Aim and Scope

The objective of the *Journal of Residuals Science & Technology* is to provide a forum for technical research on the management and disposal of residuals from pollution control activities. The Journal publishes papers that examine the characteristics, effects, and management principles of various residuals from such sources as wastewater treatment, water treatment, air pollution control, hazardous waste treatment, solid waste, industrial waste treatment, and other pollution control activities. Papers on health and the environmental effects of residuals production, management, and disposal and are also welcome.

Editor-in-Chief

P. Brent Duncan
Department of Biology
University of North Texas
Denton, TX, USA
pduncan@unt.edu

Editorial Advisory Board

C O N T E N T S

Selected Papers Presented During the European Conference on Sludge Management's (ECSM) 2010 2nd European Conference on Sludge Management: Dewatering, Drying, Thermal Valorisation, and Climate Change. This Conference was Held in Budapest, Hungary, on September 9–10, 2010.

MANAGEMENT of residual sludge has become a key issue in our global environment. Environmental regulations have tightened as production of sewage sludge has increased over the past decade and are of significant importance in countries that joined the European Union (EU) during the last two rounds of accession. New member states in Central and Eastern Europe (CEE) have developed national strategies meeting European requirements for wastewater treatment during 2010–2020.

These requirements raise issues within the region. Disposal of waste without pre-treatment is prohibited and the importance of energy recovery and waste reuse by way of land application both have increased. Also, during sewage sludge treatment it is important to take into consideration effects on climate change. One of the objectives of the conference was to raise awareness of best technologies for minimizing Greenhouse Gas (GHG) emissions related to sludge management.

This special issue of the Journal of Residuals Science and Technology (JRS&T) gathers some of the papers presented during the ECSM 2010 2nd European Conference on Sludge Management. These articles are a part of efforts designed to share experiences from old member states with new members of the EU and to display innovative technologies for sewage sludge treatment. These efforts provide guidance on how to solve some of the EU and CEE problems.

Due to rapid urbanization and population growth we have witnessed continued growth of many serious environmental issues, including degradation of water quality. For example, Hungary is making massive investments in water and wastewater treatment over the upcoming decades. The new and upgraded treatment facilities will improve water quality, but may also lead to problematic levels of sludge generation, and this prospect is now being addressed with urgency.

We must work together toward sustainable approaches for solving environmental issues worldwide in order to stay ahead of urbanization and population "exponential growth" curves. As populations and industrialization continue to grow and expand so too does the concentration, vectors, and amounts of pollution as well as increased strain on natural resources.

I would like to especially thank Dr. Angelique Leonard from the Department of Applied Chemistry at the University of Liège for gathering these articles for this special conference articles' edition of the JRS&T. I also want to take this opportunity to thank the Editorial Board for their help, their guidance, and for moving the JRS&T forward. Onwards and upwards!

Dr. P. BRENT DUNCAN
Department of Biology
University of North Texas
U.S.A.

Management of Wastewater Sludge's:
A Hot Topic at the European Level

A. LÉONARD*

Laboratory of Chemical Engineering, University of Liège, Belgium

MANAGEMENT of residual sludge has become a key issue in our global environment. Environmental regulations have tightened as production of sewage sludge has increased over the past decade and are of significant importance in countries that joined the European Union (EU) during the last two rounds of accession. New member states in Central and Eastern Europe (CEE) have developed national strategies meeting European requirements for wastewater treatment during 2010–2020.

Production of wastewater sludge is expected to reach 13.5 million tons of dry matter at the 2020 horizon within the EU27. The way to manage these growing amounts of biosolids has become a key issue as displayed by the success of the second edition of the European Conference on Sludge Management, ECSM 2010. This event was held in Budapest last 1st & 2nd of September. The location was chosen to attract participants from the new member states in Central and Eastern Europe. Indeed these states have recently set out their National Strategies on Waste Water Treatment for 2010–2020 on the development of waste water treatment facilities to meet the European requirements on waste water treatment.

The concerned community tries to find more sustainable ways of valorising sludge's. Recycling to land and energy recovery through incineration are the two most important options for now. Nevertheless, other valorisation ways may be investigated, taken into account that sludge's may include up to 50–60% of organic matter on a dry basis. In that sense it can be seen as a source of biomass that can be used to produce renewable energy or chemicals, through biomethanation or gasification for examples. Moreover, research efforts need to explore several treatment steps such as dewatering or drying in order to reduce global energy needs associated with water removal throughout sludge processing.

More than 177 specialists from 22 countries attended the conference consisting of 36 oral presentations and 24 posters. Presentations were classified under several topics: dewatering, drying, digestion-hydrolysis, gasification-combustion, and a last one entitled 'miscellaneous'. As it was realized for the first edition of ECSM, the best extended papers were selected for publication in the Journal of Residuals Science and Technology (JRS&T).

Please note the 3rd Edition will take place in September 2012 in Leon, Spain.

*Author to whom correspondence should be addressed.
E-mail: A.Leonard@ulg.ac.be

Change of the Digested Sludge Quality at Microaerobic Digestion

P. JENICEK*, C.A. CELIS, J. KOUBOVA and I. RUZICKOVA

*Department of Water Technology and Environmental Engineering, Faculty of Environmental Technology,
Institute of Chemical Technology Prague, Czech Republic*

ABSTRACT: The implementation of microaerobic conditions often improves the results of anaerobic digestion. The study was focussed on comparison of digested sludge quality after shift from strictly anaerobic to microaerobic sludge digestion. The compared parameters were: chemical composition of sludge, specific activity of methanogenic bacteria, foaming potential and dewaterability of sludge. In most parameters the quality of sludge digested at microaerobic and anaerobic conditions is comparable often the quality of microaerobic sludge is better. From the operational point of view it is very important that foaming of microaerobic digester was lower and the dewaterability was better for microaerobic sludge.

INTRODUCTION

THE quality of digested sludge is influenced significantly by specific conditions of digestion process. Some studies comparing the quality of sludge in relation to operational conditions or sludge pretreatment methods were published [1, 2]. Little is known about influence microaerobic conditions or increase of oxidation-reduction potential (ORP) on quality of digested sludge.

It has been proved in different studies that the use of microaerobic conditions in anaerobic digestion can be beneficial. The application of microaerobic technology is an efficient method of hydrogen sulphide removal from biogas [3, 4, 5, 6] and sulphide toxicity suppression [7, 8]. The implementation of microaerobic conditions has in many cases improved the efficiency of anaerobic digestion and enhanced hydrolysis and biodegradability of some organic compounds [9, 10].

In a mixed culture, even strict anaerobes can survive without any inhibition, if facultative microorganisms are able to consume the present oxygen quickly and fully. It was shown that the presence of limited amount of oxygen in digester does not destroy the digestion process even in the system where the oxygen is not consumed by sulphide oxidation [11, 12].

In this paper, microaerobic sludge means the sludge cultivated in anaerobic system with limited (trace) oxygen consumption. With respect to the ORP, the micro-

aerobic system can be marked generally as a system in which micro-consumption of oxygen causes a limited ORP increase [13].

Microaerobic conditions are obtained by dosing of a limited amount of air (oxygen can be used as well) into anaerobic reactor. There are two basic configurations of microaerobic technology. The air can be pumped into the gas space of the digester or into the mixed liquor suspension. In the first alternative, the risk of biogas pipe clogging by sulphur is higher; at the second alternative a bigger surplus of oxygen is needed. Moreover because of closer contact of sludge with oxygen the likelihood of the bacterial consortia affection is more significant in the second case.

There were published studies comparing the specific bacterial activity after a shift from strictly anaerobic to microaerobic sludge digestion. Tang [14] reported that microaeration has no obvious effect on the phylogenetic diversity of microorganisms. However the results indicated that ratio between hydrogenotrophic and acetoclastic methanogens changed due to microaeration. Zitomer and Shrout [15] reported that the methanogenic activity can sometimes be even higher under microaerobic conditions in comparison with strictly anaerobic system.

Less attention has been paid till now to comparison of digested sludge quality after anaerobic and microaerobic digestion. It is therefore the main aim of presented paper. The present paper aims to evaluate the changes in chemical composition, foaming potential, dewaterability and methanogenic activity after switch from fully anaerobic to microaerobic conditions.

*Author to whom correspondence should be addressed.
E-mail: jenicekp@vscht.cz

MATERIAL AND METHODS

The experiments were carried out in two continuously stirred lab-scale reactors with 10 L of working volume. Reactors were operated at mesophilic temperature. Total duration of parallel operation of both reactors was 208 days.

The start-up of both reactors was conducted in anaerobic conditions. After start-up, the operation of the first reactor was changed to microaerobic. To the bottom of one of them was continuously dosed air. Second reactor remained anaerobic and served as referential. Waste activated sludge of municipal wastewater treatment plant was used as the substrate for reactors; the sulphur content was increased by natrium sulphate addition. The average sludge composition was following: total suspended solids (TSS) 32.8 g/L, volatile suspended solids (VSS) 20.1 g/L, pH 7.84.

The reaction mixture was kept homogeneous by mechanical mixing and operational temperature was kept at $40 \pm 1°C$. The volumetric loading rates of digesters were 2.0 g/L.d and 0.15 g/L.d for chemical oxygen demand (COD) and SO_4^{2-} respectively. The retention time in both reactors was on average 30 days. To reach the microaerobic conditions and oxidize the sulphide produced by sulphate reducing bacteria, one reactor was connected to an air supply by peristaltic pump with flow rate fixed to 1.7 L/d.

Sludge Composition

Analytical procedures were carried out according to Standard Methods for the Examination of Water and Wastewater [16], the biogas composition and volatile fatty acids were determined by gas chromatograph GC 8000Top equipped with a heat conductivity detector HWD 800 [17].

The elemental composition of sludge was assessed by X-ray fluorescence analysis, using the ARL 9400 XP sequential WD-XRF spectrometer. It is equipped with the Rh anode end-window X-ray tube type 4GN fitted with 75 µm Be window. All peak intensity data were collected by software WinXRF in vacuum.

Specific Activity of Methanogenic Bacteria

The assessment of the specific methanogenic activity (SMA) was carried out in serum bottles under mesophilic conditions without mixing using initial loading of 1 g COD/g VSS. All tests were carried out in three replicates. The volume of produced methane was monitored and maximum methane production rate calculated according to guidelines proposed by Angelidaki *et al.*, [18]. The production of biogas was measured volumetrically using water displacement method. The biogas composition was determined by a gas chromatography. The tests were performed with acetate as organic substrate and therefore the activity of acetoclastic methanogens was assessed.

Foaming Potential and Foam Stability

These parameters were assessed as additional characteristics of digested sludge, because presence of filamentous foam forming bacteria was elevated. It has been reported, that anaerobically treated excess activated sludge exhibits the tendency to cause foaming in digester. For the description and comparison of foam quantity and quality the "bubble test" has been developed [19]. The testing was based on the test described by Pagilla *et al.*, [20] and modified with respect to the character of anaerobic sludge. The final test was carried out by bubbling of 1 liter of sludge by nitrogen with the flow rate of 1 L/min in 2 L volumetric cylinders. The level of foamy sludge is recorded after 5 minutes of bubbling and the foaming potential (FP) is calculated from this value [Equation (1)]. 5 minutes after the stopping of the gas flow the level of foamy sludge is recorded again to calculate index of stability (IS), [Equation (2)]. FP describes the capability of the sludge to create foam. IS gives the information about stability of the foam created.

$$FP = \frac{V_5}{V_0} \qquad (1)$$

where

$$IS = \frac{(V_{ST} - V_0)}{(V_5 - V_0)} \times 100 \qquad (2)$$

V_0 = volume of sludge at the beginning of measuring (usually 1 L)
V_5 = volume of foamy sludge after 5 min of bubbling
V_{ST} = volume of foamy sludge 5 min after the gas flow stop

Microscopy

The microscopic analysis of sludge samples was performed according to Jenkins *et al.* [21]. The microscopic examination in wet mounts (Olympus BH2-RFCA—125×, 250× and phase contrast 1250× mag-

nification) was aimed at basic biomass characteristics (morphological properties, structure, presence and types of protozoa/metazoa and zoogloeal colonies). Gram and Neisser staining procedures were applied to determine the abundance and to identify filamentous microorganisms.

CST

Capillary suction time (CST) is a simple parameter and method used for characterization of sludge dewaterability. Relationship between dewaterability measured by CST and a number of sludge properties has been shown in the past and it was found that the CST test is accurate, if the product of solid concentration and specific resistance to filtration is of interest [22]. The original circular setup introduced by Gale and Baskerville [23] and Whatman-17 filter paper were used for measurement.

RESULTS AND DISCUSSION

The main results of anaerobic digestion process for both reactors are shown in Table 1. It is evident that despite air dosing, the specific methane production in the microaerobic digester was higher. Significantly better quality of sludge liquor as regards soluble COD was proved. Rest of nitrogen from dosed air caused lower methane concentration in biogas of microaerobic digester, however if we balance the methane production the result of microaerobic digester is slightly better, due to suppressed inhibition by sulphides.

Table 1 shows that also variation in basic parameters of the sludges is small. Bigger difference can be found in sludge liquor composition (soluble COD). Higher concentration of total sulphide in anaerobic sludge is

not in contradiction with data about sulphur in Table 2 and indicates the oxidation of sulphide.

In the evaluated period the hydrogensulphide concentration in biogas produced in the anaerobic reactor was high (almost 7.3 g/m^3), therefore estimated H_2S removal efficiency by microaerobic digester is above 99% confirming results previously reported [11, 24].

Sludge Composition

Small differences were found in elemental composition of digested sludge from microaerobic and anaerobic reactor (Table 2). Expected increase of sulphur content due to precipitation of elemental S in sludge was confirmed, however this increase was not significant because of continuous removal of sludge (and sulphur) from digester. High Fe content is caused by Fe^{3+} addition in wastewater treatment technology where the treated activated sludge originates from.

Specific Activity of Methanogenic Bacteria

Activity of methanogenic bacteria was periodically monitored during the digesters operation. The results of activity tests are shown in Figure 1. Immediately after the start of microaeration, decrease (16%) of specific methanogenic activity (SMA) was observed. When sulphate dosing was initiated, methanogenic activity of both anaerobic and microaerobic biomass increased, possibly because of previous deficit of sulphur. During further operation, the SMA dropped again in both reactors, however, the decrease of activity of microaerobic biomass was smaller and therefore its activity was higher in comparison with the anaerobic biomass. This activity development was mainly affected by dissolved sulphide concentration probably—Figure

Table 1. Comparison of Digestion Results and Sludge Quality (Average Value and Standard Deviation).

Parameter	Microaerobic Digester	Anaerobic Digester
soluble COD (g/L)	3.44 ± 0.23	4.80 ± 0.31
specific methane production (L/kg VSS$_{added}$)	330 ± 1.5	305 ± 2.1
hydrogensulphide in biogas (mg/m³)	47 ± 14.4	7298 ± 489
TSS (g/L)	27.8 ± 0.93	27.2 ± 0.42
VSS (g/L)	16.4 ± 0.40	16.1 ± 0.28
VSS/TSS (%)	58.98 ± 0.66	59.29 ± 0.59
COD (g/L)	32.5 ± 1.5	32.7 ± 1.5
Total Sulphide (mg/L)	615 ± 46	858 ± 43
ORPH (mV)	−278 ± 11	−309 ± 2.5

Table 2. Relative Elemental Composition of Digested Sludge from Microaerobic and Anaerobic Reactor (Major Elements of Mineral Fraction), (132nd day).

Element	Microaerobic Sludge (%)	Anaerobic Sludge (%)	Ratio Anaerobic/ Aerobic
Fe	28.7 ± 0.1	29.2 ± 0.1	0.983
P	17.3 ± 0.1	17.1 ± 0.1	1.012
Ca	11.9 ± 0.09	11.9 ± 0.09	1.000
S	9.92 ± 0.09	9.68 ± 0.09	1.025
Na	9.58 ± 0.09	9.66 ± 0.09	0.992
Al	8.48 ± 0.09	8.30 ± 0.09	1.022
Si	5.60 ± 0.07	5.43 ± 0.07	1.031
K	3.67 ± 0.06	3.80 ± 0.06	0.966
Mg	1.43 ± 0.04	1.44 ± 0.04	0.993
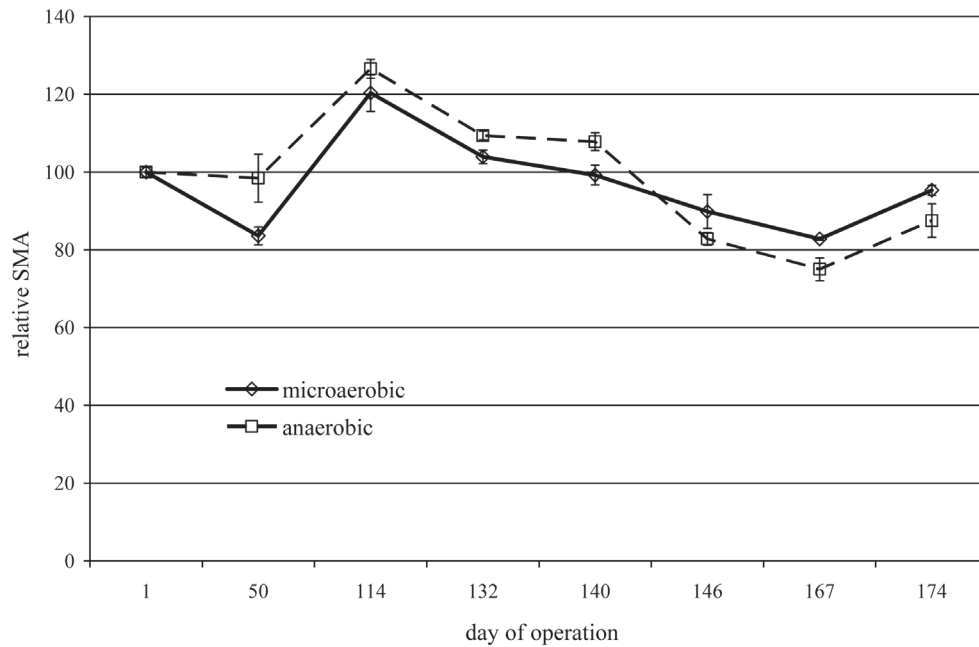
Figure 1. *The course of methanogenic bacteria activity.*

2. Sulphide concentration was gradually rising in both reactors; however, the concentration in microaerobic reactor was always significantly lower. IC_{50} (median inhibition values) reported for methanogenic activity range between 30 and 250 mg/L [25], however free H_2S is most toxic form and therefore also pH value is important. At actual pH of digesters (8.15 respectively 8.13) the maximum concentration of free H_2S was 14 and 24 mg/L for microaerobic and anaerobic digester respectively.

Foaming Problems

The waste activated sludge treated contained in some periods increased amount of filamentous bacteria and therefore the risk of digester foaming was high. A fear of stimulation of aerobic bacteria of activated sludge treated in digester existed at the start of microaerobic digester operation. The presence of oxygen could help to the survival of aerobic filamentous bacteria in microaerobic digester and consequently to stimulate the
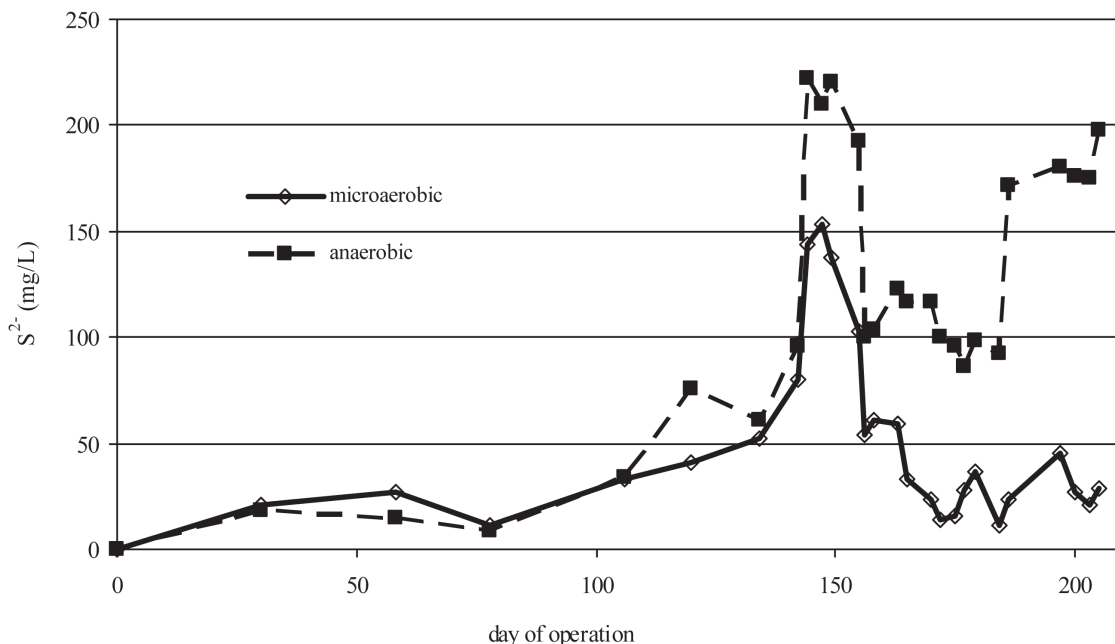
Figure 2. *The course of dissolved sulphide concentration.*

foam production. The results overcame the fear of behavior of aerobic filamentous bacteria in microaerobic conditions.

Especially during period with high foaming potential, the difference between microaerobic and anaerobic digester was high. The maximum foaming potential FP was 3.2 and 2.6 for the anaerobic and microaerobic reactor, respectively. In addition, the foam rising in anaerobic reactor was much more stable in comparison to the microaerobic reactor. The index of stability IS was 96% and 53% in the anaerobic and microaerobic reactor, respectively.

Better degradation of filaments of activated sludge (with dominance of *Microthrix parvicella*) during microaerobic digestion can be illustrated by microscopic pictures of digested sludge in Figure 3, where the fragments of filaments are in anaerobic sludge longer and more frequent and more compact.
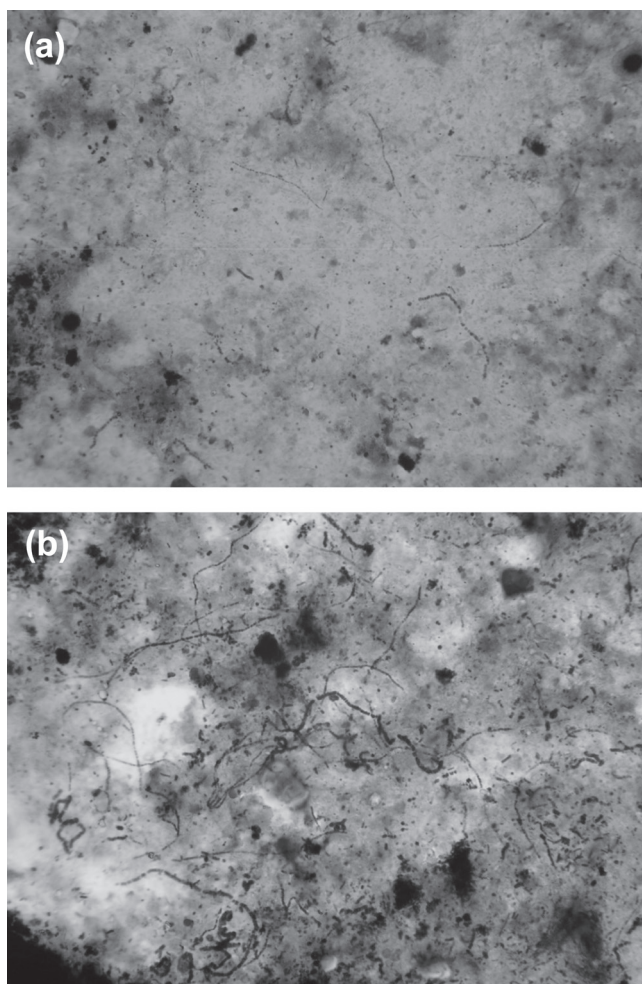
Figure 3. *Microscopic pictures of digested sludge; (a) microaerobic and (b) anaerobic, showing different abundance and morphology of filaments (Gram staining, direct light, enlargement 1250x magnification).*

Table 3. *Comparison of CST Value (in seconds) for Digested Sludge from Microaerobic and Anaerobic Reactor.*

Parameter	Average, std. dev.	Minimum	Maximum
Microaerobic digester	1484 ± 366	1176	2116
Anaerobic digester	2052 ± 672	1499	3148

Evaluation of Dewaterability by CST

Dewaterability was characterized by periodic CST measurement of sludge samples from both digesters. The results are shown in Table 3 and indicate that the dewaterability of microaerobic sludge is slightly better, however from the difference is not statistically significant. The CST values are relatively high due to specific character of treated sludge and due to high TSS and dissolved salts concentrations 27–30 g/L and 4.5–5.5 g/L respectively. Similar results were found by Ayol [26] and Wang and Dentel [27].

CONCLUSIONS

Based on the presented results of the long term monitoring of sludge quality of anaerobic and microaerobic digesters we can draw following conclusions:

- The quality of sludge in both reactors is similar, only small differences were found in composition and activities.
- Slightly higher content of total S and lower content of $S-S^{2-}$ in microaerobic sludge indicate the accumulation of elemental S.
- Methanogenic activity slightly decreased due to air dosing in the digester, however, the methanogenic activity of microaerobic biomass was higher than that of strictly anaerobic biomass at higher sulphide concentrations.
- Composition of sludge liquor differs mainly in soluble COD concentration which is significantly lower at microaerobic digestion.
- The sludge digested in microaerobic conditions manifested lower foaming potential and foam stability, dewaterability characterized by CST was not significantly better for microaerobic sludge.

ACKNOWLEDGEMENTS

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Biofiltration of Reduced Sulphur Compounds: Impact of Packing Material Inoculation with Acclimatized Microbial Communities on Performance

P. LEGRAND[1,*], L. MALHAUTIER[1], P. GEIGER[2] and J-L.FANLO[1]

[1]*Laboratoire Génie de l'Environnement Industriel, Ecole des Mines d'Alès, Avenue de Clavières 6, 30319, Alès Cedex, France*

[2]*Europe-Environnement S.A., Rue des Pins 1, 68700, Aspach-le-haut, France*

ABSTRACT: In this study, we explored the mechanisms of dimethyl sulfide (DMS) biodegradation by an acclimatized microbial community used as inoculum of an organic packing material in a gas biofilter. Firstly, experimental parameters such as temperature and pH seem to have an impact on DMS removal. DMS-polluted gas has then been treated by a laboratory-scale biofilter. DMS biodegradation activity, diversity and structure of the microbial community have been investigated. DMS elimination capacity of 4.3 ± 1.7 g DMS. m^{-3} sawmill chips. h^{-1} was achieved and 100% of bed height was required. DGGE analysis highlighted that the microbial community structure was different between each level (Bray-Curtis similarity values fluctuated between 17 and 45%) and hence did not highlight any height effect.

INTRODUCTION

THE main industrial sources of organic sulphur compounds are gaseous emissions from chemical, food processing, wastewater treatment plants and composting. Composting is more and more applied to the treatment of sludge from wastewater treatment plants, positioning as an alternative to landfill and incineration [1]. Sludge composting facilities generate atmospheric pollution and olfactive nuisances due to the emission of volatile compounds during biodegradation process [2]. Due to their obnoxious smell and low odour threshold, organic sulphur compounds especially caused odour nuisance in the surrounding areas. Legislation on emission control of volatile organic compounds and other odorous pollutants has proliferated worldwide [3, 4]. Biological processes for air pollution control have become established technology for the control of emissions of volatile organic compounds, odors, or hazardous air pollutants [5]. At present, gas biofiltration has been considered to be a cost-effective and reliable technology for control of low-concentration waste gases contaminated by volatile organic compounds and other odors [6].

Nevertheless, it has been shown that the biological removal of organic sulfur compounds from waste gases is hindered by preferential degradation of hydrogen sulphide, relatively low degradation rates and decreased removal efficiencies at acidic pH values [7–8]. It has been also suggested that the biological removal efficiency of organic sulphur compounds can potentially influence by inoculum choice [9].

In this study, Dimethyl Sulfide (DMS) was chosen as a target compound because it significantly contributes towards odorous emissions, and its degradation rate is lower than Methanethiol and Dimethyl Disulfide.

The aim of the study was thus to describe the operation of a biofilter packed with an organic packing material (sawmill chips) for the removal of DMS. This bioreactor was inoculated with an acclimatized *inoculum* operated at pH closed to neutrality. The stability and recovery of the reactor under stable operating conditions was tested during 333 days. At steady-state, the microbial community diversity and structure along bed height was assessed using Denaturating Gradient Gel Electrophoresis (DGGE).

MATERIALS AND METHODS

Inoculum

The inoculum was an acclimatized microbial suspension obtained from farmland sampling (vineyard).

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*Author to whom correspondence should be addressed.
E-mail: paul.legrand@ema.fr

The initial microbial suspension was obtained by mixing farmland sampling in a mineral medium [10]. This microbial suspension was then continuously supplied with a gaseous effluent containing DMS at 100 mg.m^{-3} with a flow rate of 100 mL.min^{-1}. To obtain complete DMS removal, pH of the microbial suspension was closed to neutrality.

BIODEGRADATION KINETICS

Experimental Apparatus

Tests were performed in 500-mL flasks containing 250 mL of buffered mineral salts medium [10] and sealed with Teflon-coated cap (Interchrom; Montluçon, France). The flasks were directly connected to a Varian CP-3800 gas chromatograph equipped with a pulse flame photometric detector via sterile 1/16″ Teflon-tubes.

Procedure

DMS (2000 µg) was added as the sole carbon and energy source. After water-air substrate equilibration according to physical laws and air-tightness control (24 hours), flasks were inoculated with a bacterial suspension (3 mL) coming from the acclimatized microbial suspension described above. A centrifugation of the microbial suspension (50 mL) with an OD (660 nm) of 0.1 (about 1.05 mg of bacterial cells per culture bottle) was previously realized and the pellet was then suspended in 3 mL of mineral salts medium. Incubation was performed at an agitation speed of 130 rpm. Six temperatures (10, 15, 20, 25, 30, and 35°C) were tested. Control flasks without inoculation were also carried out. Tests and controls were realized in triplicate for each temperature tested. The pH of the liquid medium was checked at the end of incubation by using a pH meter (Suntex TS-1, Hsi-Chih City, Taiwan).

