	37	20	1.433	0.250	2,000	465	0.48	0.53	1.71	8.95	3.13	4.92	5.34	2.65	2.68	3.29	6.53	2.20	Gavala <i>et al.</i> (2003)
30	PS+WAS	37	20	1.380	0.355	2,000	465	0.48	0.53	1.71	12.70	4.44	6.98	8.71	3.96	4.74	4.67	9.26	3.12	Bousková <i>et al.</i> (2005)
31	PS+WAS	37	20	1.380	0.385	2,000	465	0.48	0.53	1.71	13.77	4.82	7.57	9.67	4.34	5.33	5.06	10.04	3.38	
32	PS+WAS	35	27	1.300	0.250	2,700	568	0.43	0.35	1.36	8.95	3.13	4.92	5.91	2.70	3.21	4.17	7.23	2.88	De la Rubia <i>et al.</i> (2002)
33	PS+WAS	43	36	0.442	0.121	3,600	688	0.40	0.40	1.90	4.33	1.52	2.38	1.20	1.12	0.08	1.60	3.79	1.04	This work
34	PS+WAS	55	10	3.003	0.400	1,000	293	0.66	2.19	4.43	14.32	5.01	7.88	5.60	4.35	1.25	1.97	7.59	1.19	Ferrer <i>et al.</i> (2008)
35	PS+WAS	55	15	1.519	0.391	1,500	384	0.54	1.46	3.87	13.99	4.90	7.69	6.72	4.36	2.36	2.38	9.07	1.44	Benabdallah <i>et al.</i> (2006)
36	PS+WAS	55	15	2.190	0.400	1,500	384	0.54	1.46	3.87	14.32	5.01	7.88	7.02	4.47	2.54	2.44	9.28	1.48	De la Rubia <i>et al.</i> (2006)
37	PS+WAS	55	20	1.870	0.220	2,000	465	0.48	1.10	3.52	7.88	2.76	4.33	2.00	2.28	-0.28	1.55	5.74	0.94	De la Rubia <i>et al.</i> (2002)
38	PS+WAS	55	20	1.433	0.257	2,000	465	0.48	1.10	3.52	9.18	3.21	5.05	3.17	2.73	0.44	1.80	6.70	1.09	Gavala <i>et al.</i> (2003)
39	PS+WAS	55	20	1.870	0.260	2,000	465	0.48	1.10	3.52	9.31	3.26	5.12	3.28	2.78	0.51	1.83	6.79	1.11	De la Rubia <i>et al.</i> (2006)
40	PS+WAS	55	20	1.380	0.417	2,000	465	0.48	1.10	3.52	14.91	5.22	8.20	8.33	4.74	3.59	2.93	10.87	1.78	Bousková <i>et al.</i> (2005)
41	PS+WAS	55	20	1.380	0.451	2,000	465	0.48	1.10	3.52	16.14	5.65	8.88	9.43	5.17	4.26	3.17	11.77	1.92	
42	PS+WAS	55	27	1.480	0.190	2,700	568	0.43	0.81	3.18	6.80	2.38	3.74	1.70	1.95	-0.25	1.54	5.49	0.94	De la Rubia <i>et al.</i> (2006)
43	PS+WAS	55	40	0.800	0.170	4,000	738	0.39	0.55	2.79	6.09	2.13	3.35	1.75	1.74	0.01	1.63	5.46	1.00	
44	PS+WAS	55	75	0.420	0.020	7,500	1,122	0.35	0.29	2.26	0.72	0.25	0.39	-2.26	-0.10	-2.16	0.25	0.72	0.15	

**Table 16.** Calculations for the results from the literature review (Tables 6.1 -6.2), in a non-insulated digester with energy recovery from biogas and effluent sludge, at 0 °C. (Q=100 m<sup>3</sup> sludge d<sup>-1</sup>; T<sub>amb</sub>=0 °C; Heat transf coef=5)

