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Technical University of Denmark

DTU

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DTU Environment
Department of Environmental Engineering

PhD Thesis
December 2010

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Preface

The work reported in this PhD thesis, entitled 'Biogas production from food-processing industrial wastes by anaerobic digestion', was conducted at the Department of Environmental Engineering at the Technical University of Denmark from July 2007 to October 2010. Professor Irini Angelidaki was the main supervisor and Researcher Kanokwan Boe was the co-supervisor.

The thesis is organized in two parts. The first part consists of an introductive review and summary; the second part contains the papers prepared to scientific journals.

In the text, the following papers are referred to by the names of the authors and their appendix number written with roman numbers.

I Fang C, Boe K, Angelidaki I. 2010. Anaerobic co-digestion of desugared molasses with cow manure; focusing on sodium and potassium inhibition. *Bioresource Technology*, in press, doi:10.1016/j.biortech.2010.09.077

II Fang C, Boe K, Angelidaki I. 2010. Anaerobic co-digestion of by-products from sugar production with cow manure. Submitted to *Water Research*.

III Fang C, Boe K, Angelidaki I. 2010. Biogas production from potato-starch processing by-products in upflow anaerobic sludge blanket (UASB) and expanded granular sludge bed (EGSB) reactors. Submitted to *Bioresource Technology*.

IV Fang C, O-Thong S, Boe K, Angelidaki I. 2010. Comparison of UASB and EGSB performance on treating raw and deoiled palm oil mill effluent (POME). Submitted to *Journal of Hazardous Materials.*

V O-Thong S, Fang C, Angelidaki I. 2010. Thermophilic anaerobic co-digestion of pre-treated empty fruit bunch with palm oil mill effluent for efficient biogas production. In revision in *Water Science and Technology*.

In addition, the following work was conducted in PhD period, while was not included in the PhD thesis.

Fang C, Min B, Angelidaki I. 2010. Nitrate as an oxidant in the cathode chamber of a Microbial Fuel Cell for both power generation and nutrient removal purposes. In revision in *Applied Biochemistry and Biotechnology*.

Fang C, Garcia H, Angelidaki I. 2007. Bioenergy investigation of biogas potential from sugar-processing by-products. Environmental Strategies and Solutions, Ph.D. Symposium of Danish Environmental Science Schools. November 5-6, 2007 in Copenhagen. Poster presentation.

Fang C, Boe K, Angelidaki I. 2010. Biogas production from potato processing wastewaters using UASB and EGSB type reactors. *BioCycle* magazine organized BioCycle West Coast Conference 2010: Composting, Organics Recycling and Renewable Energy. April 12-15, 2010 in San Diego, California at the Town & Country Resort. Oral presentation.

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Cheng Fang

The papers **I-V** are not included in this web version but can be obtained from the library at DTU Environment.
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Acknowledgements

The outcome of this PhD project is the result of good collaboration with internal and external partners, whose efforts I greatly appreciate.

I wish to express the deepest gratitude to those who have contributed to the completion of my PhD project:

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- ❖ Mr. Hector Garcia for his technical assistance on reactor constructions and analytical advice on measurements; for his experience sharing and supporting all the time in lab works.
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- ❖ Mr. Torben Dolin, Graphic Designer, for his careful and great job in thesis formatting, illustrations and printing.
- ❖ All my former and present colleagues and students at the Department of Environmental Engineering for collaborations, contributions and sharing expertise.

- ❖ All my friends all over the world for being there, for joyful spare time, for interesting discussions, for relaxing atmosphere and for making my life a lot easier.

My heartfelt gratitude goes to all my family members (>50 persons, 4 generations), especially father 方德兴; mother 成浩慧; Li's family and Tory Li; for all their endless support and encouragement.

To know wisdom and instruction;

To perceive the words of understanding;

to receive the instruction of wisdom,

Justice, and judgment, and equity;

-Proverbs 1: 2-3

‘不受苦中苦，难为人上人’

–明·冯梦龙《警世通言》， 1624

Summary

Facing energy crisis and climate change, the world is in need of a green, efficient, carbon-neutral energy source to replace fossil fuels. Biogas, formed by anaerobic digestion of organic materials, makes sustainable, reliable, renewable energy possible.

There is potential for biogas production from food-processing industrial wastes, not only because the wastes themselves can be treated to minimise the environmental impact, but it's also known as biofuel, methane, holds promise for the future.

On this background, four issues regarding biogas production from food-processing industrial wastes were identified:

- Characteristics of different food-processing industrial wastes were analysed. Model wastes were chosen from three food-processing industries, i.e. desugared molasses (DM), sugar beet pulp (SBP), sugar beet top (SBT) and sugar beet leaves (SBL) from sugar industry; potato juice and potato pulp from potato starch industry; palm oil mill effluent (POME), deoiled POME and empty fruit bunch (EFB) from palm oil industry.

- Biochemical methane potentials from the wastes above were determined in batch experiments.

- Technical feasibility in the continuous reactor experiments using different reactor configuration i.e. continuously stirred tank reactor (CSTR), upflow anaerobic sludge blanket (UASB), expanded granular sludge bed (EGSB) was investigated for treatment of different types of wastes at different operating conditions, i.e. temperature, hydraulic retention time (HRT) and organic loading rate (OLR).

- Improvement of biogas production and solutions to overcome the inhibition by pre-treatment methods and/or co-digestion with different types of organic wastes or animal manure was tested.

This PhD project focused on addressing the above issues to get better understanding on biogas production and potential problem relating to anaerobic digestion of these wastes; performances of different reactor types; optimal operating conditions (temperature, HRT, OLR); and finally, the methods to overcome the inhibition from some specific wastes.

The results from this study showed that SBP, SBT and SBL are good substrates for biogas production and DM can inhibit biogas production, due to its high sodium and potassium concentration. However, co-digestion of DM with cow manure can overcome the ion inhibition and improve the biogas production. Both DM and SBP need proper dilution in order to optimize the methane production and cow manure can be a good co-substrate to help diluting the concentrated DM and SBP, and also to provide buffer capacity and nutrients.

Both potato juice and potato pulp from potato starch processing industry showed very good potential for producing biogas. Continuous experiments on the UASB and EGSB reactors were carried out using potato juice as model substrate. From comparison on the performance of these two reactor types, the UASB reactor could tolerate high VFA at short HRT, while the EGSB reactor could achieve higher methane yield at long HRT, however, EGSB reactor was more sensitive to VFA accumulation.

POME and deoiled POME showed good potential for biogas production at HRT 5 days in the UASB and EGSB reactors. VFA accumulation, especially propionic acid, was a good indicator for reactor instability in the EGSB reactor. The UASB reactor was found to be more stable than EGSB reactor on treating raw and deoiled POME. Another residue from palm oil industry is EFB. EFB has low potential in biogas production due to its less biodegradable components. Pre-treatment methods applied to EFB by NaOH presoaking and/or hydrothermal treatment can increase the biodegradability and therefore the methane production. The biogas production can be further enhanced by combing pre-treatment of EFB and co-digestion of EFB with POME compared to either pre-treatment or co-digestion alone.

Dansk Resumé

Konfronteret med energikrise og klimaforandringer, har verden brug for en grøn, effektiv og kulstofneutral energikilde, som kan erstatte fossile brændstoffer. Biogas, som dannes ved anaerob nedbrydning af organisk materiale, bevirker at bæredygtig, pålidelig og vedvarende energi er mulig.

Der er potentiale for biogasproduktion fra fødevareindustriens affald, ikke kun fordi selve affaldet kan behandles således, at det i sig selv reducerer miljøbelastningen, men også i form af netop biobrændstoffet metan, lover det godt for fremtiden.

På denne baggrund blev fire emner vedrørende biogasproduktion af affald genereret i fødevareindustrien identificeret og behandlet på følgende vis:

- Karakteristik af forskelligt fødevareindustriaffald blev analyseret. Modelaffald blev udvalgt fra tre fødevareforarbejdningsindustrier; afsukret melasse (DM) og sukkerroeaffald (SBP) fra forarbejdningsindustrien; kartoffelsaft og kartoffelpulp fra kartoffelstivelseforarbejdningsindustrien; palmeoliemølle spildevand (POME), deolieret POME og tomt frugtbundt (EFB) fra palmeolieindustrien.
- Biokemisk metanpotentiale fra ovenstående affald blev bestemt i batch forsøg.
- Den tekniske gennemførlighed blev testet vha. kontinuerlige reaktorforsøg med forskellige reaktorkonfigurationer; CSTR (reaktor med konstant omrøring), UASB (upflow anaerobic sludge blanket), EGSB (expanded granular sludge bed) for forskellige typer af affald på forskellige driftsbetingelser, hvilket dækker over temperatur, hydraulisk opholdstid (HRT) og organisk belastningsgrad (OLR).
- Optimering af biogasproduktionen samt løsningsstrategier for hæmning, forårsaget af forbehandlingsmetoder og/eller samudrådning af de forskellige affaldstyper for sig selv og sammen med husdyrgødning.

Denne ph.d. afhandling fokuserer på ovennævnte emner, med det formål at opnå bedre forståelse af, hvilke typer affald der har et godt biogaspotentiale og hvilke der er problematiske. For at afdække dette, er følgende fortaget; reaktortype er matchet med affaldstype, optimale driftsbetingelser (temperatur, HRT, OLR) er bestemt og strategier til at overvinde hæmning fra problematisk affald er identificeret.

Min undersøgelse viste at SBT, SBL og SBP er gode substrater til biogasproduktion, mens DM kan medføre hæmning af biogasproduktionen på grund af høje koncentrationer af natrium og kalium. Dette kan afhjælpes ved samudrådning af DM med kogødning, hvorved ionhæmningen overvindes og produktionen af biogas forbedres. Både DM og SBP har behov for korrekt fortynding hvis metanproduktionen skal forløbe optimalt, her kan kogødning fungere som fortyndingsmedium af koncentrerede substrater. En ekstra gevinst ved at benytte kogødning til fortynding, er at kogødningen i sig selv besidder både bufferkapacitet samt næringsstoffer.

Kartoffelsaft og kartoffelpulp fra kartoffelstivelseforarbejdningsindustrien viste begge potentiale for at producere biogas. Kontinuerlige forsøg i UASB og EGSB blev udført med kartoffeljuice som substrat. Sammenholdes ydeevnen af ovennævnte to reaktortyper; viser UASB sig mere tolerate overfor forhøjet VFA ved kortere HRT, mens EGSB er mere følsom overfor VFA koncentrationen, kan EGSB til gengæld opnå større metanudbytte ved længere HRT.