Biodegradation Monitoring

The gas sample was injected into the column every three hours via an automatic gas sampling valve (250 µL). A 30-metre CP-Sil 5CB apolar capillary column (Varian, USA) was used with a carrier gas (Helium) flow rate of 1 mL. min^{-1}. The temperatures of the column and the detector were 110°C and 250°C respectively. The biodegradation was monitored by on-line gas phase measurement of substrate consumption until it was exhausted.

The maximum DMS consumption rate was obtained from experimental data by using linear regression between three consecutive experimental values.

PILOT SCALE BIOFILTRATION UNIT

Biofilter Set Up

The laboratory pilot unit consisted of a glass column that was filled with sawmill chips up to a total bed height of 1 m, corresponding to a packing volume of 7.8 10^{-3} m^3 of (Figure 1). This is equipped with gas-sampling ports that were located at the inlet and outlet of the biofilter, as well as at intervals of 20 cm (10, 30, 50, 70 and 90 cm from the inlet respectively). Sawmill chips were screened between 5 and 10 mm, its thickness was between 1 to 5 mm. This organic packing material was characterized by a bulk density of 330 kg.m^{-3}, a void fraction of 67% and a pH of 6.2. It was kept at a constant moisture content of 67%.
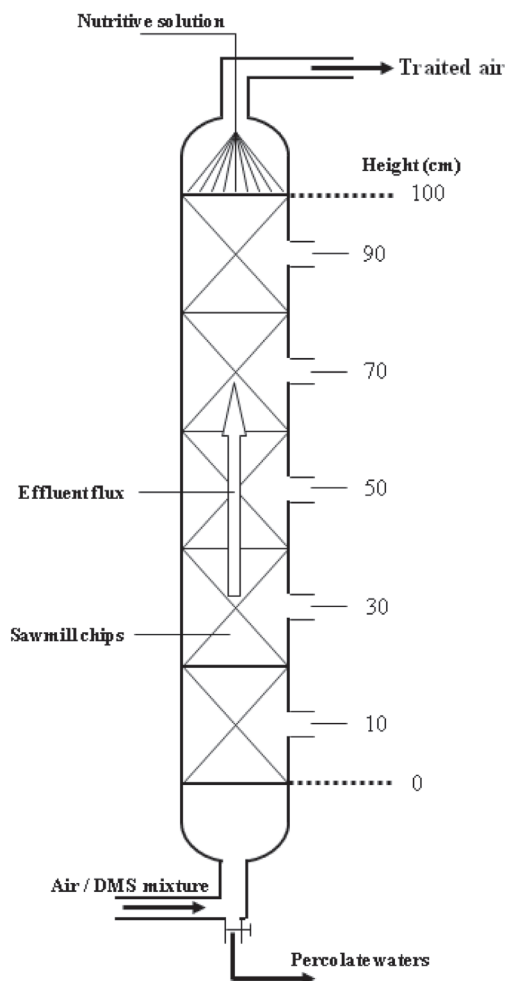

Figure 1. Diagram of the laboratory pilot scale unit of biofiltration used for DMS removal.

Procedure

The polluted gas was obtained by volatilizing liquid DMS (99+% Extra Pure, Fluka, Saint Quentin Fallavier, France) in atmospheric air via a syringe pump (Apparatus PHD 2000 Infusion, Harvard, Massachusetts, USA). The gaseous stream was then homogenized and was humidified in a scrubber (>96% relative humidity). The column was then supplied in upward flow mode with polluted air containing DMS at a concentration of 55–65 $mg.m^{-3}$ air and with a volumetric flow of 0.6 $m^3.h^{-1}$, which corresponds to a superficial gas velocity of 77 $m.h^{-1}$. The empty bed residence time (EBRT) was 47 s and the volumetric load was between 4.2–5.0 $g.m^{-3}$ sawmill chips.h^{-1}. The packing material was regularly sprayed (inflow of 750 $mL.day^{-1}$) with the mineral salts solution quoted above [10] in order to maintain a constant high humidity content and to provide essential nutrients for microbial source [11]. A buffer solution of Tris (Hydroxymethyl aminomethane) (VWR, Val Fontenay, France) at a final concentration of 5.10^{-3} $mol.L^{-1}$ was added to the mineral salt solution for pH regulation.

Inoculation

The column was seeded by recirculation of the acclimatized bacterial culture with an OD (660 nm) of 0.1 (about 52 mg of bacterial cells) during the first day of operation.

Biodegradation Activity

At the inlet and each gas-sampling port, DMS was daily detected and quantified on-line by a gas chromatograph (trace, thermo Fisher Scientific Inc., Waltham, MA) equipped with a polar capillary column (Nukol Supelco, 30 m, 0.25 mm Internal Diameter, 0.25 µm film thickness, Sigma-Aldrich, St Louis, MO) and a flame ionization detector. The gas sample was injected into the column via an automatic gas sampling valve (250 µL). The concentration of a compound was defined as follows: the area of the peak was directly related to the concentration via the calibration of the chromatograph. Based on the contaminant concentration between inlet and outlet, the estimated biodegradation was monitored by calculating the DMS removal efficiency. Monitoring of the DMS degradation in the packing material was expressed as the ratio C/C_0 (C_0: Inlet DMS concentration; C: DMS concentration) in function on the ratio H/H_0 (H_0: Column height (0 m); H: column height).

Bacterial Community Analysis

Sampling

Sawmill ships were sampled along biofilter height (10, 30, 50, 70, and 90 cm of total column height).

DNA Extraction

Each sample was duplicated and constituted of 5 g of sawmill chips suspended in 15 mL of sterile physiological serum (NaCl 0.9%). After cell detachment, the sawmill chips were discarded, and the liquid phase containing suspended cells was centrifuged at 10,000 rpm for 10 min. The cell pellet was recovered, and DNA was extracted from a pellet fraction using Power-Soil DNA Kit (MoBio, Carlsbad, CA, USA).

PCR Amplification

The V3 region (196 bp, corresponding to the Escherichia coli position F337-R533) of the eubacterial 16S rRNA gene was PCR amplified using primers V3F (5′-ACTCCTACGGGAGGCAGCAG-3′) and V3R (5′-ATTACCGCGGCTGCTGGCAC-3′), both slightly modified from Muyzer et al. (1993). A 40-base GC clamp was attached to the 5′ end of the forward primer. Amplification was performed on ~100 ng DNA template (approx. 1–2 µL) in a final volume of 50 µL containing 0.6 µM of each primer (Sigma-Aldrich), 0.8 mM of dNTP mix (0.2 mM each dNTP, Abcys, Paris, France), 10 µL of 5× Reaction Buffer (Finnzymes, Espoo, Finland), and 1.25 U of ThermoStart (VWR, Val de Fotenay, France). PCR was achieved by Tpersonal thermocycler (Biometra, Goettingen, Germany) using the following program: initial denaturation at 98°C for 4 min, 30 cycles consisting of 30 s denaturation at 98°C, 30 s annealing at 68°C and 30 s extension at 72°C, and final extension at 72°C for 10 min. Negative controls were included to verify the absence of contamination. PCR products were visualized by electrophoresis on 2% agarose gel and quantified by absorbance at 535 nm after PicoGreen staining (Quant-iT ds DNA HS reagent, Invitrogen, OR, USA).

Denaturing Gradient Gel Electrophoresis (DGGE)

Denaturing gradient gel electrophoresis (DGGE) was performed with Ingeny phor U-2 system (Goes, The Netherlands) according to the protocol of Muyzer et al. [12], using 8% polyacrylamide gels prepared with

denaturing gradient ranging from 43% to 63% (where 100% denaturant contained 7 M urea and 40% (v/v) formamide). Samples of approx. 500 ng of PCR product were loaded on the gel, along with reference standards for further pattern alignment. Electrophoresis was run at 60°C and 100 V in 1× TAE buffer, for 16 h. Gels were stained with Sybr green I (Sigma-Aldrich), washed, and photographed at 520 nm. Gels images were analyzed with Bionumerics software (Applied Maths, Gent, Belgium).

Computation of Community Diversity Indices

The species richness was defined as the DGGE band number. The Shannon index H was used as a diversity index, which took into account both the number of DGGE bands and their relative intensity:

$$H = -\sum_{i}^{n} p_1 \log(p_i)$$

where p_i is the relative abundance of the ith band of the profile.

Analysis of the Community Genetic Structure

After exclusion of the rarest bands, the initial data matrix (relative intensities according to position) was standardized and transformed by square root [13]. The pair-wise similarity index $S_{i,j}$ between community profiles i and j was calculated by the Bray-Curtis coefficient that was generated with Gelcompar II software (Applied Maths, Gent, Belgium). Hierarchical clustering, using UPGMA linking, was performed with GelcomparII software.

RESULTS AND DISCUSSION

Acclimatizing Bacterial Community

An acclimatized microbial suspension that was obtained from farmland sampling (vineyard), was used in this study. DMS was completely removed from air for several months by this microbial suspension. As sulfur-based phytosanitary products are commonly used for the treatment of vineyard, it is probable that microflora isolated from vineyard soil has some potentialities to remove sulfur-based compounds.

Influence of Temperature on DMS Biodegradation

The influence of incubation temperature on DMS degradation capacity of the acclimatized microbial suspension has been investigated. Figure 2 shows the obtained results for initial DMS quantity of 2000 µg that correspond to gas concentration of 600 mg.m^{-3} for different incubation temperatures tested. For each experiment, the thermodynamic equilibrium was reached and maintained for 24 hours before inoculating cells. For each tested temperature, DMS quantity introduced in the flask was completely biodegraded by microorganisms between 72 and 96 hours. In this figure (Figure 2), the experimental maximum DMS consumption rate obtained for triplicates has been plotted as a function of incubation temperature. For all experiments carried out, the relative standard deviation of maximum DMS consumption rate varied between 1.5 and 10.5 %.

An optimum DMS degradation capacity was observed for a temperature of 25°C with a maximal DMS consumption rate of 131.50 µg.h^{-1}. In our case, the temperature range for which the maximal DMS con-
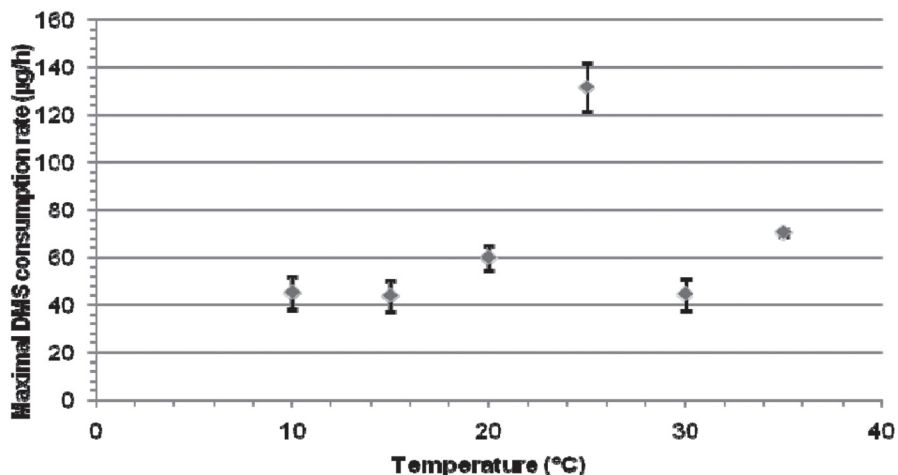

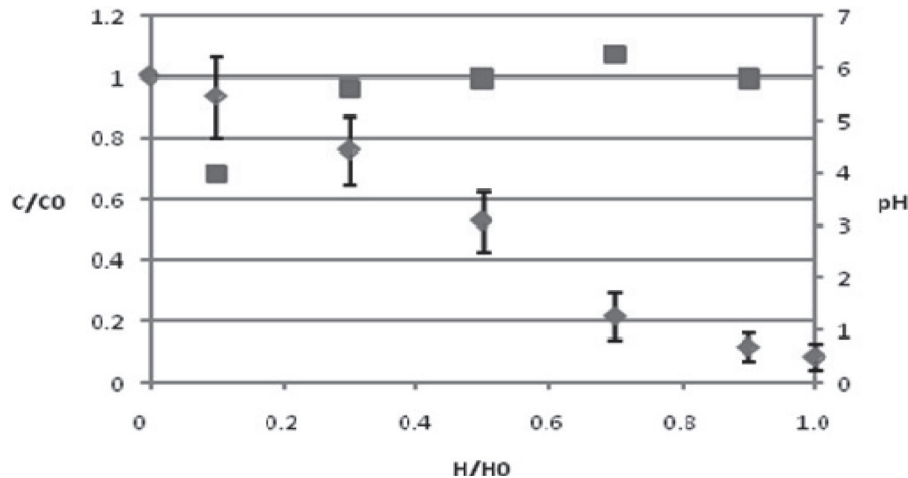**Figure 2.** *Influence of temperature on maximum DMS consumption rate.*

Figure 3. *DMS removal vs. the column height for the biofilter (average of DMS concentration values corresponding to the last 46 days of functioning). The pH of the leachate was 6.9. C_0: inlet DMS concentration; C: DMS concentration; H_0: column height (0 m); H: column height. ■ pH of packing material vs. the column height (measurement occurring at 333-day).*

sumption rate was observed is closed to 25°C. Hence, the activity of enzymes involved with the DMS metabolic pathway seemed to be optimal near 25°C. Most biofilters are colonized with mesophilic microbial communities, that is to say microbes that thrive at temperatures between 20 and 45°C [6–14]. In general, for this temperature range, enzymatic activity increases in function of temperature. DMS biodegradation activity of this planktonic microbial community would be more dependent on temperature than attached microbial community in biofilter. This difference could be attributed to growth and environmental conditions (attached biofilm *vs* planktonic biomass, substrate availability). There is little information concerning DMS metabolic pathways in literature and only a few detailed study about reaction mechanism of DMS biodegradation has been reported [15–16]. These works dealt with some bacteria such as *Hyphomicrobium* and *Thiobacillus sp.* only that converted DMS to formaldehyde and hydrogen sulphide via Methanethiol [15]. From our results, biofiltration experiments were then performed at room temperature (closed to 25°C).

Biofiltration Efficiency

DMS elimination was monitored for 333 days. At steady state, DMS elimination capacity for the applied load (4.2–5.0 g DMS. m^{-3} sawmill chips. h^{-1}) reached almost 4.3 ± 1.7 g DMS. m^{-3} sawmill chips.h^{-1}. After a short period of stabilization (about 17 days) the removal efficiency was stable throughout the experimental period.

The degradation capacity of the bacterial community attached to the packing material was visualized

according to the height of the support material (Figure 3). On this figure, the DMS concentration ratio C/C_0 was plotted against the height ratio h/h_0, where C is the outlet concentration ($mg.m^{-3}$) at the height h of the biofilter (m), C_0 the inlet concentration ($mg.m^{-3}$) and h0 the total height of the column (m). This graph represents the mean values obtained for 46 days of operation at steady state (287–333 days) and the error bars represent the corresponding standard deviations. During this period, pH of percolate waters was regulated to values closed to neutrality (pH = 6.9 ± 0.5). Complete bed height with a higher elimination between h = 0.3 and h = 0.7 m (60%) was required to attain the elimination capacity mentioned above. Measurement of pH along bed height highlighted that pH values of sawmill chips were more acid than percolate waters ones. On the last 70 cm of column height, pH values were closed to 6 while on the first 30 cm of column height, pH values were lower (around 4). It is probable that the acidification of the biofilm hindered the metabolism of the pollutant-degrading strains having colonizing the packing material on the first 30cm-column height. Our results are in accordance with those obtained by Van Langenhove *et al.* [17]. They studied the influence of pH on the DMS degradation activity of a bacterial suspension of *Hyphomicrobium* VS, widely isolated from different environments (sewage treatment plants, marine sediments, soil, and biofilters) and showed that maximum specific DMS degradation rate was reached between pH 6.0 and 7.0.

It could be suggested that spatialization of microbial communities would occur: one specialized for the oxidation of DMS to hydrogen sulphide colonized the column height between 30 and 70 cm whereas others
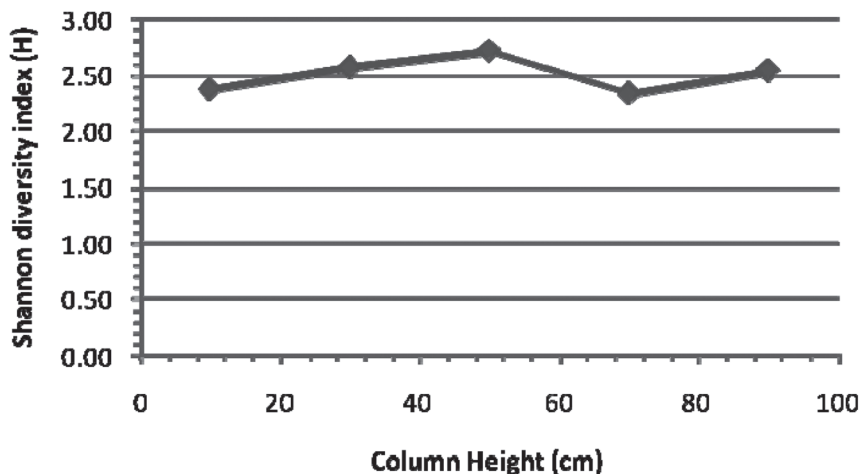
Figure 4. *Shannon Diversity Index (H) of the microbial communities along biofilter height of the DMS-degrading biofilter based on 16S rRNA analysis.*

more specialized for the biodegradation of hydrogen sulphide to sulfate colonized the section below involving acidification of this column part.

Microbiological Analysis

Methodological Validation

The reproducibility of the whole methodology-from the sampling step to the DGGE pattern analysis-was validated on biofiltration woodchips sample, for which the packing material properties were closed to sawmill chips ones (data not shown).

Diversity Along Biofilter Height

Microbiological analysis has been realized along biofilter height after maintaining steady-state for 46 days. Shannon Diversity Index was determined by DGGE pattern analysis. It remained similar, independently of bed height (Figure 4). This analysis does not seem to reveal any effect of biofilter height on biodiversity. Hence, the diversity was considered as constant (2.5 ± 0.2) in the different stages of the biofilter. This observation made for Shannon diversity index was observed for richness: on average, 15 ± 2 bands were counted on DGGE profiles of the biofilter, independently of bed height.

Microbial Stratification

The stratification of the total bacterial community has been investigated by using DGGE of the V3 region of the DNAr 16S. In the biofilter, the DGGE patterns obtained from sawmill chips sampled along the bed height are different from each other as the Bray-Curtis similarity values do not exceed 45% (Figure 5).

Moreover, the DMS degradation occurred between
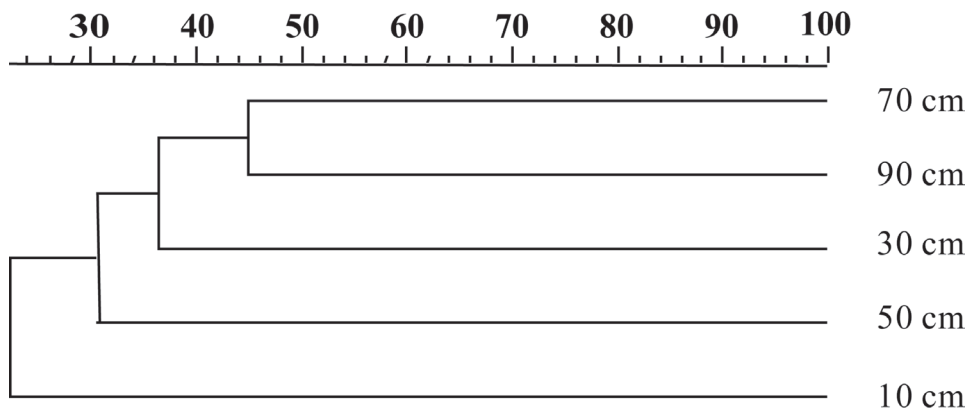
Figure 5. *UPGMA clustering based on Bray-Curtis similarities, according to biofilter depth (in cm from gas inlet); the x-axis represents the similarity.*

30 and 70 cm while the DGGE patterns obtained on this section are not similar. As this section has been supplied by a gaseous effluent containing DMS only, the longitudinal homogeneity of the total bacterial community could have been observed. Hence, in this study, it seems that the distribution of the biodegradation activity along the column is not correlated to the bacterial community structure. DMS degradation seems to not influence the microbial community structure. As it has been reported by different authors, in biofilters used for the treatment of a complex mixture of volatile compounds, the stratification of degradation activities in function of depth was observed [6–18] and this distribution of biodegradation activities correlates with the spatialization of microbial density and diversity [11–18]. Moreover, the homogeneity of the total bacterial community structure has been underlined in the column section within which a chemical family of compounds is removed.

In this study, the total bacterial community, composed not only of primary degraders but also of a lot of bacteria consuming other energy sources (packing material, saprophytes) has been investigated. This approach may be not accurate enough to detect changes within a fraction of the community. It is probable that the bacterial group degrading recalcitrant substrate such as DMS may be less diverse, and more distinct between different ecosystems.

To conclude, these results showed that the DMS removal biofilter developed here is an attractive process. It has been verified that the control of pH of packing material is of great importance to maintain DMS degradation removal. It is also necessary to gain insight into the diversity and the structure of functional microbial groups having colonized these biofilters to better control and monitor these complex ecosystems.

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Anaerobic Digestion of High Lipid Content Wastes: FOG Co-digestion and Milk Processing FAT Digestion

E.J. MARTÍNEZ, V. REDONDAS, J. FIERRO, X. GÓMEZ* and A. MORÁN

IRENA. Chemical Engineering Dept., University of Leon, Avda. de Portugal 41, 24071 Leon, Spain

ABSTRACT: The digestion of two different residues with high lipid content was investigated in the present research. The first part of the experimental work focussed on an assessment of the co-digestion of sewage sludge and fat, oil and grease (FOG) residues separated from the grease trap in the biological pre-treatments of wastewater treatment plants, as proposed in the present study. The second part of the experimental work studied the individual digestion of fat recovered from the grease trap of a milk processing plant. The digestion process was performed under batch and semi-continuous operation at mesophilic temperatures. Successful digestion of wastes was attained, with no inhibitory consequences due to the accumulation of long-chain fatty acids. An increase in biogas production was observed under batch digestion of sewage sludge when FOG was added as a co-substrate. However, the small increase reported was in accordance with the limited volume of this residue added. With regard to the digestion of the milk processing fat, although the addition of nutrients may be necessary for maintaining long-term operation, with the long hydraulic retention time tested no foam accumulation was observed and successful operation was achieved.

INTRODUCTION

THERE are numerous sources of high lipid content wastes, which include waste water treatment plants (WWTPs), animal slaughterhouses, and food processing installations [1]. Co-digestion using these kinds of wastes has become an area of interest to researchers, owing to the multiple advantages that the process offers. Co-digestion is the term used to describe the combined treatment of several wastes with complementary characteristics, this being one of the main advantages of anaerobic technology. [2]. Anaerobic digestion is a complex process which requires strict anaerobic conditions to transform organic matter into biogas, while also allowing the biochemical stabilization of sludge and reducing the amount of solids finally remaining for disposal [3]. Therefore, it is one of the most promising alternatives intended for the treatment of residues and for the production of energy by taking advantage of biogas. The process is considered an environmental friendly option [4] for the treatment of biowastes, since it not only allows stabilization of organic matter, reducing its potential to putrefy, but also contributes to the reduction of greenhouse gas emissions. Biogas

is currently seen as an important future contributor to European energy supplies [3]. The volume of biogas produced is related, among other things, to the content and quality of organic matter fed into the digester. In this way, increases in the concentration of solids in the incoming substrate may lead directly to greater biogas yields.

Lipid-rich waste which can be collected in the grease trap of wastewater treatment plants is also called fat, oil and grease (FOG) waste. This residue presents a great potential for increasing methane yield as noted by several studies [5–8]. Published reports record the benefits obtained from the co-digestion of this waste under mesophilic and thermophilic conditions can result in a doubling, or even higher increase, in methane production. Lipids are one of the major types of organic matter found in food wastes and some industrial wastewaters, such as those coming from slaughterhouses, dairy plants or fat refineries [9]. The addition of FOG, greases, or residues from slaughterhouses with high lipid contents has been evaluated in various different co-digestion processes [7, 10–12]. The addition of the co-substrate has been tested with substrates presenting volatile solid (VS) concentrations as high as 46% and successful results achieved, with no foam accumulation or inhibition due to long-chain fatty acids (LCFAs) [7–10]. The break-down of LCFAs takes place through

the β-oxidation pathway, which has been reported as the rate-limiting step of the whole anaerobic digestion process [13]. The accumulation of these components during the digestion process may cause inhibition, because of to their known toxicity affecting acetogens and methanogens [13–15]. However, recent studies have stated that inhibition caused by LCFAs could reversible, with acclimatization being a key factor in avoiding negative effects on microbial communities [8, 16–18]. Cuetos et al. [12] reported successful digestion of high lipid content wastes after a long acclimatization period, whilst recording inhibition problems and foam accumulation when the same residue was treated directly without first submitting micro-organisms to an adaptation period.

Another waste product of similar characteristics is the fat obtained from milk processing installations. The digestion of this type of waste has been studied previously [19, 20]. These studies report the hydrolysis phase of the process as the limiting factor. Under continuous operation, most reactors treating organic wastes with high loads are usually reported to work with long hydraulic retention times (HRTs). In this way, these systems may be particularly suitable for overcoming problems associated with the low hydrolysis rate of fatty milk wastes. With this in mind, the present study aimed to assess anaerobic digestion of FOG and fat obtained from a milk-processing plant. In the first part of the experimental work, the FOG obtained from the grease trap of the WWTP was co-digested with sewage sludge in volumes proportional to the production of these wastes in the plant. In the second experimental phase, the individual digestion of fat obtained from a milk-processing factory was evaluated.

MATERIAL AND METHODS

The sewage and digested sludge used was obtained from the WWTP of the city of Leon. Primary and secondary sludge (waste-activated sludge) (PS and SS) were used as the substrate for the experiments into digesting sewage sludge under batch and semi-continuous conditions. The grease used as co-substrate was obtained from the grease trap. FOG was added in a proportion of 0.2% V/V, on the basis of data from the WWTP regarding the production of wastes. Later experiments under semi-continuous operation were performed with higher volumetric proportions of the co-substrate (0.8% and 1.8% V/V). Digested sludge from the WWTP digester was used as inoculum. This digester treated a mixture of PS and waste-activated.

The temperature of the digestion process was 32°C and the average HRT was 26 days.

The fat used for the second phase of experiments was obtained from a local milk-processing factory. The digestion systems were inoculated with the same digested sludge used in the previous set of experiments. The digestion of this substrate was also evaluated under batch and semi-continuous conditions.

Batch Digestion

Batch experiments were performed to determine the biochemical gas potential of the substrates used in this study. Experiments were carried out until the cessation of gas production was observed. The batch reactors (Erlenmeyer flasks of 250 mL) were filled with inoculum and the corresponding amount of substrate in order to attain the desired proportion of VS between substrate and inoculum. Tap water was added to complete a 250 mL volume in all batch reactors. Two reactors were used for measuring gas production and composition. A batch reactor containing only inoculum was used as blank. The biogas produced by this reactor was subtracted from the corresponding tests. The temperature of digestion was 34°C, this being controlled by a water bath. Agitation was provided by means of magnetic stirrers. The gas volumes were measured using bottle gasometers and corrected to standard temperature and pressure (STP), 0ºC and 760 mmHg, respectively.

The PS used in the batch experiment presented a TS concentration of 37.6 g/L with 72% of VS. SS presented a TS content of 24.4 g/L with 75% of VS. The Erlenmeyer flasks were inoculated with 150 mL of digested sludge presenting a TS content of 20 g/L and a VS concentration of 12 g/L. Table 1 shows the characteristics of the grease collected at two different points in the grease trap (denoted FOG_1 and FOG_2). The proportion of VS between the inoculum and substrate

Table 1. Characteristics of the High Lipid Content Wastes Used in the Study for Batch and Semi-Continuous Digestion.

	FOG_1	FOG_2	GM1	GM2
Organic Matter (%)	79.8	83.7	92.5	85.5
Kjeldahl Nitrogen (%)	3.1	5.8	0.83	4.8
Grease (%)	17.6	63.5	58.5	10.8
COD (g/L)	149	92	240	117
TS (g/L)	133	55	246	109
VS (g/L)	107	48	232	91

Percentages are expressed on dry basis.

for this experiment was 1:1. Digestion systems were denoted, according to the substrate used, either PS or SS, followed by the label for the grease used, in the case of co-digestion systems.

The characteristics of the grease obtained from the milk processing factory (GM1) are also presented in Table 1. Batch digestion for this substrate was performed with several different proportions of VS between the inoculum and substrate, as follows: 0.4, 0.8, 1.0 and 1.5. The Erlenmeyer flasks were inoculated with 200 mL of digested sludge.

Semi-continuous Digestion

The digestion process was carried out in completely mixed reactors provided with mechanical stirrers. The reactor had a working volume of 3L. The systems were kept thermostatically at a temperature of 34°C ± 1°C. Reactors were provided with an outer jacket for circulating heating water that kept the system at a controlled temperature. Each reactor was provided with liquid and gas sampling ports. The reactors worked on a semi-continuous basis, being fed manually once every day. Before the reactor was fed, an equivalent volume was withdrawn. All the samples taken from the reactors were obtained after complete homogenization and previous feeding of the systems. Daily gas production was measured using a reversible device with liquid displacement and a wet-tip counter.

