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# High-rate anaerobic co-digestion of kraft mill fibre sludge and activated sludge by CSTRs with sludge recirculation



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## ABSTRACT

Kraft fibre sludge from the pulp and paper industry constitutes a new, widely available substrate for the biogas production industry, with high methane potential. In this study, anaerobic digestion of kraft fibre sludge was examined by applying continuously stirred tank reactors (CSTR) with sludge recirculation. Two lab-scale reactors (4L) were run for 800 days, one on fibre sludge (R1), and the other on fibre sludge and activated sludge (R2). Additions of Mg, K and S stabilized reactor performance. Furthermore, the Ca:Mg ratio was important, and a stable process was achieved at a ratio below 16:1. Foaming was abated by short but frequent mixing. Co-digestion of fibre sludge and activated sludge resulted in more robust conditions, and high-rate operation at stable conditions was achieved at an organic loading rate of 4 g volatile solids (VS)  $L^{-1}$  day<sup>-1</sup>, a hydraulic retention time of 4 days and a methane production of 230 ± 10 Nm L per g VS.

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# 1. Introduction

In the light of the Paris agreement on climate change (UNFCCC, 2015), it is evident that the world needs to step up its efforts to reduce greenhouse gas emissions. One way would be to expand the generation of renewable energy by producing more biogas; however, this puts a greater demand on the availability of potent substrates for biogas production.

Ekstrand et al. (2013a) showed that the fibrous fraction of kraft pulp and paper mill wastewaters contain high amounts of organic matter that is easily accessible for methane production. Since kraft pulping makes up more than 70% of the total pulp production in the world (FAOSTAT, 2011), residual kraft fibres constitute an important potential substrate that has, so far, been overlooked in the biogas industry. The anaerobic digestion of kraft fibre sludge has to the authors knowledge only been addressed in one previous

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study, however, that experiment was conducted at relatively low organic loading rates and long retention times (Bayr and Rintala, 2012). An advantage of using kraft pulp fibre sludge for anaerobic digestion (AD), in comparison to most other available lignocellulosic substrates, is that in a sense the fibres have already been pre-treated. The cooking of wood chips at high temperature and pressure in the presence of NaOH and Na<sub>2</sub>S has broken up rigid crystalline cellulose structures and dissolved most of the lignin (Bierman, 1993; Pokhrel and Viraraghavan, 2004). However, one important challenge that needs to be resolved in order to treat this type of waste at full scale is the large wastewater flows. Consequently, this study aims to investigate if high-rate AD of kraft pulp fibre sludge is possible.

Since the pulp fibres largely consist of carbohydrates, they are a nutrient-poor substrate. Consequently there is a need to supply complementary nutrients, such as nitrogen, phosphorous and trace metals, in order to sustain a growing and active biomass (Scherer et al., 1983). Still, excessive supplementation should be avoided as it leads to increased operational costs, hence the approach of this study was to keep supplements to a minimum and to implement additions only when needed.

Aside from fibre sludge, another significant waste stream at the mills is the excess sludge (activated sludge) from the conventional aerated wastewater treatment. Due to high sludge disposal costs, this treatment process is frequently optimized for low sludge



Abbreviations: COD, chemical oxygen demand; CSTR, continuous stirred tank reactor; HRT, hydraulic retention time; OLR, organic loading rate; SRT, sludge retention time; TS, total solids; VFA, volatile fatty acids; VS, volatile solids.

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production, which in turn leads to an increased demand on aeration. Aeration in biological wastewater treatment often requires more than 50% of the electricity used in a wastewater treatment plant (Stoica et al., 2009). One way of reducing the demand on low sludge production and thereby reducing the electricity requirement could be to use the activated sludge as a cosubstrate during AD of fibre sludge. Thereby, the activated sludge would be regarded as a substrate for biogas production rather than a costly waste stream.

The addition of activated sludge would also lead to a decrease in sludge disposal costs, since AD will reduce the volumes of activated sludge by converting the organic matter to CO<sub>2</sub> and CH<sub>4</sub>. Wastewater treatment sludge is generally regarded as the largest waste stream in the pulp and paper industry in terms of volume (Monte et al., 2009), which emphasises that sludge reduction is important to consider. In addition, Berg et al. (2011) showed that the dewatering-ability of the activated sludge is improved by AD. This would mean a reduced need for polymer addition and a lower electricity consumption during the dewatering of the sludges, which are often incinerated at no or low energy gains (Stoica et al., 2009).

In summary, not only would co-digestion of fibre sludge with activated sludge be of benefit for the mills, it could also decrease the need for complementary nutrients for the AD process and thus further reduce operational costs. Therefore, an important aspect of this study was to investigate whether the activated sludge can be included as a substrate during AD of fibre sludge at maintained or possibly improved process performance levels.

Typically, AD of fibrous waste would be carried out in a conventional continuously stirred tank reactor (CSTR), but the large wastewater volumes of fibre sludge and activated sludge would cause a washout of the microorganisms in this type of reactor. However, by decoupling the hydraulic retention time (HRT) from the sludge retention time (SRT), higher volumes of wastewater can be treated at shorter HRT without risking microbial washout. A way to achieve this would be to recirculate concentrated reactor sludge. Thereby, large volumes of wastewater could be treated in reasonably sized reactors, while still maintaining the necessary population of active microorganisms in the reactor.

Thus, the aim of this study was to investigate the possibility of performing AD of kraft pulp fibres at low HRT using a CSTR with sludge recirculation, both with and without the inclusion of activated sludge as co-substrate to the process.