I UASB og EGSB reaktorer viste POME og deolieret POME gode potentialer for biogasproduktion ved en opholdstid (HRT) på fem dage. Der kunne konstateres tegn på ustabilitet når koncentrationen af propionsyre forøgedes, herudfra kunne det udledes, at UASB reaktorer er mere stabile end EGSB reaktorer ved behandling af rå og deolieret POME. En anden rest fra palmeolieindustrien, kaldes tomt frugtbundt (EFB). EFB har ringe potentiale for biogasproduktion, på grund af dens indhold af mindre bionedbrydelige komponenter. Forbehandling af EFB kan øge bionedbrydeligheden og dermed forøge metanproduktionen, ligeledes kan samudrådning af forbehandlet EFB med POME forbedre produktionen af biogas.

Table of contents

1 Introduction and aim of study

The PhD project was initiated due to the needs for both energy production and waste treatment. The energy carrier in focus, in this project, was biogas, which is among the alternatives to fossil fuels. The biomass used to produce biogas was food-processing industrial wastes. By anaerobic digestion, the wastes could be treated to minimise the environmental impact and at the same time converted into methane energy.

Nowadays both energy crisis and climate change are key issues all over the world. There will be severe energy shortage in the coming 50 years. According to current research and future predictions, the crude oil will run out within 40 to 70 years, and natural gas will be finished within 50 years (Courtney and Dorman, 2003). Global average temperature is predicted to increase 1.4 to 5.8 °C by year 2100 and continue to rise long after that (Dow and Downing, 2006). Several investigations point out that this will inevitably lead to drought, flooding, increases in hurricanes and tornadoes and possibly widespread crop failures (Sen, 2009; Mills, 2009). Global warming as the result of climate change is an established fact. It is now widely accepted that it is caused by the rapidly increasing concentrations of greenhouse gas (CO_2 and others) in the atmosphere, which is emitted mainly by the combustion of fossil fuels containing carbon like coal, oil, and natural gas (Jaynes, 2010).

Security of energy supply, especially sustainable energy, and reduction of CO_2 emission are priorities on agenda worldwide. The use of biogas and biomass as an energy source is regarded as CO_2 neutral, because the CO_2 released during combustion of the biogas is the same CO_2 that the plants have assimilated during photosynthesis to create organic biomass (Jørgensen, 2009).

Renewable energy is politically demanded. The European Community has agreed targets for 2020 on renewable energy, which established a high standard for all Member States, aiming a 20% share of renewable energy by the year 2020 (European Commission Energy, 2010). Biogas is one of the most efficient and effective options among the various other alternative sources of renewable energy currently available. It is produced through anaerobic digestion processes where the microorganisms convert complex organic matter into a mixture of methane and carbon dioxide. The anaerobic digestion of biomass requires less capital investment per unit production cost compared to other renewable energy sources, such as hydro, solar and wind energy (Rao et al., 2010). It has been early demonstrated that biogas production from crop residues is economically feasible on a farm-scale level (50–500 kW) (Svensson et al., 2005).

Biogas can be produced from variety of substrates, such as animal manure, energy crops, industrial wastes etc. Biogas production is a sustainable solution to treat waste and the cost of the waste treatment is low (Verstraete et al., 2005). There is limited competition with food by using industrial wastewater and residues to produce biogas (Wellinger, 2009). The effluent from the biogas process supplies essential nutrients which can also be utilized as fertilizer (Vasudeo, 2005).

The main objective of this PhD project was to investigate the potential of biogas production from food-processing industrial wastes in batch assays and continuous reactor operations with different reactor configurations, for instance, desugared molasses (DM) and sugar beet pulp (SBP) using continuously stirred tank reactor (CSTR); potato juice, palm oil mill effluent (POME) and deoiled POME using upflow anaerobic sludge blanket (UASB) reactor and expanded granular sludge bed (EGSB) reactor (Table 1). Additional studies of different operating conditions i.e. temperature and organic loading rate (OLR), have been carried out to improve the biogas production. Co-digestion has been addressed to overcome the inhibitory effect from DM. The biogas production from SBP and empty fruit bunch (EFB) has been increased by co-digesting with manure and POME, respectively (Table 1). Furthermore, different pre-treatment methods have been applied to EFB in order to improve its biodegradability.

Table 1 Summary of food-processing industrial wastes investigated in batch tests, reactor types and co-digestion combinations.

Food-processing industry	Food-processing industrial wastes	Batch tests	Reactor experiments	Co-digestion
Sugar industry	Desugared molasses (DM)	√	CSTR	+ Manure
	Sugar beet pulp (SBP)	√	CSTR	+ DM + Manure
	Sugar beet top (SBT)	√	-	-
	Sugar beet leaves (SBL)	√	-	-
Potato starch industry	Potato juice	√	UASB & EGSB	-
	Potato pulp	√	-	-
Palm oil industry	Palm oil mill effluent (POME)	√	UASB & EGSB	-
	Deoiled POME	√	UASB & EGSB	-
	Empty fruit bunch (EFB)	√	-	+ POME

1. Sugar processing industrial wastes (*Paper I, II*)

DM and SBP are by-products from the sugar production. DM originates from de-sugaring process in the sugar production. It contains 2-3 times higher concentration of ions than normal molasses, which could inhibit the biogas process. Co-digestion strategy was tested to overcome the ion inhibition in the CSTR reactor. Cow manure was a good co-substrate, and a stable anaerobic digestion could be achieved by co-digesting DM with manure at the concentration below 15% DM.

SBT, SBL and SBP are easily degradable substrates with high methane potential, however, dilution was necessary to avoid organic overload in our study. Co-digestion of SBP with DM and/or manure could increase the methane production. Manure helped diluting the concentrated substrates and also provided buffer capacity and nutrients.

2. Potato starch processing industrial wastes (*Paper III*)

Potato juice and potato pulp are by-products from the potato starch production. Both substrates gave high methane potential determined in the batch experiments. Both UASB and EGSB reactors were applied for the treatment of potato juice in the continuous experiments. The results suggested that the UASB reactor was more tolerate to high volatile fatty acid (VFA) concentrations, while the EGSB reactor obtained higher methane yield at hydraulic retention time (HRT) longer than 8 days. The treatment of reactor effluent was investigated by acidification with sulfuric acid to pH lower than 5, almost 100% of the ammonia content could be retained, at the successive up-concentration process step, used for minimizing the effluent volume, thus withholding the fertilizing capacity of the effluent.

3. Palm oil processing industrial wastes (*Paper IV and V*)

POME and deoiled POME are suitable for methane production by high-rate anaerobic digestion. Both UASB and EGSB reactors could achieve high COD removal efficiencies; greater than 90%. The concentration of total VFA and propionic acid were the good indicators for process instability in the reactor. The UASB reactor was more stable than the EGSB reactor in anaerobic treatment of both raw and deoiled POME.

EFB is another residue from the palm oil industry. Investigation of different pre-treatment methods of EFB was carried out to increase the biodegradability and the methane production. Co-digestion strategy was applied to raw EFB and pre-treated EFB with POME. The experimental results showed that co-digestion of raw EFB with POME could increase microbial biodegradability for 25-32% compared to the digestion of EFB alone. Pre-treatment of EFB by combined hydrothermal and NaOH presoaking method plus co-digestion with POME resulted in 53% increase of the biodegradability and the methane recovery reached 91% of the theoretical potential.

2 Anaerobic digestion

Anaerobic digestion (AD) is the degradation of organic materials by micro-organisms in the absence of oxygen. It is a multi-step biological process where the organic carbon is mainly converted to carbon dioxide and methane (Angelidaki et al., 2003). The process can be divided into four steps: hydrolysis, acidogenesis, acetogenesis and methanogenesis. Figure 1 shows the pathway of anaerobic digestion.

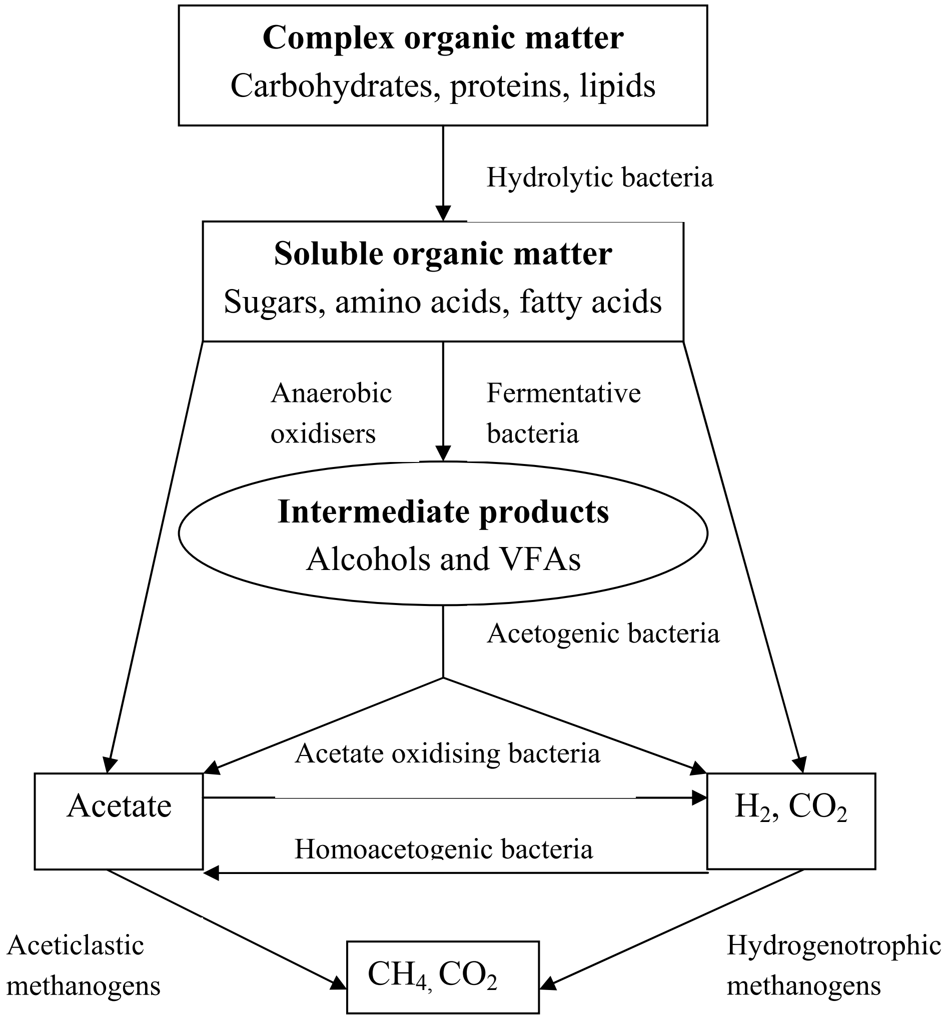

Figure 1 Pathway of anaerobic digestion (adapted from Angelidaki et al., 2002)

2.1 Hydrolysis

Hydrolysis is the first step in anaerobic digestion processes. During the hydrolysis step, complex organic matters, such as carbohydrates, proteins and lipids are hydrolyzed into soluble organic molecules such as sugars, amino acids and fatty acids by extracellular enzyme, i.e. cellulase, amylase, protease or lipase (Parawira et al., 2005). Hydrolytic bacteria, which hydrolyze the substrate with these extracellular enzymes, are facultative anaerobes. Hydrolysis can be the rate-limiting step if the substrate contains large molecules (particulates) with a low surface-to-volume ratio (Vavilin et al., 1996). While if the substrate is readily degradable, the rate-limiting step will be acetogenesis and methanogenesis (Björnsson et al., 2001). When the substrate is hydrolyzed, it becomes available for cell transport and can be degraded by fermentative bacteria in the following acidogenesis step.

