Conversion of boreal lake sedimented pulp mill fibre into biogas: a two stage hydrogen and methane production

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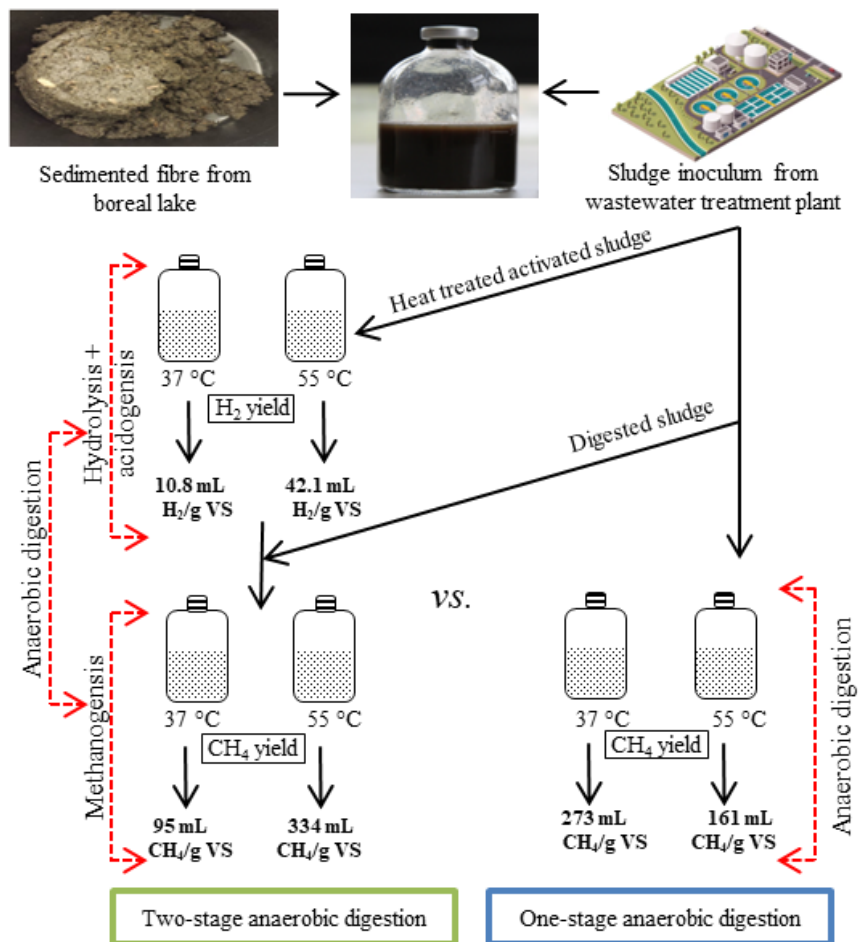
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Abstract

Possibility of producing hydrogen and methane from sedimented pulp and paper mill waste fibre was explored for the first time in a double stage process. Hydrogen and methane production was compared in batch experiments under four different conditions: two-stage hydrogen and methane production under (i) mesophilic (37 °C) and (ii) thermophilic (55 °C), and one-stage methane production under (iii) mesophilic and (iv) thermophilic conditions. Among these conditions studied, two-stage thermophilic anaerobic digestion achieved the highest hydrogen yield (42.1 ± 2.91 mL/g VS) and methane yield (334 ± 26.8 mL/g VS) at 55 °C. The experimental results were fitted to modified Gompertz equation and a strong correlation was built from the overall magnitude of the regression (R^2ranged from 0.996to 0.989) between the experimental data and the applied equation. Total energy yield from the two-stage thermophilic process was higher (3.7 kWh/L) than the one-stage process (1.7 kWh/L). The two-stage treatment also reduced the treatment time by half. Knowledge gained from this study will provide a basis for future investigation of two-stage treatment of sedimented fibres.

Keywords: Dark fermentation, two-stage digestion, sedimented fibre, mesophilic, thermophilic

Declarations

Conflict of interest: The authors declare no competing interests.

Graphical abstract

Sedimented fibre from boreal lake

Sludge inoculum from wastewater treatment plant

Heat treated activated sludge

Hydrolysis + acidogensis

37 °C   55 °C

H₂ yield

10.8 mL H₂/g VS   42.1 mL H₂/g VS

Digested sludge

Anaerobic digestion

Methanogensis

vs.