Reactors were inoculated with digested sludge. The PS used in this experiment presented a concentration of 33.5 g/L TS with 72% of VS. The SS sludge presented a TS concentration of 29 g/L with 78% of VS. In this second stage of experimentation, co-digestion with sewage sludge was evaluated using FOG_1 as co-substrate. The co-digestion of sewage sludge and FOG_1 was performed only with a mixture at 0.2% (V/V) of FOG (following the proportions tested in batch experiments). The HRT was set at 30 days. The sewage sludge in this case was composed of a mixture of PS and SS with a volumetric proportion of 30:70 based on the proportions of volumes of sewage sludge produced in the WWTP. A second reactor was used for treating a mixture with a higher volumetric proportion of FOG. The reactor was started up by applying an adaptation period where the feeding substrate had a proportion of FOG of 0.2%. This ratio was gradually increased to 0.8% and finally to 1.8%. This reactor was evaluated with an HRT of 30 days out of 120. Reactors were denoted in accordance with the substrate being digested in each case.

The digestion process for the fat from the milk-processing factory was performed using the same experimental apparatus and conditions as described above. In this phase of experimentation the fat obtained from the industry was denoted GM2. The reactor used was denoted R_GM2 and the HRT applied was 40 days.

Analytical Techniques

Nitrogen concentrations were determined by the Kjeldahl method. Organic matter was analysed in accordance with the Walkey-Black method (Walkey and Black, 1934). Grease content was determined by Soxhlet extraction using Velp Scientifica SER 148/3 in accordance with APHA Standard Methods [21]. COD, TS, VS, ammonium and pH were monitored during the digestion process. These parameters were determined in accordance with the APHA Standard Methods [21]. COD was determined using a Metrohm 862 Compact Titrosampler. The homogenized sample was digested in the presence of dichromate at 150°C for 2 h in a Hanna C9800 reactor. The composition of the biogas was analysed using a gas chromatograph (Varian CP3800 GC) equipped with a thermal conductivity detector. A packed column (HayeSep Q 80/100; 4 m) followed by a molecular sieve column (1m) was used to separate CH_4, CO_2, N_2, H_2 and O_2. The carrier gas was helium and the columns were operated at a pressure of 331kPa and a temperature of 50°C. Volatile fatty acids (VFAs) were determined on the same gas chromatograph, using a flame ionization detector (FID) equipped with a Nukol capillary column (30 m × 0.25 mm × 0.25 μm) from Supelco. The carrier gas was helium. Injector and detector temperatures were 220°C and 250°C, respectively. The oven temperature was set at 150°C for 3 min. and thereafter increased to 180°C. The detection limit for VFA analysis was 5.0 mg/L. The system was calibrated with a mixture of standard volatile acids from Supelco (for the analysis of fatty acids C2 to C7). Samples were previously centrifuged (10 min., 3500 × g) and the supernatant filtered through 0.45 μm cellulose filters. Gas chromatography was used for the analysis of the long chain fatty acids (LCFAs). Samples for LCFA analysis were extracted as described by Fernández et al. [22]. Samples were mixed with n-heptane, the solution was then centrifuged for 30 min. at 3500 × g and filtered through a 0.2 μm Millipore Millex-FGS filter. The sample was injected into a Perkin-Elmer AutoSystem XL chromatograph equipped with a FID detector and a PEG (100% Polyethylene Glycol) column (15 m × 0.53 mm × 0.5 μm). The carrier gas

was helium. The initial oven temperature of 100°C was maintained for 1 minute, and then increased to 250°C, with a ramp of 5°C per minute, this temperature being maintained for 5 min. Injector and detector temperatures were 250°C and 275°C, respectively. The system was calibrated using a mixture of LCFAs from individual acids with concentrations in the range of 0 to 100mg/L. The detection limit for LCFA analysis was 5.0mg/L. The acids analysed were C6:C24 (with even numbers of carbon atoms) all from Sigma.

RESULTS

Sewage Sludge and FOG digestion

The results obtained from batch digestion assays are presented in Figure 1 for PS and SS systems. Since the addition of co-substrate was limited to the volumetric proportions of production in the WWTP, the benefits of this addition were scarcely noticeable in the minor increase in cumulative methane production in the PS systems. The specific methane production obtained for the individual digestion of PS was 462 mL/g VS, whilst this value increased to 542 mL/g VS (average values for both co-digestion systems). The small addition of complex wastes resulted in a decrease in the biogas production rate, as may be observed from the sigmoid behaviour of the cumulative methane graph. With regard to the SS system, no improvement was observed, all systems presenting an average specific methane production of 358 mL/g VS. Contrary to what was the case for the previous system evaluated, the addition of high lipid content waste did not affect in any significant way the methane production of the co-

digestion system. Although an increase in the total volume of biogas produced was obtained in the first case, modification of the rate of biogas production was also observed. This delay may be rationalized as an adaptation of micro-organisms to the presence of the complex substrate, resulting in sigmoid curves of cumulative methane production.

Under semi-continuous operation FOG_1 was selected for the assessment of the digestion process, because of to its higher solid contents. Additionally, the residue denominated FOG_2 presented particles of greater size which might render normal operation difficult. Daily biogas production is presented in Figure 2(a). As may be observed, the gas evolution was practically constant with similar values for both reactors evaluated. The addition of the co-substrate in the proportions applied did not translate into an increase in the biogas rate. As it might be expected from batch experiments, the low addition of volatile solids did not represent any significant increase in the organic load supplied to the co-digestion reactor. VS measured during experimentation showed stable behaviour with no significant modifications.

The effect of increasing the volumetric proportions in the mixture may be seen in Figure 2(b). The gradual increase in FOG addition resulted in poor performance from the reactor. Table 2 shows the values obtained for the parameters monitored during digestion. The specific methane production calculated for this second reactor treating the mixture of FOG at 1.8% was lower than that obtained for the previous reactor. This value was calculated for the period between 75 and 120 days of operation. Owing to the small amounts of the co-substrate added to the first system (0.2% FOG) no ob-
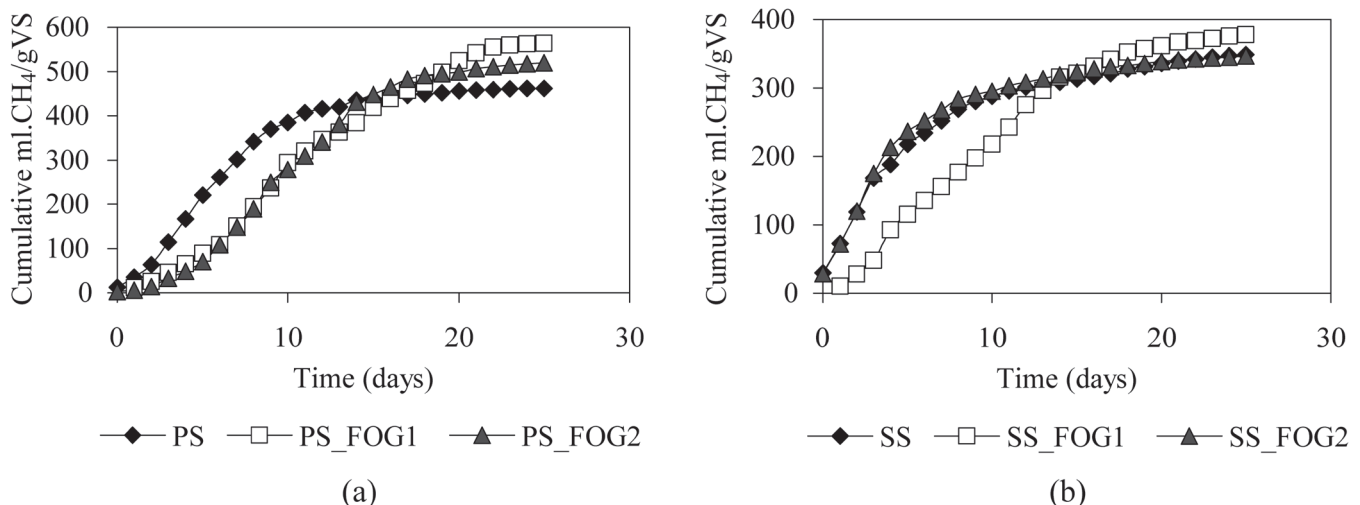

(a)   (b)

Figure 1. *Gas Production from batch digestion test of Sewage Sludge and FOG for (a) PS Systems and (b) SS Systems.*
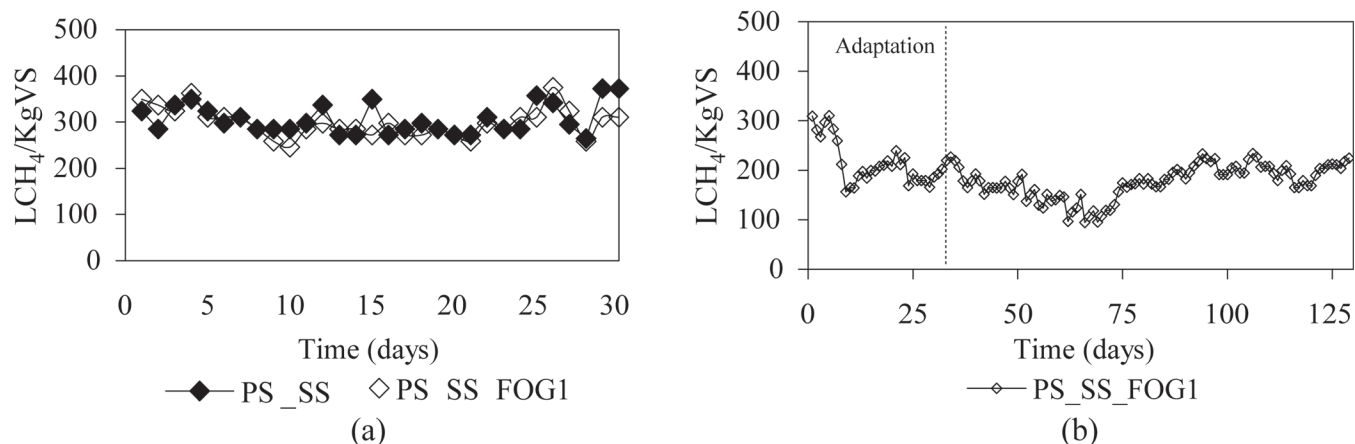
Figure 2. *(a) Specific methane production of reactor treating the mixture of primary sludge and secondary sludge (PS_SS) and the co-digestion mixture (PS_SS_FOG1) at 0.2 % (b) Daily gas production of reactor treating the mixture at 1.8%.*

servable effects were measured. Results obtained for this reactor presented low values for VFA concentrations. Additionally, the LCFAs detected showed low concentrations of octanoic (C8), decanoic (C10), and myristic (C14) acids, with values below 50 mg/L. Increasing the FOG content of the mixture did not result in higher VFA values. This may be rationalized by the mechanisms of inhibition of LCFAs. An accumulation of LCFAs may inhibit anaerobic digestion because of direct toxicity to acetogens and methanogens, the two main groups involved in LCFA breakdown [13]. Another inhibiting mechanism is the result of the adsorption of surface active acids onto the cell wall [23], thus affecting the processes of transportation and protection. Figure 3 shows the LCFA concentrations measured in the reactor treating the FOG mixture at 1.8%. Values obtained here were lower than those reported in the literature as causing inhibition [24]. Thus, the lower gas yield of the reactor was probably due to adsorption of the FOG components onto cell walls.

The addition of fat residues to digesters has been recommended by several authors who have evaluated the co-digestion of greases by applying either continuous supplementation or pulsed addition of waste [11, 25–27]. The addition of high lipid content wastes seems a plausible option for increasing biogas production in already existing digestion systems, as has been demonstrated by the practical implantation of this option in WWTPs [28]. However, in the present study an increase in the FOG concentration resulted in inhibition of the digestion process, highlighting the relevance of testing modifications under pilot scale conditions prior to undertaking operational changes in industrial plants. Another relevant aspect deals with operating considerations which should also be carefully evaluated, to

avoid clogging the process piping when delivering this co-substrate [29].

Milk Processing Waste Digestion

Figure 4 shows the results for the gas production obtained under batch digestion of milk-processing waste. The increase in substrate concentration results in an inhibitory effect, as may be observed from the lower production of gas obtained as the Inoculum to Substrate ratio (I/S) increases. Inhibition associated with the concentration of LCFAs has been reported under continuous operation and digestion assays [30–32]. From the results obtained here, it may be rationalized that increasing amounts of the substrate resulted in higher concentrations of LCFAs, which in turn decreased the biogas yield.

Sage et al. [20] studied the degradation of milk fat, reporting a lag phase of several days prior to degradation of fat by anaerobic micro-organisms, with this lag phase before biogas production being mainly due to unsaturated free fatty acids (FFA).Conversion to
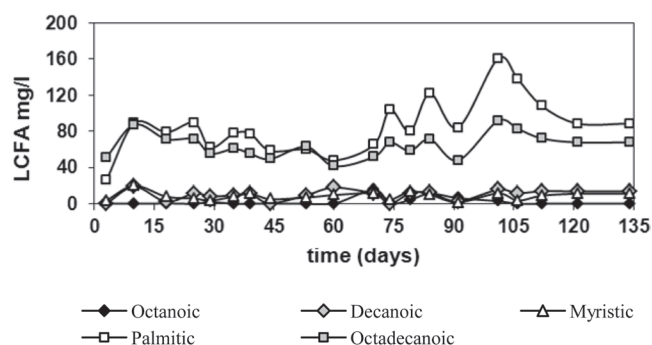

Figure 3. *LCFA concentration measured from the reactor treating co-digestion mixture (PS_SS_FOG1) with increasing proportion of FOG.*

Table 2. Parameters of Reactors for Semi-continuous Digestion.

Parameters	PS_SS	PS_SS_FOG1 0.2%	PS_SS_FOG1 1.8%	R_GM2
Organic loading rate (g VS/m^3/day)	771	776	790	652
Specific CH$_4$ production mL/g VS/day	304	298	200	440
Average daily gas production (L/day)	1.08	1.06	0.70	1.9
% CH$_4$ in biogas	65	65	67	63
VS (g/L)	14.4	12.6	12.9	9.0
TS (g/L)	21.8	19.4	18.5	14.4
COD (g/L)	13.8	12.2	12.5	13.8
T NH$_4^+$ (mg/L)	1103	1114	824	509.2
VFA concentration: Acetic (mg/L)	126	84		135
Propionic (mg/L)	13	—	—	23
Butyric (mg/L)	—	—	—	5

biogas occurred at a lower rate for saturated than for unsaturated FFA.

Taking into consideration the low methane yield obtained with the increase in substrate concentration, semi-continuous digestion was evaluated applying a low organic loading rate. Figure 5(a) shows the daily biogas production of the reactor working under semi-continuous operation (R_GM2). The reactor was daily fed with an organic loading rate of 0.65g VS/L/day with an HRT of 40 days. Under these conditions, steady production of gas was observed, presenting an average methane content of 63%. Data relating to the performance of this reactor are also presented in Table 2. The specific methane production obtained was higher than the value reported for the sludge digestion systems, corroborating the high methane potential of this waste. Although inhibition was probably the cause of the limited production of methane under batch digestion in some of the experiments carried out, this situation may be circumvented under semi-continuous operation by the application of a low organic loading rate

to the reactor. In this way, the concentration of LCFAs was low during the period of experimentation, with the main acids detected being palmitic, stearic and arachidic, their average concentrations being 89, 80 and 50 mg/L respectively. Although steady gas production was attained, one of the main problems when considering the digestion of food processing wastes is the concentration of nutrients needed to maintain stable operation of biological treatment processes during long-term performance. In the present study, the concentration of ammonium in the reactor was initially that of the inoculum used for starting-up the digestion process. However, as the time of experimentation grew, a significant reduction was observed, as may be seen in Figure 5(b).

CONCLUSIONS

Digestion of high lipid content waste was successfully attained for both substrates evaluated. The studies undertaken using sewage sludge as the main substrate resulted in no observable modifications to specific methane production with a FOG content of 0.2% (V/V). However, an increase in the addition of FOG to 1.8% resulted in significant detriment to the performance of the process. On the other hand, when digesting fat obtained from a milk-processing factory, the results showed successful operation of the semi-continuous reactor operating with an HRT of 40 days, although at high inhibition was reported from batch tests performed.
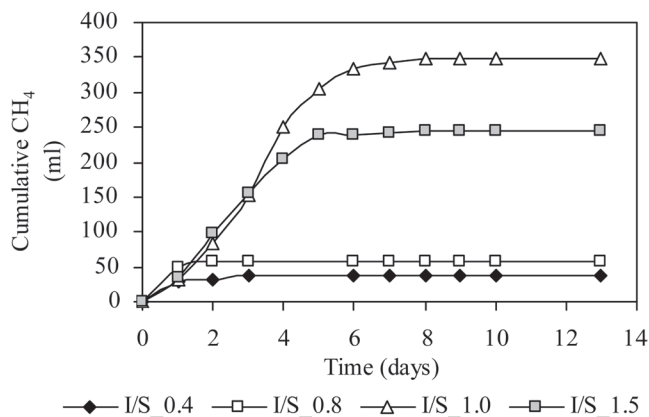
Figure 4. Cumulative methane production of batch reactors treating the milk processing waste at different ratios of VS of inoculum/substrate (I/S).
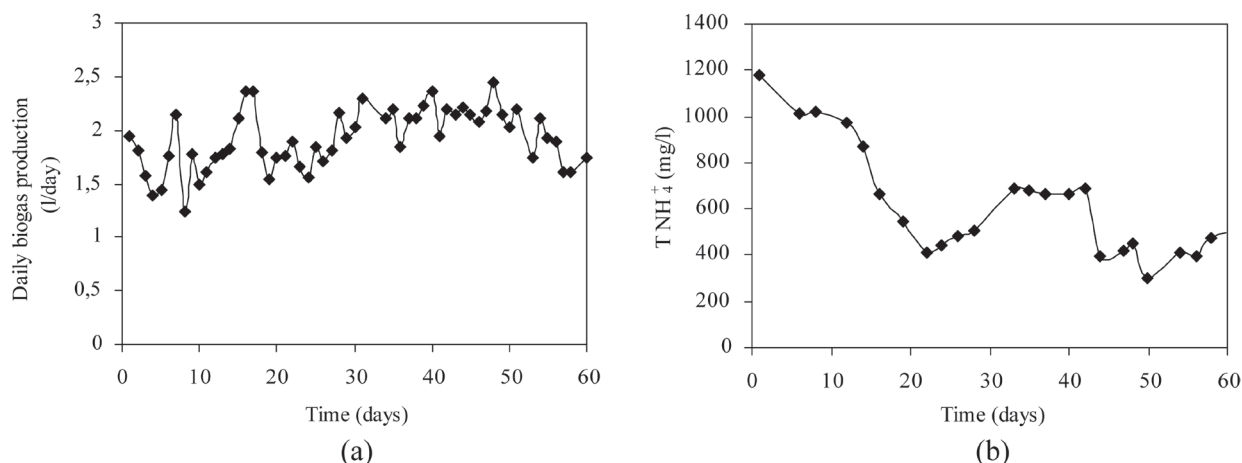
Figure 5. *(a) Daily gas production of reactor treating the waste obtained from the processing milk factory (R_GM2), (b) Total ammonium concentration inside the reactor.*

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The Distribution of Heavy Metals Following Sewage Sludge Gasification

H. SAVEYN[1,2,4], J.H. FERRASSE[2,*], A. B. HERNANDEZ[2], J. ROSE[3], P. VAN DER MEEREN[1] and NICOLAS ROCHE[2]

[1]*Particle and Interfacial Technology Group, Ghent University, Belgium*
[2]*Laboratoire de Mécanique, Modélisation et Procédés Propres, Aix-Marseille University, ECCOREV, CNRS, UMR 6181, Aix-en-Provence, France*
[3]*CEREGE, Aix-Marseille University, ECCOREV, CNRS, UMR 6635, Aix-en-Provence, France*
[4]*European Commission, Institute for Prospective Technological Studies, Seville, Spain*

ABSTRACT: Limited knowledge exists about the fate of inorganic components, and especially metallic species, present in sewage sludge, upon gasification. This study shows that major elements are mainly retained within the sludge char, whereas minor elements such as Cd, Pb, Zn, As and Hg are partially or completely lost due to volatilization or melting from the sludge matrix. Hence, this work stresses the importance of monitoring the metal distribution during the gasification process.

INTRODUCTION

SLUDGE disposal is becoming a problematic theme in many industrialised countries due to more stringent regulations and concerns for pollutant spreading to the ecosystem. Yet, sewage sludge, characterized by a relatively high energy content comparable to that of brown coal (17.5 MJ/kg dry matter; [1]), has a good potential to become a renewable energy resource. Biochemical conversion techniques like anaerobic digestion are already very common, but are not able to tackle recalcitrant biomass such as lignins or toxic persistent organic pollutants (POPs). Direct combustion is the most straightforward and best known technology, but has some disadvantages. As such, the volume of exhaust gases to treat is much higher and the energy conversion efficiency that can be obtained is lower [2]. A promising alternative process is gasification, which has been in use for the transformation of wood or coal to energy gases and is getting increased attention for other raw materials [3, 4, 5, 6]. Most of the existing research work on gasification of biomass focuses on improving product yields and energy efficiency of the process. Yet there is limited knowledge about the fate of inorganic components, and especially metallic species, present in the feed stock that is being gasified. Sewage sludge is known to be under scrutiny for its high content of heavy metals, such as Zn, Pb, Cu, Cr, Ni, Cd, and Hg, which are present in concentrations from less than 1 part per million (ppm) to more than 1000 ppm [7]. The presence of such high levels of metallic species may entail a series of technological issues (e.g. catalyst poisoning) and ecotoxicological problems (e.g. pollution of condensate) downstream of the gasification reactor. Therefore, this work aims at investigating the fate of metallic species during sewage sludge gasification.

MATERIALS AND METHODS

Dewatered sewage sludge (17% dry matter) was sampled from a municipal wastewater treatment plant of 200 000 pe in the south of France and subsequently frozen (–18°C) in 50 g samples. Before each measurement, a sample was thawed prior to gasification in a bench scale gasifier, consisting of a semi-batch updraft reactor, supplied by superheated steam pushed by Argon as carrier gas operating close to atmospheric pressure. For this study, all the presented experiments have been realised with two different gasification atmospheres: the first one containing more than 99% of water vapour on volume basis and complemented by Argon and the second one with an air/steam mixture in the ratio of 1 mole per mole.

The samples were inserted in the reactor on a porous metal foam grid (RCMPL-NC-4753.016, Recemat, The Netherlands) and the same material was used as a

particulate gas filter [8].The sample was then heated to the desired temperature and exposed to this temperature for a set time. Downstream of the gasification reactor was a condenser unit for the recovery of steam and condensable gas products. The system is schematically depicted in Figure 1. On-line gas measurements were complemented by off-line (metal) analyses on the original sludge material, the char residue, deposits on the metal foam filters and the condensate. Metal foam filters were subject to 1h extraction by a 5% HNO_3 solution. Metal analyses were performed by inductively coupled plasma atomic emission spectroscopy (ICP-AES, Horiba Ultima-C 2000), after either acid digestion with HNO_3 and H_2O_2 or alkaline hydrolysis with $LiBO_2$. The ICP-AES instrument was calibrated with standard solutions prior to all measurements and after every 15 samples. Furthermore, externally purchased reference materials were used for testing the accuracy of the instrument. Cotton filters were prepared for metal analysis by a method based on the study by Rezic et al. [9]: after adding 20 ml of a 5% HNO_3 solution to a cotton sample of about 0.5g, cotton samples were subjected to sonication (2×15 minutes) in a sonicator bath and subsequent 16h reaction. Measurement of As and

Hg was done by in situ hydride formation by HCl and $NaBH_4$ in order to improve measurement sensitivity.

RESULTS AND CONCLUSIONS

Validation of the Measurement Methodology

In a first instance, the measurement methodology applied in this study was validated by comparing the measurement results of the sampled sludge with results from an independent certified laboratory, hired by the treatment plant.

This was done for both a series of major and minor elements and the results are depicted in Figure 2 and Figure 3.

According to Richaud et al. [10] the estimated uncertainty of trace metal analysis by ICP is about 20 %. Taking into account this error margin, it is clear that the own measurements do not differ from the independent laboratory, except for Hg. It must be stated though that the later element was analysed by standard injection in the ICP plasma and that the results were below the limit of quantification. When measuring Hg by the method of in situ hydride formation, a value of 0.69 ppm was
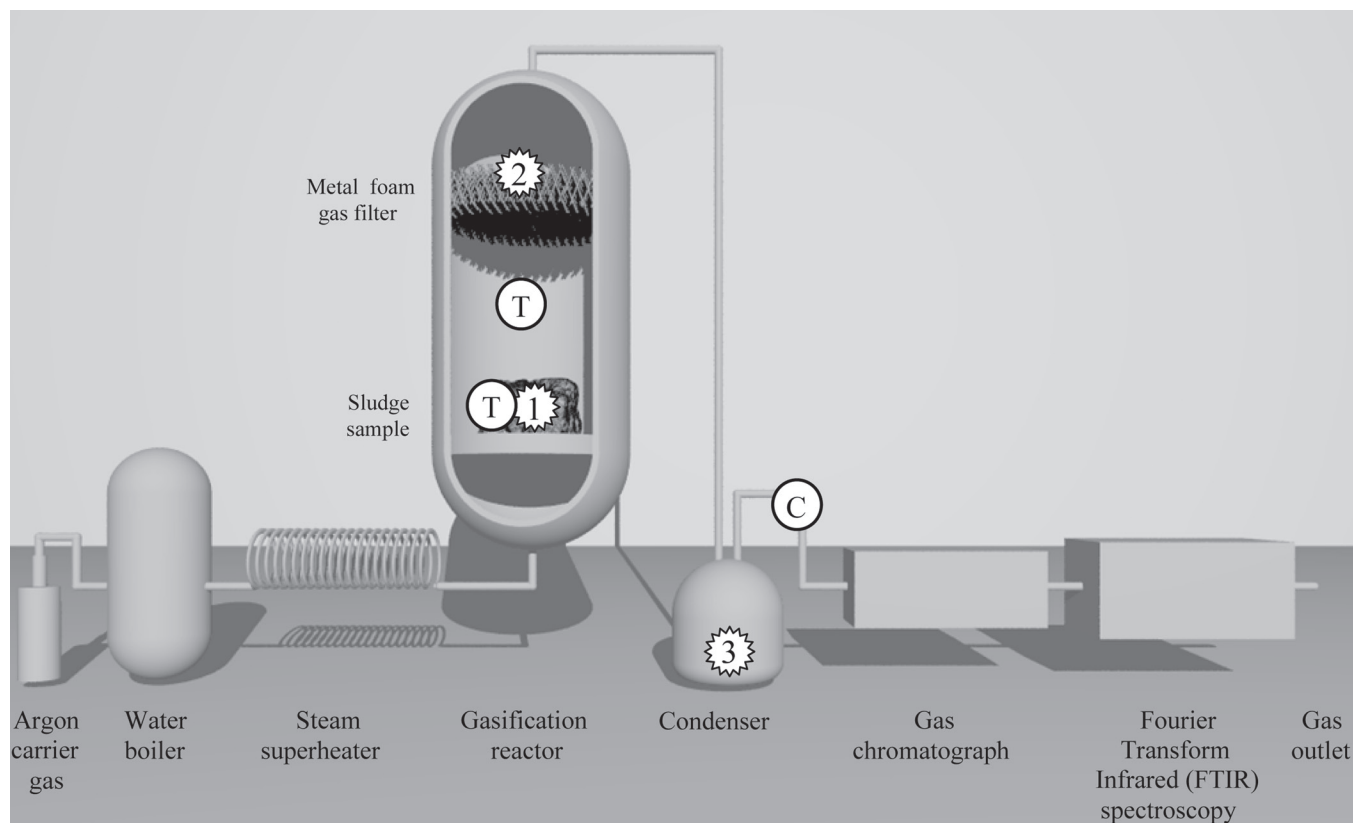
Figure 1. *Schematic representation of bench scale gasification set-up. The numbered stars indicate sampling points for off-line analyses (1 sludge char residue; 2 = metal foam filter deposits; 3 = condensate). The symbol T represents a temperature sensor position, symbol C represents the position of the cotton filter operating at ambient temperature.*
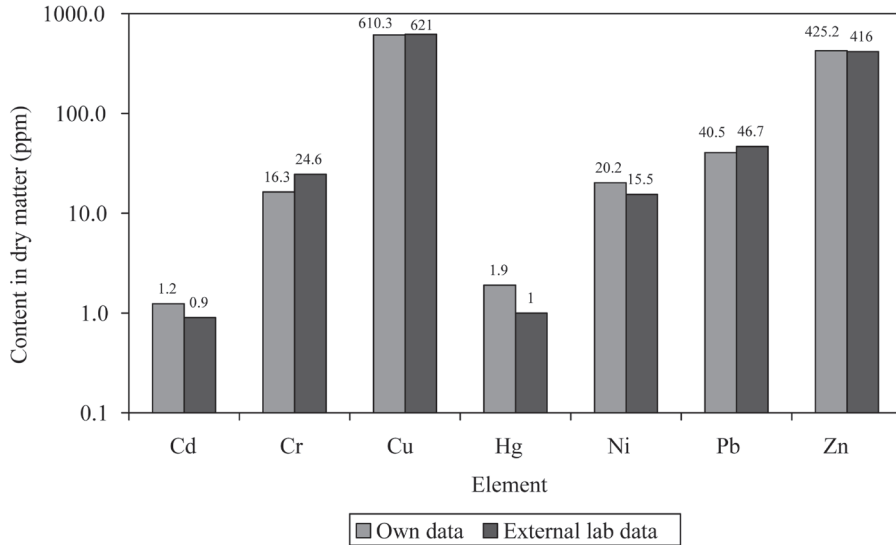
Figure 2. *Comparison of own measurement data with external lab data on the sludge sample collected for this study.*

obtained, which is in agreement with the 1.0 ppm value of the independent laboratory, taking into account the 20% estimated uncertainty.

Evolution of Metal Content

Preliminary Study: Effect of Temperature on Metal Loss from Sludge Sample

Figure 4 shows the results of the metal analyses performed on the char residues obtained following gasification of a series of sludge samples at different temperatures (105, 350, 650 and 850°C). The measurement data have been represented as function of the metal mass quantities initially present in the sludge sample,

for a series of minor elements. As such, a value of 1 represents 100% retention of a metal in the sludge char, lower values represent metal loss and values higher than one represent metal enrichment. The initial concentrations (expressed in mg/kg dry sludge mass) of the different metals Cd, Cr, Cu, Hg, Ni, Pb and Zn were 1.18, 21.25, 466.42, 2.79, 17.78, 54.03 and 476.97, respectively.