2.2 Acidogenesis

In the acidogenesis step, the soluble organic molecules from hydrolysis are utilized by fermentative bacteria or anaerobic oxidizers (Garcia-Heras, 2003). These microorganisms are both obligate and facultative anaerobes. In a stable anaerobic digester, the main degradation path way results in acetate, carbon dioxide and hydrogen. The intermediates, such as volatile fatty acids and alcohols, play a minor role. This degradation path way gives higher energy yield for the microorganisms and the products can be utilized directly by methanogenic microorganisms (Schink, 1997). However, when the concentration of hydrogen and formate is high, the fermentative bacteria will shift the path way to produce more reduced metabolites (Angelidaki et al., 2002). The products from acidogenesis step consist of approximately 51% acetate, 19% H_2/CO_2, and 30% reduced products, such as higher VFA, alcohols or lactate (Angelidaki et al., 2002). Acidogensis step is usually considered the fastest step in anaerobic digestion of complex organic matter (Vavilin et al., 1996).

2.3 Acetogenesis

Intermediates formed during acidogenesis, consist of fatty acids longer than two carbon atoms, alcohols longer than one carbon atom and branched-chain and aromatic fatty acids. These products cannot be directly used in methanogenesis and have to be further oxidized to acetate and H_2 in acetogenesis step by obligated proton reducing bacteria in a syntrophic relationship with hydrogen utilisers. Low H_2 partial pressure is essential for acetogenic reactions to be thermodynamically favorable (Schink, 1997). The products from acetogenesis are then the substrates for the last step of anaerobic digestion, which is called methanogenesis.

2.4 Methanogenesis

In methanogenesis step, acetate and H_2/CO_2 are converted to CH_4 and CO_2 by methanogenic archaea. The methanogenic archaea are able to grow directly on H_2/CO_2, acetate and other one-carbon compound, such as formate and methanol (Schink, 1997). In the normal anaerobic digesters, acetate is the precursor for up to 70% of total methane formation while the remaining 30% originates from H_2/CO_2 (Klass, 1984). Moreover, the inter-conversion between hydrogen and acetate, catalyzed by homoacetogenic bacteria, also plays an important role in the methane formation pathway. Homoacetogens can either oxidize or synthesize acetate depending on the hydrogen concentration in the system (Kotsyurbenko, 2005). Hydrogenotrophic methanogenesis functions better at high hydrogen partial pressure, while aceticlastic methanogenesis is independent on hydrogen partial pressure. At higher temperatures, the acetate oxidation pathway becomes more favorable (Schink, 1997). It has been reported that methane formation through acetate oxidation can contribute up to 14% of total acetate conversion to methane under thermophilic conditions (60 °C) (Petersen and Ahring, 1991).

3 Food-processing industrial wastes

Food processing comprises the methods and techniques used to transform raw ingredients into food; or to transform food into other forms for consumption by humans or animals, either at home or in the food processing industries (Kaushik, et al., 2009). The processes often produce large amounts of wastes, so called by-products, which have been evaluated in many studies for their potential utilization and their suitability for chemical and biological treatments. Since these by-products contain relatively high concentrations of organic contents, anaerobic digestion is a preferable method for treatment of these materials.

3.1 Sugar processing waste

Sugar is produced in 121 Countries and the overall global sugar production was approx. 160 million tons in year 2009, which was 4.5% higher than in year 2008 (World sugar market review, 2010). Approximately 70% of sugar is produced from sugar cane, while the remaining 30% is produced from sugar beet. Beet-sugar production generates several streams of organic wastes and the process scheme is shown in Figure 2. The circles mark the model wastes from sugar production, used in our research (*Paper I, II*). The three main waste-streams are molasses, beet pulp and cutoffs (beet top and beet leaves). Molasses is a syrup residue from the sugar extraction process, which can contain up to 48% sugar (Satyawali and Balakrishnan, 2007). Technological advances in the sugar industry have made it possible to extract more sugar from the normal molasses. Desugared molasses is a residue from the desugaring process of normal molasses (Olbrich, 1963). From the factory data (DANISCO, Denmark), every ton of beet sugar produced generates 0.24 ton of DM, 0.33 ton of beet pulp, and 0.53 ton of grass cut-offs (Sugar production, 2001).

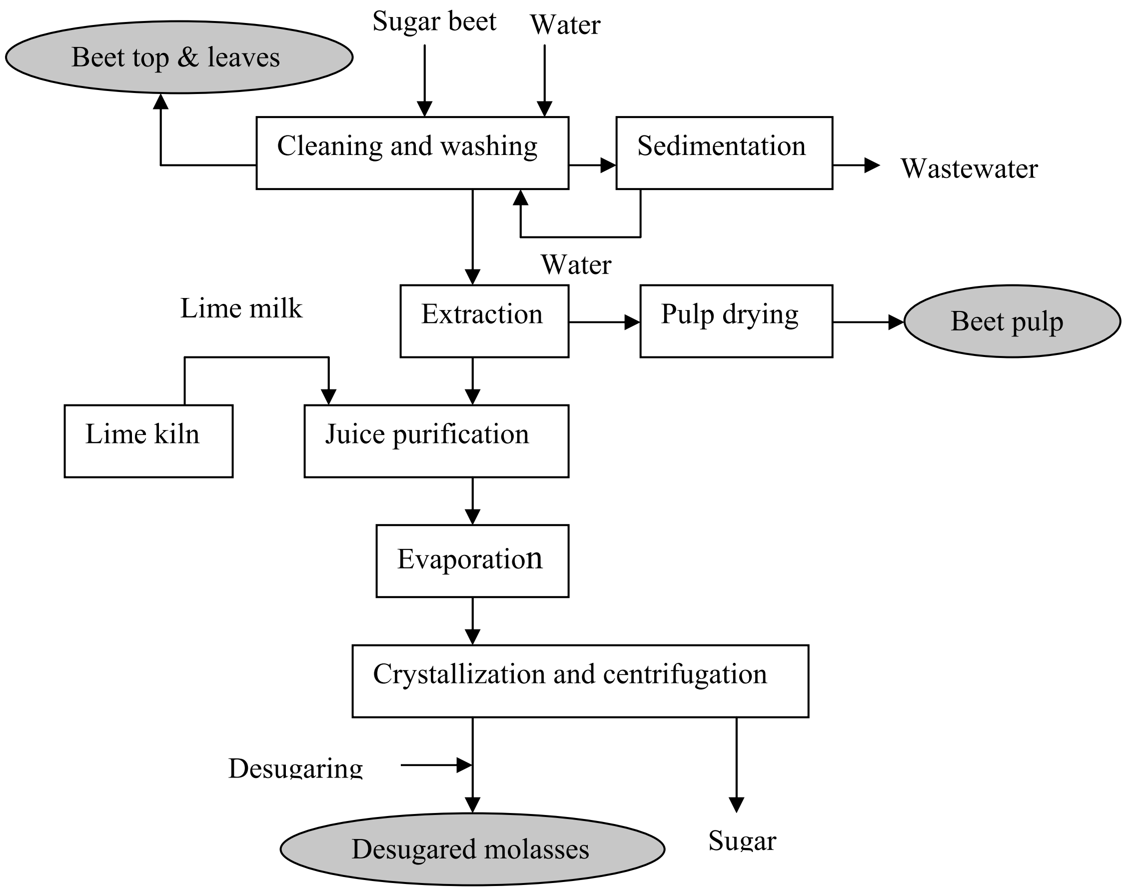

Figure 2 Scheme of beet-sugar processing (adapted from Danisco sugar process scheme; the circles mark the model wastes from sugar production used in our research)

3.2 Potato starch processing waste

World potato production is steadily increasing, from 268 million tons in 1991 to 314 million tons in 2008 (FAOSTAT, 2010). Since the 1990s, production has dramatically increased in Asia, Africa and Latin America, with over a fivefold increase in the past 40 years. These countries now account for half of the world's production, with China and India accounting for one third of the total production. Denmark was the thirteenth largest potato producer in Europe in 2007, typically contributing 1.5-2 million tons per year, depending on the season. 75% of the Danish potatoes are used in potato starch production and 85% of Danish potato starch is exported to more than 40 countries all over the world (International starch institute, 2010). Potato starch production generates several streams of organic wastes and the process scheme is shown in Figure 3. The circles show the model wastes from potato starch production relevant to our research (Paper

III). Per ton of potato flour (80% potato starch and 20% water) produced, 6.6 m^3 of potato juice and 0.73 ton of potato pulp are produced as by-products (Potato flour production, 2002). These two by-products contain biodegradable components such as starch and proteins, which could be used for biogas production through anaerobic digestion.