37 °C   55 °C

CH₄ yield

95 mL CH₄/g VS   334 mL CH₄/g VS

37 °C   55 °C

CH₄ yield

273 mL CH₄/g VS   161 mL CH₄/g VS

Anaerobic digestion

Two-stage anaerobic digestion

One-stage anaerobic digestion

1. Introduction

Pulp and paper industry started in Finland about 150 years ago, and is now the fifth largest pulp and paper producer in the world (Finnish Forest Industries). This industry consumes huge amounts of fresh water in different stages of the process (300–2600 m^3 of fresh water per ton of paper produced) [1], which results in huge quantity of wastewaters. For decades, pulp and paper wastewaters containing fibre residues were discharged without any treatment, which resulted in the sedimentation of solid fibres in the nearby natural water reservoirs. Depending on the pulping process, the constituents of the sedimented fibres are different but cellulose, hemicellulose and lignin are the main components [2]. Kokko et al., and Chatterjee et al., [3, 4] have successfully treated sedimented fibres, from pulp and paper mill waste, excavated from the bay of the Lake Näsijärvi (Tampere, Finland) with anaerobic digestion along with simultaneous methane production. There is 1.5 million m^3 of sedimented fibres in the bay of Lake Näsijärvi and anaerobic treatment with simultaneous biogas production is a potential approach for treating the sedimented fibres [4].

Lignocellulosic materials are recognized as the largest source of monosugars (C5 and C6), which are the main fermentable substrates for biofuels and biochemicals production [5]. Anaerobic digestion is accomplished through four consecutive steps: hydrolysis, acidogenesis, acetogenesis and methanogenesis by different species of microorganisms to degrade the complex substances into biogas [6]. This process can be separated into two-stages for sequential hydrogen fermentation, involving hydrolysis and acidogenesis (volatile fatty acids (VFA) production), and methane production (VFA utilization), involving acetogenesis and methanogenesis, under controlled conditions. Higher organic loading rates (OLRs) and process stability via separation of hydrolysis and acidogenesis from the methanogenesis process can be achieved in a two-stage process [7]. In addition, it has been reported that two-stage process achieves higher energy conversion efficiency and a possibility for higher waste treatment efficiency compared to the one-stage process [8]. Two-stage process allows running each step at different operational conditions favourable for the microbial community at each stage. The OLR can be increased in the first hydrogen fermentation stage and the VFA-rich effluent of this stage can be optimized for the second methanogenic stage [9]. The second stage can have higher hydraulic retention time (HRT) (larger reactor volume) that ensures that the VFA load is not too high. The produced methane could be upgraded and used as a fuel or injected to the natural gas grid [10]. Hydrogen, produced in the first stage, could be mixed with methane to form hythane [11], burned in fuel cell for electricity generation [12], or used as a feedstock for many industries [13]. Different substrates have been tested for the two-stage biohydrogen and biomethane production processes, such as food waste [7], cassava stillage [14] and water hyacinth [15], with hydrogen and methane yields of 65 and 546 mL/g volatile solids (VS), 14 and 249 mL/g VS, and 51.7 and 143.4 mL/g VS, respectively.

Temperature is one of the key parameters affecting fermentation process as different temperatures can enhance or inhibit the growth of microbiota involved in the process [16]. In addition, kinetics and thermodynamics of the hydrogen production reactions depend on the reaction temperature [17]. Some hydrogen-consuming bacteria, such as homoacetogens, are inhibited at higher temperatures [18]. In addition, high temperature decreases the hydrogen solubility in the liquid phase and consequently decreases the hydrogen partial pressure leading to higher hydrogen yield [19]. While thermophilic conditions positively affect reaction kinetics resulting in higher

hydrolysis rates and consequently higher hydrogen production [20], mesophilic fermentation is often applied due to smaller energy requirements and suitability for the growth of most microorganisms [21].

The objective of the current work was to compare the efficiency of conversion the organic matter contained in boreal lake sedimented pulp mill fibre into biogas through four different scenarios. Here, we tested mesophilic vs, thermophilic anaerobic treatment of sedimented fibres (SF) for hydrogen production. In the next step, the effluents of the hydrogen production were converted into methane in anaerobic digestion. The hydrogen and methane yields as well as the VS removal of the two-stage process were compared to the one-stage conversion of sedimented fibres to methane at mesophilic and thermophilic conditions. Besides, we documented the treatment performance of the four tested scenarios.