The data clearly show that an element such as Cu exhibits a relative stable behvaiour, meaning it does not volatilize or melt and hence is retained within the char. Other metals, such as Hg and Cd are rapidly lost from the sludge char during the gasification process. The point at which the major losses occur is clearly related to the boiling point of the different metals, be-
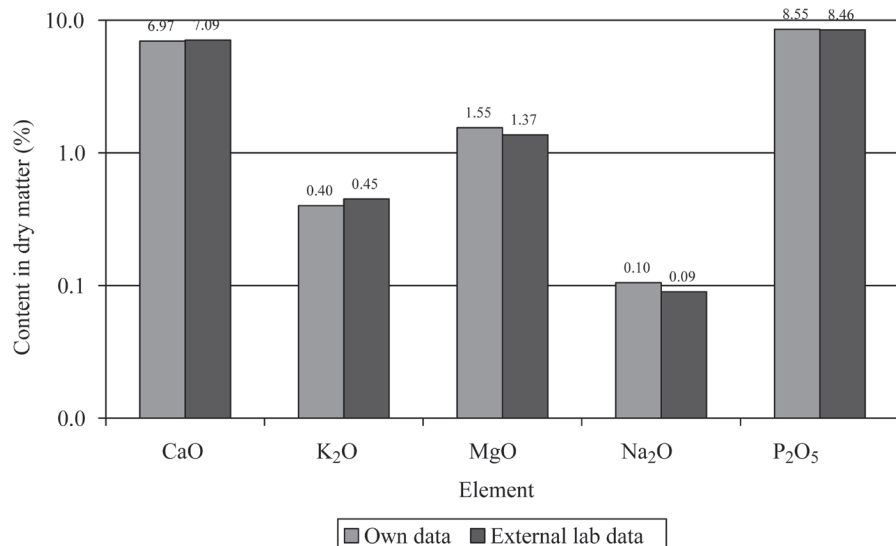
Figure 3. *Comparison of own measurement data with external lab data on the sludge sample collected for this study.*
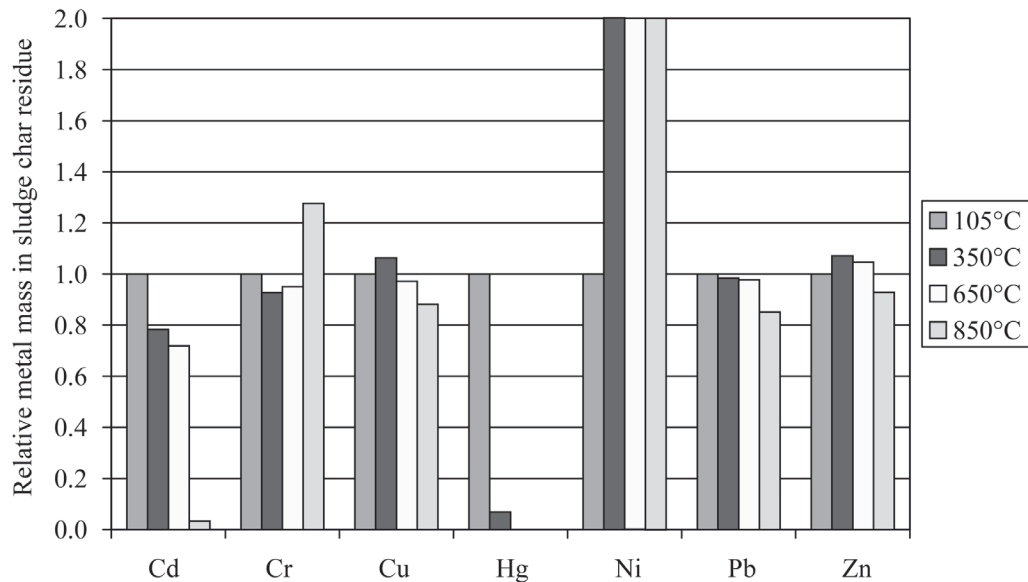
Figure 4. *Relative metal content (based on initial dry sludge mass) in sludge char residue resulting from gasification during 1 hour at different temperatures and using an atmosphere of 99% water vapour on volume basis and complemented by Argon.*

ing 357°C for Hg and 765°C for Cd [11]. These findings are similar to those of Bool and Helble [12] and Helble et al. [13] who worked on gasification of coal. Two metallic species, Cr and Ni show a sharp relative increase in concentration. It is believed that this was due to pollution of the sludge sample by the metallic foams used as sample supports. As such, for Ni, the contamination of the sample led to a 12-fold increase in relative concentration at 350°C. The char contents of Pb and Zn showed a tendency to decline at higher temperatures, although this was not fully clear. Therefore, a more extensive gasification study was performed in the second part of this work.

Advanced Study: Fractionation of Elements

In this part of the study, gasification was performed at conventional gasification temperatures (between 700 and 900°C) under different conditions of steam addition, residence time and air supply, in an experiment consisting of 20 tests.

Major Elements

Analysis of the chars after gasification revealed that major elements such as Al, Ca, Fe, K, Mg, Na, P, Si and Ti were nearly all retrieved in the char (Figure 5). The data shown in Figure 5 represent average values from twenty experiments. Error bars represent the standard deviation on the whole experiments. No clear tendencies due to temperature or atmosphere were observed.

Two elements show a somewhat different recovery, namely Ca and Na. Ca has a tendency to lower recovery ratios, most probably due to leaching inside the gasification reactor. It was noticed that the char in the gasification reactor was still wet at the end of the gasification reaction and subsequent cooling, most probably from condensation of the steam vapour and moisture released from the sludge sample. As for the Na element, overestimations are thought to be linked to the reliability of the ICP measurement, given the large standard deviation encountered for this measurement.

Minor Elements

As for the minor elements, the recovery rates were clearly different (Figure 6). Chromium and nickel were often retrieved at ratios of more than 100% with very high variations, most probably due to contamination from the metal support foam, which consisted of a nickel-chromium alloy. Copper was recovered at nearly 100%, whereas lead and zinc showed recovery values of 66.2 ± 20.9 % and 71.7 ± 10.6 % (average ± standard deviation), respectively. These recovery ratios can clearly be linked to the melting and boiling points of these elements. Melting temperatures are 1084.62, 327.46 and 419.53°C for Cu, Pb and Zn respectively, whereas the respective boiling temperatures are 2562, 1749 and 907°C [11]. The loss of Pb and Zn was proven by analysis of the downstream fractions of these elements. As such, the support foam that held the sample,

Figure 5. *Recovery of major elements in the char upon gasification. The error bars represent standard deviations for all temperatures, residence times and atmospheres from the experimental design.*

contained on average 1.7% of the Pb and 1.7% of Zn from the sludge sample with individual measurements up to 10.9% and 11.9%, respectively. The foams used to retain gas particles leaving the reactor contained on average 2.9% of the Pb and 1.2% of Zn from the sludge sample with individual measurements up to 12.3% and 6.5%, respectively. Finally, the condensate downstream of the gasification reactor contained on average 1.3% of the Pb and 0.4% of Zn from the sludge sample with individual measurements up to 5.5% and 1.9%, respectively. This clearly indicates that metals can be released from the sludge sample and be recovered at different stages of the reactor system. It should also be

mentioned that closure of the Pb and Zn balance was not obtained, with 10 to 50% of the elements missing. Although this may be partially due to measurement uncertainties, it is believed that some of the Pb or Zn could have been collected elsewhere, such as on the reactor walls. For the sake of completeness, it must be stated that Cd analyses demonstrated that Cd levels in the sludge chars were above detection limits, but below quantification limits, indicating a partial but not a full loss of Cd from the original sludge sample. For most of the other fractions, such as condensate or metal foam extracts, Cd values were below detection or quantification limits.
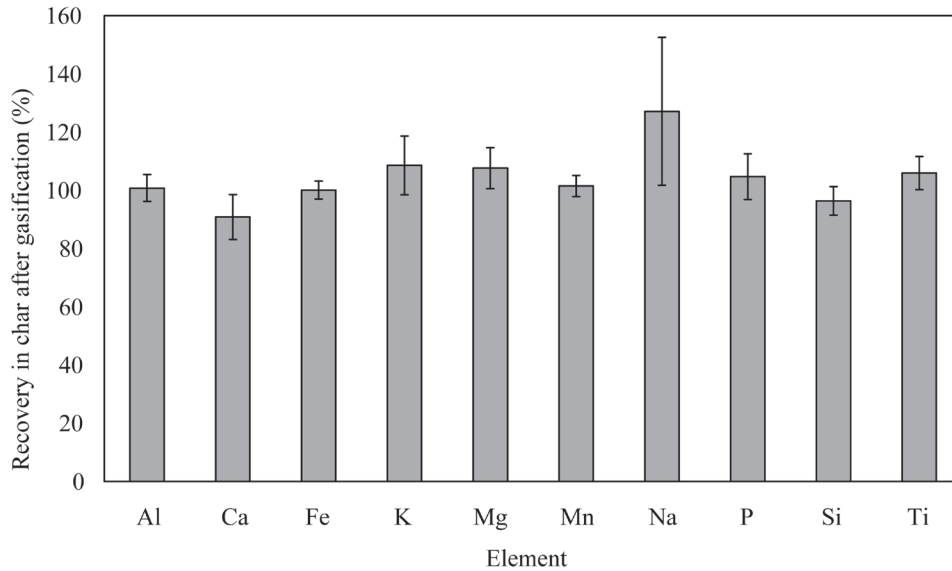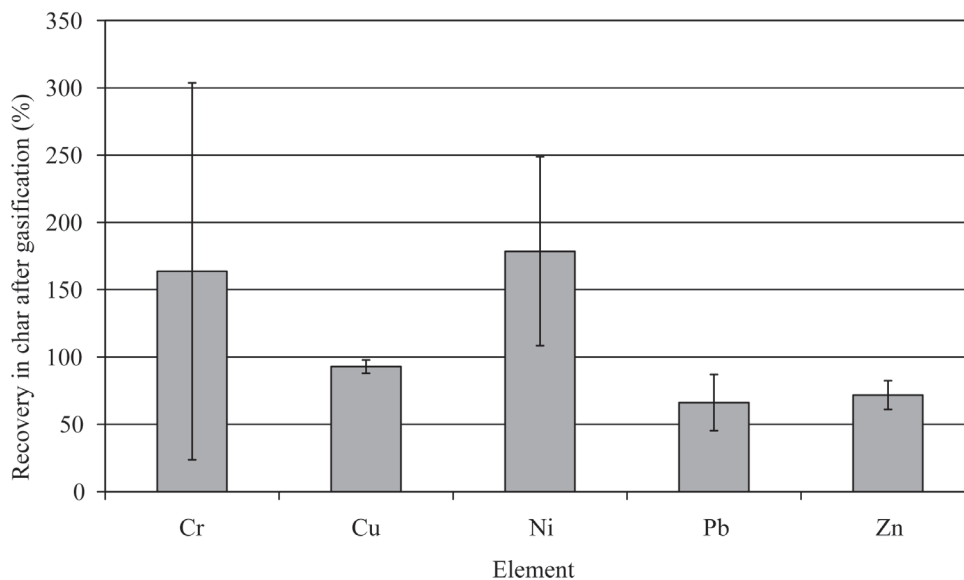

Figure 6. *Recovery of major elements in the char upon gasification. The error bars represent standard deviations for all temperatures, residence times and atmospheres from the experimental design.*

The use of air during gasification appeared to have a positive influence on the Pb and Zn recovery in the char. As such, Pb and Zn contents in the char were 63.3% and 67.7%, respectively in a test with air injection, whereas they were only 34.1% and 55.9% under the same test conditions but for the absence of air in the reactor. This is most probably due to the formation of oxides, by reaction of the oxygen present in the air, which stabilize the elements in the char.

It appeared that both residence time and temperature had an effect on the metal recovery within the char, whereby the temperature effect seemed predominant. Two tests were performed at 0 minutes residence time, i.e. a heating stage immediately followed by cooling, at either 800 or 900°C. In this case, the recovery for Pb and Zn in the char was 81.7% and 80.0%, respectively, at 800°C, whereas it dropped to 46.0% and 61.0% at 900°C. When keeping the temperature at 900°C but extending the residence time from 0 to 270 minutes, the recovery of Pb and Zn slightly decreased further to 34.1 and 55.9, respectively.

Finally, the influence of steam supply on the metal content could not clearly be established.

Mercury and Arsenic

Hg and As are known for their high volatilities. Elemental mercury is already in the liquid state at room temperature and has a boiling temperature of 356.73°C. Arsenic undergoes sublimation at 614°C and has a triple point at 817°C [11].

Char samples were analysed from gasification experiments between 700 and 900°C. The char samples contained between 41.0% and 60.6% of the original As present in the sludge, but none of the initial Hg. Also here, large metal amounts were retrieved downstream of the gasifier. The condensate contained on average 16.0% and 15.4% of the initial As and Hg, respectively, whereas the gas filter foams contained 3.6% As and 2.0% Hg, respectively. The metal balance remained very incomplete, especially in the case of Hg, where no more than 40% of the initial mercury was recovered in the different fractions. It is supposed that due to the high volatility of this element, a large fraction ends up in the product gas, despite filtering and condensation of the gas. This hypothesis is supported by analysis of a cotton filter, placed after the metal foam filter and condenser, before the gas chromatograph. It was found that, on average, 0.3% of the initial As and 2.6% of Hg was deposited on this cotton filter. With As and Hg being retrieved so far downstream of the reactor, it is clearly feasible that a major fraction escapes with the product gas.

CONCLUSIONS

The results from this study clearly demonstrate the possible risk of both metal loss from sludge and metal contamination towards sludge during gasification and hence the importance of monitoring the metal distribution during the gasification process and selection of the reactor materials. It appeared that especially Cd, Hg, As as well as Pb and Zn are susceptible to be released from the sludge sample, ending up in the gas cleaning sections downstream of the reactor or even escaping through the product gas.

DISCLAIMER

The views expressed are purely those of the authors and may not in any circumstances be regarded as stating an official position of the European Commission.

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Characterization and Validation of Liquid Fuels for WWTP Sludge Pyrolysis

A. LOZANO[1,*], A.FULLANA[1] and V. GARCÍA[2]

[1]*Chemical Engineering Department, University of Alicante, Spain*
[2]*Environmental department of Isolux Corsan Group*

ABSTRACT: The present work evaluated the production of liquid fuels by slow pyrolysis of sludges from sewage treatment plants. The pyrolysis process was carried out at 550ºC in a batch reactor at pilot plant scale. In these conditions the pyrolysis liquid yields varied between 10 and 30% by weight. The heating value of liquid oils ranged between 28 and 32 MJ/kg. The content of metals and sulfur in the resulting oils was also analyzed and results showed that these compounds were present in bio-oil at low yields. Results show the pyrolyzed liquids can be used as fuel in energy applications.

INTRODUCTION

SUPPLY and reuse or recycling of quality water are becoming one of the greatest challenges of the 21st century. Innovative technology and services are needed for monitoring, managing, treating and restoring both consumption water and wastewater. However, the reuse of wastewater generates an additional residue which presents environmental problems and requires proper management and treatment [1, 2].

For example, in Spain the production of that waste has been growing steadily in recent years. In 2007 1.2 million tones of dry sludge were produced, according to the Ministry of Agriculture and Environment (MARM) [3]. The increased production of sludge from wastewater treatment plants (WWTP) is raising problems of proper management, treatment and, above all, of disposal.

In Spain 66% of the total volume of sludge goes to agricultural use. The remaining sludge is disposed in landfills or incinerated. However, each of the above solutions involves problems which often hinder their use in certain applications.

One of the main disadvantages entailed by using sewage sludge as fertilizers is its high metal content.

The use of treated sludge on agricultural land is only allowed when the concentration of heavy metals in the sludge and in the receiving soil does not exceed certain limits and providing that the accumulation of metals in the receiving plots is being controlled [4-5].

Energy recovery from sludge coming from wastewater treatment plants may be a good alternative since energy is recovered at the same time as the sludge is treated. Currently, the energy recovery method most commonly used is incineration. However, direct burning of sludge has major drawbacks such as the generation of gaseous emissions and the release of metals from the sludge [4, 6].

An alternative to incineration, which avoids some of its shortcomings, is pyrolysis. Sludge pyrolysis produces non-condensable gases (NCG) (CO, CO_2, H_2, CH_4, C_2H_4, C_2H_2, C_2H_6, etc . . .), a carbonaceous solid called char and a black, viscous liquid known as bio-oil. The non-condensable gases produced during pyrolysis can be used directly as energy source or they can be used to provide the heat necessary to carry out the pyrolysis process. The char also contains a high percentage of nitrogen which can be used in fertilizers or as an adsorbent for use in soils [7]. Finally the bio-oil is a value-added product resulting from the pyrolysis process that can be used as fuel or as raw material for the synthesis of organic compounds [4].

The aims of the present work were to obtain a liquid fuel by slow pyrolysis of sludge from wastewater treatment plants and evaluate the suitability of this fuel for energy applications. The effects of the sewage sludge

*Author to whom correspondence should be addressed.
E-mail: agustin.lozano@ua.es

digestion degree on the obtained results were also studied.

EXPERIMENTAL

Experimental Procedure

Figure 1 presents a schematic of the experimental setup in which the pyrolysis of the sludge in discontinuous mode took place.

The reactor is a hollow cylinder where the sludge load to be pyrolized is introduced. It has a pipe where nitrogen is introduced through, generating an inert atmosphere and contributing to drag the vapors and gases formed during the pyrolysis process. The energy required is provided by an electric furnace, with an opening at the top through which the reactor is introduced. The heat transfer from the furnace to the reactor occurs mainly by radiation. The volatile compounds and gases generated during the process flow through the sludge and are evacuated to a fluid collector through a pipe. From the collector the gas rises up to a condenser where the vapors are condensed with solid CO_2 and the resulting liquid falls by gravity to the liquid collector. Non-condensable gases go out through a chimney to the exterior after passing through a butane burner. The condensed liquids are evacuated through a valve at the bottom of the collector and the bio-oil and the aqueous phase are separated by gravity.
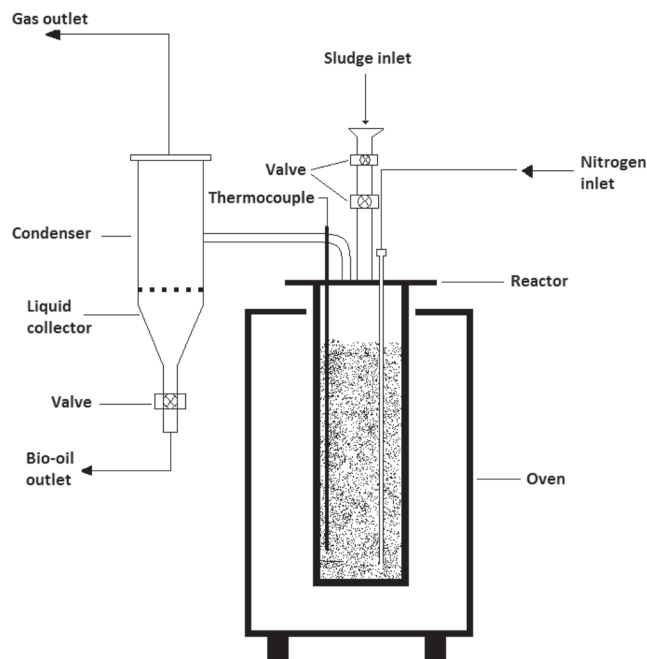
Figure 1. *General view of the sewage sludge pyrolytic reactor.*

The experimental setup has thermocouples that control the temperature of the oven, the output gas (after burner) and inside the reactor, and a flow meter that regulates the amount of inert gas which is introduced into the reactor.

All experiments were carried out by slow pyrolylsis, typically used in the production of charcoal. In this process, the raw material is heated up to 500–600ºC, and the residence time of the gases and volatiles is several minutes [8].

Pyrolysis experiments were carried out in an inert atmosphere of nitrogen with a flow rate of 5 l/min measured at 25ºC and 1 atm. The flow of nitrogen was introduced at the bottom of the reactor through a pipe. Previously to each experiment, the sludge was ground in order to homogenize particle size. The dried and ground sludge was loaded into the reactor and placed in the oven. A set up temperature of 550ºC was programmed and the N_2 began to pass through the reactor. The sludge was heated from room temperature to 550ºC at an average speed of 10°C/min. Figure 2 shows the variation of oven and reactor temperatures versus time for the experiment with sludge L_1.

The resulting pyrolyzed liquid underwent vacuum filtration at a pore size of 13 μm. The phases obtained had different densities, so they could be separated by decantation.

Following each experiment, the condenser, collector and pipes where the vapors flowed through were cleaned with acetone. The liquid resulting from this cleaning was filtered and underwent vacuum distillation in order to remove acetone. The obtained bio-oil was blended with the organic phase resulting after the decantation process.

Characterization of Sludges and Products Obtained by Pyrolysis

To carry out the experiments three sewage sludge samples (L_1, L_2, L_3), from three different WWTP were chosen. The samples had different degrees of anaerobic digestion, being L_1 and L_2 digested sludges and L_3 an undigested sludge.

The methodology described in the following pages was used for each of the three samples and for the products obtained by pyrolysis. The low heating value (LHV) of the samples was determined with a LECO calorimeter, model AC-350. The elemental composition was obtained with an elemental analyzer, Model Carlo Erba CHNS-O EA1108. The removable material was determined with a Dionex ASE 200 extractor us-
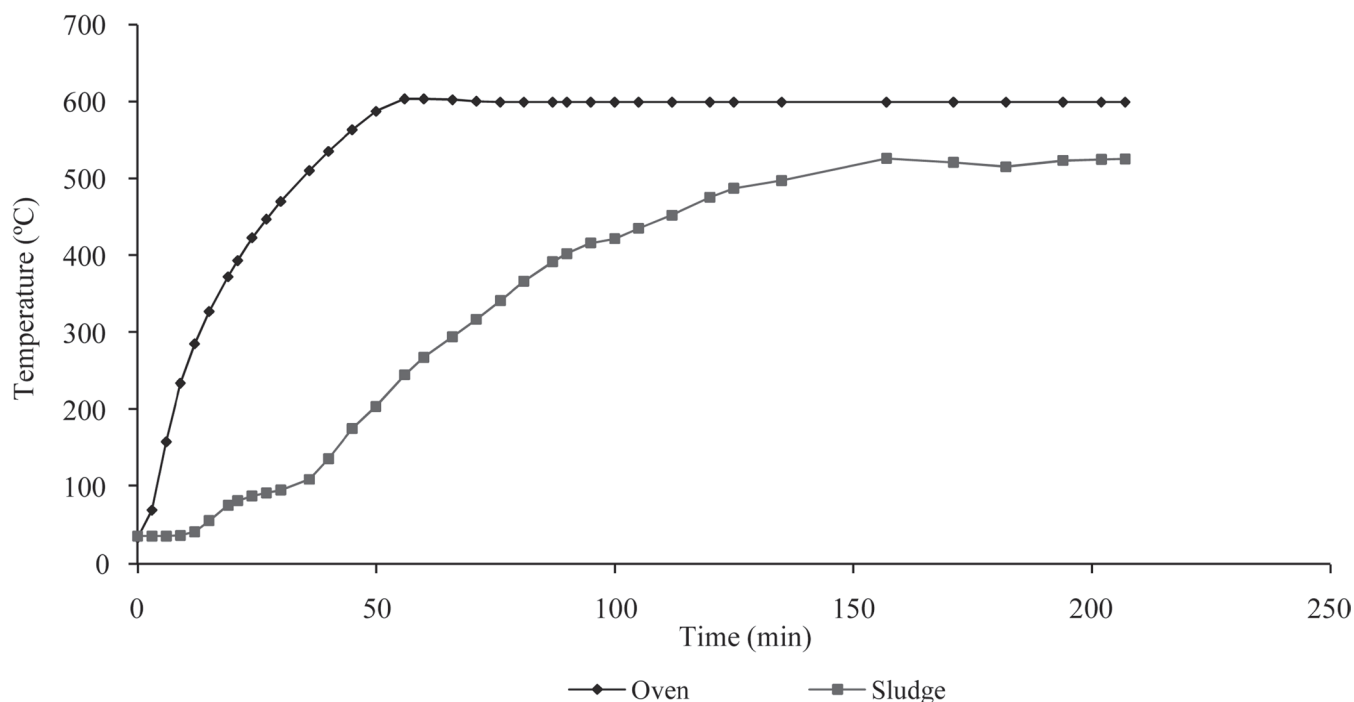
Figure 2. *Variation of the heating oven and reactor temperatures.*

ing methanol as a solvent. The extraction took place in two cycles at 80ºC and 110 bar. The extract was concentrated in a Büchi rotavapour, model R-205 until the weight was constant. The mineralogical composition of the material was analyzed with X-ray fluorescence spectroscopy using a sequential X-ray PW2400 Philips Magix Pro. Table 1 shows the characterization properties of each sludge.

RESULTS AND DISCUSSION

In the present work, the production of bio-oil by slow pyrolysis of sludges from different sources and processes was compared. The conversions obtained for each sludge are shown in Table 2.

The yields of each fraction varied depending on the degree of digestion. As the sludge digestion decreased the proportion of bio-oil phase obtained increased while the fraction of NCG decreased. Sludges L_1 and L_2 presented different levels of anaerobic digestion, being L_1 a more digested sludge than L_2. The pyrolyzed product

yields obtained from sewage sludges were determined as dry ash free mass and the NCG fraction was calculated by mass balance as shown in Equation (1):

$$m_{NCG} = m_{ds} - (m_{char} + m_{oil} + m_{water}) \tag{1}$$

Where "m" refers to the mass and the subscript "*ds*" refers to dry sludge.

It was proved experimentally that there was a relation between the yield of methanol extract from the sludge and the yield of bio-oil obtained by slow pyrolysis. The lower the digestion degree of the sludge, the higher the yield of both the extract and the obtained bio-oil (see Figure 3).

With regard to the low heating value (LHV) obtained for the bio-oil fraction (see Table 3), an increase of the LHV was observed as the sludge digestion degree increased. This behavior can be explained since the volatile compounds generated during the pyrolysis of digested sludge are less polar than those obtained

Table 1. Sludge Sample Characterization.

Property	L_1	L_2	L_3
LHV (MJ/kg)	9.5 ± 0.3	12.1 ± 0.4	15.7 ± 0.5
Extractives in methanol (wt.%)	8.4 ± 0.2	11.8 ± 0.3	25.7 ± 0.7
Water content (wt.%)	8.6 ± 0.2	8.5 ± 0.4	4.4 ± 0.1
Ash (wt.%)	51.0 ± 1.0	36.6 ± 2.1	29.6 ± 1.2

Table 2. Yields of Pyrolysis Fractions.

Sludge	Char (wt.%)	Bio-oil (wt.%)	Water Fraction (wt.%)	NCG (wt.%)
L_1	13.9 ± 0.7	17.2 ± 0.5	36.5 ± 2.1	32.4 ± 0.5
L_2	21.0 ± 0.7	18.7 ± 0.4	30.8 ± 3.0	29.5 ± 0.9
L_3	20.8 ± 0.3	36.2 ± 0.8	19.4 ± 1.4	23.6 ± 1.2

NCG—Non condensable gas.

Table 3. Low Heating Value for Each Obtained Fraction.

LHV (MJ/kg)	L_1	L_2	L_3
Char	4.9 ± 0.2	8.5 ± 0.3	9.9 ± 0.6
Bio-oil	32.8 ± 1.3	30.5 ± 0.9	28.8 ± 1.4
NCG	24.8 ± 0.8	27.3 ± 1.1	23.8 ± 0.7

from non-digested sludge [7]. This affects the solubility of water in the bio-oil, that is, a more digested sludge leads to a bio-oil with less water and therefore more LHV.

The NCG low heating value was determined by an energy balance as shown in Equation (2):

$$LHV_{NCG} = \frac{m_{ds}LHV_{ds} - (m_{char}LHV_{char} + m_{oil}LHV_{oil})}{m_{NCG}}$$

(2)

Table 3 shows the LHV values for each obtained fraction.

A heat treatment at 100ºC was applied to the bio-oil obtained from L^3 in order to remove volatile compounds and water. After this treatment the LHV reached a value of 37.6 MJ/kg. The high content of water contributes negatively to some properties of the oil like decreasing the LHV and the flame temperature, and favours corrosion in environments rich in sulfur [9].

The metal content of the bio-oil was determined by X-ray fluorescence, obtaining the results shown in Table 4. It can be concluded that most of the metals from the sludge were retained in the structure of the carbonaceous char phase, so they did not form part of the bio-oil phase.

It was noticed that some compounds such as Fe, P, Si were partly transported by the gas phase to the bio-oil phase. However, the metals were mainly retained in the solid phase.

The sulfur content of the bio-oil never exceeded 1%, being lower than the sulfur content of fuel oil from petroleum. This is an important advantage, the sulfur can cause corrosion as well as SO_2 emissions. The sulfur content of the fuel should be kept at values lower than 1% since in the presence of water it can promote the corrosion of energy equipment [9].

The energy performance of the different fractions produced in relation with the total energy contained in the sludge was also evaluated. The calculation was based on the energy per unitary mass of dried sludge, and the energy associated with the other three fractions [2]. For the least digested sludges, the energy recovery from the bio-oil fraction reached almost 50%. The total recoverable energy from the process is equal to the sum of the energy contained in the bio-oil and the one in the NCG phases, and it reached values close to 70% in the different sludges. Table 5 shows the energy balance for each sludge.