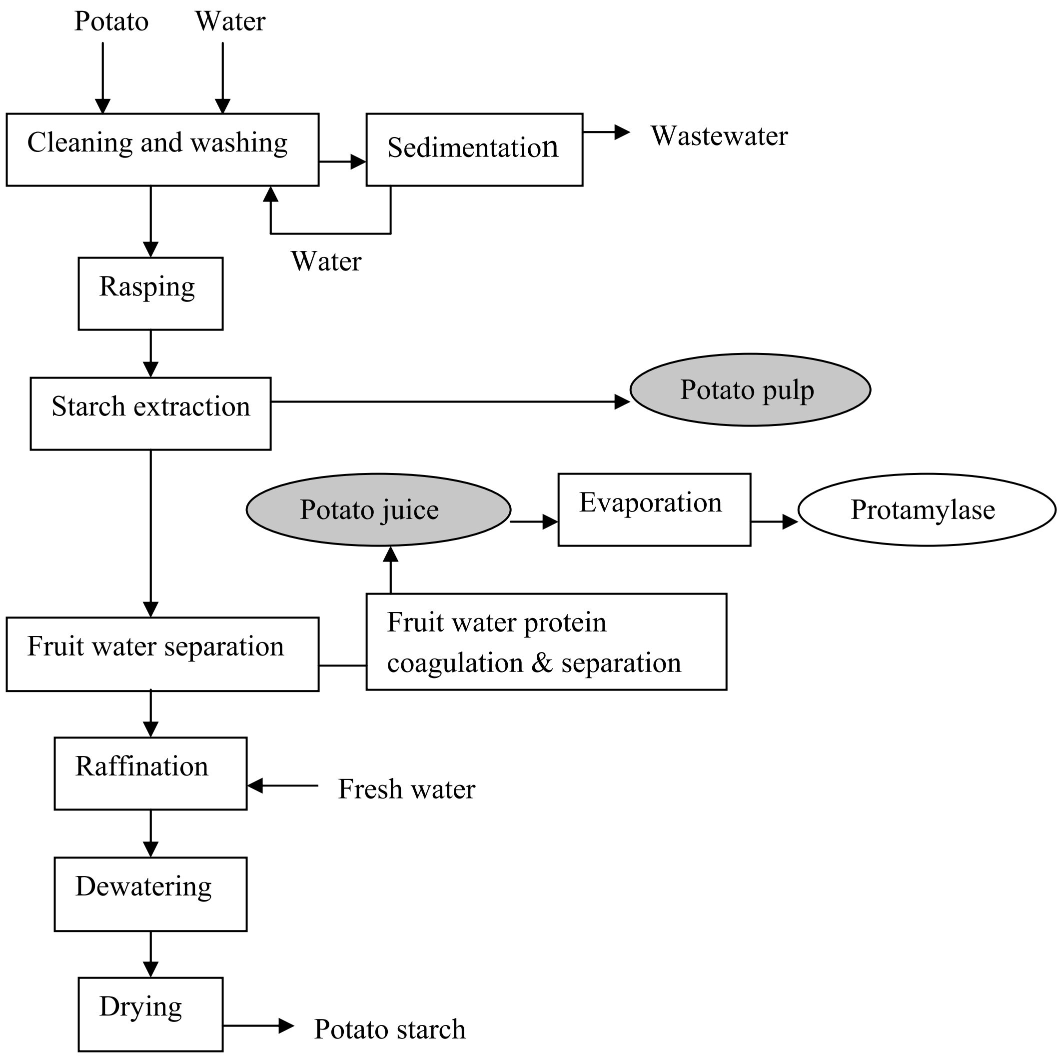

Figure 3 Scheme of potato starch processing (adapted from Zuckerforschung Tulln potato starch extraction scheme; the circles show the model wastes from potato starch production relevant to our research)

3.3 Palm oil processing waste

The production of palm oil is increasing every year since the 1960s. Crude palm oil is the main product in the palm oil industry. However, large amounts of wastes are also generated, such as POME produced through a multistep oil extraction processes, EFB produced after sterilization, and deoiled POME produced after clarification of POME (Poh and Chong, 2009). Palm oil production generates several streams of organic wastes and the process scheme is shown in Figure 4. The circles mark the model wastes from palm oil production in our research (*Paper IV, V*). For every ton of palm oil extracted, 2.5 tons of POME and 1.3 tons of empty fruit bunch are generated. At least 44 million tons of POME was produced in Malaysia in year 2008, which led to high demand for proper treatments (Wu et al., 2010).

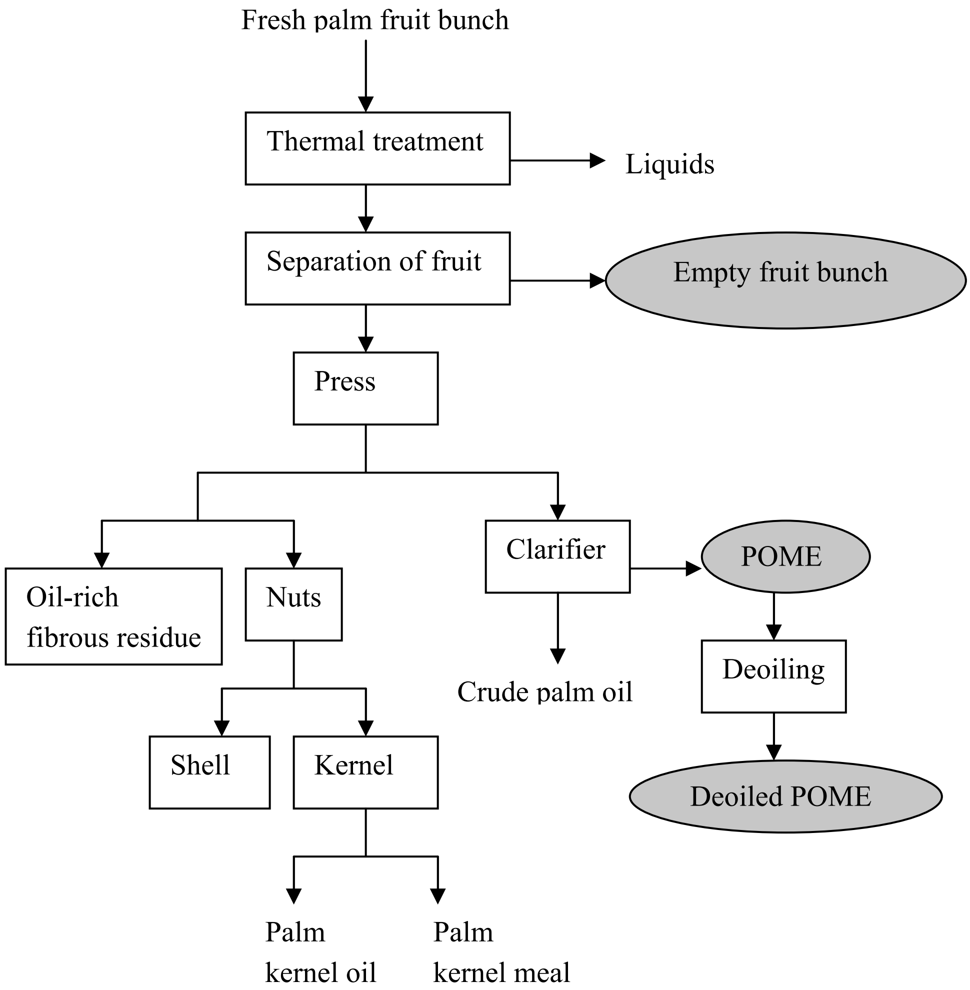

Figure 4 Scheme of palm oil processing (adapted from Solano, 1986; the circles mark the model wastes from palm oil production in our research)

4 Biogas in batch and reactor experiments

Anaerobic batch digestion is a traditional way to determine biodegradability and ultimate methane potential during the digestion of the waste (Angelidaki and Sanders, 2004; Parawira et al., 2004). Semi-continuous and continuous operations are also among the conventional solutions in the anaerobic digesters. Semi-continuous/continuous operation is preferable in AD processes, because the maximum growth rate can be achieved constantly at steady state by controlling the feeding rate. Choice of reactor type in the continuous operation is determined according to the waste characteristics, especially particulate solid contents. Solid and slurry wastes are mainly treated in CSTRs, while soluble organic wastes are more suitable to high-rate biofilm systems, such as UASB and EGSB reactors (Angelidaki et al., 2002; Kato et al., 1997).

4.1 Biological methane potential (BMP) assays

The BMP assay is a method based on the product formation where biogas, methane and/or intermediates production are monitored from closed vials containing the selected waste and methanogenic inoculum incubated at a specific temperature (Angelidaki and Sanders, 2004; Angelidaki et al., 2009). The methane potential can be determined as the specific methane production for indefinite degradation time. The rate of ultimate biodegradation of the waste can also be determined by monitoring methane at pre-set time intervals until the specific methane activity is no longer observed (Maya-Altamira, 2008). Nine batch experiments were performed in four types of sugar processing wastes, i.e. DM, SBP, SBT and SBL; two types of potato starch processing wastes, i.e. potato juice and potato pulp; and three types of palm oil processing wastes, i.e. POME, deoiled POME and EFB (*Paper I, II, III, IV, V*). All batch experiments were designed following the method from Angelidaki et al. (2009) and the BMP test bottle setup is shown in Figure 5 as an example.

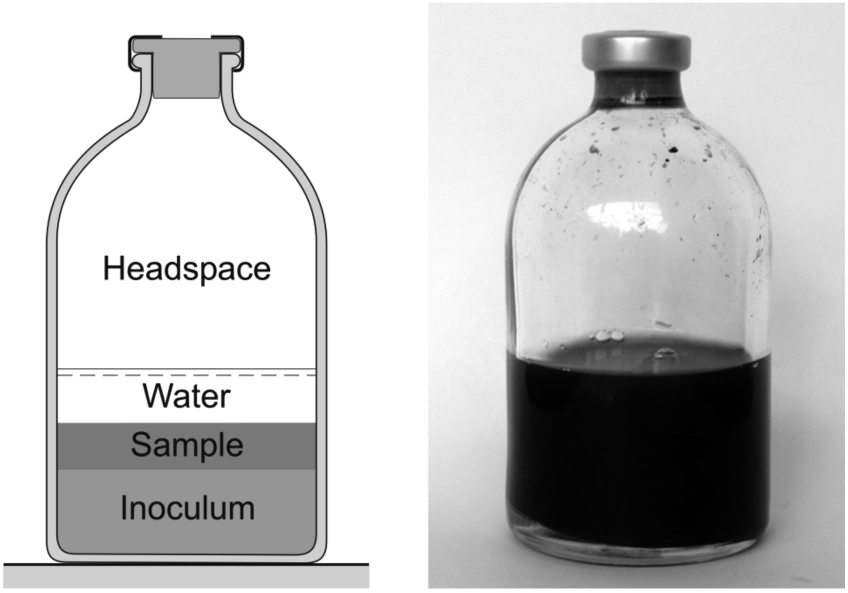

Figure 5 BMP test bottle setup (adapted from Angelidaki and Garcia, 2007)

4.2 Continuously stirred tank reactor (CSTR)

The CSTR reactor is widely used in industrial scale biogas production and in wastewater treatment units (i.e. activated sludge reactors). CSTR has been successfully applied to anaerobic digestion of energy crops and food residues (Fu et al., 2010; Demirel and Scherer, 2008). CSTR configuration has several advantages, considered to be practical and simple to operate (Kaparaju et al., 2009). In CSTR systems, the biomass is suspended in the main liquid and will be removed together with the effluent, so that sludge retention time is equal to HRT. This makes it necessary to run at long HRTs, usually 10-20 days, to avoid washing out the slow growing methanogens (Boe, 2006). Most studies on methane production from manure or industrial slurries have been conducted in one phase CSTR or CSTR in series (i.e. reactors where the different biomasses involved in the biogas process are present in a mixed culture), the serial CSTRs achieve higher methane yield compare to single CSTR with same total volume (Boe and Angelidaki, 2009). CSTR configuration used in our research is shown in Figure 6.