2. Materials and methods

2.1. Sedimented fibres and inocula

The substrate, sedimented fibres, was obtained near a discharge point of an old pulp and paper facility in the Lake Näsijärvi (Tampere, Finland). A sampling ferry (Ramboll Finland Oy) excavated the samples from the bottom of the bay in the late summer of 2018 [4]. The sample was kept in a dark cold room at 4 °C. The sample was mixed manually and coarse wood pieces were removed before use. Total solids (TS), VS and pH of the sedimented fibre samples used in this experiment were 10.5±0.16%, 9.4±0.17% and 4.7±0.3, respectively (Table 1).

Activated sludge and digested sludge collected from Viinikanlahti municipal wastewater treatment plant (Tampere, Finland) were used as inoculum for biohydrogen and biomethane production, respectively. The activated sludge was settled (supernatant was discarded after settling for two hours) and boiled in 10 mL tubes containing 5 mL activated sludge for 30 minutes to kill methanogens according to El-Qelish et al., [22]. The digested sludge was collected from a mesophilic (35 °C) anaerobic digester treating the surplus sewage sludge and was settled for one hour (supernatant was discarded) before use as inoculum for methane production. The characteristics of both inocula after pretreatments are given in Table 1.

Table 1

Characteristics of sedimented fibre and inoculum sludge.

2.2. Batch assays

Two-stage biohydrogen production potential (BHP) and biomethane production potential (BMP) assays of sedimented fibre were conducted using 560 mL serum bottles in mesophilic (37 °C) and thermophilic (55 °C) conditions. In hydrogen production step, 100 g (55.2 g VS) of pretreated activated sludge, 24 g (21.6 g VS) of sedimented fibre and 4.5 mL of phosphate buffer (10.7 g NaH_2PO_4 and 3.2 g Na_2HPO_4 per liter) were added to the bottles and completed with Milli-Q water up to 200 mL. A control set was done with the same constituents, where the sedimented fibre was replaced by Milli-Q water to keep the liquid volume of all bottles at 200 mL.

The initial pH was adjusted to 6.4 ± 0.1 using 6 M HCl and 6 M NaOH solution. After 7 days when the hydrogen yield stabilized, 15 mL liquid sample was collected from each bottle to analyse the pH, TS, VS, total chemical oxygen demand (COD_{tot}), soluble chemical oxygen demand (COD_{sol}) and VFA. After liquid samples were taken, bottles were closed, sealed anaerobically and kept in dark at 4 °C for one night before being subjected to biomethane production step.

The residual of BHP assays (185 mL), including the control set, were subjected to BMP assays. This was done by adding 185 mL of the BHP assay effluent to 100 mL of digested sludge. BMP of sedimented fibres without BHP step was also determined by adding 28 g of sedimented fibre to 100 mL of digested sludge and 67 mL of $NaHCO_3$ buffer (42 g/L solution) and completed up to 285 mL with 90 mL of Milli-Q water. A control set for the biomethane production from the digested sludge was done with the same constituents, where the sedimented fibre was replaced by Milli-Q water to keep the liquid volume of all bottles at 285 mL. The pH of all bottles was between 7.1-8.0.

All bottles, both for BHP and BMP, were purged with nitrogen gas for three minutes, closed with butyl rubber stoppers and sealed with aluminium lid. Two static incubators at mesophilic (37 °C) and thermophilic (55 °C) conditions were used to house the bottles. All the batch assays were conducted in triplicate.

2.3. Analyses and calculations

The volume of the gas (H_2 and CO_2) produced in the BHP assays was quantified every day by syringe method [23]. The gas constituents were analyzed with a Shimadzu gas chromatograph (GC-2014) with a thermal conductivity detector according to Nissilä et al., [24]. Cumulative H_2 production were determined using the mass balance equation of Logan et al., [25], after the hydrogen or methane content of the controls was excluded. The CH_4 produced in the BMP assays was measured three times a week with a Perkin Elmer Clarus 500 with flame ionization detector (GC-FID) with a Mol-Sieve 5A PLOT column [3]. Methane volume was quantified considering the CH_4 percentage in the headspace according to Angelidaki et al., [26]. The actual methane production was estimated by subtracting the methane produced of the test samples from the methane produced from the control. A weather monitoring station was used to determine the temperature and pressure during the analysis process (wireless digital weather station-WH1080). The gas volumes were converted to standard temperature and pressure (0 °C, 1 bar). Both hydrogen and methane yields were calculated against the VS concentration added to the BHP assays (for BHP or two-stage anaerobic treatment) or against the VS concentration added to the BMP assays.