CONCLUSIONS

The pyrolysis process of sludges from urban water treatment plants produces a bio-oil with characteristics similar to those of fuel from oil; thus, they can be used
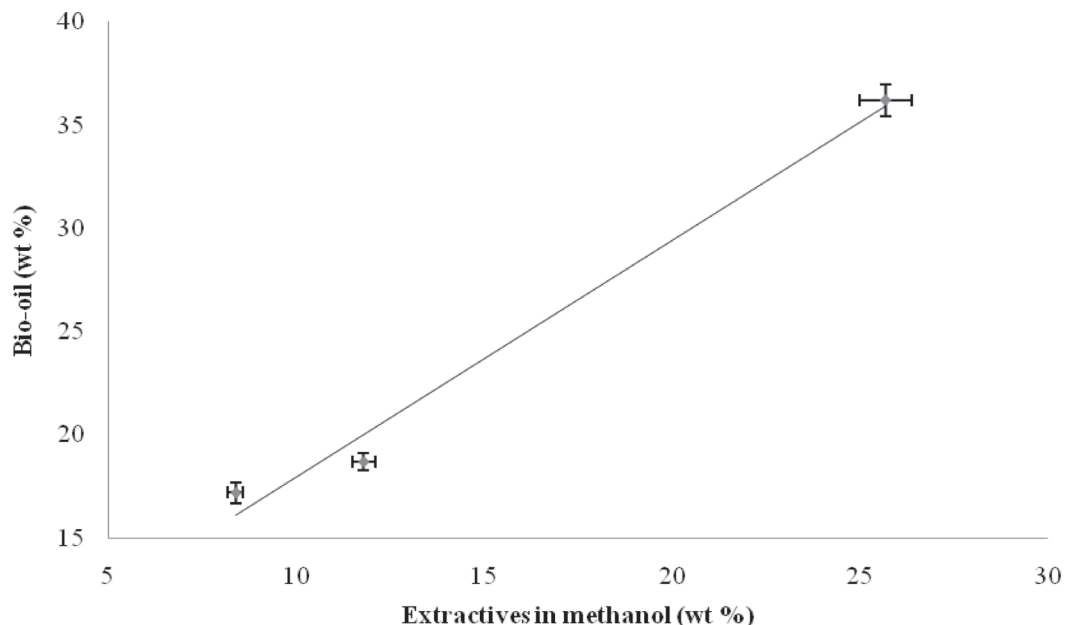
Figure 3. *Relation between the yields of bio-oil and the extractives in methanol.*

Table 4. Elemental Composition of the Sludges and Their Corresponding Bio-oils.

Elemental Analysis (wt.%)	L_1		L_2		L_3	
	Sludge	Bio-oil	Sludge	Bio-oil	Sludge	Bio-oil
C	25.9	77.4	31.6	71.0	37.2	66.8
N	4.0	8.8	5.1	6.3	3.9	4.9
H	4.4	10.8	5.1	9.9	5.5	11.2
S	0.8	0.5	0.5	0.7	1.1	nd
O	13.8	2.1	20.9	12.3	22.7	17.1
Ca	10.2	0.1	7.3	0.1	5.2	0.2
Si	8.7	0.1	7.9	0.2	4.1	0.1
Fe	4.6	0.1	3.0	0.1	4.0	0.1
P	3.8	0.2	3.4	0.2	2.3	0.2
Al	3.8	nd	2.5	nd	1.6	0.02
K	1.4	nd	1.5	nd	0.7	nd
Na	0.3	nd	n.m.	nd	n.m.	nd
Mg	1.2	0.03	0.6	nd	0.4	nd
Ti	0.5	nd	0.4	nd	0.3	nd
Cl	0.3	0.3	0.4	0.3	0.3	0.2
Zn	0.2	nd	0.3	nd	0.3	nd
Cu	0.1	nd	0.1	nd	0.1	nd
Sr	0.1	nd	0.2	nd	0.1	nd
Ba	nd	nd	nd	nd	0.1	nd
Mn	0.1	nd	0.03	nd	0.04	nd
Pb	nd	nd	0.02	nd	0.03	nd
Ni	n.m.	nd	nd	nd	0.02	nd
Zr	0.03	nd	0.03	nd	0.02	nd
Rb	0.01	nd	0.01	nd	0.01	nd
Br	0.02	nd	0.03	nd	0.01	nd
As	nd	nd	0.004	nd	0.003	nd
Y	0.003	nd	nd	nd	0.001	nd
Ce	0.04	nd	nd	nd	nd	nd
Cr	0.03	nd	nd	nd	nd	nd
I	0.02	nd	nd	nd	nd	nd
Sn	0.02	nd	0.1	nd	nd	nd

n.d. not detected, n.m. not measured.

in the production of energy. Sludges with low degrees of digestion are the most suitable ones for bio-oil production. On the other hand, the fraction of NCG produced can be used in the process itself, minimizing the energy consumption required to carry out pyrolysis, or it can be used for other purposes, like drying the biomass.

The metal content of the bio-oil is low; thus, its use has the environmental advantage that heavy metal emissions to the environment are low.

Finally, by means of pyrolysis, the original sludge is transformed into an inert product in the form of char. Although the char has a high metal content, these are retained in the structure and are not released into the environment.

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Table 5. Energy Recovery for Each Fraction.

Sludge	Bio-oil (% rec.)	NCG (% rec.)	Char (% rec.)
L_1	29.1	41.4	29.5
L_2	29.8	35.0	35.2
L_3	46.8	25.2	28.0

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Ozonation for Sludge Reduction and Improved Biological Nutrient Removal

A. INCHAUSTE-DAZA[1], D. SAROJ[2,*], C.M. LOPEZ-VAZQUEZ[1] and D. BRDJANOVIC[1,3]

[1]Department of Urban Water and Sanitation, UNESCO-IHE Institute for Water Education, Westvest 7, 2611 AX Delft, The Netherlands
[2]Centre for Environmental Health Engineering (CEHE), Faculty of Engineering and Physical Sciences, University of Surrey, Surrey GU2 7XH, United Kingdom
[3]Department of Biotechnology, Delft University of Technology. Julianalaan 67, 2628 BC Delft, The Netherlands

ABSTRACT: Municipal wastewater is often treated at a centralized treatment facility based on Activated Sludge Process (ASP) where the production of (excess) activated sludge (bio-solids) is mostly not desired and further sludge handling often contributes to a large fraction of the total operational cost, depending upon the applicable legislation and/or sludge treatment and disposal practices. Further, the Biological Nutrient Removal (BNR) based on ASPs often pose extra cost due to the need for external carbon source (acetate, methanol etc.) for the optimal performance of denitrification and biological phosphorous removal. In this research, the excess sludge was ozonated and the filtrates were used in activity batch tests for anaerobic P-release, aerobic P-uptake and denitrification. Experimental results have shown that the ozone dose in the range of 0.1–0.2 g O_3/g MLSS leads to a good degree of bio-solids destruction (sludge reduction) resulting in highly biodegradable organics. The use of ozonated sludge at those ozone doses as carbon source in the activity tests revealed that the soluble biodegradable organics can be successfully used for improved denitrification and biological phosphorous removal. Further, the process cost analysis showed the economic feasibility under complete sludge treatment scenario, where about 42% of sludge reduction is achieved and about 30% of the total operational cost reduction may be possible under assumed conditions.

INTRODUCTION

ONE of the most important problems that municipal wastewater treatment plants (WWTPs) have been facing during last decades is the production of excess sludge, to be treated and disposed. The sludge treatment and disposal can contribute to a large fraction the total costs of WWTPs [13, 15, 19, 23]. Further, the Biological Nutrient Removal (BNR) based activated sludge systems often pose extra cost due to the need for external carbon source (Acetate, methanol etc.) for optimal performance of denitrification and biological phosphorous removal. Several advance oxidation methods, including ozonation, have been studied for their sludge reduction and biodegradable materials production capacity which can be used to support the need for external carbon source in the denitrification processes [18].

The lysis-cryptic growth concept has been widely

*Author to whom correspondence should be addressed.
E-mail: d.saroj@surrey.ac.uk

applied to study the sludge yield reduction. Several techniques for sludge disintegration to induce cryptic growth have been studied including thermal, chemical, and advanced oxidation treatment. However, sludge ozonation is considered to be one of the most successfully applied technology at both, lab and full-scale, for sludge reduction purposes [1, 15, 19]. Due to the strong oxidative character of ozone, the bacterial cell membranes get ruptured during sludge ozonation and the internal materials are released into the bulk liquid. This lysis process produces a new substrate available to be degraded in subsequent biological processes, so that the overall sludge yield in the system is reduced. The development of the new integrated process i.e. ozonation of sludge and further biological degradation has been studied and applied showing promising results on the reduction of excess sludge from WWTPs [6, 9, 12, 21]. As a consequence of the good performance of the new process approach, the ozonation of sludge has been also effectively included in activated sludge systems at full-scale [16, 20]. The reduction of activated sludge and various facets of the process integration have been

recently studied [4, 5, 7, 14, 17]; however, the investigation of the use of ozonated sludge as substrate in nutrient removal is limited. Also, little is known about the activity of the microorganisms regarding biological nutrient (nitrogen and Bio-P) removal. This paper aims to show the potential of sludge ozonation to reduce the excess sludge production at WWTPs, and the simultaneous use of the ozonated sludge as carbon source in nutrient removal processes.

MATERIALS AND METHODS

The activated sludge used in the experiments was taken from the *Hoek van Holland* municipal WWTP located in the Netherlands. The sludge was stored at 4°C to prevent any change in its composition. Prior to any experiments, the sludge was refreshed by stirring and aeration with a small volume of influent from the same WWTP. In the first phase of the experimental work, sludge ozonation tests were carried out. In the second phase of the experimental work, biological activity tests were carried out to study the influence of using ozonated sludge filtrate on biological nutrient removal processes.

Ozonation of Activated Sludge

The ozonation setup (Figure 1) consisted of an ozone generator (Trailigaz Ozonizer, LABO LO) and an ozone contactor including 1 L reactor, a diffuser, a magnetic stirrer, a metal stirring device, two ozone trap bottles with 4% KI solution each for the determination of the ozone gas strength going into the sludge sample (trap bottle A), and for the determination of the ozone gas that did not react with the sample (trap bottle B). The properties of sludge and the ozonation parameters have been summarized in Table 1.

Two ozonation experiments (Run 1 and Run 2) were carried out up to a maximum duration of 6 hours (Table 1). The results from first ozonation experiment were used to investigate the influence of ozonation on the properties of mixed liquor at various ozone doses. At the end of first ozonation experiment, up to a maximum dose of 0.17 g O_3/g MLSS, ozonated sludge was collected to be used for the biological activity tests related with biological phosphorous removal. The second ozonation experiment was carried out up to a maximum dose of 0.16 g O_3/g MLSS. For the second ozonation experiment, the analyses of intermediate samples are not reported in this paper. At the end of second ozonation experiment, ozonated sludge was collected to be used for the biological activity tests related with biological nitrogen removal (Nitrification-Denitrification).

The ozonated sludge samples (at doses 0.17 g O_3/g MLSS and 0.16 g O_3/g MLSS) were filtered (0.45 micron GF/C whatman filter) and the filtrates were subsequently used for the biological activity tests as carbon source (Bio-P and denitrification). Prior to the activ-
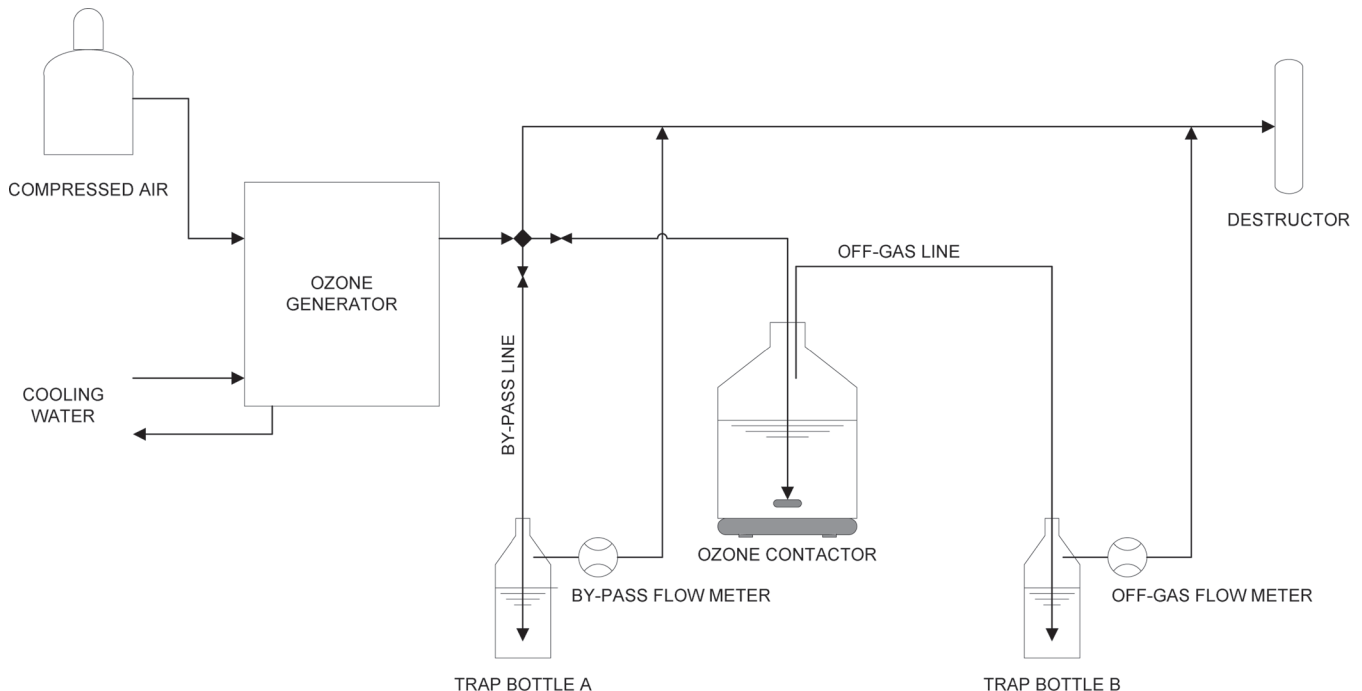

Figure 1. *Schematic diagram of gas flow for the ozonation setup.*

Table 1. Properties of Activated Sludge and Ozonation Parameters.

Sludge Properties		Ozonation Parameter	Run 1	Run 2
MLSS, mg/L	4,010	O_3 Concentration, mg/L	19.1	19.1
MLVSS, mg/L	3,000	Transfer efficinecy, %	66.3	61.9
Total COD, mg/L	5,477	O_3 flow rate, L/h	9.08	9.08
Filtrate COD, mg/L	155	Ozonation duration, h	6	6
		Ozone dose, g O_3/g		
MLSS/MLVSS	0.75	MLSS	0.17	0.16

ity tests, the filtrate of ozonated sludge was subject to chemical precipitation with $FeCl_3$ to remove all the phosphorus released during ozonation. Single analysis for each parameter was carried out and all the parameters were analyzed as per standard methods [3].

Activity Tests

The filtrates of ozonated sludge samples were used for the biological activity tests as source of carbon for Bio-P and denitrification activities. A double jacketed laboratory glass batch reactor with a working capacity of 1 L was used for the activity tests which were run at laboratory hall temperature (≈20°C) due to the practical purposes of this research. The bio-reactor was provided with ventilation, nitrogen gas and compressed air supply (depending on the need for anoxic or aerobic condition), a stirring device and the corresponding sampling point. Prior to the activity tests, the filtrate of ozonated sludge was subject to chemical precipitation with $FeCl_3$ to remove all the phosphorus released during ozonation. All the parameters were analyzed as per standard methods ([3]). Two types of biological activity tests were carried out to study the biological phosphorous removal (Bio-P) and biological nitrogen removal. The ozonated sample with a dose of 0.17 g O_3/g MLSS had been used for Bio-P tests and that with a dose of 0.16 g O_3/g MLSS had been used for nitrogen removal tests.

The biological phosphorus removal batch test consisted of an anaerobic phase where the substrate is fully consumed as the phosphorus is released, followed by an aerobic phase where the phosphorus is uptaken. Each phase lasted for 2 hours and the mixed liquor was continuously sampled and analysed to determine the acetate (for the blank test) and phosphorus concentrations. In case of the nitrogen removal batch tests, it consisted of an aerobic phase (nitrification) where ammonia is oxidised to nitrate, followed by an anoxic phase (denitrification) where the nitrate produced be-

fore is reduced to nitrogen gas. Each phase lasted for 1 hour and the mixed liquor was continuously sampled and analysed to determine the ammonia and nitrate concentrations. The anoxic and anaerobic conditions were maintained by means of nitrogen gas supply. The substrate was fed in a pulse mode at the beginning of the anaerobic phase for the phosphorus release, and at the beginning of the denitrification step. The initial substrate concentrations set in the reactor were the same in all cases (16 mg COD/L). For the first part of the nitrogen removal test i.e. nitrification, the ammonium concentration was set at 15.4 mg NH_4-N/L in the reactor to promote nitrate formation. The artificial ammonia (as $(NH_4)_2CO_3$) was added externally to supply sufficient nitrogen for the nitrification activity test. The details of the experimental protocol have been described elsewhere [10].

RESULTS

Ozonation of Activated Sludge

As a result of first sludge ozonation, the ozone doses varied from 0.03 to 0.17 g O_3/g MLSS. The lysis of the sludge induced by ozone is reflected in the evident reduction of the solids content in the activated sludge, and the increase in the soluble fraction of the sludge with ozone dose. In effect, the reduction of the amount of solids in the mixed liquor was up to 42% at 0.13 g O_3/g MLSS dosage.

Nevertheless, the efficiency of the sludge reduction is achieved at certain optimal ozone beyond which the ozone not only disintegrates the biomass cells but also mineralizes the soluble organics. Figure 2(a) shows the total and soluble COD trends during ozonation of sludge at different dosages. It can be observed that the total COD of the ozonated sludge decreases with ozone dose resulting in a higher soluble COD fraction. The total COD decreased from 5,477 mg/L to 3,910 mg/L at 0.13 g O_3/g MLSS, while the soluble COD content increased from 153 mg/L to 1,327 mg/L at the same dose. If a COD mass balance is applied to the ozonated sludge samples, the total COD decrease with ozone dose increase indicates that part of it was mineralized into CO_2. Regarding other soluble components present in the filtrate of ozonated sludge, Figure 2(b) depicts the trends for the ammonia, nitrate, phosphate and sulphate concentrations found in the filtrates at different doses. As it can be seen, the most remarkable changes took place over the ammonia and phosphate components. Their concentrations peaked at 0.03 g O_3/g MLSS dose
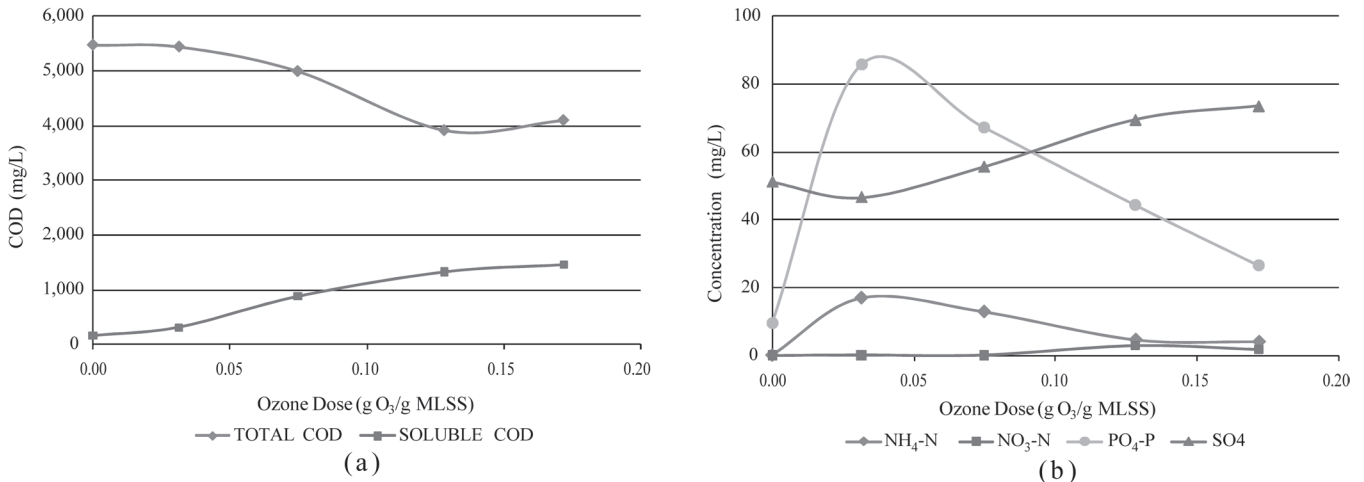
Figure 2. Concentrations variation during ozonation (a) COD, (b) NH_4-N, NO_3-N, PO_4-P, SO_4.

achieving values of about 17 mg NH_4-N/L and 86 mg PO_4-P/L, respectively. On the other hand, the nitrate and sulphate concentrations remained almost the same, showing very little variation with ozone dose increase.

Activity Tests

Although the optimal ozone dose appeared to be 0.13 g O_3/g MLSS (Figure 2), the filtrate obtained from ozonated samples at higher ozone dose were used in the biological activity tests. The activity tests solely aimed at the investigation of the influence of using ozonated sludge (as carbon source) on the performance of biological nutrient removal processes. The filtrates of ozonated sludge at 0.17 g O_3/g MLSS and 0.16 g O_3/g MLSS ozone doses were used as substrate for biological phosphorus removal and denitrification batch tests, respectively. The results from the activity tests show that the filtrates were efficiently used as carbon

source for the P-release and nitrate reduction. The results shown in Figure 3(a) indicates that the filtrate of ozonated sludge at 0.17 g O_3/g MLSS dose lead to the release of orthophosphates into the bulk liquid from 4.96 mg PO_4-P/L to 9.34 mg PO_4-P/L at the end of the anaerobic phase. The specific anaerobic P-release was found to be 1.46 mg PO_4-P/g VSS, and the specific P-uptake was 2.55 mg PO_4-P/g VSS during the aerobic stage. Therefore, the net P removal in the process was 1.09 mg PO_4-P/g VSS. For the denitrification batch test using ozonated sludge as carbon source, the test was carried out just after the corresponding nitrification test using a combined ammonia substrate (ammonia from a synthetic medium mixed with ozonated sludge filtrate, as described earlier). Accordingly, Figure 3(b) confirms that the nitrate formed during nitrification was reduced from 12.08 mg NO_3-N/L to 5.26 mg NO_3-N/L with organic carbon from the ozonated sludge filtrate as electron donor.

Figure 3. Activity tests (a) Anaerobic P-release and aerobic P-uptake trend with ozonated sludge as substrate (0.17 g O_3/g MLSS); (b) NH_4-N oxidation (nitrification) and NO_3-N reduction (denitrification) trend with ozonated sludge as substrate (0.16 g O_3/g MLSS).

Process Analysis for Sludge Reduction

A process analysis for the assessment of the potential sludge reduction by means of activated sludge ozonation was carried out. A typical nitrification-denitrificatio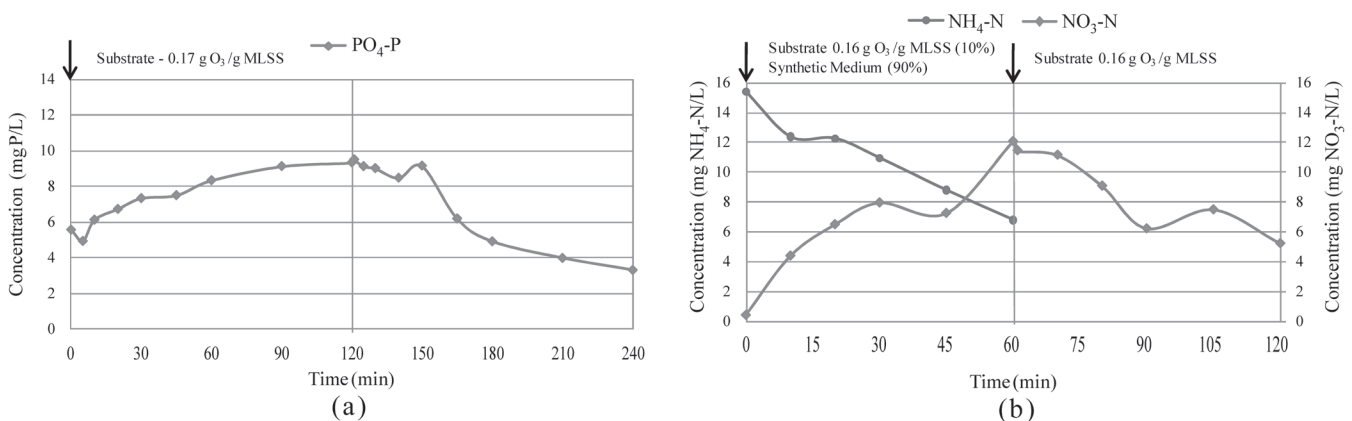n enhanced biological phosphorus removal process (NDEBPR) was selected with an A2O (anaerobic/anoxic/aerobic) process configuration ([8]). It corresponds to a 75,000 P.E. (15000 m^3/d) fictitious WWTP treating municipal sewage. The process analysis was carried out using the typical data for the design of NDEBPR system [8] and cost calculations correspond to Western European situation with data from various sources (e.g. [4]; electricity cost 0.07 €/kWh etc.). In this context, three systems were conceived: a typical NDEBPR, a NDEBPR with external carbon addition (acetate), and a NDEBPR with carbon addition from ozonated sludge. In the latter, a chemical phosphorus precipitation unit was included to avoid the recirculation of the phosphorus released during ozonation into the biological process (Figure 4). Finally, the operational costs with regard to energy consumption, external carbon source, ozone production and maintenance, and sludge handling costs of the three systems were calculated for comparison. The processes were designed following the principles and assumptions of the mixed culture steady state model [8], where the main control parameter was the phosphorus concentration in the effluent (<1 mg P/L). Further, the sludge handling in all the systems included a complete sludge treatment scenario viz. conditioning, dewatering, drying, incineration and ash disposal. Figure 4 depicts the NDEBPR system including the recirculation of the ozonated excess sludge into the biological process at the dose of 0.13 g O_3/g MLSS.

Table 2 shows the cost analysis for the three systems (Options 1, 2 and 3) considered in this research. It can be seen that when ozonation of sludge is integrated, there is a mass reduction of 41.9% in terms of kg MLSS/day. On the other hand, although the COD load increased for systems 2 and 3 in order to achieve a phosphorus concentration in the effluent lower than 1 mg/L, the oxygen consumption and the energy required for the biological treatment does not increase significantly. Regarding the operational costs, the item for ozone production was differentiated with regard to the oxygen source i.e. compressed air or liquid oxygen. In case the ozone is produced from air, the energy required is 54% higher than that from pure oxygen. However, the two options where ozonation is included resulted more economical than the conventional system. In the first case, the operational cost of the system with ozonation from air was 31.4% lower, while in the second case 30.6%. Moreover, the NDEBPR + O_3 systems were even cheaper than the system where acetate was added as external carbon source. The chemical phosphorus precipitation unit included in the last system was not taken into account in the analysis, since the P-rich sludge produced may be seen as a P-source from which it can be recovered resulting in additional benefit.
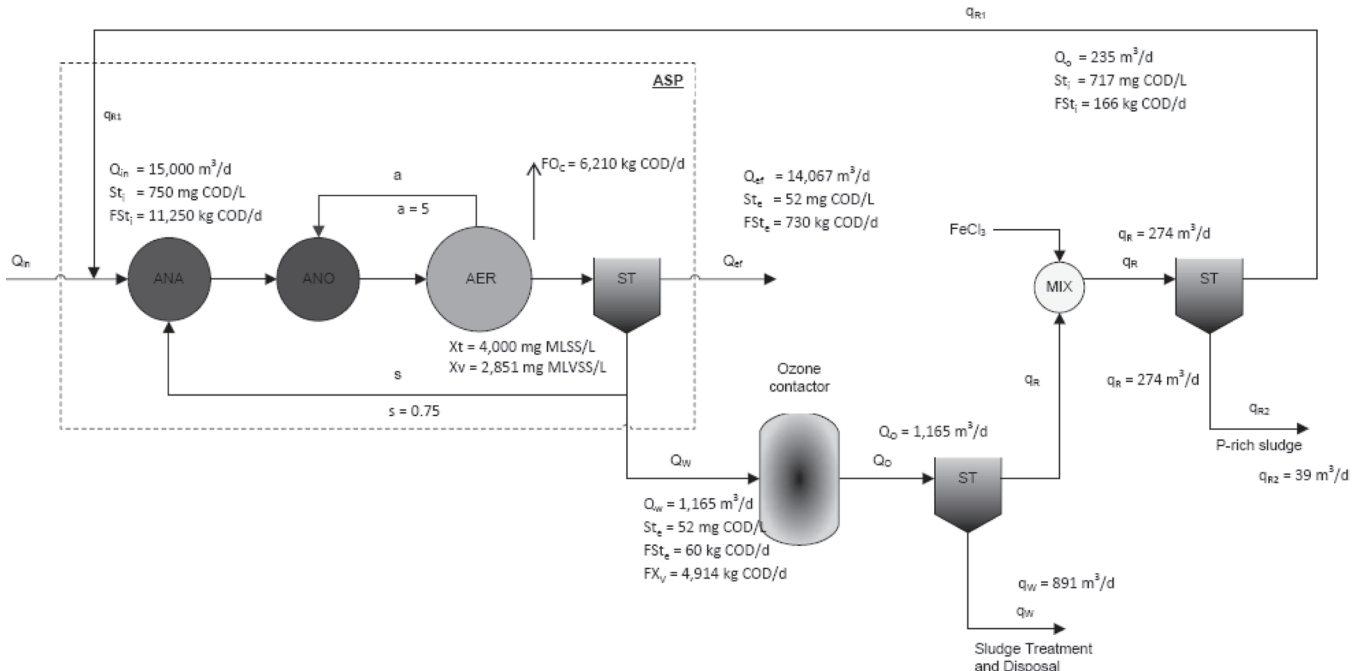

Figure 4. *NDEBPR system with addition of ozonated sludge filtrate (option 3).*

Table 2. The Summary of Operational Costs for an A2O Process Configuration.