N°	Sludge	T °C	SRT d	OLR kg VS m <sup>-3</sup> d <sup>-1</sup>	CH4 rate m <sup>3</sup> CH <sub>4</sub> m <sup>-3</sup> d <sup>-1</sup>	V m <sup>3</sup>	Surface area m <sup>2</sup>	Input electricity MJ d <sup>-1</sup> m <sup>-3</sup>	Input heat MJ d <sup>-1</sup> m <sup>-3</sup>	Heat loss MJ d <sup>-1</sup> m <sup>-3</sup>	Output energy MJ d <sup>-1</sup> m <sup>-3</sup>	Output electricity MJ d <sup>-1</sup> m <sup>-3</sup>	Output heat MJ d <sup>-1</sup> m <sup>-3</sup>	Energy balance MJ d <sup>-1</sup> m <sup>-3</sup>	Electricity balance MJ d <sup>-1</sup> m <sup>-3</sup>	Heat balance MJ d <sup>-1</sup> m <sup>-3</sup>	Energy ratio	Electricity ratio	Heat ratio	Reference
15	WAS	35	8	n.d.	0.055	800	252	0.75	1.96	4.77	1.95	0.68	1.08	-5.72	-0.07	-5.66	0.26	0.91	0.16	Lafitte-Trouqué and Forster (2002)
16	WAS	35	10	n.d.	0.057	1,000	293	0.66	1.57	4.43	2.05	0.72	1.13	-4.81	0.06	-4.87	0.31	1.09	0.19	
17	WAS	37.6	21	1.000	0.117	2,100	480	0.47	0.82	3.72	4.19	1.47	2.30	-1.24	0.99	-2.24	0.84	3.11	0.51	
18	WAS	38.5	20	0.800	0.046	2,000	465	0.48	0.89	3.87	1.63	0.57	0.90	-3.77	0.09	-3.86	0.31	1.19	0.19	Bolzonella <i>et al.</i> (2005)
19	WAS	36	22	0.800	0.098	2,200	495	0.46	0.74	3.50	3.49	1.22	1.92	-1.57	0.76	-2.32	0.74	2.63	0.45	
20	WAS	35	33	0.700	0.052	3,300	649	0.41	0.48	2.97	1.86	0.65	1.02	-2.18	0.24	-2.43	0.48	1.59	0.30	
21	WAS	34.5	37.2	0.530	0.068	3,720	703	0.40	0.41	2.82	2.43	0.85	1.34	-1.44	0.46	-1.89	0.67	2.15	0.41	Mininni <i>et al.</i> (2006)
22	WAS	34	40	1.000	0.026	4,000	738	0.39	0.38	2.71	0.93	0.33	0.51	-2.64	-0.06	-2.57	0.27	0.84	0.17	Bolzonella <i>et al.</i> (2005)
23	WAS	55	8	n.d.	0.160	800	252	0.75	3.53	7.50	5.72	2.00	3.15	-6.62	1.25	-7.88	0.49	2.67	0.29	Lafitte-Trouqué and Forster (2002)
24	WAS	55	10	n.d.	0.112	1,000	293	0.66	2.82	6.96	4.00	1.40	2.20	-6.84	0.74	-7.58	0.38	2.12	0.23	
25	WAS	55	18	n.d.	0.080	1,800	433	0.50	1.57	5.72	2.86	1.00	1.58	-5.21	0.50	-5.71	0.37	2.00	0.22	Pavan <i>et al.</i> (2006)
26	PS	55	15	0.693	0.146	1,500	384	0.54	1.88	6.08	5.23	1.83	2.88	-3.79	1.29	-5.08	0.62	3.39	0.36	Lu <i>et al.</i> (2007)
27	n.d.	38	19	1.130	0.241	1,900	449	0.49	0.92	3.88	8.61	3.01	4.74	2.45	2.52	-0.07	1.63	6.16	0.99	Zábranská <i>et al.</i> (2006)