Modified Gompertz equation was applied to correlate cumulative hydrogen (equation 1) and methane (equation 2) production potentials as follows:

Equation 1:

$$H(t) = p \, x \, \exp\left\{-\exp\left[\frac{R_m e}{p}\left(\lambda - t\right) + 1\right]\right\}$$

Where H, represents cumulative hydrogen production (mL) at reaction time (t); P is the hydrogen production potential; R_m, maximum rate of hydrogen formation (mL-H_2/h) and λ, the duration of lag phase (h) [27].

Equation 2:

$$CH4(t) = p \, x \, \exp\left\{-\exp\left[\frac{R_m e}{p}\,(\lambda - t) + 1\right]\right\}$$

Where CH_4, is the cumulative methane production (mL) at time (t); P is the methane production potential; R_m, maximum methane production rate (mL-CH_4/d) and λ, is the duration of lag phase (d) [28].

Liquid samples were analyzed for VFA content (acetate, propionate, butyrate, isobutyrate and valerate) using a Shimadzu gas chromatograph GC-2010 Plus equipped with a flame ionization (FID) detector according to Kokko et al., [3]. COD_{tot} and COD_{sol} were measured according to the Finnish Standard SFS-5504 (1988). COD_{sol} was measured after filtering the samples with 0.45 μm using filter paper (Whatman). TS and VS were measured according to APHA standard procedures [29].

In order to assess significant differences between mesophilic and thermophilic inoculations, statistical analysis of experimental data was performed using analysis of variance (ANOVA) and the Tukey test at $p < 0.05$ applying the IBM SPSS Statistics package 24 [30].

2.4. Energy calculations

Energy yield of the one- and two-stage anaerobic treatment process was evaluated based on the hydrogen and methane yields. Energy value of 3.5 Wh/L was used to convert hydrogen yield to energy [31] and 10.5 Wh/L for converting methane yield to energy [32]. As the biodegradable-organic content of the sedimented fibre is the fraction which is converted into biogas (H_2 and CH_4) and according to our previous publication [3], it is calculated that only 12% (0.19 million m^3) of the whole amount of the sediment fibre (1.5 million m^3) could be converted into biogas. Based on these conditions, the energy yield of biodegradable-organic content of sedimented fibres was compared at mesophilic and thermophilic conditions. The energy used for heating and mixing has not been taken into account.

3. Results and discussion

3.1. Hydrogen production from sedimented fibres

Hydrogen production started after a lag time of 2 and 4 days at thermophilic and mesophilic conditions, respectively (Fig. 1). Hydrogen yield at thermophilic conditions was 4 times (42.1 ± 2.91 mL H_2/gVS) the yield at mesophilic conditions (10.8 ± 2.89 mL H_2/gVS) in 7 days for the sedimented fibre with an initial VS of 9.4%. No methane was detected in any of the BHP assays in the present study. Earlier, Lin et al., [33] obtained a hydrogen yield of 64.5 mL/g VS of hydrogen from mesophilic co-digestion of pulp and paper mill sludge and food waste, where the VS of the pulp and paper mill sludge and food waste were 18.38 and 25.59 %, respectively. Thermophilic conditions are considered advantageous for hydrogen production [34, 35] as they affect positively the kinetics of the hydrogen metabolic pathway and increases the growth rate of the hydrogen producing microorganisms [36]. In addition, thermophilic conditions could inhibit some hydrogen consumers,

such as homoacetogenic bacteria that consume hydrogen and carbon dioxide to produce acetate [18, 37, 38]. To evaluate the effect of fermentation temperature on the hydrogen production from SF, kinetic parameters P, R_m and λ were determined by fitting the cumulative hydrogen production using the modified Gompertz equation (Table 2). Comparing the kinetic parameters of our study with other studies, the P, R_m, λ and R^2 were 200 mL, 0.9 mL H_2/h, 50 h and 0.98 for H_2 production from black-liquor [39], 20.73 mL, 2.05 mL H_2/h, 5.51 h and 0.994 for hydrogen fermentation of grass [40], 180 mL, 0.8 mL H_2/h, 30 h and 0.97 for hydrogen production from pulp and paper mill liquor [41] and 12.8 mL, 2.0 mL H_2/h, 4.8 h and 0.99 for biohydrogen production from food waste [10]. A strong correlation could be built from the overall magnitude of the regression (R^2=0.996-0.989) between the experimental data and the applied equation in the current study compared to the other studies.