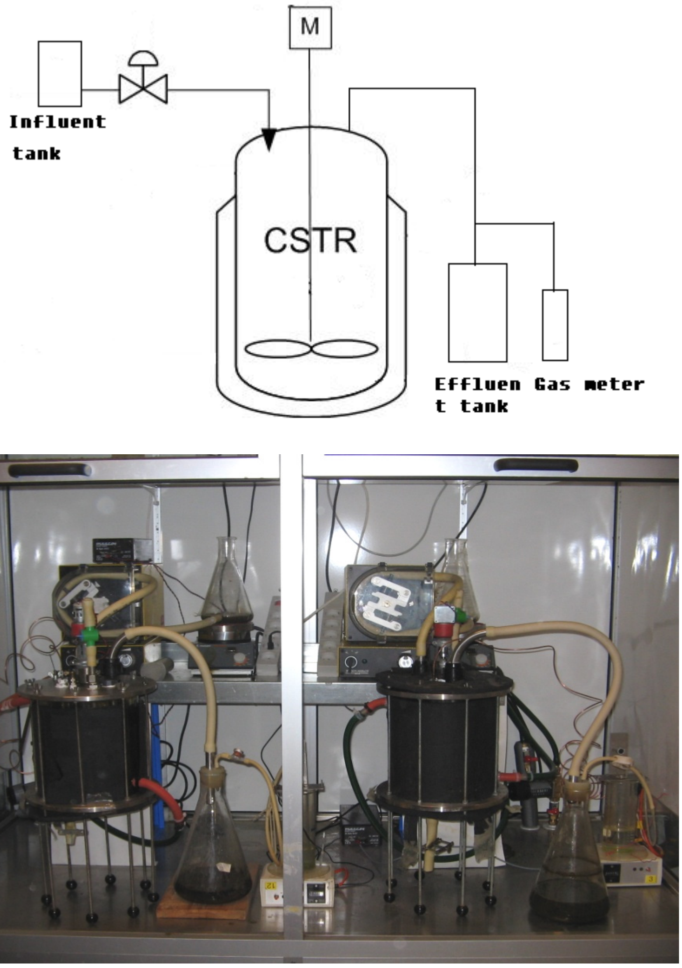

Figure 6 CSTR reactor setup (the picture was taken at Biotech lab, Department of Environmental Engineering, Technical University of Denmark)

A CSTR reactor operated under both mesophilic and thermophilic conditions, with a HRT of 20 days was found to be feasible for biogas production by feeding with DM, with methane yield of 300 mL-CH_4/gVS-added, at a mixture of 5% DM in cow manure (*Paper I*). However, the biogas production was inhibited when digesting DM alone in CSTR due to the 2-3 times higher cation concentrations (Na^+, K^+) in DM, compared to normal molasses. Another CSTR reactor was operated with SBP under the same operating condition as DM (*Paper II*). SBP was shown to be a good substrate for biogas production and the methane yield of 280 mL-CH_4/gVS-added was obtained in a thermophilic CSTR, co-digesting 50% of SBP with cow manure.

4.3 Upflow anaerobic sludge blanket (UASB)

The UASB reactor was developed in the early 1970s (Lettinga et al., 1980) and is nowadays widely used for treatment of several types of wastewaters (Shastry et al., 2010; Sevilla-Espinosa et al., 2010). In the UASB reactor, the immobilized cell is used, where the biomass is retained while the substrate is pumped through, allowing a high organic loading rate (Kaparaju et al., 2009). The success of the UASB concept relies on the establishment of a dense sludge bed in the bottom of the reactor, in which the biological processes take place. This sludge bed is basically formed by accumulation of the incoming suspended solids and the growth of bacteria (Seghezzo et al., 1998). Natural turbulence caused by the influent flow and the biogas formed inside the reactor provides good wastewater-biomass contact in UASB systems. The setup of UASB in our study is shown in Figure 7.

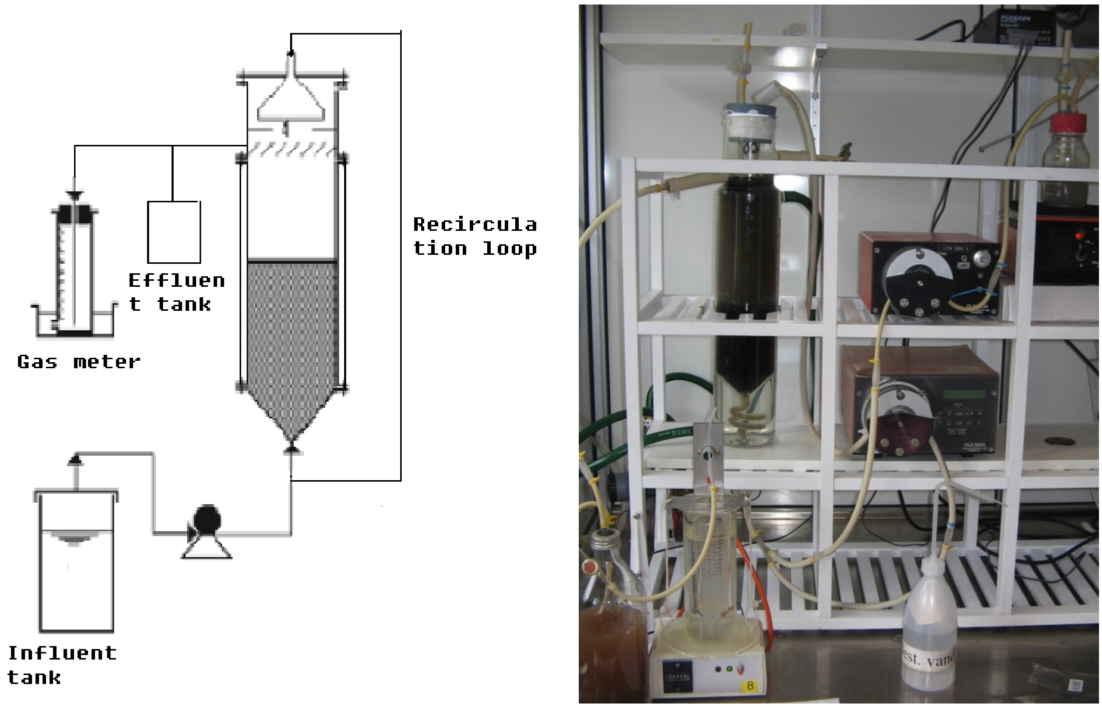

Figure 7 UASB reactor setup (Adapted from Gonçalves et al., 2005; the picture was taken at Biotech lab, Department of Environmental Engineering, Technical University of Denmark)

Stable methane yield of 240 mL-CH_4/gVS-added has been achieved in UASB reactor operated at HRT 5 days at 37 ºC with OLR 5.1 gCOD/L-reactor.d of potato juice wastewater (*Paper III*). Maximum methane yield of 438 mL-

CH_4/gVS-added, which accounts for 98% of the theoretical yield, has been achieved in UASB reactor operated at HRT 5 days at 55 ºC with OLR 5.8 gVS/L-reactor.d of POME (*Paper IV*). The high and stable methane yield achieved in UASB, could suggest that the UASB type of reactor is suitable for continuous biogas production at high OLR. However, UASB reactor allows organic substrates with low content of suspended solids (Angenent et al., 2004). In potato juice, the influent suspended solids concentration was 1.97 g/L and the wastewater COD was 25 g/L, much higher than in the literature (Kalogo and Verstraete, 1999; von Sperling et al., 2001; Cheng et al., 2010), which could result in lower methane yield.

4.4 Expanded granular sludge bed (EGSB)

The concept of EGSB reactor was introduced by de Man et al. (1988) and it is a modification of the conventional UASB reactor. Both EGSB and UASB are inoculated with granular sludge, but the hydrodynamic conditions are different. Superficial velocity in EGSB is 5–10 m/h, which is 5 to 10 times higher than in UASB, due to a high height to diameter ratio and a high recirculation rate (Kalogo and Verstraete, 1999). These characteristics improve the mixing and the contact between the wastewater and the sludge in the EGSB reactor (Puyol et al., 2009). It decreases the apparent Ks (substrate saturation constant) of the granular biomass and makes the EGSB reactor a particularly suitable system for loading conditions <1 gCOD/L-reactor.d (Kalogo and Verstraete, 1999). This is also proved by many researchers; that the EGSB is very attractive for low-strength wastewater, such as influent concentration of COD less than 1000 ~ 2000 mg /L (Kato et al., 1997; Lettinga, 1996). The setup of EGSB is shown in Figure 8.