28	PS+WAS	35	20	1.140	0.229	2,000	465	0.48	0.78	3.52	8.20	2.87	4.51	2.60	2.39	0.21	1.72	5.98	1.05	Benabdallah <i>et al.</i> (2006)
28	PS+WAS	37	20	1.433	0.250	2,000	465	0.48	0.85	3.72	8.95	3.13	4.92	3.02	2.65	0.36	1.78	6.53	1.08	Gavala <i>et al.</i> (2003)
30	PS+WAS	37	20	1.380	0.355	2,000	465	0.48	0.85	3.72	12.70	4.44	6.98	6.38	3.96	2.42	2.52	9.26	1.53	Bousková <i>et al.</i> (2005)
31	PS+WAS	37	20	1.380	0.385	2,000	465	0.48	0.85	3.72	13.77	4.82	7.57	7.35	4.34	3.01	2.73	10.04	1.66	
32	PS+WAS	35	27	1.300	0.250	2,700	568	0.43	0.58	3.18	8.95	3.13	4.92	3.86	2.70	1.16	2.13	7.23	1.31	De la Rubia <i>et al.</i> (2002)
33	PS+WAS	43	36	0.442	0.121	3,600	688	0.40	0.57	3.55	4.33	1.52	2.38	-0.63	1.12	-1.74	0.96	3.79	0.58	This work
34	PS+WAS	55	10	3.003	0.400	1,000	293	0.66	2.82	6.96	14.32	5.01	7.88	2.45	4.35	-1.91	1.37	7.59	0.81	Ferrer <i>et al.</i> (2008)
35	PS+WAS	55	15	1.519	0.391	1,500	384	0.54	1.88	6.08	13.99	4.90	7.69	4.09	4.36	-0.27	1.65	9.07	0.97	Benabdallah <i>et al.</i> (2006)
36	PS+WAS	55	15	2.190	0.400	1,500	384	0.54	1.88	6.08	14.32	5.01	7.88	4.39	4.47	-0.08	1.68	9.28	0.99	De la Rubia <i>et al.</i> (2006)
37	PS+WAS	55	20	1.870	0.220	2,000	465	0.48	1.41	5.52	7.88	2.76	4.33	-0.33	2.28	-2.60	1.06	5.74	0.62	De la Rubia <i>et al.</i> (2002)
38	PS+WAS	55	20	1.433	0.257	2,000	465	0.48	1.41	5.52	9.18	3.21	5.05	0.85	2.73	-1.88	1.24	6.70	0.73	Gavala <i>et al.</i> (2003)
39	PS+WAS	55	20	1.870	0.260	2,000	465	0.48	1.41	5.52	9.31	3.26	5.12	0.96	2.78	-1.82	1.26	6.79	0.74	De la Rubia <i>et al.</i> (2006)
40	PS+WAS	55	20	1.380	0.417	2,000	465	0.48	1.41	5.52	14.91	5.22	8.20	6.01	4.74	1.27	2.01	10.87	1.18	
41	PS+WAS	55	20	1.380	0.451	2,000	465	0.48	1.41	5.52	16.14	5.65	8.88	7.11	5.17	1.94	2.18	11.77	1.28	Bousková <i>et al.</i> (2005)
42	PS+WAS	55	27	1.480	0.190	2,700	568	0.43	1.05	5.00	6.80	2.38	3.74	-0.35	1.95	-2.30	1.05	5.49	0.62	De la Rubia <i>et al.</i> (2006)
43	PS+WAS	55	40	0.800	0.170	4,000	738	0.39	0.71	4.38	6.09	2.13	3.35	0.00	1.74	-1.74	1.11	5.46	0.66	
44	PS+WAS	55	75	0.420	0.020	7,500	1,122	0.35	0.38	3.56	0.72	0.25	0.39	-3.64	-0.10	-3.54	0.17	0.72	0.10	