Item	Unit	NDEBPR (1)	NDEBPR + HAc (2)	NDEBPR + O₃ (3) Air	NDEBPR + O₃ (3) Pure Oxygen
Sludge waste flow	m^3/d	1,165	1,165	891	891
Daily sludge waste production	kg MLSS/d	11,421	11,421	6,636	6,636
Operational Costs					
Energy for biological treatment	€/year	153,811	153,811	155,978	155,978
External carbon source	€/year	–	8,343	–	–
Sludge treatment and disposal	€/year	4,796,766	4,796,766	2,787,148	2,787,148
Energy for ozone production	€/year	–	–	477,847	220,714
Oxygen for ozone production	€/year	–	–	–	223,817
Ozonation yearly maintenance	€/year	–	–	15,576	8,397
Total Operational Costs	**€/year**	**4,950,577**	**4,958,920**	**3,436,549**	**3,396,055**

DISCUSSION

Ozonation of Activated Sludge

It can be observed in the Figure 2(a) that as the ozone dose increases, the soluble COD concentration rises and the total COD concentration drops. Nevertheless, at ozone doses as low as 0.03 g O₃/g MLSS the soluble COD concentration increase rate was very slow and so was the total COD decrease. It seems that the initial soluble COD concentration in the untreated sludge was considerable (153 mg/L) so that the ozone first mineralized this soluble fraction, and subsequently, it acted over the particulate fraction or biomass. At this stage, when dosages ranged from 0.03 to 0.13 g O₃/g MLSS the soluble COD had a significant increase. However, at ozone doses above 0.13 g O₃/g MLSS the soluble COD concentration increase was not significant anymore. The results suggest that at the dosages higher than 0.13 g O₃/g MLSS, the ozone not only solubilises the sludge, but it starts to mineralize the existing soluble organics in the medium, decreasing its efficiency for sludge disintegration.

Activity Tests

Regarding the P-removal batch tests using substrate produced during sludge ozonation, the results show that the ozonated sludge filtrate was effectively used as carbon source in the anaerobic stage of the Bio-P tests [Figure 3(a)]. The objective of ozonation should aim at the RBCOD formation optimization avoiding its mineralization as much as possible. The results shown in Figure 3a indicate undeniably that there has been P-release in the anaerobic phase using ozonated sludge. In addition, under anaerobic conditions the phosphorous accumulating organisms (PAOs) can only store volatile fatty acids (VFAs) as internal PHA [8]. Presumably, the RBCOD present in the supernatant of ozonated sludge is apparently similar to the fermentable COD in sewage. Since the acetate formation or acidogenesis is the most rapid conversion step in the anaerobic food chain ([8]), any VFA formed by fermentative bacteria is accumulated in the process, being rapidly stored by PAOs. Therefore, the only answer to why there was P-release in the anaerobic stage, even though VFA was not measured in the ozonated sludge supernatant, is that when ozonated sludge is used as substrate during the P-release process, the fermentation of soluble and colloidal organics as result of ozonation might be the key step for PAOs P-release. However, more research will be required to clearly understand the types of organics generated from the ozonation and the possible formation of VFAs supporting the metabolic activities of PAOs.

Regarding the nitrogen removal process, organic carbon source is certainly needed for denitrification purposes. Earlier studies have obtained good denitrification rates using thermally or chemically disintegrated sludge i.e. hydrolysate as carbon source [11]. In this study, the ozonated sludge was confirmed to be a suitable carbon source for the nitrate reduction into nitrogen gas. Moreover, a relationship between the ozone dose and the denitrification degree can be expected during the denitrification experiments, as the ozone dose increases, the nitrate removal could be more significant due to enhancement in biodegradability [10]. However, since the suitability of ozonated sludge for denitrification is due to its biodegradability (high RBCOD fraction), the optimal ozone

dose must be well determined. Some researchers indicated that the supernatant of ozonated sludge including the colloidal and soluble COD is well accepted as carbon source for denitrification processes regardless the ozone dose [2, 14, 22]. The influence of ozone dose on nature of generated organics that can be used as carbon source in the denitrificaiton process needs further research. Further, during denitrification in real WWTPs, three phases are recognized each one with its respective nitrate removal rate: the denitrification based on the RBCOD, the second rate is determined by the slowly biodegradable COD (SBCOD), and the slowest rate takes place under endogenous conditions. As it can be observed in Figure 3(b), the first and second denitrification rates are visible, indicating that at certain point in time during the anoxic phase, the RBCOD supplied either acetate or ozonated sludge supernatant was finished, and the SBCOD available in the mixed liquor started to be consumed by denitrifiers. A complete understanding of the various phases of SBOD consumption during denitrification process needs further dedicated research.

Process Analysis for Sludge Reduction

From the ozonation experiment at various dosages [Figure 2(a)], the ozone dose of 0.13 g O_3/g MLSS was reasoned to be optimal. Following the calculation at this dose, the amount of energy required to produce ozone from air was found to be higher than when pure oxygen is used (Table 2). In addition, the need of oxygen source for the latter is offset by the energy consumption. It is also clear from Table 2 that the most affected parameter when ozonation is part of the system is the amount of solids generated. Regardless the type of gas source to produce ozone, the integrated system 3 produced lower amount of solids per day. Therefore, the costs for sludge treatment and disposal (complete process) decreased significantly when compared to the typical NDEBPR system without ozonation. Hence, taking into account all the items from the operational cost analysis in this study, the NDEBPR + O_3 systems (option 3) had significantly lower operational costs.

The analysis shows that the ozonation of sludge is still a feasible option in terms of operational costs in small WWTPs, provided that the sludge handling comprehends a complete treatment and final ashes disposal. This is attributed to the fact that the sludge management at full-scale WWTPs can make up to 60% of the total operational costs [13, 15, 18, 23] and if the excess sludge to be treated can be reduced significantly, the overall operational costs will also be reduced significantly. However, this study does not take into account various other factors such as capital cost of required infrastructure if the presented concept is to be applied at a full scale wastewater treatment plant. Moreover, the operation and control systems would require improvement or adaptation. Though, these concerns are very much common when retrofitting of existing wastewater treatment facilities is considered aiming at advanced wastewater treatment and process optimisation.

CONCLUSIONS

Following conclusions can be drawn:

- The reduction of excess sludge by means of ozonation appears to be feasible. The sludge solubilisation increases up to an optimum ozone dose after which its efficiency starts to deteriorate. The optimal ozone dose for the most efficient sludge solubilization was found to be about 0.13 g O_3/g MLSS. Further, it appears that at dosages higher than 0.13 g O_3/g MLSS, the ozone not only solubilises the sludge, but also starts to mineralize the existing soluble organics in the bulk MLSS, decreasing its efficiency for sludge disintegration.

- The filtrate of ozonated sludge was effectively used as carbon source for biological phosphorus removal and denitrification processes. Activity test conducted with ozonated sludge filtrate at 0.17 g O_3/g MLSS ozone dose leads to anaerobic phosphorus release indicating that the possible presence of VFAs in the ozonated sludge. The use of ozonated sludge filtrate as organic carbon source for denitrification showed the nitrate removal (ozone dose of 0.16 g O_3/g MLSS).

- In the process analysis for sludge reduction, activated sludge recirculation into the anaerobic reactor via ozonation, at an ozone dose of 0.13 g O^3/g MLSS, leads to an excess sludge reduction of 41.9%. Furthermore, as a result of the operational costs analysis a reduction of 31% was estimated regardless the oxygen source for the ozone production. It can be reasoned from the literature and this research that the overall optimum ozone dose (for the integrated system discussed) should fall in the range of 0.1 to 0.2 g $O3$/g MLSS for the typical excess sludge from municipal WWTP.

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Comparison of the Mesophilic and Thermophilic Anaerobic Sludge Digestion from an Energy Perspective

I. FERRER[1],[*], F. VÁZQUEZ[2] and X. FONT[3]

[1]Environmental Engineering Division, Department of Hydraulic, Maritime and Environmental Engineering,
Universitat Politècnica de Catalunya, c/ Jordi Girona 1-3, Building D-1, E-08034 Barcelona, Spain
[2]Institute of Biotechnology and Biomedicine, UAB, Bellaterra, Cerdanyola del Vallès, 08193 Barcelona, Spain
[3]Composting Research Group, Department of Chemical Engineering, Escola Tècnica Superior d'Enginyeria,
Universitat Autònoma de Barcelona (UAB), Bellaterra, Cerdanyola del Vallès, 08193 Barcelona, Spain

ABSTRACT: Anaerobic digestion promotes simultaneous sludge stabilisation and bio-energy generation. Increasing the net energy production would contribute in reducing the energy demand of sewage treatment plants. Thermophilic digestion may be used to upgrade (conventional) mesophilic digestion, a major drawback being increased energy requirements. The objective of this study is to compare the mesophilic and thermophilic sludge digestion from an energy perspective. Data from laboratory-, pilot- and full-scale digesters are used to compare the energy balance and ratio of full-scale systems. The results highlight the importance of sludge characteristics on the effectiveness of the process, and the need to recover energy from digestates in thermophilic digesters. Net energy production is comparable in thermophilic with half the retention time of mesophilic systems (10–15 vs. 20–30 days).

INTRODUCTION

ANAEROBIC digestion enhances 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 demand, although electricity is required for sludge pumping and mixing. Energy production is defined by the methane production rate, hence by the organic solids removal; which depends on the substrate biodegradability and process parameters, including temperature, sludge retention time (SRT) and organic loading rate (OLR), amongst others.

Thermophilic anaerobic sludge digestion, in one or two-stage systems, is a successful approach to upgrade (conventional) mesophilic digestion. It increases the reaction rate and enhances pathogen destruction [1]. A major drawback is increased energy consumption. According to Zupančič and Roš (2003) [2], heat requirements in thermophilic digesters are about twice those of mesophilic digesters; but they may be covered by combined heat and power (CHP) generation from the biogas produced and heat recovery from digested

sludge. Zábranská *et al.* (2000) [3] reported that heat requirements for two-stage thermophilic digesters are fully covered by increased biogas production; additionally surplus electricity is generated.

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 poor biogas production and increased heat requirements [4]. In such a case, digesters may not be able to self-sustain even mesophilic operation [5]. With solids contents above 4 %, mesophilic and thermophilic single-stage digesters should have a positive heat balance, which would be improved by implementing a two-stage hyperthermophilic-thermophilic process (70ºC + 55ºC) [6].

The objective of this study is to compare the mesophilic and thermophilic anaerobic sludge digestion from an energy perspective. To this end, data from laboratory-, pilot- and full-scale sludge digesters are used to estimate energy production and consumption (i.e. energy balance and ratio) of full-scale systems, under a range of operating conditions.

FUNDAMENTALS OF THE ENERGY BALANCE

In anaerobic digesters, organic matter is converted

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into a primary fuel source (biogas). This fuel source may then be converted into usable energy through different processes, including the combustion in boilers or in combined heat and power units. In the present study, the second alternative is considered, resulting in two forms of output energy (electricity and heat).

The anaerobic digesters considered are completely stirred tank reactors (CSTR), which means that input electricity is needed for sludge mixing and pumping. Since sludge digesters operate in the mesophilic (30–40ºC) or thermophilic (50–60ºC) range of temperature, input heat is needed to raise sludge temperature from ambient (0–20 ºC) to process temperature; and to compensate for the heat loss through the walls of the digester and piping. Heat losses depend on the insulation of the tank, the heat transfer coefficient being 1 and 5 W m^{-2} ºC^{-1} with and without insulation, respectively [7].

A schematic diagram of the energy balance in the anaerobic digester considered is shown in Figure 1. The system and energy balance proposed are described in detail by Ferrer et al. (2009) [8].

The energy ratio between output and input energy, electricity or heat, is calculated according to Equations 1 through 3. This value enables to compare the efficiency of different reactors and processes [9, 10].

$$\text{Energy ratio} = \frac{E(\text{output})}{E(\text{input, electricity}) + E(\text{input, heat})} \quad (1)$$

$$\text{Energy ratio} = \frac{E(\text{output, electricity})}{E(\text{input, electricity})} \quad (2)$$

$$\text{Heat ratio} = \frac{E(\text{output, heat})}{E(\text{input, heat})} \quad (3)$$
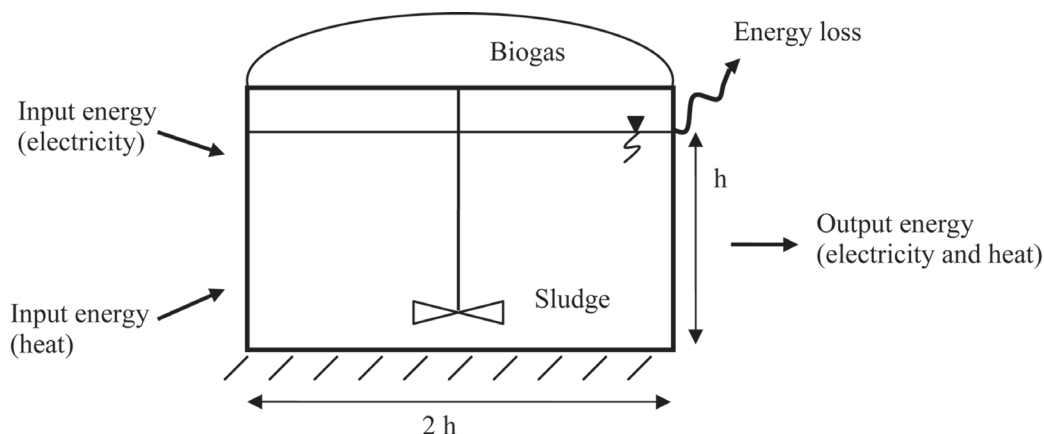
RESULTS

Process Performance

Tables 1 and 2 summarise results from the literature on the performance of laboratory-, pilot- and full-scale reactors treating sewage sludge. Generally, the comparison of data from different studies is not straightforward due to the variability between operating parameters. Some authors have compared the efficiency of mesophilic and thermophilic reactors operating under the same conditions. Similar results are observed with SRT above 20 days, regardless of process temperature: biogas production rates around 0.3–0.4 m$^3_{\text{biogas}}$ m^{-3} d^{-1} and volatile solids (VS) removals around 53% [11, 12]. On the other hand, in thermophilic reactors with low SRT of 15 days biogas production rate is increased by 60% (from 0.36 to 0.6 m$^3_{\text{biogas}}$ m^{-3} d^{-1}) and VS removal by 12% (from 41.6 to 46.3%) compared to mesophilic ones with 20 days SRT [13, 14]. At even lower SRT of 10 and 8 days, biogas production rate is 100% and 200% higher in thermophilic compared to mesophilic systems [15]. Therefore, by operating within the thermophilic range of temperature, it is feasible to reduce the SRT, while increasing methane production. Methane content in biogas is 60–70% (Tables 1–2).

Volatile solids removal ranges between 30–60 %. Values below 30% correspond to digesters treating waste activated sludge (WAS), in which gas production rate is also the lowest, below 0.2 m$^3_{\text{biogas}}$ m^{-3} d^{-1} [5, 15]. The methane yield is defined by the substrate composition, thus for sludge it should be constant. However, literature results clearly show some variability between 0.1 and to 0.8 m$^3_{\text{CH4}}$ kg VS$_{\text{removed}}^{-1}$ (Tables 1–2). This is a consequence of sludge heterogeneity, resulting from several factors like the proportion of

Figure 1. *Schematic diagram of the energy balance of the anaerobic digesters considered.*

primary sludge (PS) and WAS in the mixture, and the SRT of activated sludge units in the case of WAS [5], amongst others. Methane yields are consistently higher with PS (0.4–0.8 m^3_{CH4} kg $VS_{removed}^{-1}$) compared to WAS (0.17–0.43 m^3_{CH4} kg $VS_{removed}^{-1}$) or to the mixture of PS and WAS, both in mesophilic (0.8 vs. 0.3–0.5 m^3CH_4 kg $VS_{removed}^{-1}$) [11, 14, 16] and thermophilic systems (0.4–0.6 m^3_{CH4} kg $VS_{removed}^{-1}$) [13, 14, 17].

Energy Ratios

Theoretical energy ratios of mesophilic and thermophilic single-stage digesters with energy recovery from biogas, and from biogas and digested sludge are shown in Tables 1 and 2, respectively. Values above 1 indicate excess (or net) energy production, while values below 1 indicate insufficient energy generation to fulfil the system's consumption.

According to the results, sludge digestion always yields surplus electricity (electricity ratios > 1). Indeed, output electricity from cogeneration with the biogas produced is much higher than input electricity for sludge pumping and mixing. Electricity ratios basically depend on the methane production rate, and the best ratios are obtained with the lowest SRT and highest OLR (Nº 19–22 in Tables 1 and 2).

On the other hand, heat ratios depend on ambient temperature, and thus on tank insulation. Heat requirements are defined by the difference between influent sludge and process temperature; and heat losses through the walls of the tank by the difference between process and ambient temperature. As shown in Table 1, only mesophilic digesters treating PS and WAS are capable of self-sustaining process temperature with energy recovery from biogas (Nº 13–17 in Table 1). Thermophilic reactors and mesophilic treating WAS do not fulfil the heat demand with residual heat from cogeneration engines (heat ratios < 1).

However, if heat is also recovered from digested sludge by means of a sludge-to-sludge heat exchanger [16], heat ratios increase to values above 1 in digesters treating PS and WAS, both under mesophilic and thermophilic conditions (Nº 13–28 in Table 2). In general, reactors treating WAS are not capable of self-sustaining process temperature in this case either. This suggests that cogeneration is not appropriate when WAS is digested as a sole substrate.

Overall energy ratios are consistently higher for digesters treating PS and WAS, compared to digesters treating only WAS, both under mesophilic and thermophilic conditions (Tables 1 and 2). The proportion be-

tween PS and WAS in the mixture may account for the differences between energy ratios of reactors operating at the same temperature, SRT and OLR; but with different sludge composition. Also, long SRT during the activated sludge process decrease WAS biodegradability and specific biogas production. With reduced OLR and specific methane production, even mesophilic temperature cannot be self-sustained, especially during cold seasons [5].

It is important to highlight that most systems operate at low OLR (< 3 kg VS m^{-3} d^{-1}), because the total solids concentration of thickened sludge is generally below 5% (data not shown). However, 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) [4], 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.

Energy Balances

Comparing the energy ratios of mesophilic and thermophilic digesters, similar net energy production is expected from thermophilic digesters with SRT of 10–20 days and mesophilic with SRT of 20 days. Therefore, thermophilic systems can either be smaller (i.e. reactor volume) or have a higher treatment capacity (i.e. sludge flow rate) being as energy efficient as mesophilic ones.

To exemplify this, Figure 2 shows a comparison between mesophilic and thermophilic digesters with the same working volume. In Figure 2(a) the energy balance of a mesophilic reactor treating a sludge flow rate Q (100 m^3 d^{-1}) at 20 days SRT, is plot beside the energy balance of a thermophilic reactor treating a sludge flow rate 2Q (200 m^3 d^{-1}) at 10 SRT. In Figure 2(b), the energy balance of a mesophilic reactor treating a sludge flow rate Q (100 m^3 d^{-1}) at 30 days SRT, is plot beside the energy balance of a thermophilic reactor treating a sludge flow rate 2Q (200 m^3 d^{-1}) at 15 SRT. 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 an energy perspective, thermophilic reactors treating twice the sludge flow rate (2Q) are as efficient as mesophilic reactors (Q), with the same working vol-

Table 1. Energy Ratios for Anaerobic Digesters with Energy Recovery from Biogas.

| | | Experimental Conditions and Results | | | | 100 m³ sludge d⁻¹ | | Energy Ratio | | | | Heat Ratio | | | | Electricity Ratio | |
| | | | | | | | | Insulated 20°C | Insulated 0°C | Non-Insul. 20°C | Non-Insul. 0°C | Insulated 20°C | Insulated 0°C | Non-Insul. 20°C | Non-Insul. 0°C | All | Reference |
N°	Sludge	T (°C)	SRT (d)	OLR (kg VS m⁻³ d⁻¹)	CH₄ Rate (m³ CH₄ m⁻³ d⁻¹)	V (m³)	Surface Area (m²)										
1	WAS	35	8	–	0.055	800	252	0.22	0.13	0.19	0.11	0.13	0.08	0.11	0.06	**1.30**	[15]
2	WAS	35	10	–	0.057	1,000	293	0.29	0.17	0.24	0.13	0.17	0.10	0.14	0.08	**1.49**	[15]
3	WAS	37.6	21	1.000	0.117	2,100	480	**1.00**	0.63	0.74	0.44	0.60	0.37	0.44	0.25	**3.80**	[5]
4	WAS	38.5	20	0.800	0.046	2,000	465	0.35	0.23	0.27	0.16	0.21	0.13	0.16	0.09	**1.46**	[5]
5	WAS	36	22	0.800	0.098	2,200	495	0.93	0.58	0.70	0.40	0.57	0.34	0.42	0.23	**3.20**	[5]
6	WAS	35	33	0.700	0.052	3,300	649	0.74	0.45	0.53	0.29	0.48	0.27	0.32	0.17	**1.84**	[5]
7	WAS	34.5	37.2	0.530	0.068	3,720	703	**1.10**	0.66	0.77	0.41	0.72	0.40	0.48	0.24	**2.45**	[18]
8	WAS	34	40	1.000	0.026	4,000	738	0.46	0.27	0.32	0.17	0.30	0.17	0.20	0.10	0.94	[5]
9	WAS	55	8	–	0.160	800	252	0.29	0.22	0.24	0.18	0.16	0.13	0.14	0.10	**3.82**	[15]
10	WAS	55	10	–	0.112	1,000	293	0.25	0.19	0.20	0.15	0.14	0.11	0.12	0.09	**2.92**	[15]
11	WAS	55	18	–	0.080	1,800	433	0.31	0.24	0.24	0.17	0.18	0.14	0.13	0.10	**2.51**	[19]
12	PS	55	15	0.693	0.146	1,500	384	0.48	0.37	0.37	0.27	0.27	0.21	0.21	0.15	**4.36**	[17]
13	PS+WAS	35	20	1.140	0.229	2,000	465	**2.14**	**1.30**	**1.63**	0.09	**1.31**	0.76	**1.00**	0.52	**7.36**	[14]
14	PS+WAS	37	20	1.433	0.250	2,000	465	**2.09**	**1.32**	**1.58**	0.92	**1.26**	0.77	0.94	0.53	**8.04**	[11]
15	PS+WAS	37	20	1.380	0.355	2,000	465	**2.96**	**1.87**	**2.25**	**1.30**	**1.79**	**1.09**	**1.33**	0.75	**11.39**	[20]
16	PS+WAS	37	20	1.380	0.385	2,000	465	**3.21**	**2.03**	**2.44**	**1.41**	**1.94**	**1.19**	**1.44**	0.81	**12.36**	[20]
17	PS+WAS	35	27	1.300	0.250	2,700	568	**3.02**	**1.84**	**2.21**	**1.21**	**1.90**	**1.09**	**1.34**	0.70	**8.45**	[12]
18	PS+WAS	43	36	0.442	0.121	3,600	688	**1.27**	0.89	0.88	0.56	0.78	0.52	0.52	0.32	**4.33**	[21]
19	PS+WAS	55	10	3.034	0.400	997	292	0.89	0.69	0.73	0.54	0.51	0.39	0.41	0.30	**10.43**	[22]
20	PS+WAS	55	15	2.063	0.404	1,504	384	**1.32**	**1.02**	**1.03**	0.76	0.76	0.58	0.59	0.43	**12.06**	[21]
21	PS+WAS	55	15	1.519	0.391	1,500	384	**1.28**	**1.00**	**1.00**	0.73	0.73	0.56	0.56	0.41	**11.66**	[14]
22	PS+WAS	55	15	2.190	0.400	1,500	384	**1.31**	**1.01**	**1.02**	0.75	0.75	0.57	0.58	0.42	**11.93**	[13]
23	PS+WAS	55	20	1.870	0.220	2,000	465	0.94	0.72	0.70	0.51	0.54	0.41	0.40	0.29	**7.07**	[12]
24	PS+WAS	55	20	1.433	0.257	2,000	465	**1.09**	0.84	0.82	0.60	0.63	0.48	0.47	0.34	**8.24**	[11]
25	PS+WAS	55	20	1.870	0.260	2,000	465	**1.11**	0.85	0.83	0.61	0.64	0.49	0.47	0.34	**8.35**	[13]
26	PS+WAS	55	27	1.480	0.190	2,700	568	**1.06**	0.82	0.76	0.55	0.62	0.47	0.44	0.31	**6.49**	[13]
27	PS+WAS	55	31	0.643	0.161	3,087	621	**1.01**	0.78	0.71	0.51	0.59	0.45	0.41	0.29	**5.61**	[21]
28	PS+WAS	55	40	0.800	0.170	4,000	738	**1.33**	**1.03**	0.90	0.65	0.79	0.60	0.52	0.37	**6.17**	[13]
29	PS+WAS	55	75	0.420	0.020	7,500	1,122	0.26	0.20	0.16	0.11	0.16	0.12	0.09	0.06	0.77	[13]

Numbers in bold highlight energy ratios ≥ 1.

Table 2. Energy Ratios for Anaerobic Digesters with Energy Recovery from Biogas and Digested Sludge

[5] N°	Sludge	T (°C)	SRT (d)	OLR (kg VS m⁻³ d⁻¹)	CH₄ Rate (m³ CH₄ m⁻³ d⁻¹)	V (m³)	Surface Area (m²)	Energy Ratio Insulated 20°C	Energy Ratio Insulated 0°C	Energy Ratio Non-Insul. 20°C	Energy Ratio Non-Insul. 0°C	Heat Ratio Insulated 20°C	Heat Ratio Insulated 0°C	Heat Ratio Non-Insul. 20°C	Heat Ratio Non-Insul. 0°C	Electricity Ratio All	Reference
1	WAS	35	8	–	0.055	800	252	0.84	0.53	0.49	0.26	0.68	0.37	0.33	0.16	0.91	[15]
2	WAS	35	10	–	0.057	1,000	293	**1.03**	0.66	0.59	0.31	0.85	0.46	0.40	0.19	**1.09**	[15]
3	WAS	37.6	21	1.000	0.117	2,100	480	**3.11**	**2.05**	**1.53**	0.84	**2.64**	**1.47**	**1.02**	0.51	**3.11**	[5]
4	WAS	38.5	20	0.800	0.046	2,000	465	**1.14**	0.76	0.56	0.31	0.94	0.54	0.37	0.19	**1.19**	[5]
5	WAS	36	22	0.800	0.098	2,200	495	**2.84**	**1.83**	**1.41**	0.74	**2.50**	**1.33**	0.95	0.45	**2.63**	[5]
6	WAS	35	33	0.700	0.052	3,300	649	**1.96**	**1.26**	0.95	0.48	**1.90**	0.96	0.66	0.30	**1.59**	[5]
7	WAS	34.5	37.2	0.530	0.068	3,720	703	**2.77**	**1.77**	**1.33**	0.67	**2.78**	**1.37**	0.94	0.41	**2.15**	[18]
8	WAS	34	40	1.000	0.026	4,000	738	**1.12**	0.71	0.54	0.27	**1.16**	0.56	0.38	0.17	0.84	[5]
9	WAS	55	8	–	0.160	800	252	**1.29**	**1.00**	0.69	0.49	0.85	0.63	0.42	0.29	**2.67**	[15]
10	WAS	55	10	–	0.112	1,000	293	**1.07**	0.82	0.55	0.38	0.71	0.52	0.33	0.23	**2.12**	[15]
11	WAS	55	18	–	0.080	1,800	433	**1.17**	0.89	0.53	0.37	0.81	0.58	0.32	0.22	**2.00**	[19]
12	PS	55	15	0.693	0.146	1,500	384	**1.88**	**1.44**	0.89	0.62	**1.29**	0.93	0.54	0.36	**3.39**	[17]
13	PS+WAS	35	20	1.140	0.229	2,000	465	**6.55**	**4.17**	**3.34**	**1.72**	**5.84**	**3.03**	**2.28**	**1.05**	**5.98**	[14]
14	PS+WAS	37	20	1.433	0.250	2,000	465	**6.61**	**4.33**	**3.29**	**1.78**	**5.63**	**3.10**	**2.20**	**1.08**	**6.53**	[11]
15	PS+WAS	37	20	1.380	0.355	2,000	465	**9.37**	**6.13**	**4.67**	**2.52**	**7.99**	**4.39**	**3.12**	**1.53**	**9.26**	[20]
16	PS+WAS	37	20	1.380	0.385	2,000	465	**10.17**	**6.65**	**5.06**	**2.73**	**8.66**	**4.76**	**3.38**	**1.66**	**10.04**	[20]
17	PS+WAS	35	27	1.300	0.250	2,700	568	**8.49**	**5.42**	**4.17**	**2.13**	**7.93**	**4.05**	**2.88**	**1.31**	**7.23**	[12]
18	PS+WAS	43	36	0.442	0.121	3,600	688	**3.67**	**2.57**	**1.60**	0.96	**3.05**	**1.85**	**1.04**	0.58	**3.79**	[21]
19	PS+WAS	55	10	3.034	0.400	997	292	**3.82**	**2.93**	**1.96**	**1.37**	**2.55**	**1.86**	**1.19**	0.80	**7.58**	[22]
20	PS+WAS	55	15	2.063	0.404	1,504	384	**5.22**	**3.98**	**2.47**	**1.70**	**3.56**	**2.57**	**1.49**	**1.00**	**9.39**	[21]
21	PS+WAS	55	15	1.519	0.391	1,500	384	**5.04**	**3.85**	**2.38**	**1.65**	**3.44**	**2.48**	**1.44**	0.97	**9.07**	[14]
22	PS+WAS	55	15	2.190	0.400	1,500	384	**5.16**	**3.94**	**2.44**	**1.68**	**3.52**	**2.54**	**1.48**	**1.00**	**9.28**	[13]
23	PS+WAS	55	20	1.870	0.220	2,000	465	**3.45**	**2.63**	**1.55**	**1.06**	**2.41**	**1.72**	0.94	0.62	**5.74**	[12]
24	PS+WAS	55	20	1.433	0.257	2,000	465	**4.03**	**3.07**	**1.80**	**1.24**	**2.81**	**2.01**	**1.09**	0.73	**6.70**	[11]
25	PS+WAS	55	20	1.870	0.260	2,000	465	**4.08**	**3.11**	**1.83**	**1.26**	**2.84**	**2.04**	**1.11**	0.74	**6.79**	[13]
26	PS+WAS	55	27	1.480	0.190	2,700	568	**3.61**	**2.75**	**1.54**	**1.05**	**2.58**	**1.83**	0.94	0.62	**5.49**	[13]
27	PS+WAS	55	31	0.643	0.161	3,087	621	**3.31**	**2.51**	**1.38**	0.94	**2.40**	**1.69**	0.84	0.56	**4.83**	[21]
28	PS+WAS	55	40	0.800	0.170	4,000	738	**4.07**	**3.09**	**1.63**	**1.11**	**3.02**	**2.12**	**1.00**	0.66	**5.46**	[13]
29	PS+WAS	55	75	0.420	0.020	7,500	1,122	0.66	0.50	0.25	0.17	0.53	0.36	0.15	0.10	0.72	[13]

Numbers in bold highlight energy ratios ≥ 1.