Fig. 1. Hydrogen yield from sedimented fibres at 37 and 55 °C, error bars represent standard deviations.

Table 2

Modified Gompertz equation parameters for hydrogen production from sedimented fibre at 37 and 55 °C.

Hydrogen production is associated with acetate and butyrate production [34]. Acetate was the main metabolite, with the concentrations of 5.1±0.8 gCOD/L at 37 °C and 5.0±0.4 gCOD/L at 55 °C, followed by butyrate, 0.4±0.04 gCOD/L at 37 °C and 1.4±0.04 gCOD/L at 55 °C, and propionate, 0.2±0.04 gCOD/L at 37 °C and at 55 °C (Table 3). It has been reported in literature that at 55 °C, hydrogen production through acetate pathway is feasible only at low hydrogen partial pressure (1kPa), while at a higher hydrogen partial pressure the metabolic pathway shifts to the butyrate pathway [17]. Therefore, the hydrogen production at 55 °C firstly starts by the acetate pathway then shifts to the butyrate pathway when the hydrogen partial pressure was increased which explains the higher butyrate production at thermophilic condition [34]. Final pH by the end of the incubations were 5.6 ± 0.1 and 5.8 ± 0.3, and total VFA concentrations were 6.5 and 5.6 gCOD/L for the thermophilic and mesophilic conditions, respectively. Soluble COD significantly increased (P<0.05) from 2.6 to 7.8 ± 0.9 and 6.8 ± 1.5 g/L in thermophilic and mesophilic conditions, respectively.

The TS and VS removals of the hydrogen production process were insignificant (P> 0.05) for both mesophilic (8.7 ± 0.9 and 11.1 ± 0.1 %) and the thermophilic conditions (13 ± 1.0 and 16.6 ± 0.6 %) , respectively (Table 3). VS removals of 6 to 10% has been reported during mesophilic hydrogen production for different other type of wastes [42, 43]. While a VS removal of 7.9 % has been reported from the thermophilic H_2 production from cassava stillage [44]. Low VS removal is expected as the hydrogen production process is run at low hydraulic retention time [45]. In addition, the pH drop and high concentration VFA could inhibit the microbial activity and consequently its ability on removal of the TS and VS content [46].

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Table 3

pH, TS, VS, COD_{tot}, COD_{sol} and VFA before and after hydrogen production from sedimented fibres at 37 and 55 °C.

3.2. Methane production from sedimented fibres

Methane yield of one-stage anaerobic digestion was 273 ± 11.0 and 161 ± 10.5 mL-CH$_4$/g VS at 37 and 55 °C, respectively (Fig. 2). This could be attributed to the high activity of the acidogenic bacteria at 55 °C, which resulted in accumulation of VFA (drop of the pH) [46]. In addition, methanogens have been reported to be very sensitive to VFA [47]. Methane has been successfully produced from sedimented fibres both in batch bottles [48] and in continuous reactor experiments [4] at 35 °C with yields of 250 and 201 mL CH$_4$/gVS, respectively. In contrast, two-stage anaerobic treatment produced 95.0 ± 12.2 mL-CH$_4$/kg VS at 37 °C and 334.3 ± 26.8 mL-CH$_4$/g VS at 55 °C (Fig. 2). At 37 °C, biomethane yield of the one-stage process (273 mL-CH$_4$/g VS) was three times the methane yield in the two-stage (95 mL-CH$_4$/g VS) since part of the VS has been converted into H$_2$ in the first stage. All VFA were consumed by the end of the one- and two-stage anaerobic treatment processes except for very tiny acetate peaks in some of the samples (mostly the thermophilic ones) but they were also basically under the detection limit. Methane production at two-stage process at 55 °C was enhanced as methanogens take the advantage of converting the hydrolysed and pre-fermented organics rather than the untreated feed in one-stage process [49, 50]. Besides, the feed of the second stage process was rich of soluble, easily fermentable organics and VFA which means lower organic loading rate into second stage digesters [51]. The COD_{sol} concentrations remained high, 5.2 and 7.6 g COD_{sol}/L after one- and two-stage processes; respectively, indicating that high fraction of the soluble COD is not anaerobically biodegradable [48]. The major part of the soluble COD is intermediate metabolites such as long chain fatty acids, alcohols, phenols and ketones and these compounds are indicated as recalcitrant or possible inhibitors for the methanogenic activity [52].