**Table 17.** Calculations for 2 stage results from Chapter 5 (Table 6.4) and literature review (Tables 6.1 –6.2), in insulated digesters with energy recovery from biogas, at 20 °C (Q=100 m<sup>3</sup> sludge d<sup>-1</sup>; T<sub>amb</sub>=20 °C; Heat transf coef=1)

N°	Sludge	T	SRT	OLR	CH4 rate	V	Surface area	Input electricity	Input heat	Heat loss	Output energy	Output electricity	Output heat	Energy balance	Electricity balance	Heat balance	Energy ratio	Electricity ratio	Heat ratio	Reference
		°C	d	kg VS m <sup>3</sup> d <sup>-1</sup>	m <sup>3</sup> CH <sub>4</sub> m <sup>3</sup> d <sup>-1</sup>	m <sup>3</sup>	m <sup>2</sup>	MJ d <sup>-1</sup> m <sup>-3</sup>												
45	PS+WAS	70/55	0.375+10	3.045	0.560	37.5+1,000	33+293	0.65	20.14	2.07	19.32	6.76	10.63	-5.47	6.12	-11.59	0.85	10.45	0.48	This work (Chapter 5) Ferrer <i>et al.</i> (2008)
46	PS+WAS	70/55	1+10	2.659	0.480	100+1,000	63+293	0.63	19.00	1.96	15.62	5.47	8.59	-7.52	4.84	-12.36	0.72	8.72	0.41	
47	PS+WAS	70/55	2+10	2.778	0.590	200+1,000	100+293	0.60	17.42	1.79	17.60	6.16	9.68	-3.97	5.56	-9.53	0.89	10.27	0.50	
48	PS+WAS	38/35	12+11		0.320	1,200+1,100	331+312	0.46	3.27	0.39	5.48	1.92	3.01	0.81	1.46	-0.65	1.33	4.20	0.82	Zabranska <i>et al.</i> (2000)
49	PS+WAS	55/52	12+11		0.407	1,200+1,100	331+312	0.46	6.36	0.79	6.96	2.44	3.83	-1.34	1.98	-3.32	0.92	5.34	0.54	
50	PS+WAS	38/35	6.7+18.6		0.231	670+1,860	224+443	0.44	2.97	0.50	6.07	2.13	3.34	1.55	1.68	-0.13	1.55	4.81	0.96	Zabranska <i>et al.</i> (2006)
51	PS	70/55	2+13	0.35	0.242	200+1,300	100+349	0.54	13.93	1.71	7.49	2.62	4.12	-9.44	2.08	-11.52	0.46	4.86	0.26	Lu <i>et al.</i> (2007)

**Table 18.** Calculations for 2 stage results from Chapter 5 (Table 6.4) and literature review (Tables 6.1 –6.2), in insulated digesters with energy recovery from biogas, at 0 °C (Q=100 m<sup>3</sup> sludge d<sup>-1</sup>; T<sub>amb</sub>=0 °C; Heat transf coef=1)

N°	Sludge	T	SRT	OLR	CH4 rate	V	Surface area	Input electricity	Input heat	Heat loss	Output energy	Output electricity	Output heat	Energy balance	Electricity balance	Heat balance	Energy ratio	Electricity ratio	Heat ratio	Reference
		°C	d	kg VS m <sup>3</sup> d <sup>-1</sup>	m <sup>3</sup> CH <sub>4</sub> m <sup>3</sup> d <sup>-1</sup>	m <sup>3</sup>	m <sup>2</sup>	MJ d <sup>-1</sup> m <sup>-3</sup>												
45	PS+WAS	70/55	0.375+10	3.045	0.560	37.5+1,000	33+293	0.65	24.17	3.05	19.32	6.76	10.63	-10.48	6.12	-16.59	0.69	10.45	0.39	This work (Chapter 5) Ferrer <i>et al.</i> (2008)
46	PS+WAS	70/55	1+10	2.659	0.480	100+1,000	63+293	0.63	22.80	2.88	15.62	5.47	8.59	-12.24	4.84	-17.08	0.59	8.72	0.33	
47	PS+WAS	70/55	2+10	2.778	0.590	200+1,000	100+293	0.60	20.90	2.64	17.60	6.16	9.68	-8.29	5.56	-13.86	0.73	10.27	0.41	
48	PS+WAS	38/35	12+11		0.320	1,200+1,100	331+312	0.46	5.09	0.86	5.48	1.92	3.01	-1.47	1.46	-2.93	0.86	4.20	0.51	Zabranska <i>et al.</i> (2000)
49	PS+WAS	55/52	12+11		0.407	1,200+1,100	331+312	0.46	8.18	1.25	6.96	2.44	3.83	-3.62	1.98	-5.60	0.70	5.34	0.41	
50	PS+WAS	38/35	6.7+18.6		0.231	670+1,860	224+443	0.44	4.63	1.10	6.07	2.13	3.34	-0.71	1.68	-2.39	0.98	4.81	0.58	Zabranska <i>et al.</i> (2006)
51	PS	70/55	2+13	0.35	0.242	200+1,300	100+349	0.54	16.72	2.51	7.49	2.62	4.12	-13.03	2.08	-15.11	0.38	4.86	0.21	Lu <i>et al.</i> (2007)