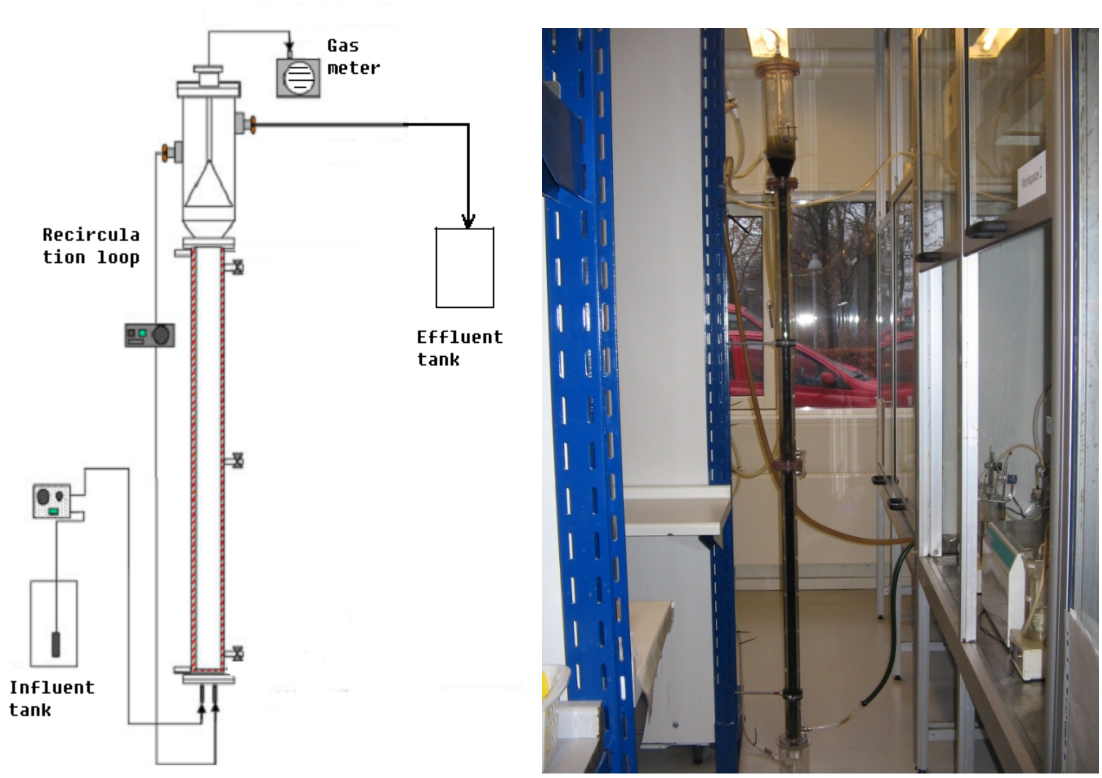

Figure 8 EGSB reactor setup (adapted from Costa et al., 2009; the picture was taken at Biotech lab, Department of Environmental Engineering, Technical University of Denmark)

Stable methane yield of 380 mL-CH_4/gVS-added has been achieved in EGSB reactor operated at HRT 8 days at 37 ºC with OLR 3.2 gCOD/L-reactor.d of potato juice wastewater (*Paper III*). We found out in EGSB performance, that total VFA concentrations could better reflects the alarm status of the process compared to methane yield. There is possibility of inhibition from the un-ionized volatile fatty acids (UVFAs) which may lead to the disintegration of the granules because the methanogens die (Tiwari et al., 2006). We observed the granular disintegration at total VFA concentrations of 30 mM at pH 7, corresponding to 12 mg/L of UVFAs. This concentration exceeded the inhibition level of 10 mg/L reported by Duarte and Anderson (1982) and Kroeker et al. (1979). When treating raw and deoiled POME, the EGSB reactor was found to be less stable than the UASB reactor under the same OLR. It could be seen from higher VFA concentrations, especially propionic acid, compared to the UASB reactor (*Paper IV*).

5 Factors affecting the biogas process

The factors affecting the biogas production are mainly caused by the characteristics of the feedstock and operating condition of the process. Sometimes feedstock itself can contain inhibitors such as high concentrations of cations (*Paper I*). Other times toxic compounds are not initially present in the feed, but are produced during the anaerobic digestion process, such as VFAs (*Paper III, IV*). Factors from the feedstock (i.e. nutrients, pH, buffering capacity and inhibitory compounds), and operating conditions (i.e. temperature and OLR), influence directly on the performance of microorganisms.

5.1 Temperature

Anaerobic digestion can be applied in a wide range of temperatures from psychrophilic (<20 °C) to extreme-thermophilic conditions (>60 °C) (Kashyap et al., 2003, van Lier, 1997, Lepistö and Rintala, 1999). Increasing temperature has several advantages: it can increase solubility of organic compounds; increase chemical and biological reaction rates; improve diffusivity of soluble substrate; increase death rate of pathogenic bacteria, especially under thermophilic condition; increase the degradation of long chain fatty acids, VFAs and other intermediates etc. (Boe, 2006). The disadvantage of high temperature can be that it decreases pKa of ammonia, thus increases the fraction of free-ammonia which is inhibitory to microorganisms and increases pKa of VFA, which increases its un-dissociated fraction, especially at low pH (4-5) such as in the acidogenic reactor (Boe, 2006). This is the reason why, the thermophilic process is in general more sensitive to inhibition.

In our study, while digesting POME and deoiled POME, we applied 55 °C to UASB and EGSB reactor operation (*Paper IV*). Despite the high temperature advantages, another important reason is that POME has an initial temperature of 80-90 ºC (Najafpour et al., 2006) from oiling process and deoiled POME can have the temperature 45-50 ºC after de-oiling from POME. Therefore operating the reactor under thermophilic condition will be more economical than mesophilic condition, in terms of the ability to use a smaller digester and obtain a better methane production. For example, we observed a methane yield of 600 mL-CH_4/gVS-added with deoiled POME in UASB reactor, which accounted for

98% of the theoretical methane yield. In this case, almost all the organics in the deoiled POME was digested at 55 °C in high-rate system.

5.2 Nutrients

Efficient biodegradation requires nutrients and sufficient nutrients are therefore important to microbial cell growth. Macro- nutrients such as carbon, nitrogen, potassium phosphorus, sulphur (Kayhanian and Rich, 1995) and micro-nutrients such as Fe, Ni, Zn and Co in smaller amount (Cresson et al., 2006) are required for optimal anaerobic microbial growth.

In our study, all nutrients were generally mixed as basic anaerobic (BA) medium (Angelidaki and Sanders, 2004) which was introduced in the digestion of POME and deoiled POME (*Paper IV*) to provide enough nutrient for starting up the AD process. However, from the economical point of view, in the large industrial scale operation, the need for these supplements according to different waste characteristics should be further investigated, in order to reduce the operational cost.

5.3 pH and buffering capacity

Many groups of microorganisms have the same optimal pH range, while each group has a specific pH region for optimal growth in anaerobic degradation. Methanogenic archea can function in quite narrow pH interval from 5.5-8.5 with an optimal range of 6.5-8.0 (Boe, 2006). Fermentative bacteria can function in wider pH range pH 4 to 8.5 (Hwang et al., 2004) and have different optimal pH in respect to the fermentation products (Horiuchi et al., 2003). In a mixed-culture anaerobic digester, the optimal pH range is 6.6-7.8 (Lay et al., 1997). Knowledge in pH and factors causing or resisting to pH change is essential to control and secure a successful operation in an AD system.

Buffering capacity (also called alkalinity) is an important factor for process stability, in terms of resistance to pH change. The main buffer in anaerobic digesters is bicarbonate (HCO_3^-), with a pKa of 6.3, and the main generated acids are VFAs, with an aggregate pKa around 4.8 (Boe, 2006). Other compounds such as hydrogen sulphide (pKa 7.1), dihydrogen phosphate (pKa 7.2), and ammonium ion (pKa 9.3) are commonly found in the digester which influence the pH balance if present at high concentrations (Björnsson, 2000).

Raw POME and deoiled POME are very low in alkalinity (*Paper IV*) and they have the initial pH 4.3 and pH 4.7 respectively, which were too acidic to start up the AD process. Therefore bicarbonate was added to the substrate in order to increase the buffering capacity and bring pH up to 6 to be able to meet the optimal pH in a mixed-culture anaerobic digester (*Paper IV*).

5.4 Volatile fatty acids (VFA)

VFAs are some of the most important intermediates in the anaerobic biogas process; it is the conversion from VFA into methane and carbon dioxide which is important (Pind et al, 2003). The increase of VFA concentration in the biogas process is well-known, as a result of process imbalance. Thus, it has been commonly suggested as an indicator in the anaerobic digester (Björnsson et al., 2000; Boe, et al., 2007). The un-ionized fraction of volatile fatty acids (UVFAs) has been found to contribute to the inhibition of methanogenesis. The un-ionized also called free fatty acid can pass through cell membranes and dissociate, which disrupts cell homeostasis (Russell and Diez-Gonzalez, 1997). In order to determine the observed effect of UVFA; pH and the total VFA concentration are the two key parameters. Fifty percent of methane production inhibition was reported when the UVFA concentration exceeded 10 mg/L in acetic and glucose-fed digesters (Duarte and Anderson, 1982). The same concentration level was mentioned by Kroeker et al. (1979) that a definite trend toward digester failure as UVFA concentration increases above 10 mg/L as acetic acid. The differential growth of fermentative bacteria and methanogens may cause pH to change if UVFA concentration exceeds the buffering capacity of the reactor content (Florencio et al. 1995), under most circumstances lead to disintegration of the granules because the methanogens die (Tiwari et al., 2006).

In our study on different configurations of high-rate reactor systems, comparing UASB and EGSB, we found, that the UASB reactor could tolerate higher VFA concentration than the EGSB reactor under the treatment of potato juice (*Paper III*). In both reactors, VFA concentration increased, as a result of increased organic load. Total VFA concentrations of 30 mM at pH 7, corresponding to 12 mg/L of free VFAs led to granular disintegration in EGSB reactor (Figure 9). However, the UASB reactor can tolerate total VFA concentrations up to 100 mM, it can be explained, that the granules in the UASB reactor were more packed than those in the EGSB reactor, thus they were less exposed to toxic compounds, compared to the EGSB reactor. This indicated that the EGSB reactor was more sensitive to high VFA concentration than the UASB reactor. Additionally, under anaerobic digestion of POME and deoiled POME, the VFA concentration and especially propionic acid were substantially higher in the EGSB reactor, compared to the UASB reactor (Paper IV). This agreement on propionic acid as a sole process indicator with Hansson et al. (2002) and Nielson et al. (2007) also supports the conclusion that the UASB reactor is more robust and can better achieve stable operation, compared to the EGSB reactor.