Fig. 2. Methane yield of one- vs. two-stage anaerobic digestion at 37 °C and 55°C, error bars represent standard deviations.

Results of modified Gompertz equation of one-stage treatment were appropriate for describing the kinetic parameters and showed that the lag time (λ) at 55 °C was five times that the required at 37 °C as the inoculum source comes from mesophilic (35 °C) anaerobic digester [27]. In addition, the value of regression coefficient (R^2) was ranging from 0.991 to 0.969 (Table 4) which indicates that the applied modified Gompertz equation was fit for simulating the experimental data. Similarly, for the two-stage treatment, the regression coefficient (R^2) ranged between 0.88 to 0.889 and the maximum methane production rate (R_m) was 30.1 mL-CH$_4$/d at 37 °C and 41.5 mL-CH$_4$/d at 55 °C which reflects the higher methane yield at 55 °C [53]. Ware and Power, [54] applied modified Gompertz equation for the methane potential from complex poultry slaughterhouse wastes and the kinetic parameters (P, R_m, λ and R^2) were 634.2 mL, 22.92 mL CH$_4$/h, 3.02 h and 0.996; respectively. The application of Gompertz equation provided an accurate description of the experimental data. Application of

Gompertz equation has been also applied for determination the kinetic parameters of the biomethane methane potential of co-digestion of pig manure and dead pigs [55]. The kinetic parameters (P, R_m, λ and R^2) of Gompertz equation for codigestion of pig manure and dead pigs (15%) were 334.1 mL, 8.04 mL CH_4/h, 19.7 h and 0.955; respectively, indicating that the model fitted the experimental results and a good correlation could be built between the experimental results and the relevant parameters. In another study, biochemical methane potential of anaerobic digestion of vegetable crop residues was excellently fitted to modified Gompertz equation giving a regression coefficient 0.986-0.998 [56].

Table 4

Modified Gompertz equation parameters for methane production from sedimented fibre at 37 and 55 °C.

In one-stage process, 80% of the methane was produced in the first 17 days (224.3 mL-CH_4/g-VS) at 37 °C and in day 10 (161±10.5 mL-CH_4/g-VS) at 55 °C. In the two-stage process, 80% of the biogas (76.4 mL-CH_4/g-VS) was produced in day 8 at 37 °C, and 80% in the first 22 days (299 mL-CH_4/g-VS) at 55 °C. Kokko et al., [3] reported that more than 80% of the methane yield from total sedimented fibre was produced in the first two weeks at 35 °C, which is similar to the results obtained in the present study. Hydrogen production step before the methane production reduced the time required for methane production in the current study at mesophilic conditions. Fu et al., [42] obtained 80% of the methane yield from vinasse at 33 and 29 days with one- and two-stage anaerobic digestion, respectively (Table 5). The anaerobic treatment of pulp and paper mill and Kraft pulp mill took 55 days [57] and 40 days [58], respectively, to get the 80% yield of the methane. The lag time in thermophilic one- and two-stage digestion was 5 and 10 days, respectively (methane yield < 50 mL-CH_4/g-VS) compared to <3 days in mesophilic conditions. This could be attributed to the adaptation of the inoculum sludge for the thermophilic temperature (inoculum was sampled from a mesophilic digester at 35 °C) [59].

Table 5

Two-stage hydrogen and methane yields obtained from various feedstocks.

TS removal of the one-stage anaerobic digestion was 49.8 ± 0.2 and 47.7 ± 1.9% at 37 °C and 55 °C ($p>0.05$), respectively, while VS removal was 59 ± 0.8% at both 37 °C and 55 °C ($p>0.05$) (Table 6). The deteriorated TS removal of one-stage at 55 °C could be attributed to the drop of the pH due to high activity of the acidogenic bacteria at thermophilic conditions [46].Two-stage anaerobic digestion resulted in TS and VS removals of 45.7 ± 0.6 and 64.9 ± 0.5% at 37 °C ($p>0.05$) and 42.9 ± 0.1 and 58.2 ± 0.1% at 55 °C ($p>0.05$). The VS removal efficiency of two-stage anaerobic digestion was significantly higher ($P<0.05$) higher than that of one-stage (Table 6). A VS removal of 63% and 54% was reported in the anaerobic digestion of sedimented fibre by Kokko et al., [3] and Chatterjee et al., [4] in batch experiment and continuous reactor, respectively. Two-stage anaerobic digestion achieved COD_{sol} removal efficiency of 20.2% at 37 °C and 29.2 % at 55 °C ($p>0.05$).

