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# Volatile fatty acids production from sewage organic matter by combined bioflocculation and anaerobic fermentation



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## HIGHLIGHTS

- Bioflocculation anaerobic VFA system shows high potential for organic recovery.
- High-loaded MBR bioflocculation concentrated more than 75% of sewage COD.
- At an SRT of 5 days VFA yield was 15% of sewage COD, but more than 24% is feasible.

#### GRAPHICAL ABSTRACT



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### ABSTRACT

This work aims at exploring the feasibility of a combined process bioflocculation to concentrate sewage organic matter and anaerobic fermentation to produce volatile fatty acids (VFA). Bioflocculation, using a high-loaded aerobic membrane bioreactor (HL-MBR), was operated at an HRT of 1 h and an SRT of 1 day. The HL-MBR process removed on average 83% of sewage COD, while only 10% of nitrogen and phosphorus was removed. During anaerobic fermentation of HL-MBR concentrate at an SRT of 5 days and 35 °C, specific VFA production rate of 282 mg VFA-COD/g VSS could be reached and consisted of 50% acetate, 40% propionate and 10% butyrate. More than 75% of sewage COD was diverted to the concentrate, but only 15% sewage COD was recovered as VFA, due to incomplete VSS degradation at the short treatment time applied. This shows that combined process for the VFA production is technologically feasible and needs further optimization.

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## 1. Introduction

Conventional (aerobic) municipal wastewater treatment plants are designed to remove organic matter and nutrients such that an effluent quality is produced that meets the discharge guidelines. Instead of a waste, municipal wastewater recently has started to be considered a valuable resource in terms of reusable water,

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energy and nutrients (Stowa, 2010; Wang et al., 2012). Unfortunately, generally the concentration of organic matter in sewage is low and sewage has a relatively low temperature of e.g. 10-20 °C (Metcalf and Eddy, 2004). These characteristics prevent the direct production of valuable resources from organic matter, such as methane or volatile fatty acids (VFA), and makes pre-treatment to concentrate the organic matter necessary. Aerobic bioflocculation of raw sewage in a high-loaded membrane bioreactor (HL-MBR) is a promising technique to accomplish such a concentration step, while at the same time it can produce a water quality that is fit for reuse (Akanyeti et al., 2010; Faust et al., 2014).





Often anaerobic digestion is applied to reduce the amount of primary sludge (PS) and secondary activated sludge (AS) and to produce methane from these solids (Lettinga, 1995). This process consists of four subsequent steps: hydrolysis, acidogenesis, acetogenesis and methanogenesis. Complete digestion results in the production of methane, whereas the first three steps have VFA as the main end product. Production of VFA is useful, as they are the starting compounds for subsequent production of a wide range of higher value products, such as electricity, hydrogen gas, medium chain fatty acids, and bioplastics. The VFA can also be used to enhance biological nutrient removal (Lee et al., 2014). However, to produce VFA, methanogenesis should be avoided. This can be accomplished by applying a short solid retention time (SRT) to wash-out the methanogenic microorganisms and/or by operating the anaerobic reactors at extreme pH values. For example, no detectable methane production was found in anaerobic fermenters operated at extremely low pH (pH 4) or extremely high pH (pH 10-11) (Chen et al., 2007; Yu et al., 2013; Yuan et al., 2006).

Previous studies on solids hydrolysis and VFA production from sewage organic matter were conducted with PS, AS, or a mixture of these. The higher fraction of biodegradable organic matter in PS gives a higher VFA yield per gram of solids compared to AS or a mixture of PS and AS (Ucisik and Henze, 2008; Yuan et al., 2009). Ucisik and Henze (2008) reported a specific VFA production of 270 mg chemical oxygen demand (COD)/g of volatile suspended solids (VSS) from PS and this VFA was composed of 50% acetate, 35% propionate, 10% butyrate, and 5% other VFA. This result is in line with the study by Ferreiro and Soto (2003), who found a specific VFA production of 170 –370 mg COD/g VSS of PS and a VFA composition of 37–60% acetate, 30–55% propionate and 8–20% butyrate.