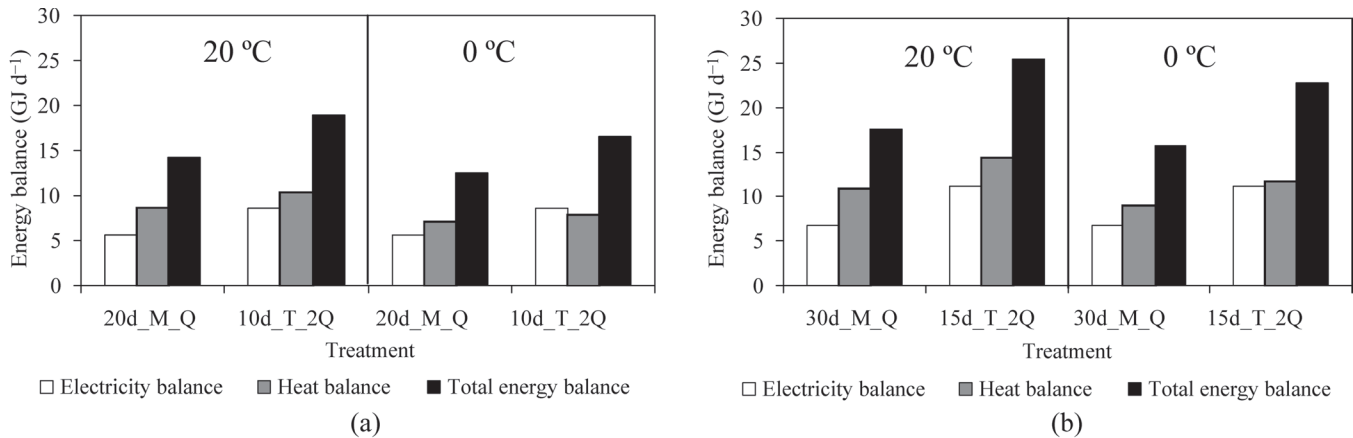**Figure 2.** Electricity, heat and total energy balance of anaerobic digesters treating 100 m^3_{sludge} d^{-1} (Q) at SRT of 20 or 30 days under mesophilic conditions; and treating 200 m^3_{sludge} d^{-1} (2Q) at SRT of 10 or 15 days under thermophilic conditions. Energy is recovered from the biogas and digested sludge. The digesters are insulated and ambient temperature is 0ºC or 20ºC.

ume. Notice that in this example insulated digesters with energy recovery from biogas and digested sludge are considered.

CONCLUSIONS

The energy assessment of the anaerobic sludge digestion highlights the following conclusions:

1. Anaerobic digesters are net electricity producers, since output electricity from cogeneration with biogas is higher than input electricity for sludge pumping and mixing. The best results are obtained with low SRT (10–15 days) and high OLR (2–3 kg VS m^{-3} d^{-1}).

2. Residual heat from cogeneration fulfils the heat requirements of mesophilic digesters treating mixed PS and WAS. Thermophilic digesters self-sustain process temperature with additional heat recovery from digested sludge.

3. Digesters treating WAS as a sole substrate are not capable of self-sustaining process temperature with residual heat from cogeneration. In this case, cogeneration with biogas does not seem a good option.

4. The energy efficiency increases with the ORL, resulting from decreased SRT and concentrated feed sludge. Thus, increasing the solids concentration of thickened sludge entering digestion is a way of increasing the net energy production.

5. From an energy perspective, the performance of thermophilic digesters working with half the SRT (10–15 days) of mesophilic digesters (20–30 days) is comparable. In this way, it is possible to reduce

the size or increase the treatment capacity of the system, with subsequent savings in terms of sludge and wastewater treatment costs.

ACKNOWLEDGEMENTS

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Main Factors Influencing Anaerobic Digestion of Sludge and Energy Efficiency at Several Large WWTP in Central Europe

P. CHUDOBA[1,*], C. SARDET[2], G. PALKO[3] and E. GUIBELIN[4]

[1]Veolia Voda Continental Europe, Praha, Czech republic
[2]Veolia Wasser Germany, Leipzig, Germany
[3]FCsM, Budapest, Hungary
[4]Technical direction Veolia Eau, St. Maurice, France

ABSTRACT: Energy efficiency is one of the most important performance indicators of WWTP operation. It is closely related to all aspects of sustainable development and the level of energy consumption influences directly the environmental impact of a WWTP. It is thus evident that the interest of plant operator is to optimise (decrease) the energy consumption and at the same time increase the energy production. The strategy "consume less & produce more" has been implemented by the Veolia Water group in Europe at 22 larges WWTPs with anaerobic digestion of sludge and energy recovery from biogas. The energy production is "boosted" by switching to thermophilic digestion, applying sludge pre-treatment, optimising sludge thickening and implementing biowastes co-digestion. These 22 WWTPs consume together 280 Gwh$_{el}$./y, and produce 160 Gwh$_{el}$./y, which shows an average electricity self-sufficiency of approx. 60%. Three WWTPs with co-digestion of biowastes are approaching even 100% of energy self-sufficiency.

INTRODUCTION

CURRENT energetical trends start to influence more and more different fields of human activities, including wastewater treatment. Energy savings became one of the most important performance indicators of a WWTP operation, and the concept of "consume less and produce more" presents a real challenge to become more and more independent on local energy supplier. To optimise the energy consumption, different approaches were experienced, aiming namely replace old, high-energy consuming motors (blowers, pumps, . . .), by modern, low-energy consuming material. The energy efficiency optimisation is closely related to enhanced *in-situ* energy production from waste sludge. The use of biogas resulting from the anaerobic digestion of sludge for combined heat and energy production (CHP) by cogeneration is well known and widely described process [1,2]. Veolia Water group operates in Europe approx. 22 larges WWTPs with a capacity of more than 100,000 PE, anaerobic digestion of sludge and CHP from biogas. In order to optimise both anaerobic digestion process and energy recovery from

sludge, to exchange the experiences and to share best practices, a common ANaerobic Digestion Commission (ANDC) was created in 2005. This commission, involving 25 operators of larges WWTPs and experts from Technical direction and R&D department, defined all main parameters influencing both anaerobic digestion and energy recovery efficiencies, and established commonly used best practices for the energy recovery from sludge. Main influencing factors have been then optimized at practically all operated large WWTPs, resulting in both higher anaerobic digestion and energy efficiencies. The anaerobic digestion of sludge followed by energy recovery from biogas is thus from the energetical point of view one of the most interesting technologies of sludge treatment.

MAIN FACTORS INFLUENCING THE ANAEROBIC DIGESTION

The ANDC created a databasis of 22 larges WWTPs with the main focus on efficiency of sludge treatment and energy consumption/production. Several WWTPs are approaching 90% of the energetical self-sufficiency, some of them have potential to reach a complete self-sufficiency. To improve the anaerobic digestion process, the ANDC formulated main influencing fac-

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*Author to whom correspondence should be addressed.
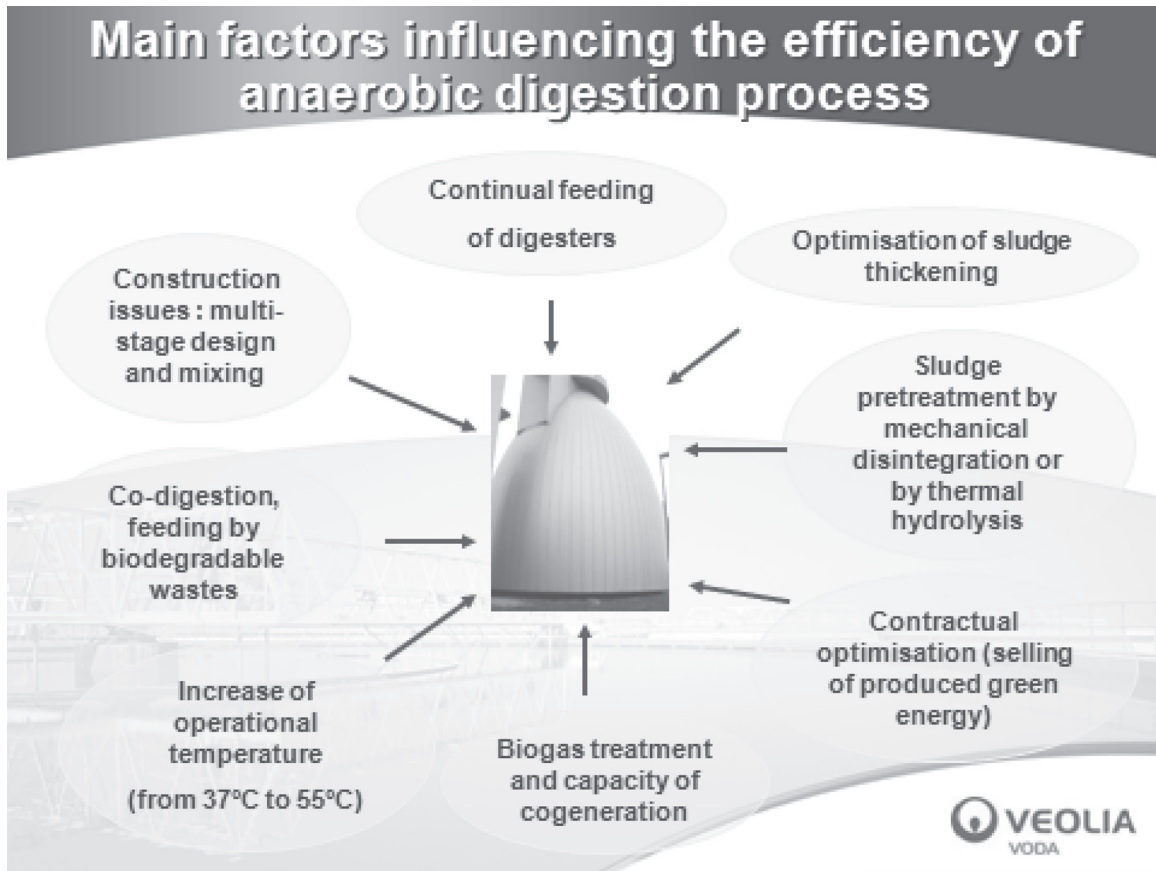E-mail: pavel.chudoba@veoliavoda.cz

Figure 1. *Main factors influencing both anaerobic digestion and energy production efficiency.*

tors, whose optimisation will contribute to both higher process efficiency and energy production (Figure 1). Most of these factors have allready been discussed in the literature [2].

Some of these factors have allready been practically improved at several large European WWTPs :

* *Sludge pre-treatment:* Lysatec mechanical disintegration (Prague WWTP), thermal hydrolysis (Brussels WWTP).
* *Optimisation of sludge thickening:* replacing the thickening centrifuges by low-energy consuming belt-thickener (Pest-South and Pest-North WWTPs).
* *Construction issues:* 2-stage anaerobic digesters design is used frequently (21 from 22 monitored larges WWTPs operated by Veolia Water Europe)
* *Co-digestion of external biowastes:* 10 from 22 monitored WWTPs implemented co-digestion (Pest-South, Pest-North, Pilsen, Braunschweig, Gera, Görlitz, Berlin WWTPs, . . .).
* *Thermophilic anaerobic digestion:* 4 from 22 monitored WWTPs use the thermophilic digestion (Prague, Pilsen, Braunschweig and Pest-South WWTPs).

Since 2005, the ANDC is monitoring main operational performances of selected WWTPs with a capacity of more than 100,000 PE and anaerobic digestion with energy recovery from biogas by cogeneration. The list of main monitored WWTPs is presented in Table 1. This monitoring enabled ANDC to establish a databasis of main performance indicators, which is used then for operational benchmarking, process improvements and optimisations, especially focused on anaerobic digestion of sludge and energy production from biogas. Some of these operational optimisations and improvements will be presented here-below.

PRACTICAL APPLICATIONS, CASE STUDIES

Optimisation of Sludge Thickening

Properly designed and operated mechanical sludge thickening is one of the most important conditions for an efficient anaerobic digestion of sludge. Several mechanical devices with various efficiencies and energy consumptions are used—centrifuges, belt-filters, rotating sieves, flotation, etc. The usual DS content of

Table 1. List of Main Monitored Larges WWTPs (data from 2008).

WWTP (Country)	Capacity in PE	Load in kg COD/d	Capacity of Concentration in kWh	Production of Biogas in Nm³/y	Biogas to Cogeneration in Nm³/y	Produced Electricity in Wh/y	Efficiency kWh/Nm³ of Biogas	Electricity Self-sufficiency in %	Remark
Görlitz (D)	140,000	8,923	570	949,858	940,086	1,536,586	1.63	72	M, co-dig
Schönebeck (D)	90,000	7,890	210	576,542	443,971	862,001	1.94	53	M
Gera (D)	200,000	15,099	380	1,099,716	903,761	1,705,205	1.89	68	M, co-dig
Den Hague—Houtrust (NL)	487,000	31,393	3,600	2,427,094	2,421,946	4,549,204	1.88	30	M
Den Hague—Harnaschpolder (NL)	1,473,000	95,096	3,750	4,976,878	4,967,487	12,612,500	2.54	43	M
Prague (CZ)	1,641,600	210,800	5,400	17,878,058	13,868,369	27,863,300	2.01	75	T, co-dig, MD
Pest-South (H)	293,300	77,484	1,300	6,824,005	4,526,581	9,037,587	2	70	T, co-dig
Pilsen (CZ)	380,000	54,508	1,410	4,170,116	3,989,299	7,020,512	1.76	75	T, co-dig
Braunschweig (D)	275,000	54,400	2,760	3,708,000	3,590,200	8,537,000	2.38	66	T, co-dig
Szeged (H)	230,000	23,919	660	1,371,657	1,247,007	3,026,556	2.43	49	M
Seafield (UK)	800,000	75,000	2,329	10,380,600	5,784,309	12,725,479	2.2	53	M
Olomouc (UK)	259,500	15,183	900	1,695,252	802,381	1,794,500	2.24	32	M, co-dig
Hrdec Kralove (CZ)	141,000	9,622	876	1,149,020	940,073	1,248,000	1.33	32	M, co-dig
Teplice (CZ)	130,000	7,086	280	620,999	575,010	1,083,051	1.88	49	M
Ústi (CZ)	180,000	16,875	470	1,077,299	854,462	1,375,299	1.61	32	M, co-dig
Liberec (CZ)	190,000	12,122	475	1,266,245	1,205,941	1,927,317	1.6	46	M
Berlin Wassmansdorf (D)	1,767,000	176,672	4,000	14,302,069	14,093,068	28,261,147	2.01	64	M, co-dig
Berlin Shonerlinde (D)	675,000	83,795	530	6,228,528	1,721,5485	3,579,170	2.08	15	M
Madrid Sur (Esp)	3,007,950	169,242	6,110	12,171,800	10,170,321	18,555,443	1.82	59	M

M: mesophilic; T: thermophilic; Co-dig: codigestion; MD: mechanical disintegration.

thickened sludge amounts to 4–6%, lower values are considered as unsufficient, higher values are obtained only exceptionally, usually thanks to a specific pre-treatment of sludge (mechanical disintegration, thermal hydrolysis), main limiting factor for these higher DS contents is viscosity of sludge and possible pumping problems.

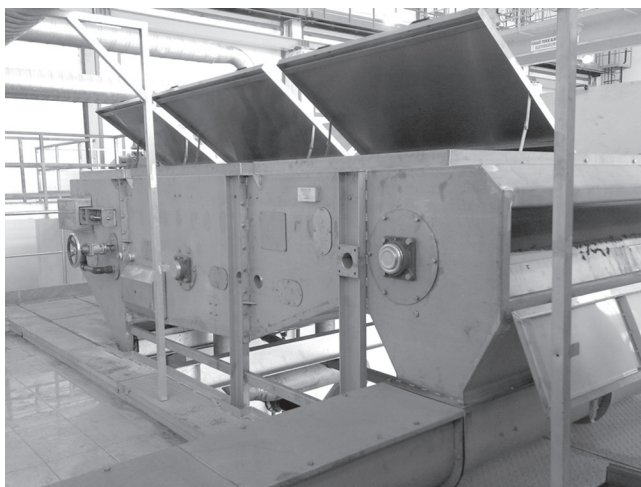The energy consumption of these thickening devices varies, and its optimisation can contribute to a substan-

Figure 3. *Gravity belt-thickener Flavy, implemented at the Pest-South and Pest-North WWTPs.*

tially improved energy balance of the whole WWTP. At Pest-South and Pest-North WWTPs (Hungary), plant operator decided to replace the old thickening centrifuges Guinard by new gravity belt-thickener Flavy (Figure 2). The energy consumption has been reduced by 950 kWh/d and by 5,070 kWh/d, respectively. At the same time, the DS content of thickened sludge increased from 5.5 to 7,0% and from 4.5 to 4,8%, respectively. Another positive results were observed by plant operator—the HRT in Pest-South WWTP digesters increased from 12.5 to 13.7 d, the OM degradation efficiency increased from 62 to 65% and the volume of dewatered sludge decreased by 2%.

Thermophilic Digestion

Thermophilic anaerobic digestion (at 55°C) is used usually in multi-stage digesters configuration as 1st rapid acetogenesis phase [3]. In some cases, the overloaded mesophilic digester has been transformed to thermophilic in order to increase its capacity. This is the case of Prague WWTP [4,5]. After progressive switching of all 12 digesters from mesophilic to thermophilic operational conditions (completed in 2003), the capacity of anaerobic digestion increased, which enabled plant operator to apply a higher load and as
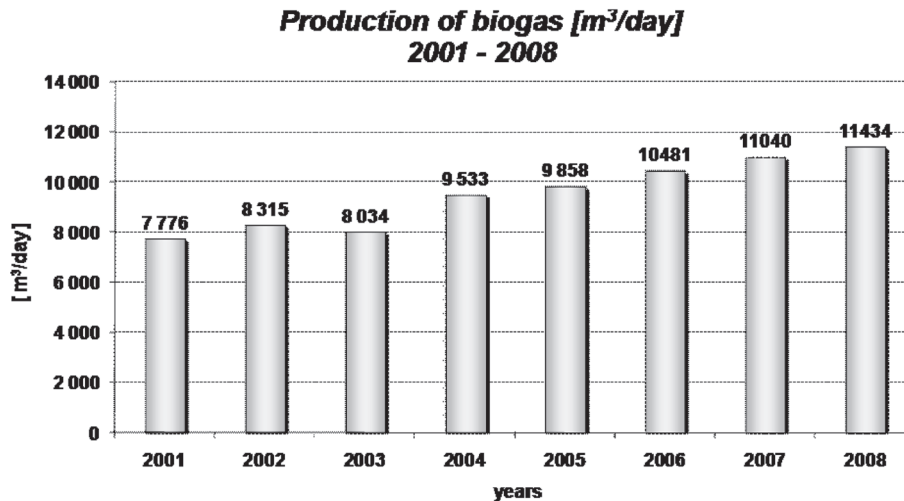
Production of biogas [m³/day]
2001 - 2008

Figure 3. Biogas production at Pilsen WWTP (fully thermophilic digestion since 2005).

the main result, the biogas production increased by 80–100%. The total electricity production increased from an average value of 20,000 MWh/y up to 30,000 MWh/y, which represents approx. 74% of the electricity needs of the whole WWTP. The heat produced by cogeneration covers 100% of the WWTP needs. The amount of produced biogas is sufficient for a complete energy independency of WWTP, but the capacity of cogeneration currently available (5 motors, whole capacity 5.5 MW) is not sufficient. Two additional cogeneration units are needed to reach 7 MW and full energetical self-sufficiency.

Similar situation is in Pilsen, where plant operator decided to increase the capacity of the anaerobic digesters by switching them form mesophilic to thermophilic operation [6]. The main reason for this optimisation was the fact that the Pilsen WWTP receives both wastewater and solid wastes from Pilsner-Urquell

brewery, the solid wastes being directly fed in the digester. Consequently, with a higher digestion capacity, plant operator can both optimise co-digestion of biowastes and improve the global energy balance of WWTP. The thermophilic operation started in 2004, and resulted in an increase of both biogas and electricity production (Figures 3 and 4). The energy produced by cogeneration of biogas at the Pilsen WWTP is sufficient to cover 100% of heat needs and 74% of electricity needs, respectively.

From 22 monitored WWTPs, thermophilic digestion is implemented at 4 WWTPs: Prague, Pilsen, Braunschweig and Pest-South WWTPs. The ANDC benchmarking shows, that some performance indicators reached by these WWTPs are better, than those obtained by WWTPs with mesophilic digestion, particularly sludge production, efficiency of OM removal and energy self-sufficiency.
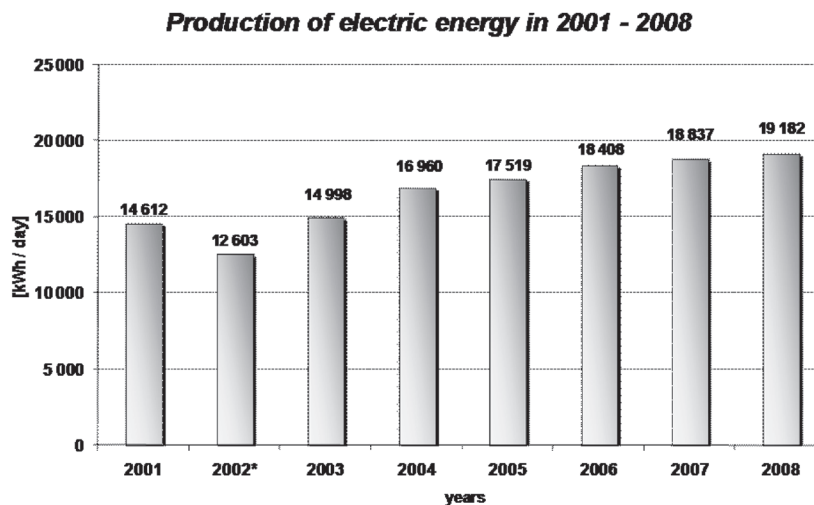
Production of electric energy in 2001 - 2008

Figure 4. Electricity production at Pilsen WWTP (fully thermophilic digestion since 2005).
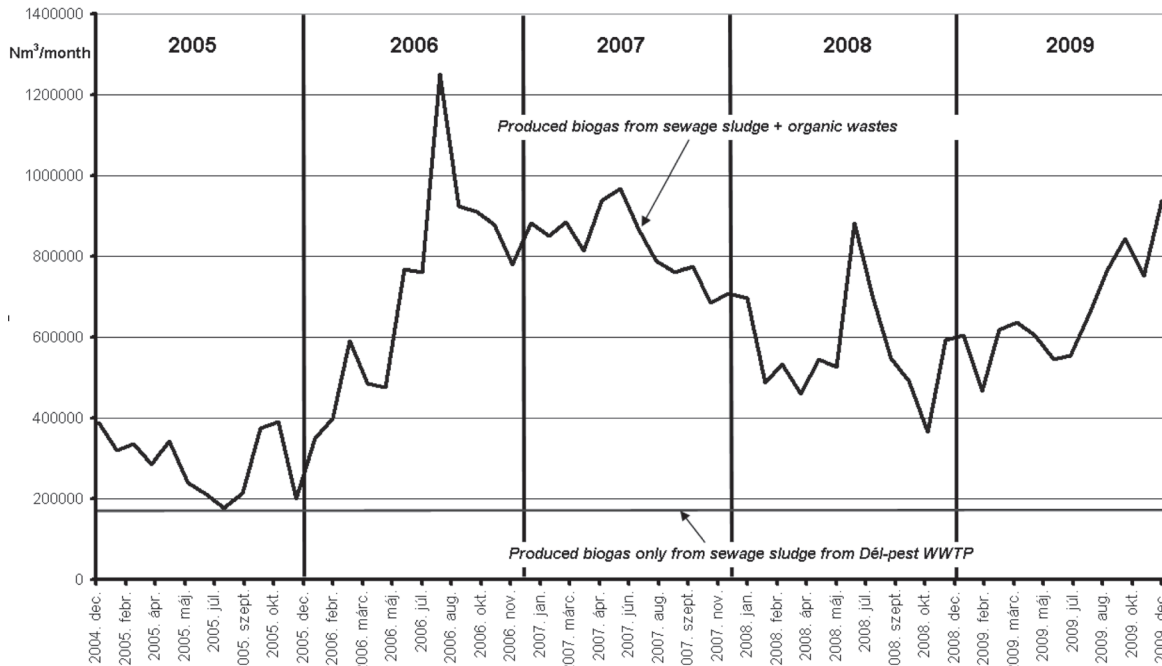
Figure 5. *Increase of biogas production at the Pest-South WWTP (from 200,000 Nm³/month without co-digestion up to 600,000–1,000,000 Nm³/ month with co-digestion).*

Co-digestion—Case of Pest-South and Pest-North WWTPs

It is evident from available energetical balances, that an absolute energetical self-sufficiency of an existing WWTP cannot be reached without an external organic carbon input (sludges from other WWTPs, biowastes, organic substrates, . . .). From this point of view, the only possibility is to implement co-digestion of such an external carbon source [7]. Some practical cases show, that the co-digestion of a properly selected external carbon source (often greases or biowastes) can result in a very high increase of both biogas and electricity production [8,9]. For example at the Pest-South WWTP (293,300 PE) the co-digestion of biowastes was implemented in 2004–2006. The evolution of biogas production presented at Figure 5 shows, that the co-digestion of external biowastes resulted in an increase of biogas production up to 500%.

The Pest-South WWTP co-digestion concept was implemented also at the Pest-North WWTP (775,000 PE) in the period 2007 to 2009. The existing sludge thickening and dewatering has been completed by new mesophilic anaerobic digesters (2 × 12,000 m³), membrane gas holder (2,000 m³), 3 cogeneration units (1× Jenbacher + 2× Caterpillar with a total installed capacity of 3 MW) and reception technology for external liquid biowastes. The evolution of biogas production and energy consumption & production are presented in fig-

ures 6 and 7, respectively. It can be seen, that the input of external liquid biowastes increased biogas production by 50%, and a complementary operational optimisation (reduction of electricity consumption) resulted in an increased energetical self-sufficiency of the plant, which reached up 90% in the first quarter of 2010 (Figure 7: yellow part of columns—purchased electricity, green part of columns—produced electricity).

Another concrete example of energy efficiency increase is the case of Görlitz WWTP, Germany (140,000 PE). By a progressive optimisation of energy consumption and increase of energy production by co-digestion of greases, the electrical self-sufficiency of this WWTP was increased from 30 to 72 % in the period 2004–2009 (Figures 8 and 9).
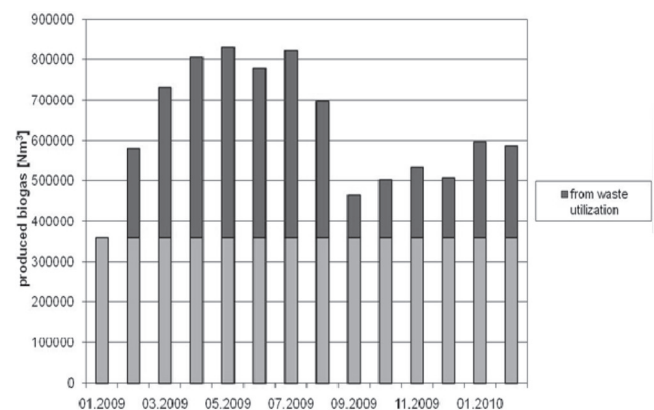

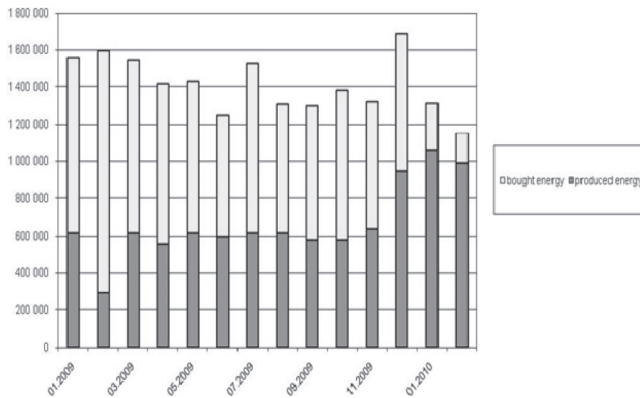**Figure 6.** *Evolution of biogas production (Pest-North WWTP, 2009–2010).*

Figure 7. *Electrical energy self-sufficiency (Pest-North WWTP, 2009–2010).*

The above mentioned examples show, that the energetical self-sufficiency of a WWTP can be reached by implementation of co-digestion. Nevertheless, there are some risks and limitations, which have to be considered, minimized and controlled before any implementation:

- Sufficient available capacity of digesters (avoid organic overloading).
- Sufficient HRT in digesters (recommended mimimum HRT = 20 d).
- Sufficient capacity of biogas network, treatment, storage and cogeneration.
- Sufficient available capacity of wastewater treatment technology to remove the excess N and P, introduced by external biowastes (quantity and quality of sludge water).
- Availability, constant supply and long-term contractual relationship with biowaste suppliers.
- Optimized mixture of biowastes (minimize the risk of inhibition).
- Control over the sludge composition (dewaterability, polymer consumption).
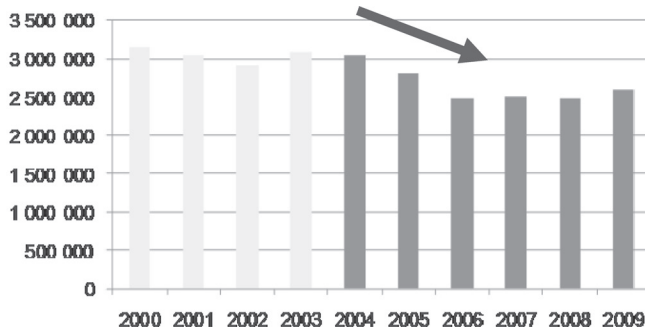- Suitable technology for external biowaste reception, handling and pre-treatment.