Table 6

Characteristics of the influents and effluents of the one- and two-stage anaerobic digestion at 37 and 55 °C.

3.3. *Energy recovery of one- and two-stage anaerobic treatment of sedimented fibres*

Energy yields of the one- and two-stage anaerobic digestion processes were evaluated at mesophilic and thermophilic conditions (Table 7). Two-stage thermophilic process achieved the highest overall energy yield of 3.7 kWh/L, which is two times the one-stage thermophilic anaerobic digestion (1.7 kWh/L), 3.7 times higher than the two-stage mesophilic anaerobic digestion (1.0 kWh/L) and 1.3 times higher than the one-stage mesophilic digestion (2.9 kWh/L). Similar results were reported by Mamimin et al. [60], who obtained a 34% higher energy yield (4.3 kWh/L) in the two-stage anaerobic digestion (thermophilic hydrogen production followed by mesophilic methane production) of palm oil mill effluent than the one-stage mesophilic methane anaerobic digestion (2.8 kWh). Fu et al., [42] reported a 12.9% increase in the energy recovery from vinasse in two-stage process than the one-stage process. Other studies found that there is no significant difference between the energy yield of the single and two-stage process [47]. This process depends on many factors including the nature of the feedstock and the different operational conditions (pH, HRT, OLR, Temperature and inoculum).

Table 7

Net energy recovery from sedimented fibres in one- and two-stage anaerobic digestion at 37 and 55 °C.

The biodegradable fraction (VS) (180 000 m^3) of the sedimented fibres (total volume of 1.5 million m^3) that could be converted to biogas, could result in 520 or 660 million kWh, if subjected to one-stage mesophilic or two-stage thermophilic anaerobic digestion conditions, respectively. The estimated energy yield of the mesophilic one-stage anaerobic digestion of 1.5 million m^3 sedimented fibres (520 million kWh) is comparable to the yield obtained from the batch experiments of [3] (476 million kWh).

Conclusions

This study demonstrated for the first time two-stage anaerobic treatment for hydrogen and methane production from boreal sedimented pulp mill fibres which represents a sustainable and environmentally-friendly treatment approach. Two-stage thermophilic anaerobic treatment of sedimented fibres achieved the highest energy yield and treatment performance of the four tested scenarios. Though the contribution of hydrogen on the overall energy production from the anaerobic digestion of the sedimented fibres was 3.7% at 37 °C (0.04 kWh) and 4.1 % at 55 °C (0.15 kWh), the fermentation step played an important role in hydrolysis of sedimented fibres for the second step where the COD$_{sol}$ was increased from 2.6 ± 1.4 to 6.8 ± 1.5 at 37 °C and to 7.8 ± 0.9 at 55 °C. The estimated energy yield of the mesophilic one-stage anaerobic digestion of 1.5 million m^3 sedimented fibres is 520 GWh, while the thermophilic two-stage anaerobic digestion could produce 660 GWh. In addition, lag phase

of the hydrogen production step decreased by 4 days at 37 °C and 2 days at 55 °C. Digestion time at 37 °C of two-stage treatment was decreased to 8 days as compared to 17 days for one-stage treatment. The saved time would help in the reduction of the required volume of the treatment facilities and times of operation. However, pulp and paper industry depending on thermomechanical pulping process, produces huge amounts of hot wastewater (50-70 °C) which could be used for heating the anaerobic reactors avoiding energy losses and need for cooling this hot wastewater before treatment or discharge. The effluent of our proposed treatment could be subjected to filter press where the solid fraction is used as a soil amendment and the liquid fraction is recycled and mixed with the treatment plant influent.

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Declarations

Ethical Approval

Not applicable

Competing interests

The authors declare no competing interests.