**Table 19.** Calculations for 2 stage results from Chapter 5 (Table 6.4) and literature review (Tables 6.1 -6.2), in non-insulated digesters with energy recovery from biogas, at 20 °C (Q=100 m<sup>3</sup> sludge d<sup>-1</sup>; T<sub>amb</sub>=20 °C; Heat transf coef=5)

N°	Sludge	T	SRT	OLR	CH4 rate	V	Surface area	Input electricity	Input heat	Heat loss	Output energy	Output electricity	Output heat	Energy balance	Electricity balance	Heat balance	Energy ratio	Electricity ratio	Heat ratio	Reference
		°C	d	kg VS m <sup>-3</sup> d <sup>-1</sup>	m <sup>3</sup> CH <sub>4</sub> m <sup>-3</sup> d <sup>-1</sup>	m <sup>3</sup>	m <sup>2</sup>	MJ d <sup>-1</sup> m <sup>-3</sup>												
45	PS+WAS	70/55	0.375+10	3.045	0.560	37.5+1,000	33+293	0.65	20.14	10.37	19.32	6.76	10.63	-13.77	6.12	-19.88	0.62	10.45	0.35	This work (Chapter 5) Ferrer <i>et al.</i> (2008)
46	PS+WAS	70/55	1+10	2.659	0.480	100+1,000	63+293	0.63	19.00	9.78	15.62	5.47	8.59	-15.35	4.84	-20.19	0.53	8.72	0.30	
47	PS+WAS	70/55	2+10	2.778	0.590	200+1,000	100+293	0.60	17.42	8.96	17.60	6.16	9.68	-11.14	5.56	-16.70	0.65	10.27	0.37	
48	PS+WAS	38/35	12+11		0.320	1,200+1,100	331+312	0.46	3.27	1.93	5.48	1.92	3.01	-0.73	1.46	-2.19	0.97	4.20	0.58	Zabranska <i>et al.</i> (2000)
49	PS+WAS	55/52	12+11		0.407	1,200+1,100	331+312	0.46	6.36	3.93	6.96	2.44	3.83	-4.48	1.98	-6.46	0.65	5.34	0.37	
50	PS+WAS	38/35	6.7+18.6		0.231	670+1,860	224+443	0.44	2.97	2.50	6.07	2.13	3.34	-0.45	1.68	-2.13	1.03	4.81	0.61	Zabranska <i>et al.</i> (2006)
51	PS	70/55	2+13	0.35	0.242	200+1,300	100+349	0.54	13.93	8.54	7.49	2.62	4.12	-16.27	2.08	-18.35	0.33	4.86	0.18	Lu <i>et al.</i> (2007)

**Table 20.** Calculations for 2 stage results from Chapter 5 (Table 6.4) and literature review (Tables 6.1 -6.2), in non-insulated digesters with energy recovery from biogas, at 0 °C (Q=100 m<sup>3</sup> sludge d<sup>-1</sup>; T<sub>amb</sub>=0 °C; Heat transf coef=5)