Akanyeti et al. (2010) reported that with a combination of aerobic bioflocculation and subsequent anaerobic digestion at least 35% of sewage COD can be converted to methane. This yield is much higher than a methane recovery of 18% when PS and/or a mixture of PS and AS are digested (Cao, 2011). This is because the bioflocculation process not only concentrates the COD that is contained in the settleable solids, but also all of the suspended COD, colloidal COD and even part of the soluble COD. Besides, with bioflocculation aerobic mineralization of organic matter, taking place in conventional activated sludge (CAS) systems, is largely avoided. For example, Faust et al. (2014) showed that, given proper operational conditions, excellent bioflocculation is possible, as only 10-15% of the COD load is lost by mineralization. Model calculations by Khiewwijit et al. (2015) also showed a high potential COD recovery. However, the model calculations focussed on methane production rather than on the production of more valuable VFA.

In the literature no information is available about the COD recovery that can be achieved by combined bioflocculation and VFA production. Therefore, in this study the performance of this combination was further investigated (Fig. 1), focusing on solids degradation, VFA production, VFA composition, and nitrogen (N) and phosphorus (P) release. For this purpose an HL-MBR was used for the bioflocculation process and an anaerobic sequencing batch reactor (SBR) for subsequent VFA production from the concentrate that was produced by the HL-MBR.

#### 2. Methods

#### 2.1. Municipal wastewater characteristics

Municipal wastewater was collected from a school and a few households nearby this school (Van Hall School Leeuwarden, The Netherlands). The wastewater first passed a sedimentation column



Fig. 1. Combined bioflocculation using a high-loaded MBR (HL-MBR) to concentrate sewage organic matter and sequencing batch reactor VFA production reactor (SBR).

#### Table 1

Average characteristics of municipal wastewater fed to HL-MBR process. (Concentrations are average values calculated from 56 grab samples taken over a period of 195 days. Standard deviations are shown between brackets.)

Analysis	Unit	Average value
Total COD	mg COD/L	310 (113)
Suspended COD	mg COD/L	162 (88)
Colloidal COD	mg COD/L	64 (49)
Soluble COD, COD <sub>s</sub>	mg COD/L	84 (40)
NH <sub>4</sub> -N	mg N/L	34 (13)
PO <sub>4</sub> -P	mg P/L	5 (2)

to remove heavy inert particles like sand and was stored in a stirred buffer tank.

Table 1 gives a summary of the most important characteristics of the wastewater that was collected in the buffer tank. Occasional comparison of these characteristics with those of the raw wastewater confirmed that no significant changes in total COD, NH<sub>4</sub>-N and PO<sub>4</sub>-P took place in the sedimentation column. In the column less than 3% of the suspended COD was removed.

#### 2.2. HL-MBR bioflocculation

The HL-MBR was operated at a hydraulic retention time (HRT) of  $1.0 \pm 0.1$  h and an SRT of  $1.0 \pm 0.1$  days to optimize flocculation and at the same time to minimize (aerobic) organic matter mineralization. The reactor design was the same as used by Faust et al. (2014) and by Akanyeti et al. (2010). The working volume of the reactor was 2.6 L and the reactor was equipped with two submerged flat sheet membranes (Kubota Corporation, UK). The chlorinated polyethylene membrane sheets had a surface area of  $0.124 \text{ m}^2$  and an average nominal pore-size of  $0.2 \ \mu\text{m}$ . Aeration and mixing were accomplished by pressurized air to maintain the minimum dissolved oxygen (DO) concentration of  $2 \text{ mg O}_2/L$ . This was checked with an online oxygen sensor (Oxymax COS22D, Endress + Hauser). Peristaltic pumps (Masterflex L/S, Cole-Parmer) were used to feed the wastewater and for permeate and concentrate production. To reduce membrane fouling the permeate pump was operated in cycles of 15 min permeation followed by 5 min relaxation. The concentrate pump was operated in cycles of 1 min concentrate production, followed by 59 min relaxation. A PVC pipe of 3.5 cm diameter and 30 cm height was used to control the liquid level in the reactor. The membranes were cleaned mechanically by milli-Q water spraying at least once a day in order to remove a gel layer that was formed on the membrane surface during filtration.

#### 2.3. Fermentation of HL-MBR concentrate

Approximately 400 mL sludge (19 g VSS/L) from an anaerobic digester treating a mixture of PS and AS (wastewater treatment

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