#### 2. Material and methods

#### 2.1. Experimental set-up

Two glass CSTRs (R1 and R2) with a working volume of 4L were run at 37 °C for 800 days. The inoculum was a mixture of AD sludge from a municipal wastewater treatment plant (Linköping, Sweden), activated sludge from a pulp and paper mill, and sludge from a lab-scale reactor treating fibre sludge under anaerobic conditions.

To prolong the SRT of the experimental reactors, reactor sludge was withdrawn from the CSTRs once a day and centrifuged (2–4 min at 2300 RCF; Heraeus Megafuge 16, Thermo Scientific). Part of the centrifuge reject was discarded, and concentrated sludge was re-suspended together with the substrate and returned to the CSTRs to obtain a total solids (TS) level of 3.0–3.5%. This procedure gave a HRT of 8 days and a SRT of about 16 days, which was altered stepwise during Phase IV (see below) to give a HRT of 4 days and a SRT of about 10 days. For details regarding feeding volumes, sludge recirculation and discarding of sludge, see Table S1.

The experiment was divided into four phases. During Phase I (days 1-36), both reactors were given fibre sludge at organic loading rates (OLRs) of 0.5–1 g volatile solids (VS)  $L^{-1}$  day<sup>-1</sup>. During Phase II (days 37-283), activated sludge was introduced as a cosubstrate to R2, but not to R1, to investigate whether there were any positive or negative effects of co-digestion in comparison to mono-digestion. Both reactors were supplied with the same amount of fibre sludge (OLR ranging  $0.5-4 \text{ g VS } \text{L}^{-1} \text{ day}^{-1}$ ) throughout the period, except during process disturbances. The addition of activated sludge was based on the actual sludge production at the pulp and paper mill from which the substrates were collected (TS ratio of 11:1 for fibre sludge and activated sludge), and corresponded to a 0.08–0.2 g VS L<sup>-1</sup> day<sup>-1</sup> higher OLR for R2 compared to R1. From day 284 (Phase III), both reactors were fed with fibre sludge and activated sludge, and the possibility of running the co-digestion process at a higher OLR was investigated. After 24 days of co-digestion at an OLR of  $3 \text{ g VS } \text{L}^{-1} \text{ dav}^{-1}$ . the OLR of fibre sludge in both reactors was increased to 4 g VS L<sup>-1</sup> day<sup>-1</sup> for 3 days. This increase was repeated after another 43 days and lasted between days 326-359. At the end of this phase, the OLR was returned to 3 g VS  $L^{-1}$  day<sup>-1</sup>, in preparation for Phase IV. The HRT was kept constant at 8 days. During days 462-800 (Phase IV), the HRT was lowered stepwise to 4 days in both reactors, followed by an increase in OLR to 4 g VS L<sup>-1</sup> day<sup>-1</sup> of fibre sludge in R2. For this period, R1 worked as a control for R2, meaning that each change was first implemented in R2, then in R1. To initiate this phase, sludge from both reactors was withdrawn, mixed and returned to the reactors on days 457 and 458, in order to ensure equal system properties before starting the alterations. Then, the HRT was reduced to 6 days (on day 460 for R2 and 542 for R1) and to 4 days (on day 658 for R2, 688 for R1). This meant a decrease in SRT from  $16 \pm 1$  to  $12 \pm 1$  days for both reactors, due to the increased amount of centrifuge reject leaving the system (Table S1). Lastly, the OLR in R2 was increased to  $3.5 \text{ g VS } \text{L}^{-1} \text{ day}^{-1}$  on day 690 and to 4 g VS  $\text{L}^{-1} \text{ day}^{-1}$  on day 703.

The fibre sludge was initially added to the reactors together with water to maintain a HRT of 8 days. However, upon increasing the OLR during Phase II, less water was needed. Starting from day 111 the fibre sludge was thickened by filtration (125  $\mu$ m, Test Sieve, Retsch, Germany) prior to feeding, to allow for an increase in OLR without altering the HRT. The resulting fibre sludge filtrate was used to adjust the feeding volume in order to maintain the desired HRT. When decreasing the HRT to 6 days, the feed was temporarily supplemented with tap water, 71 days for R1 and 87 days for R2, to be able to separate the effect of reducing the HRT from the increase in feed of fibre sludge filtrate. Due to a process disturbance at the mill, one of the fibre batches had to be discarded, and during days 76–85 the reactors were fed with pure pulp instead of fibre sludge (Fig. 1).

Intermittent mixing of the reactors was conducted by an internal impeller ( $\varnothing$  70 mm, height 30 mm) driven by a servomotor (MAC050-A1; All motion Technology). Initially, the reactors were mixed 4–5 times a day at 150–400 RPM for 15 min, but the duration and frequency was adjusted to 4-min intervals at 400 RPM 20 times a day from day 248 to avoid fibre accumulation at the surface.

The pH of the digester liquid was controlled by adding,  $Ca(OH)_2$  at a rate of 0.1–1.0 g  $Ca(OH)_2/L$ , to buffer the acidity of the inoculum and degradation products of the substrate. From day 49, part of the  $Ca(OH)_2$  was replaced by MgO, to achieve a mass ratio of calcium to magnesium of 2.5:1.0, considered optimum for growth of methanogens (Zehnder et al., 1980). As a result, 0.30 g  $Ca(OH)_2/L$  and 0.11 g MgO/L were added to R1 and 0.38 g  $Ca(OH)_2/L$  and 0.14 g MgO/L were added to R2. The higher amount of alkalinity added to R2 was needed to compensate for the low pH of the activated sludge (pH 6.5). From day 153, the mass ratio of Ca:Mg was

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