N°	Sludge	T	SRT	OLR	CH4 rate	V	Surface area	Input electricity	Input heat	Heat loss	Output energy	Output electricity	Output heat	Energy balance	Electricity balance	Heat balance	Energy ratio	Electricity ratio	Heat ratio	Reference
		°C	d	kg VS m <sup>-3</sup> d <sup>-1</sup>	m <sup>3</sup> CH <sub>4</sub> m <sup>-3</sup> d <sup>-1</sup>	m <sup>3</sup>	m <sup>2</sup>	MJ d <sup>-1</sup> m <sup>-3</sup>												
45	PS+WAS	70/55	0.375+10	3.045	0.560	37.5+1,000	33+293	0.65	24.17	15.25	19.32	6.76	10.63	-22.68	6.12	-28.79	0.48	10.45	0.27	This work (Chapter 5) Ferrer <i>et al.</i> (2008)
46	PS+WAS	70/55	1+10	2.659	0.480	100+1,000	63+293	0.63	22.80	14.38	15.62	5.47	8.59	-23.75	4.84	-28.59	0.41	8.72	0.23	
47	PS+WAS	70/55	2+10	2.778	0.590	200+1,000	100+293	0.60	20.90	13.18	17.60	6.16	9.68	-18.84	5.56	-24.40	0.51	10.27	0.28	
48	PS+WAS	38/35	12+11		0.320	1,200+1,100	331+312	0.46	5.09	4.28	5.48	1.92	3.01	-4.90	1.46	-6.36	0.56	4.20	0.32	Zabranska <i>et al.</i> (2000)
49	PS+WAS	55/52	12+11		0.407	1,200+1,100	331+312	0.46	8.18	6.27	6.96	2.44	3.83	-8.64	1.98	-10.62	0.47	5.34	0.26	
50	PS+WAS	38/35	6.7+18.6		0.231	670+1,860	224+443	0.44	4.63	5.52	6.07	2.13	3.34	-5.12	1.68	-6.81	0.57	4.81	0.33	Zabranska <i>et al.</i> (2006)
51	PS	70/55	2+13	0.35	0.242	200+1,300	100+349	0.54	16.72	12.56	7.49	2.62	4.12	-23.08	2.08	-25.16	0.25	4.86	0.14	Lu <i>et al.</i> (2007)

**Table 21.** Calculations for 2 stage results from Chapter 5 (Table 6.4) and literature review (Tables 6.1 –6.2), in insulated digesters with energy recovery from biogas and effluent sludge, at 20 °C (Q=100 m<sup>3</sup> sludge d<sup>-1</sup>; T<sub>amb</sub>=20 °C; Heat transf coef=1)

N°	Sludge	T	SRT	OLR	CH4 rate	V	Surface area	Input electricity	Input heat	Heat loss	Output energy	Output electricity	Output heat	Energy balance	Electricity balance	Heat balance	Energy ratio	Electricity ratio	Heat ratio	Reference	
		°C	d	kg VS CH <sub>4</sub> m <sup>3</sup> d <sup>-1</sup>	m <sup>3</sup> CH <sub>4</sub> m <sup>3</sup> d <sup>-1</sup>	m <sup>3</sup>	m <sup>2</sup>	MJ d <sup>-1</sup> m <sup>-3</sup>													
45	PS+WAS	70/55	0.375+10	3.045	0.560	37.5+1,000	33+293	0.82	-3.02	2.07	19.32	6.76	10.63	17.52	5.94	11.58	193.23	8.24	106.28	This work (Chapter 5) Ferrer <i>et al.</i> (2008)	
46	PS+WAS	70/55	1+10	2.659	0.480	100+1,000	63+293	0.79	-2.85	1.96	15.62	5.47	8.59	14.16	4.68	9.49	156.22	6.91	85.92		
47	PS+WAS	70/55	2+10	2.778	0.590	200+1,000	100+293	0.75	-2.61	1.79	17.60	6.16	9.68	15.91	5.41	10.50	176.02	8.21	96.81		
48	PS+WAS	38/35	12+11		0.320	1,200+1,100	331+312	0.53	-0.05	0.39	5.48	1.92	3.01	4.06	1.38	2.68	6.31	3.58	9.06		Zabranska <i>et al.</i> (2000)
49	PS+WAS	55/52	12+11		0.407	1,200+1,100	331+312	0.53	0.41	0.79	6.96	2.44	3.83	4.54	1.90	2.63	4.03	4.56	3.21		Zabranska <i>et al.</i> (2006)
50	PS+WAS	38/35	6.7+18.6		0.231	670+1,860	224+443	0.51	-0.05	0.50	6.07	2.13	3.34	4.50	1.61	2.89	6.31	4.14	7.43	Zabranska <i>et al.</i> (2006)	
51	PS	70/55	2+13	0.35	0.242	200+1,300	100+349	0.66	-2.09	1.71	7.49	2.62	4.12	6.47	1.96	4.50	74.93	3.97	41.21	Lu <i>et al.</i> (2007)	