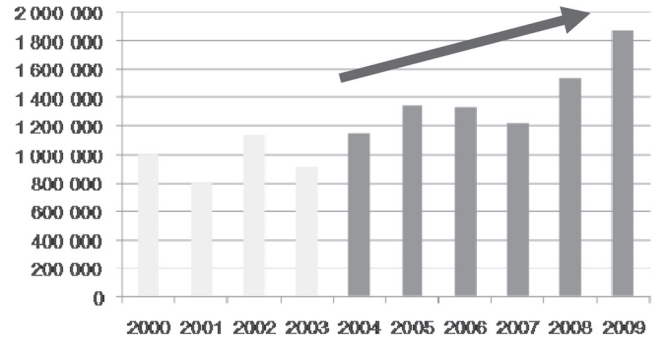***Figure 8.*** *Energy consumption (Görlitz WWTP).*

Figure 9. *Energy production (Görlitz WWTP).*

Energetically self-sufficient WWTP— Dream or Reality?

Parallel decrease of the consumption and increase of the production of energy from biogas leads to a higher independency of a WWTP on external energy supply (local energy distributor). The question is what are the limits and what is the maximum level of energetical self-sufficiency, which can be reached? From a theoretical point of view, there is no such threshold value. Some literature data show, that a properly implemented co-digestion can result in more than 100% of energy self-sufficiency [8,9]. In such a case, the WWTP becomes an independent energy source. Some examples of WWTPs with implemented co-digestion are summarized in Table 2. The data presented in this table represent the result of both reduction of energy consumption and incerase of biogas and energy production by implementing thermophilic digestion or co-digestion of biowastes. These practical examples showed that thanks to co-digestion and other operational improvements, biogas production can be increased several times. Also, the electricity production is increased, for example co-digestion of waste greases led at Gera WWTP (200,000 PE) and Görlitz WWTP (140,000 PE) during the period 2008–2010 to an increase of produced electricity from 1.8 to 2.7 GWh per year and 1.1 to 1.9 GWh per year, respectively. Despite very good performances achieved by these WWTPs, there are still margins for improvements, in order to reach a full energetical self-sufficiency.

Sludge pre-treatment is also one of a possible way to improve the anaerobic digestion performance and increase both biogas and energy production [2] (Figure 1). From the existing practical applications, it can be pointed out mechanical disintegration by technology Lysatec, implemented at Prague WWTP [4,5] and thermal hydrolysis by technology Biothelys or Cambi, implemented at Brussels WWTP [10]. Both technolo-

Table 2. Example of Several European WWTPs with Implemented Co-digestion.

WWTP	Capacity (PE)	Co-substrate	Result	Remark
Pest-South (H)	293,300	100,000 t biowastes/y	90% energy self-sufficiency	2–4× more biogas
Grevesmuhlen (D)	40,000	greases	113% energy self-sufficiency	
Gera (D)	200,000	1.1 M m^3/y (greases, glycerine)	additional 2.8 M m^3 biogas/y	increase of electricity production: 1.8 → 2.7 GWh/y
Görlitz (D)	140,000	1,200 m^3 greases/y	additional 1 M m^3 biogas/y	increase of electricity production: 1.1 → 1.9 GWh/y
Hradec Králové (CZ)	141,000	25 t G-phase/month	24% more biogas	savings 21,000 €/y
Viareggio (I)	100,000	5 t municipal biowaset/d	50% more biogas	
Treviso (I)	75,000	10 t municipal biowaste/d	400% more biogas	

gies increase the VS degradation, and consequently the biogas production. It is reported that Lysatec technology increases the biogas production by 15–20% [4].

Wastewater represents a very precious source of water and elements to be reused and recycled. Organic carbon has to be considered as a possible source of energy. Nitrogen and phosphorus are recognized for their fertilising properties. By introducing external biowastes for co-digestion in WWTPs digesters, the input of all here-above mentioned sources can be increased. Consequently, the WWTP will not be only a wastewater treatment plant, but has to be considered as a center for secondary sources recovery and reuse. The optimal situation will be a complete energetical self-sufficiency (in both heat and electricity), and production of valuable secondary matter: water for re-use, electricity and heat, fertilizer (MAP) and biomass (digested sludge for further energy recovery in power-plants). This concept has to be involved in future municipal strategy of integrated wastewater, biowaste and energy management. All locally available ressources can be thus re-used and recycled, without needs of any further transportation and non-ecological destruction. Moreover, the application of such an integrated strategy will result in both lower carbon footprint and environmental impact.

CONCLUSION

The anaerobic digestion is one of the most suitable technology for sludge treatment, stabilization and energy recovery. In order to increase the process efficiency, several "boosting" strategies have been set-up, as optimisation of sludge thickening, sludge pre-treatment and conditioning, optimisation of the anaerobic digestion (thermophilic regime, optimal mixing, continuous feeding) and implementation of co-digestion. The Anaerobic Digestion Commission of Veolia Water works on these subjects, proposes best practices, facilitates

the exchanges, monitors the operational performances of large WWTP with energy recovery from sludge, and provides plant operators and sludge experts with an up-dated databasis of main performance indicators. Thanks to this commission and the European technical network, various operational innovations and improvements have been realized, resulting in increased energy efficiency of several WWTPs, some from which are currently approaching 100% of energetical self-sufficiency. The main recommendation of this commission is to implement the anaerobic digestion in all WWTPs where it is possible, to optimize its operation according to the ANDC standards, and eventually implement co-digestion of biowastes, with an enhanced energy production. Future trend is to enlarge the function of a WWTP from simple wastewater treatment to a complex recycling and reusing processes.

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New Laboratory Developments for Sludge Flocculation

P. GINISTY* and C. PEUCHOT

IFTS, BP 292, 47007 Agen, France

ABSTRACT: The characterisation of sludge to assess in laboratory its amenability to thickening and dewatering is an operation highly sensitive to the operating procedure adopted for the chemical conditioning step.

The definition of a standardized procedure is needed for the selection of type and dose of a conditioning product at laboratory scale. A new device was developed to ensure the repeatability of sludge flocculation tests independently of the operator, and to prepare a homogenous thickened flocculated sludge for subsequent dewatering tests.

Results obtained with different kind of sludges, polymers and mixing conditions are presented in this paper.

INTRODUCTION

SLUDGE conditioning is generally used to facilitate water removal during subsequent thickening and dewatering operations. It enables to neutralize or destabilize the chemical or physical forces acting on colloidal and particulate matters suspended in the sludge with different mechanisms [1, 2]. This destabilization process brings about the growth of small visible sized particles into larger aggregates [3] known as flocs which have a non defined shape [4]. Chemical conditioning of sludge includes the use of organic polymers, inorganic salts and a combination of both [5]. The choice of suitable chemical agents and optimum dose are very important factors for effective conditioning and subsequent dewatering but another major factor is the degree of mixing required to distribute chemical conditioner in the sludge and to form flocs [6]. Sludge flocculation requires first high velocity (energy) mixing for polymer dispersion then gentle mixing with sufficient time to build the floc size and maximise water release. High energy mixing or excessive mixing time will degrade the floc and generate poor solid-liquid separation [7]. No generalized ranking of products, in order of effectiveness, can be given since the ranking changes with sludge type, dosage and nature of conditioning agent, degree of shearing, time of mixing, etc. . . . .

It is necessary to flocculate sludges in repeatable and quantified conditions for good comparison of products, considering the strong influence of its char-

acteristics (physico-chemical form, charge distribution and density, average molecular weight, ramification degree) and mixing conditions (mixer shape and position, time and energy mixing, polymer injection point and flow rate . . .). In wastewater treatment, an apparatus conventionally called a "jar tester" has been used for many years and is simply a four or six position gang stirrer for agitating the biosolids wastewater in 1 L beakers at a controlled speed (and shear) while polymer flocculants or coagulants are added. Three steps are used: high speed then low speed mixing then no mixing to allow floc settling. The device and protocol can't be applied to sludge conditioning owing to high solids concentration and higher viscosity of flocculated suspension and different chemical kinetics. Each supplier has its own procedure to flocculate sludge. For instance, some consist in pouring the sludge back and forth between 2 beakers to gently flocculate the biosolids [8]. Nevertheless, no standard protocol and equipment still exist.

The importance of this problem has been already recognised by CEN/TC308 that published a Technical Report (CEN/TR 14742, 2006) specifying a procedure for the chemical conditioning of sludges [9]. The corresponding recommended device [10] is a standard stirred beaker with the control of mixing speed and time. To produce a standard, validation through interlaboratory trials, is a necessary step and it is needed to evaluate the efficiency of flocculation to evaluate the repeatability and reproductibility of the procedure on fresh sludges. The drainability of the flocculated sludge could be a parameter, easily and quickly obtainable and a recent published standard (EN 14701-4,

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2010) showed the repeatability and reproductibility of this characterization method specifically.

A new device, based on this principle, was developed to ensure the repeatability of sludge flocculation tests independently of the operator, and to prepare a homogenous thickened flocculated sludge for subsequent dewatering tests. The paper discusses about the repeatability of flocculation step with biological sludges and its dependence to chemical and hydrodynamic parameters.

MATERIALS AND METHODS

Sludge Flocculation

Sludge flocculation experiments were made with a new kind of device called "boo-test" developed by IFTS. It is composed of two sludge flocculators (diameter: 90 mm) and drainage cells with automatic transfer from one to another (Figure 1). The two flocculators are stirred in the same conditions. Time and speed mixing, impeller position (function of the sludge volume) are measured and controlled. Drainage cells are equipped with filter cloth reference Si030904 from Rai-Tillières manufacturer. Filtrate mass during drainage is recorded by a weight sensor connected to a computer.

Figure 1. *Sludge flocculation device ("boo-test").*

Drainage kinetics and separated phases characteristics were chosen to evaluate the repeatability and sensitivity of flocculation. The parameters for drainage kinetics are the ratio filtrate mass/sludge initial mass at 30, 90 and 600 s and the time required to collect a mass of filtrate equal to 50% of the initial sludge. Filtrate was characterized by suspended matters measurement (according to EN 872) and cake by its dryness (according to EN 12880). Floc growth/breakage can be evaluated by different in-line systems (granulometers, rheometers) to determinate the best mixing conditions [11, 12] and dewaterability of flocculated sludges can be quantified by other laboratory tests [13].

Sludge

The experiments were made with an activated urban sludge from a wastewater treatment plant near Agen (France). Sludges were sampled upstream and downstream a gravity thickener to study 2 samples of different dry matter concentration: 1 and 3% w/w. The size of particules, measured by a laser diffraction granulometer (Cilas Instrument) was on average 40 µm.

The sludge was stored at 4°C until maximum 4 days after sampling. Quantity of 350 mL was sampled for each test, and let to reach ambient temperature before conditioning.

Polyelectrolytes

The polyelectrolytes tested (EM 540 BD, 640 CT, 640 MBL, FO 4990 SH) were supplied by SNF FLO-ERGER and were prepared by dilution at 5 g/L. Quantity of polyelectrolyte solution was fixed according to the wanted dose of polyelectrolyte by quantity of dry sludge (kg/T DS).

RESULTS AND DISCUSSION

Repeatibility

The tests were made with the 2 sludges: thickened and not thickened the polyelectrolyte used in the wastewater treatment plant: EM 540 BD at a 14 kg/T DS dosage. Preliminary tests showed that largest flocs are produced with a mixing time of 20 s. A mixing speed of 700 tr/min and an impeller position at 20 mm from the bottom enable to flocculate the whole sludge volume. Flocs formed have a mean size of about 1 mm (Figure 2).

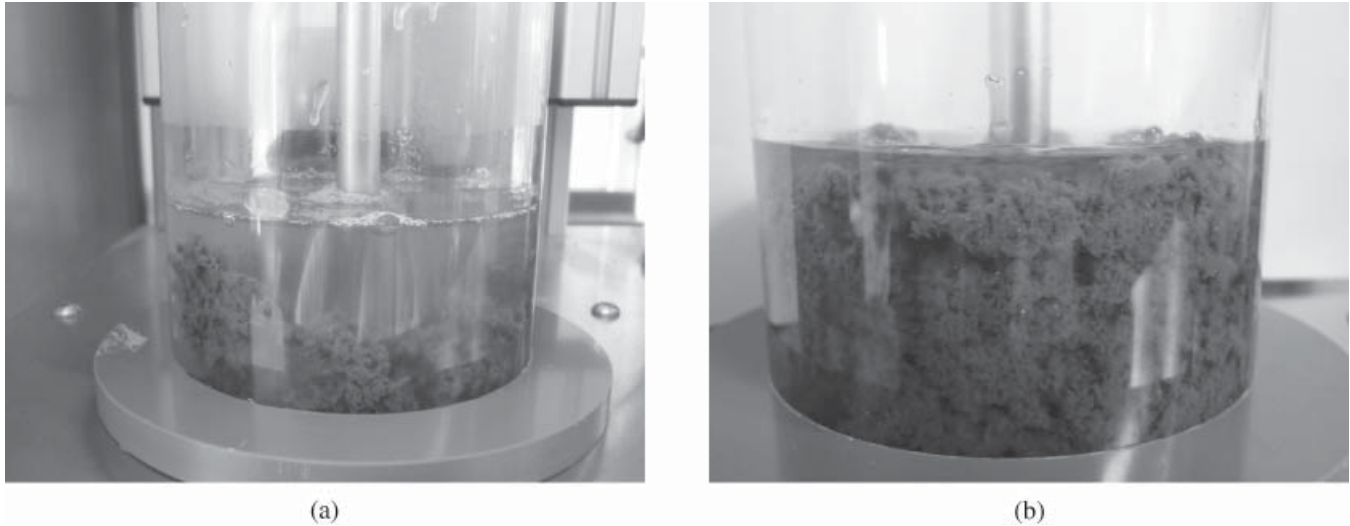Table 1 highlights the excellent repeatability of floc-

Figure 2. *Flocs formed with EM 540 BD and 2 types of sludges: (a) not thickened (b) thickened.*

culation between 2 flocculation cells and 2 types of sludge in comparison with simple drainage tests with transfer of flocculated sludge in an external drainage cell by operator.

Sensitivity

Sludge flocculation requires good mixing with sufficient time to build the floc size and maximize water release. Figure 3 shows that flocs obtained with EM 540 BD achieve their largest size at 20 seconds and

excessive mixing time decreases their mean diameter even if successive flocs degradation/reflocculation mechanisms happen and maintain flocs of lower size during a long period of time (> 130 s).

Mixing speed should be chosen to homogeneously distribute the polymer in the sludge volume without breaking the flocs in formation. A value of 700 rpm is usually sufficient but improvements can be obtained by using higher mixing speed (1700 tr/min) in a very short time (2 s) especially if the sludge is highly concentrated.

Table 1. *Comparison of Repeatability of Flocculation of Not Thickened and Thickened Sludges (EM 540 BD, 14 kg/T MS) on the Basis of Drainage Tests Results (tests made 3 times).*

| Parameter	Statistics	Not Thickened Sludge		Thickened Sludge		
		Cell 1	Cell 2	Cell 1	Cell 2	External Cell
M/M_0 30 s	Average	0.830	0.829	0.565	0.564	0.561
	Std	0.003	0.003	0.004	0.002	0.016
	P (%)	0.316	0.349	0.653	0.328	2.86
M/M_0 90 s	Average	0.835	0.834	0.582	0.581	0.592
	Std	0.002	0.004	0.002	0.002	0.012
	P (%)	0.256	0.423	0.397	0.301	2.03
M/M_0 600 s	Average	0.844	0.844	0.617	0.615	0.619
	Std	0.003	0.003	0.001	0.003	0.010
	P (%)	0.303	0.316	0.171	0.418	1.56
t_e (s)	Average	< 1	< 1	10	10	11.5
	Std	< 1	< 1	1	1	2
	P (%)	< 1	< 1	11	11	17.8
Dryness (%)	Average	6.89	6.78	7.19	7.21	6.5
	Std	0.08	0.03	0.08	0.11	0.16
	P (%)	1.19	0.51	1.16	1.50	2.49
MES (mg/L)	Average	95	95	230	260	104
	Std	6	4	5	20	20
	P (%)	6.80	3.76	2.09	8.96	19
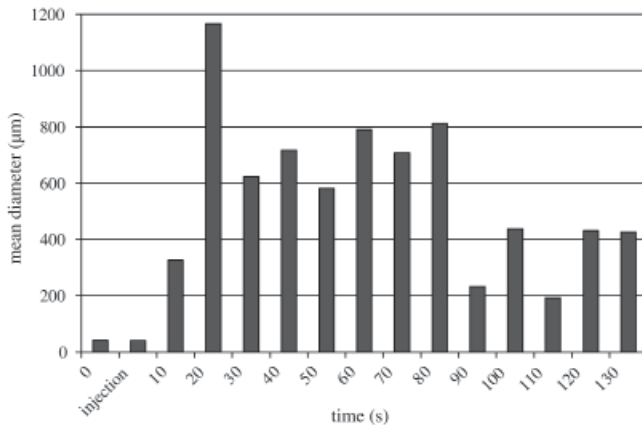
Figure 3. *Flocs formation kinetics with EM540 BD polymer at 14 kg/T DS (mean diameter measured by laser diffraction granulometer).*

The stirrer influence was studied with 3 types of geometry: 3 inclinated blades (120°), 2 horizontal blades (180°) and 4 horizontal blades (90°). Figure 4 shows the influence of the impeller geometry on drainage curves for high concentration sludge and highlights that flocs are less sheared with the 2 horizontal blade stirrer. For low concentration sludge, no significant differences were obtained between the 3 geometries.

The polymer injection parameters are also important, and the polymer injection flow rate during sludge mixing was studied. Figure 5 shows the longer the injection time, the slower the drainage kinetics. A low quantity of polymer allows the formation of small flocs that bridge in larger flocs during polymer injection but

they are less resistant to shearing than flocs immediately formed with a higher quantity of polymer. It is sometimes difficult for the operator to control the flow rate of polymer injection and it is recommended to distribute polymer at the sludge surface before mixing, even if it can diffuse less or more quickly in the sludge and begin to agglomerate particles in microflocs. Automatic polymer injector is under development for the "boo-test" to ensure the control of this parameter.

The influence of polymer injection point was also studied. It was found no significant, provided that mixing is sufficient enough to disperse polymer quickly in the whole sludge volume.

If all the hydrodynamics parameters are controlled and quantified so as to be repeated, the device enables to differentiate the efficiency of chemical parameters (nature of polyelectrolyte, dosing).

For instance Figure 6 presents the volume of filtrate versus time for the thickened sludge treated by different doses of EM 540 BD. The drainage kinetics curves show that filtrate flow rate increases with the polyelectrolyte dose until 14 kg / T DS. The same behaviour was observed for the not thickened sludge.

Figure 7 points out that fastest drainage kinetics are obtained with polymers with cross-linked backbone (EM 540 BD, 640 MBL) comparatively to linear chain polymers (FO 4490 SH, EM 640 CT).

Flocs obtained with cross-linked backbone polymer looked more compact and more resistant to shearing
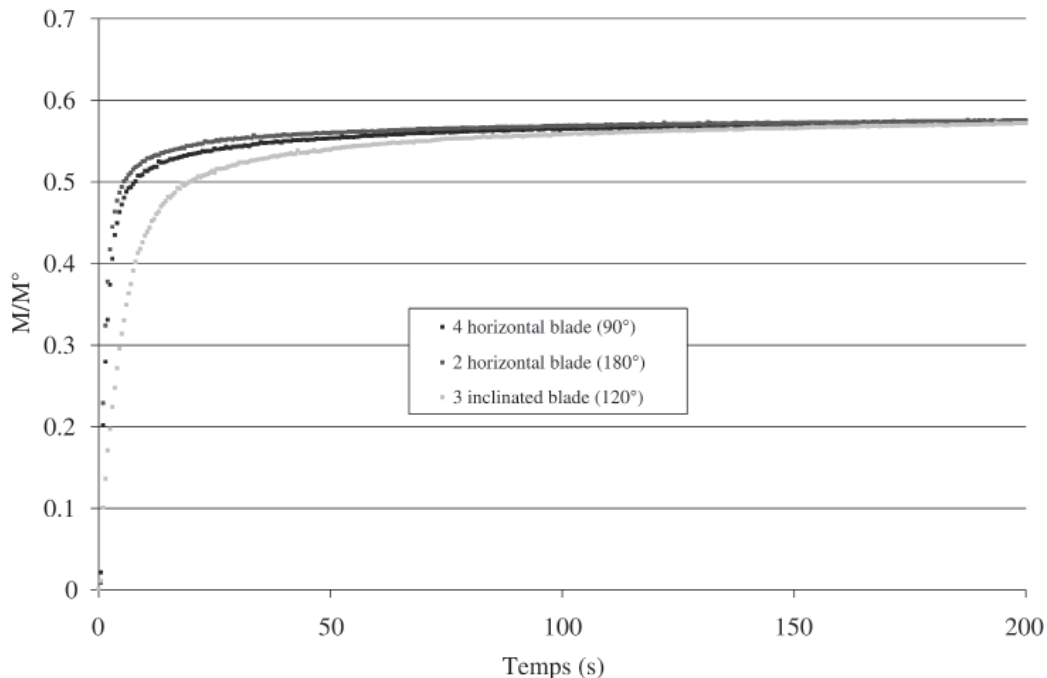

Figure 4. *Influence of impeller geometry on drainage curves of sludge (thickened) flocculated with EM 540 BD at 14 kg/T DS.*
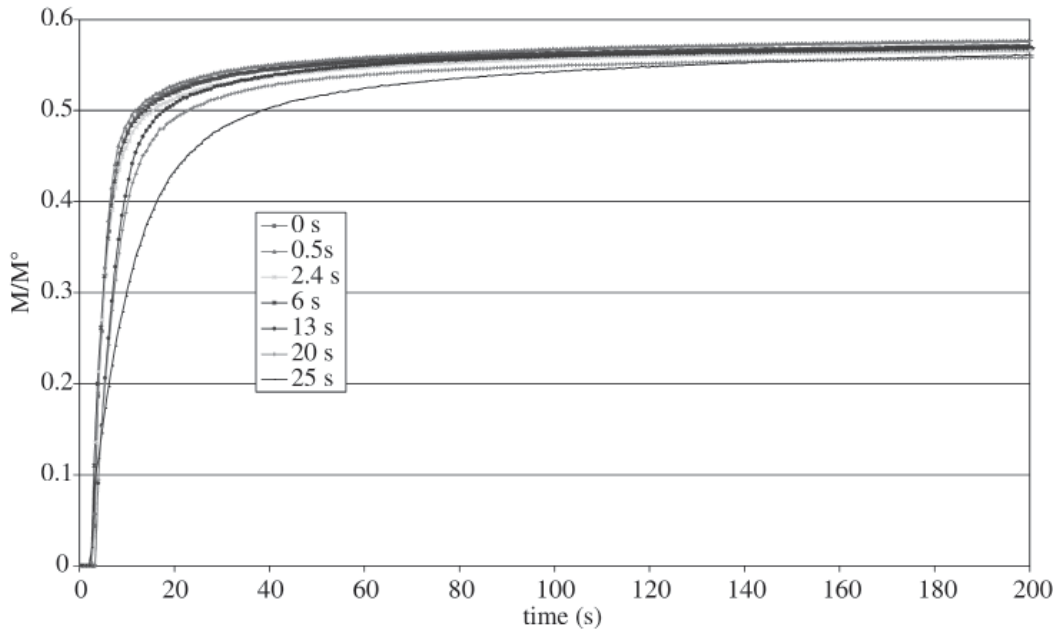
Figure 5. *Influence of polymer injection time during sludge mixing on drainage kinetics of sludge (thickened) flocculated with EM 540 BD at 14 kg/T DS.*

than linear chain polymers and the consequence is a faster release of water during drainage.

CONCLUSION

The paper pointed out the role of main hydrodynamic parameters in sludge flocculation and showed the interest of the device called "boo-test" recently developed by IFTS, necessary for the development of a new standard which requires a good repeatability independently of the operator. The aim is to produce an effective standardized procedure for the conditioning operation when selecting a conditioning product, coagulant or polyelectrolyte, at laboratory scale in view of full scale application.

The application of this standardised procedure will allow the effectiveness of different sludge chemical conditioning agents to be evaluated and their optimal
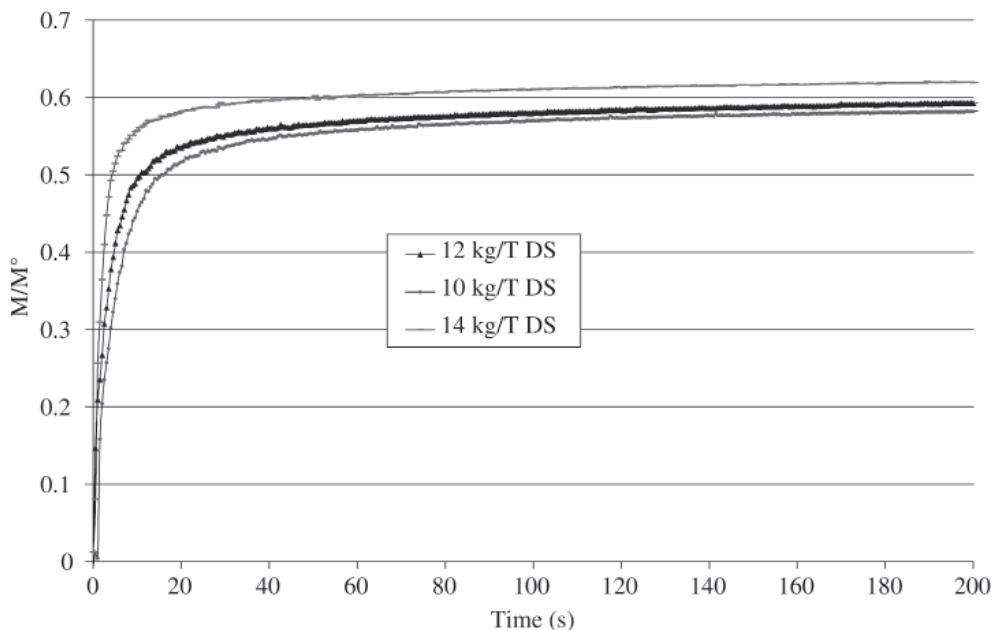

Figure 6. *Influence of EM 540 BD dose on drainage kinetics of thickened sludge.*
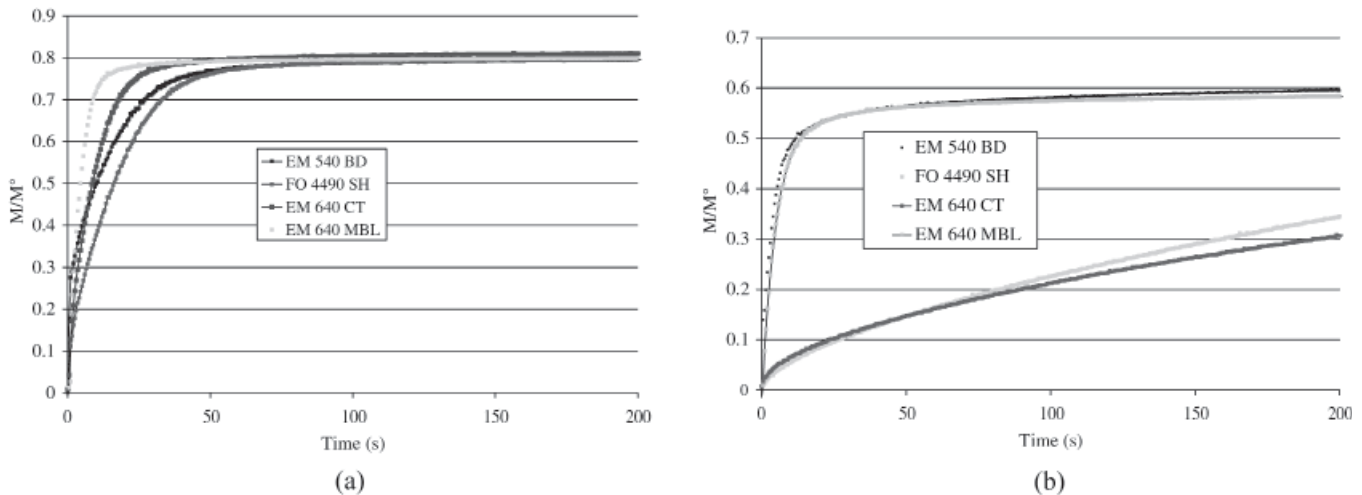
Figure 7. *Flocs formed with EM 540 BD and 2 types of sludges [(a) not thickened, (b) thickened] flocculated with polymers at 14 kg /T DS.*

dosage to be determined for the thickening of sludge in a drainage table. It produces, in a repeatable manner, a homogeneous flocculated thickened sludge that can be used for further tests for sludge dewatering assessment.

Further work is needed to test the equipment and procedure with other sludges in the scope of CEN/TC 308 (digested sludge, waterworks sludge . . .) or not (particular industrial sludge).

ACKNOWLEDGEMENTS

SYMBOLS

DS: dry sludge

M: Mass of filtrate

M_0: initial mass of sludge

MES: suspended matters

P: precision (ratio between standard deviation and average).

Std: standard deviation

T_e: filtration time corresponding to collection of 50% of the initial sludge volume

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Table 5. Comparison of state-of-the-art matrix resins with VPSP/BMI copolymers.

Resin System	Core Temp. (DSC peak)	T_E	Char Yield, %
Epoxy (MY720)	235	250	30
Bismaleimide (H795)	282	>400	48
VPSP/Bismaleimide copolymer			
C379: H795 = 1.9	245	>400	50
C379: H795 = 1.4	285	>400	53

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