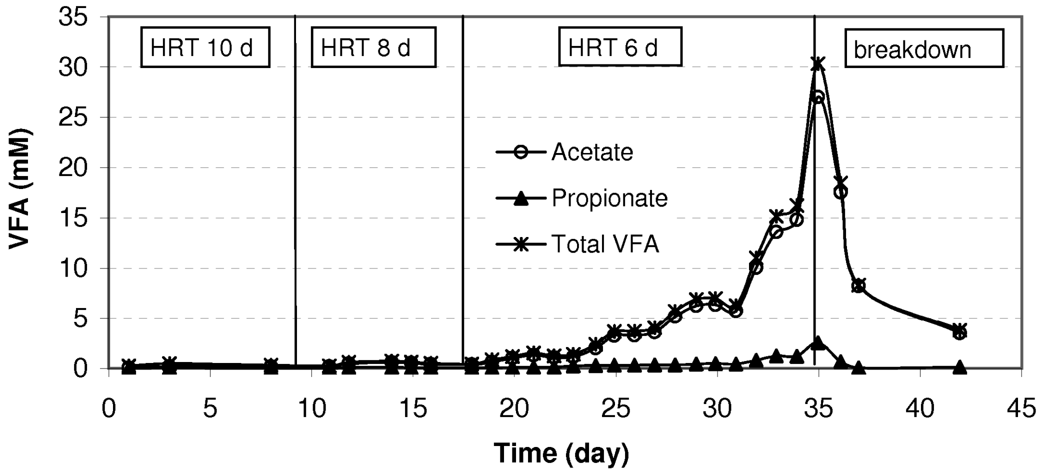

Figure 9 VFA concentrations from potato juice at different HRTs in the EGSB reactor at mesophilic temperature

5.5 Organic loading rate (OLR)

Most industrial organic wastes contain a high fraction of easily degradable organic matters, which results in high methane yield, however also leads to high VFA production. It is therefore important to control OLR to maximize the biogas production. Under-loading the process (with low feeding rate) gives low biogas production rate. It is of course safer to run under-loading to prevent process failure, however it is also uneconomical because the capacity of the process is not fully utilized. Increasing the organic load leads to more biogas production, but also risk of overloading. Overloading of the reactor, normally results in VFA accumulation. Thus, high concentration of VFA decreases pH and makes VFA become more toxic to the methanogens, which can terminate the AD process. That is to say, both under-load and overload introduces process imbalance in the anaerobic digester (Stamatelatou et al., 1997).

Biogas from our research on potato juice achieved 380 mL-CH_4/gVS-added in the EGSB reactor at HRT 8 days, with the OLR 3.2 gCOD/L-reactor.d and increase OLR to 4.2 gCOD/L-reactor.d led to process failure, presumably due to the fast VFA development and high VFA accumulation from the overloading (*Paper III*). In Figure 10, we observed an increase of methane production rate, along with the increasing organic loading rate. However at HRT 6 days, OLR 4.2 gCOD/L-reactor.d finally terminated the AD process. A similar phenomenon was seen while treating POME (*Paper IV*). When the OLR was increased to 10.4 gVS/L-reactor.d, both the UASB and the EGSB reactor performance began to be overloaded and biogas process failed, seen by fast decreasing in methane yield.

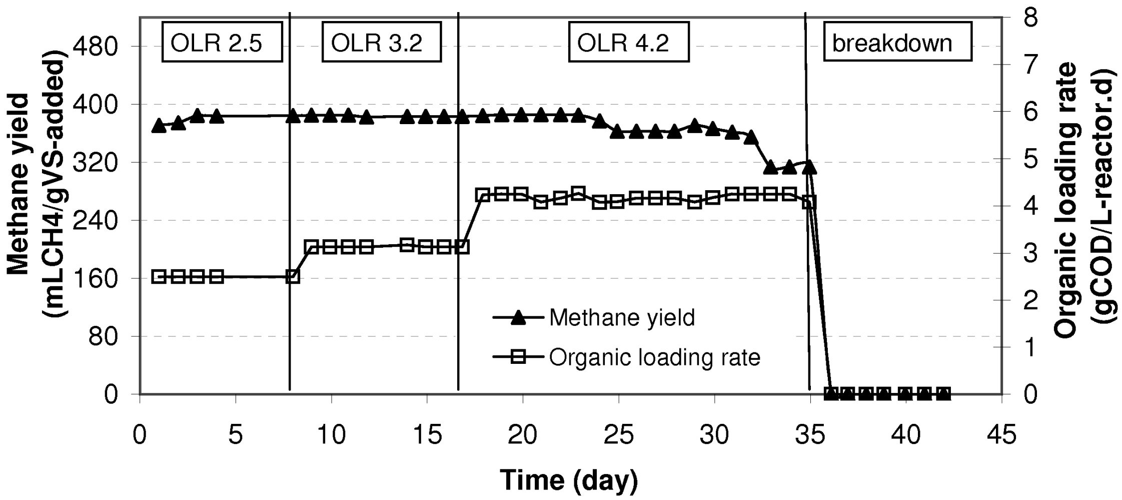

Figure 10 Methane yield of potato juice at different OLRs in the EGSB reactor at mesophilic temperature

5.6 Ion inhibition

The high ion concentration could cause inhibition in the biogas process (McCarty and McKinney, 1961). He et al. (2006) investigated the effect of sodium and potassium at the concentration of 25 and 50 g/L on anaerobic hydrolysis and acidogenesis of vegetable wastes. They observed that acidogenesis was more sensitive than hydrolysis and it was necessary to control pH when the cation concentration was high, in order to ensure successful acidogenesis. Sodium cation has been reported to cause moderate inhibition at 3.5-5.5 g/L and strong inhibition at 8 g/L (Kugelman and McCarty, 1965). De Baere et al. (1984) reported initial inhibition by Na^+ at 30 g/L in a biofilm reactor and suggested that the high tolerance of Na^+ in their study was due to the protection of the microbial communities in biofilm, which mediated concentration gradient, resulting in lower concentrations in the vicinity of the microorganisms.

In general, DM contains lower concentration of sugar and higher concentration of ions, such as sodium and potassium, than normal molasses due to the desugaring process. Due to its relatively high organic contents, DM would constitute an attractive substrate for AD for production of biogas. However, its high ions concentration caused problem for the AD process (*Paper I*). From the correlation in Figure 11, 50% inhibition occurred at a cation concentration of approx. 11 and 28 g/L for sodium and potassium, respectively. 50% inhibition was the cation concentration resulting in half methane yield compared to the methane yield without the cation. In general, the activity of the methanogenic archaea is influenced by several parameters such as temperature, pH, volatile acids, salts and other toxic compounds. The results from this study showed that high concentration of sodium and potassium can also affect the anaerobic digestion process.

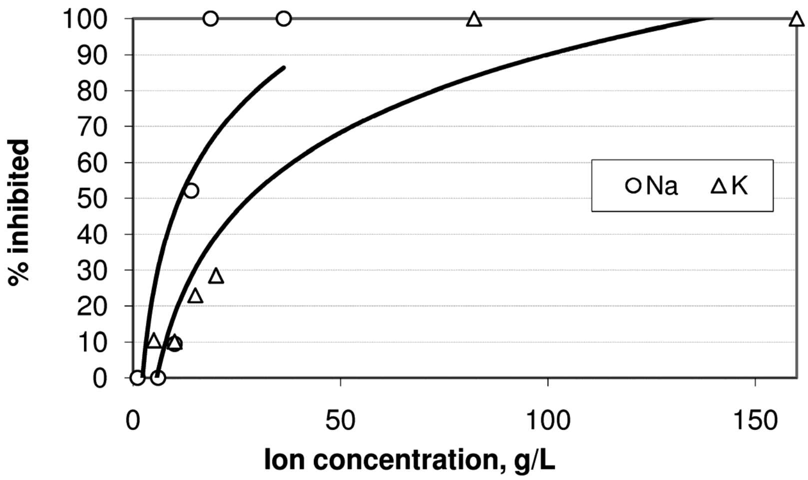

Figure 11 Inhibition effects of sodium and potassium

6 Improvement of the biogas process

Biogas process optimization through better monitoring and control is one way of improving process efficiency (Boe, 2006). Other ways can be through pre-treatment of the substrate to release more biodegradable compounds, or co-digestion with different wastes and/or with animal manure. This will limit the inhibition from the substrate and enhance the biogas production.

6.1 Pre-treatment of substrate

Oil palm mill plants produce large amounts of solid wastes such as EFB (23%), mesocarp fibers (12%) and shells (5%) for every ton of fresh fruit bunch been processed in the mills (Baharuddin et al., 2010). Huge quantity of oil palm biomass, especially 17.1 million tons of EFB was generated by the Malaysia oil palm industry in year 2005 (Chew and Bhatia, 2008). Thus, the treatment of EFB has gained interests from many researchers (Misson et al. 2009; Tan et al., 2010).

6.1.1 Chemical treatment

Chemical treatments include acid, base and oxidant treatments. Acid treatment can significantly improve the reaction rate of the subsequent process of cellulose hydrolysis, while treatment with base increases the internal surface by swelling; decrease of polymerization degree and crystallinity; destruction of links between lignin and other polymers; breakdown of lignin (Sanchez and Cardona, 2008). Alkaline hydrolysis with NaOH has been successfully applied to treat lignocellulosic materials by Sun and Cheng (2002). Pre-treatment of EFB in this study was investigated on NaOH addition and the methane yield in batch experiment was improved by 37% compared to non-treated EFB, and reached 52% of the theoretical value of 422 mL-CH_4/gVS-added (Figure 12, *Paper V*).

6.1.2 Hydrothermal treatment

Hydrothermal, also called steam treatment, is performed at high temperature and pressure. During pre-treatment, the biomass is often mixed with water and heated to around 180-200 ºC for 5-15 minutes in order to destroy the protecting lignin structure and make the cellulose available for the enzymes. It is proved that the hydrothermal pre-treatment can significantly improve biodegradability by achieving sufficient solubilization of the lignocellulose to enhance hydrolysis in the AD process, which results in the increase in biogas production (Bruni et al.

2010). In our study, hydrothermal treatment of EFB was performed and methane yield improved by 29% compared to non-treated EFB, reaching 49% of the theoretical value (Figure 12, *Paper V*).

6.1.3 Combined chemical and hydrothermal treatment

Biofibers treated with steam and catalyst NaOH, resulted in 26% higher methane yield, compared to untreated biofibers (Bruni, 2010). Hydrothermal treatment with NaOH addition may have converted part of the lignin into acetic acid, while hydrothermal treatment with H_3PO_4 addition may have just reallocated lignin (Kaparaju and Felby, 2010). Hydrothermal treatment with NaOH presoaking has been reported to increase the biogas production of sorted municipal solid waste by 50% (Wang et al. 2009). In our study of pre-treatment of EFB, combined hydrothermal and NaOH presoaking was carried out and methane yield was improved by 57% compared to non-treated EFB, and thereby reached 60% of the theoretical value. From the results of three different pre-treatment methods, we concluded that combined hydrothermal and chemical treatment achieved the best methane yield (Figure 12, *Paper V*).