**Table 22.** Calculations for 2 stage results from Chapter 5 (Table 6.4) and literature review (Tables 6.1 –6.2), in insulated digesters with energy recovery from biogas and effluent sludge, at 0 °C (Q=100 m<sup>3</sup> sludge d<sup>-1</sup>; T<sub>amb</sub>=0 °C; Heat transf coef=1)

N°	Sludge	T	SRT	OLR	CH4 rate	V	Surface area	Input electricity	Input heat	Heat loss	Output energy	Output electricity	Output heat	Energy balance	Electricity balance	Heat balance	Energy ratio	Electricity ratio	Heat ratio	Reference	
		°C	d	kg VS CH <sub>4</sub> m <sup>3</sup> d <sup>-1</sup>	m <sup>3</sup> CH <sub>4</sub> m <sup>3</sup> d <sup>-1</sup>	m <sup>3</sup>	m <sup>2</sup>	MJ d <sup>-1</sup> m <sup>-3</sup>													
45	PS+WAS	70/55	0.375+10	3.045	0.560	37.5+1,000	33+293	0.82	-2.42	3.05	19.32	6.76	10.63	15.94	5.94	10.00	13.31	8.24	16.82	This work (Chapter 5) Ferrer <i>et al.</i> (2008)	
46	PS+WAS	70/55	1+10	2.659	0.480	100+1,000	63+293	0.79	-2.28	2.88	15.62	5.47	8.59	12.67	4.68	8.00	11.26	6.91	14.42		
47	PS+WAS	70/55	2+10	2.778	0.590	200+1,000	100+293	0.75	-2.09	2.64	17.60	6.16	9.68	14.55	5.41	9.13	13.58	8.21	17.72		
48	PS+WAS	38/35	12+11		0.320	1,200+1,100	331+312	0.53	0.22	0.86	5.48	1.92	3.01	3.32	1.38	1.94	3.40	3.58	2.80		Zabranska <i>et al.</i> (2000)
49	PS+WAS	55/52	12+11		0.407	1,200+1,100	331+312	0.53	0.68	1.25	6.96	2.44	3.83	3.79	1.90	1.89	2.82	4.56	1.98		Zabranska <i>et al.</i> (2006)
50	PS+WAS	38/35	6.7+18.6		0.231	670+1,860	224+443	0.51	0.20	1.10	6.07	2.13	3.34	3.65	1.61	2.04	3.34	4.14	2.56	Zabranska <i>et al.</i> (2006)	
51	PS	70/55	2+13	0.35	0.242	200+1,300	100+349	0.66	-1.67	2.51	7.49	2.62	4.12	5.24	1.96	3.28	4.99	3.97	4.91	Lu <i>et al.</i> (2007)	

**Table 23.** Calculations for 2 stage results from Chapter 5 (Table 6.4) and literature review (Tables 6.1 -6.2), in non-insulated digesters with energy recovery from biogas and effluent sludge, at 20 °C (Q=100 m<sup>3</sup> sludge d<sup>-1</sup>; T<sub>amb</sub>=20 °C; Heat transf coef=5)

N°	Sludge	T	SRT	OLR	CH4 rate	V	Surface area	Input electricity	Input heat	Heat loss	Output energy	Output electricity	Output heat	Energy balance	Electricity balance	Heat balance	Energy ratio	Electricity ratio	Heat ratio	Reference
		°C	d	kg VS m <sup>3</sup> d <sup>-1</sup>	m <sup>3</sup> CH <sub>4</sub> m <sup>3</sup> d <sup>-1</sup>	m <sup>3</sup>	m <sup>2</sup>	MJ d <sup>-1</sup> m <sup>3</sup>												
45	PS+WAS	70/55	0.375+10	3.045	0.560	37.5+1,000	33+293	0.82	-3.02	10.37	19.32	6.76	10.63	9.23	5.94	3.28	2.37	8.24	1.45	This work (Chapter 5) Ferrer <i>et al.</i> (2008)
46	PS+WAS	70/55	1+10	2.659	0.480	100+1,000	63+293	0.79	-2.85	9.78	15.62	5.47	8.59	6.34	4.68	1.66	2.02	6.91	1.24	
47	PS+WAS	70/55	2+10	2.778	0.590	200+1,000	100+293	0.75	-2.61	8.96	17.60	6.16	9.68	8.74	5.41	3.33	2.48	8.21	1.52	
48	PS+WAS	38/35	12+11		0.320	1,200+1,100	331+312	0.53	-0.05	1.93	5.48	1.92	3.01	2.51	1.38	1.13	2.27	3.58	1.60	Zabranska <i>et al.</i> (2000)
49	PS+WAS	55/52	12+11		0.407	1,200+1,100	331+312	0.53	0.41	3.93	6.96	2.44	3.83	1.39	1.90	-0.51	1.43	4.56	0.88	
50	PS+WAS	38/35	6.7+18.6		0.231	670+1,860	224+443	0.51	-0.05	2.50	6.07	2.13	3.34	2.51	1.61	0.89	2.05	4.14	1.37	Zabranska <i>et al.</i> (2006)
51	PS	70/55	2+13	0.35	0.242	200+1,300	100+349	0.66	-2.09	8.54	7.49	2.62	4.12	-0.37	1.96	-2.33	1.05	3.97	0.64	Lu <i>et al.</i> (2007)

**Table 24.** Calculations for 2 stage results from Chapter 5 (Table 6.4) and literature review (Tables 6.1 -6.2), in non-insulated digesters with energy recovery from biogas and effluent sludge, at 0 °C (Q=100 m<sup>3</sup> sludge d<sup>-1</sup>; T<sub>amb</sub>=0 °C; Heat transf coef=5)

N°	Sludge	T	SRT	OLR	CH4 rate	V	Surface area	Input electricity	Input heat	Heat loss	Output energy	Output electricity	Output heat	Energy balance	Electricity balance	Heat balance	Energy ratio	Electricity ratio	Heat ratio	Reference
		°C	d	kg VS m <sup>3</sup> d <sup>-1</sup>	m <sup>3</sup> CH <sub>4</sub> m <sup>3</sup> d <sup>-1</sup>	m <sup>3</sup>	m <sup>2</sup>	MJ d <sup>-1</sup> m <sup>3</sup>												
45	PS+WAS	70/55	0.375+10	3.045	0.560	37.5+1,000	33+293	0.82	-2.42	15.25	19.32	6.76	10.63	3.74	5.94	-2.20	1.42	8.24	0.83	This work (Chapter 5) Ferrer <i>et al.</i> (2008)
46	PS+WAS	70/55	1+10	2.659	0.480	100+1,000	63+293	0.79	-2.28	14.38	15.62	5.47	8.59	1.17	4.68	-3.51	1.21	6.91	0.71	
47	PS+WAS	70/55	2+10	2.778	0.590	200+1,000	100+293	0.75	-2.09	13.18	17.60	6.16	9.68	4.00	5.41	-1.41	1.49	8.21	0.87	
48	PS+WAS	38/35	12+11		0.320	1,200+1,100	331+312	0.53	0.22	4.28	5.48	1.92	3.01	-0.10	1.38	-1.49	1.09	3.58	0.67	Zabranska <i>et al.</i> (2000)
49	PS+WAS	55/52	12+11		0.407	1,200+1,100	331+312	0.53	0.68	6.27	6.96	2.44	3.83	-1.22	1.90	-3.13	0.93	4.56	0.55	Zabranska <i>et al.</i> (2006)
50	PS+WAS	38/35	6.7+18.6		0.231	670+1,860	224+443	0.51	0.20	5.52	6.07	2.13	3.34	-0.77	1.61	-2.38	0.97	4.14	0.58	
51	PS	70/55	2+13	0.35	0.242	200+1,300	100+349	0.66	-1.67	12.56	7.49	2.62	4.12	-4.80	1.96	-6.77	0.65	3.97	0.38	Lu <i>et al.</i> (2007)