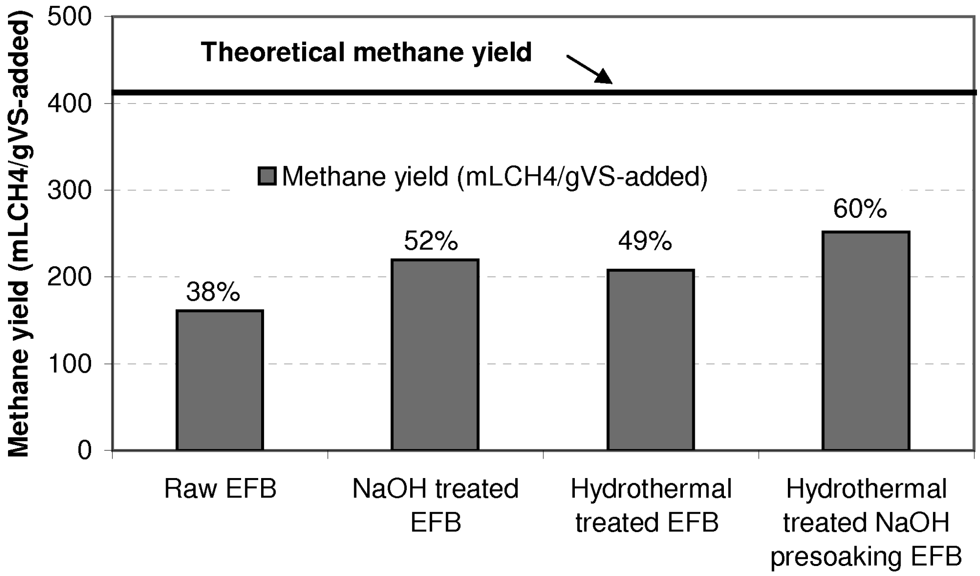

Figure 12 Methane yield under different pre-treatment methods applied to EFB

6.2 Co-digestion strategy

The co-digestion concept, applied in Denmark in the middle 1980's, is based on co-digestion of manure together with other organic wastes. Many centralized full scale biogas plants have been built in Denmark in accordance with this concept

(Raven and Gregersen, 2005). Co-digestion of manure and organic wastes from industry and households has been successfully applied for biogas production (Tafdrup, 1994). Meanwhile co-digestion offers economic and environmental benefits due to cost-sharing by processing multiple waste streams in a single facility (Margarita et al., 2009). There are three main advantages using animal manure for co-digestion. First; it is a source for nutrients, trace metals, vitamins and other compounds necessary for microbial growth. Second; it plays a role in neutralizing pH and improving buffering capacity (Angelidaki and Ellegaard, 2005). Third; the high water content in manure helps dilute the concentrated organic wastes, which would be inhibitory and difficult to treat separately. Moreover, a high buffering capacity in manure makes the process more resistant to the effect of VFA accumulation (Angelidaki and Ellegaard, 2003). Several studies have reported that the biogas process could be improved and stabilized by applying of co-digestion strategy (Totzke, 2009). Gelegenis et al. (2007) observed 10% improvement in biogas yield when applying co-digestion of olive-oil mill wastewater with diluted poultry manure, compared to digestion of poultry manure alone.

In our study of the biogas production from DM, co-digestion with cow manure helped dilute the cations (i.e. sodium and potassium) that we proved the inhibitory compound in high concentrations (see chapter 5.6, *Paper I*). Often, alkali such as $NaHCO_3$ or $KHCO_3$ were added to maintain pH above 4.8, in the anaerobic digestion of pure substrate, such as dry pulps (Hutnan et al., 2000), sugar beet silage (Demirel and Scherer, 2008), or SBP (Hutnan et al., 2001). While co-digestion of SBP with cow manure would avoid alkaline addition, since manure can provide adequate buffering capacity under co-digestion, and at the same time achieve good methane yields (*Paper II*). The methane yield we obtained at 1% DM, diluted with water, was identical to higher DM concentration up to 5%, when mixed with manure. This indicated that higher organic load from DM could be applied, when co-digested with manure (*Paper I*). Co-digestion of SBP with DM helped decrease inhibition from DM, and thus, improve the reactor performance, compared to digest DM alone (*Paper II*). In addition, it would not be sustainable to add water, which would result in larger wastewater volumes, while co-digestion with manure would not cause this problem as manure already exists. Co-digestion of raw EFB with POME was also investigated and the study showed that co-digestion could enhance microbial biodegradability and result in 25-32% higher specific methane production compared to digest EFB alone (*Paper V*).

6.3 Combined pre-treatment and co-digestion

Neves et al., (2006) reported their research on enhancement of the biogas production from industrial waste composed of 100% barley, the waste from production of instant coffee substitutes, which was considered very poor for biogas production. They found out that when the waste was subjected to alkaline hydrolysis pre-treatment before co-digestion with activated sludge, the methane production increased 67%, while, if co-digested with kitchen waste, the methane production increased 61%.

EFB was low in biodegradability due to its lignocellulosic composition (39% cellulose, 22% hemicellulose and 23% lignin). The structure of EFB needs to be significantly improved by pre-treatment before it can be efficiently used for biogas production (*Paper V*). Co-digestion of EFB and POME in a single treatment step would simplify the technical and economical requirements for the conversion of both wastes into biogas. EFB, pre-treated with NaOH presoaking and hydrothermal treatment, co-digested with POME resulted in 140% increase of methane yield compared to digest raw EFB alone and the methane recovery reached 91% of the theoretical potential (Figure 13, *Paper V*).

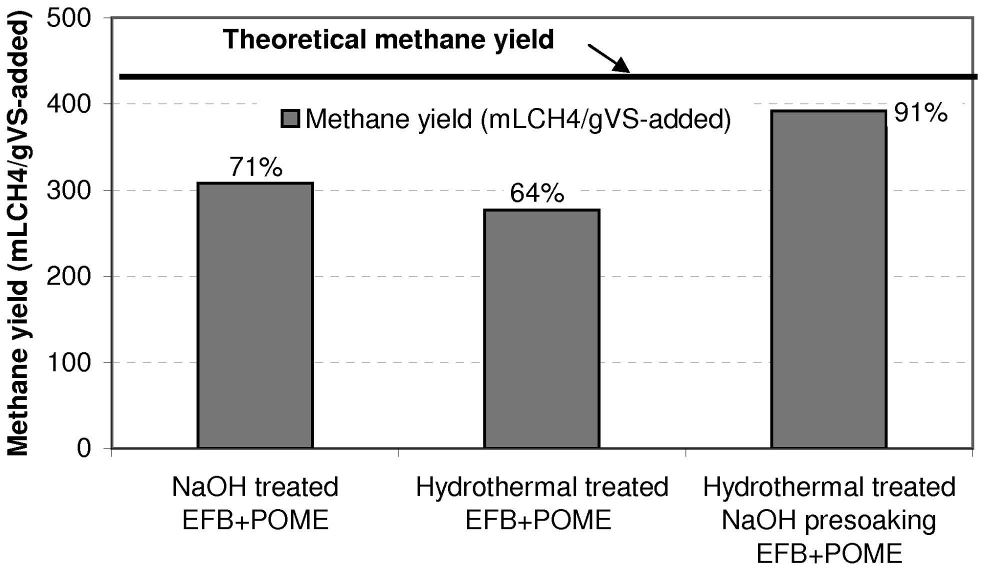

Figure 13 Methane yield under different pre-treatment methods applied to EFB and POME (mixing ratio of 6.8:1 on VS basis; 1:1 on volume basis)

7 Conclusions

This thesis has mainly focused on biogas production from food-processing industrial wastes, including desugared molasses (DM), sugar beet pulp (SBP), sugar beet top (SBT), sugar beet leaves (SBL), potato juice, palm oil mill effluent (POME), deoiled POME and empty fruit bunch (EFB) by anaerobic digestion. The co-digestion strategy in between sugar processing wastes themselves or with animal manure was applied to overcome inhibition and improve the biogas process. Combined pretreatment with co-digestion was investigated in palm oil processing wastes. The major contributions of this thesis work are summarized as follows:

- Methane potentials of food-processing industrial wastes, such as DM, SBP, SBT, SBL; potato juice, potato pulp; POME, deoiled POME and EFB were determined in batch assays. All of them are attractive substrates for biogas production.

- DM, SBP, potato juice, POME and deoiled POME were chosen as model wastes to run the continuous reactor experiments in the continuously stirred tank reactor (CSTR), upflow anaerobic sludge blanket (UASB) and expanded granular sludge bed (EGSB) reactors. The latter four substrates were proved easily degradable substrates, with good methane potential. However, dilution was necessary to avoid organic overload.

- Co-digestion was applied to DM, SBP with animal manure. DM contains more than 2-3 times higher concentration of ions than normal molasses, especially sodium and potassium, which could strongly inhibit the biogas process. In order to minimize the inhibition, co-digestion with manure was applied and resulted in maximum methane yield of DM of 300 mL-CH_4/gVS-added in the CSTR reactor. Successful anaerobic digestion of a mixture of 5% DM with cow manure was achieved and a stable methane production could be obtained at concentrations lower than 15% DM. The average methane yield of 280 mL-CH_4/gVS-added was achieved in a thermophilic reactor, co-digesting 50% SBP with cow manure. Manure proved to be a very good substrate for co-digestion with sugar processing industrial wastes, as it helps diluting the concentrated substrates and also provides buffer capacity and nutrients.

- High-rate reactor configurations, i.e. an UASB and an EGSB reactor, were introduced to potato juice, POME and deoiled POME. HRT of 5 days with OLR 5.1 gCOD/L-reactor.d and methane yield of 240 mL-CH_4/gVS-added in the UASB reactor and HRT 8 days with OLR 3.2 gCOD/L-reactor.d and methane yield 380 mL-CH_4/gVS-added in the EGSB reactor were obtained in steady state when treating potato juice. Both UASB and EGSB are capable of producing biogas at mesophilic conditions; UASB is more tolerate to VFA concentrations, while produces lower methane yield than EGSB. The effluent from the UASB and EGSB reactors could be treated by ammonia retainment method and almost 100% of ammonia has been retained by decreasing pH below 5. Using POME and deoiled POME as feedstock, the UASB and the EGSB reactors were reliably operated at the same HRT of 5 days, OLR 5.8 and 2.6 gVS/L-reactor.d respectively. Both reactors achieved comparably high COD removal efficiencies above 90%.

- Pre-treatment of EFB by NaOH presoaking and hydrothermal treatment, co-digested with POME, at mixing ratios of 6.8:1 on VS basis, corresponding to 1:1 on volume basis, had a high synergetic effect with highest methane potential of 392 mL-CH_4/gVS-added corresponding to 82.7 m^3CH_4/m^3 mixture. Considering an energy content of 36 MJ per m^3 CH_4, the energy content in the produced methane from pre-treated EFB co-digested with POME was 2977 MJ/m^3.

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9 Appendices

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III Fang C, Boe K, Angelidaki I. 2010. Biogas production from potato-starch processing by-products in upflow anaerobic sludge blanket (UASB) and expanded granular sludge bed (EGSB) reactors. Submitted to *Bioresource Technology*.

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