Authors' contributions

Mohamed El-Qelish: Methodology, Investigation, Formal analysis, Visualization, Writing – original draft. **Pritha Chatterjee:** Methodology, Investigation, Visualization, Writing – original draft, Writing – review & editing, Supervision. **Marika Kokko**: Investigation, Visualization, Writing – original draft, Writing – review & editing, Supervision. **Fatma El-Gohary:** Writing – review & editing, Supervision. **Mohamed Abo-Aly**: Writing – review & editing, Supervision. **Jukka Rintala**: Visualization, Writing – original draft, Writing – review & editing, Supervision, Funding acquisition, Project administration.

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Availability of data and materials

All data are available in the manuscript

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Table 1

Characteristics of sedimented fibre and inoculum sludge.

Sample	pH	TS (%)	VS (%)	COD$_{tot}$ (g/L)	COD$_{sol}$ (g/L)
Sedimented fibre	4.7±0.3	10.5±0.16	9.4±0.17	NM	NM
Inoculum for hydrogen production	6.8±0.2	2.0±0.02	1.1±0.0	17.5±0.2	0.9±0.7
Inoculum for methane production	7.2±0.5	4.8±0.37	2.7±0.13	37.7±1.1	8.0±0.5

NM – not measured; Total solids (TS), Volatile solids (VS), Total chemical oxygen demand (COD$_{tot}$), Soluble chemical oxygen demand (COD$_{sol}$)

Table 2

Modified Gompertz equation parameters for hydrogen production from sedimented fibre at 37 and 55 °C.

Parameters	Hydrogen production at 37 °C	Hydrogen production at 55 °C
P (mL-H_2)	10.4	42.0
R_{max} (mL-H_2/h)	0.4	0.7
λ (h)	67.0	35.0
R^2	0.989	0.996

Table 3

pH, TS, VS, COD_{tot}, COD_{sol} and VFA before and after hydrogen production from sedimented fibres at 37 and 55 °C.

Parameter	Beginning of the experiments	Effluent after biohydrogen production	
		37 °C	55 °C
pH	6.5 ± 0.1a	5.8 ± 0.3ab	5.6 ± 0.1b
COD_{tot} (g/l)	23.7 ± 0.25a	21.3 ± 0.3b	20.9 ± 2.0ab
COD_{sol} (g/l)	2.6 ± 1.4a	6.8 ± 1.5ab	7.8 ± 0.9b
TS %	2.3±0.17a	2.1 ± 0.2a	2.0 ± 0.01a
VS %	1.8±0.1a	1.6 ± 0.1b	1.5 ± 0.2ab
Acetate (gCOD/L)	0.5 ± 0.05a	5.1 ± 0.8b	5.0 ± 0.4b
Propionate (gCOD/L)	0.2 ± 0.01a	0.2 ± 0.04a	0.2 ± 0.04a
Butyrate (gCOD/L)	0. 3 ± 0.03a	0.4 ± 0.04a	1.4 ± 0.04b
VFA total (gCOD/L)	1.0 ± 0.03a	5.6 ± 0.3b	6.5 ± 0.2b

Statistical analysis was between beginning of the experiments and effluents of 37 and 55 °C and between effluents of 37 and 55 °C

Compared results in the same row with same superscript letters are statistically not significant different (P<0.05).

Table 4

Modified Gompertz equation parameters for methane production from sedimented fibre at 37 and 55 °C.

Parameters	One stage 37 °C	One stage 55 °C	Two stage 37 °C	Two stage 55 °C
P (mL-CH_4)	273.3	161.3	95.9	334.5
R_{max} (mL-CH_4/d)	20.1	28.4	30.1	41.5
λ (d)	1.0	5.0	1.0	11.0
R^2	0.991	0.969	0.880	0.889

Table 5

Two-stage hydrogen and methane yields obtained from various feedstocks.

Feedstock	Reactor type	T (°C)	H_2 yield	CH_4 yield	Reference
Household solid waste	CSTR	37	43 mL/gVS	500 mL/gVS	[61]
Swine manure + market biowaste	CSTR	55	140 mL/ g VS	351 mL/ g VS	[47]
Food waste	CSTR	37	292.7 mL/ gVS	391.6 mL /gVS	[62]
Vinasse	Batch	37	14.8 mL/gVS	274 mL/gVS	[9]
Food waste+ sewage sludge+ raw glycerol	Batch	35	179.3 mL /gVS	342 mL /gVS	[43]
Oil palm trunk	Batch	50 H_2, 35 CH_4	60.22 mL /g	309.4 mL/g	[63]
Sugarcane bagasse	Semi-continuous	50 H_2, 37 CH_4	226 mL/g	341 mL/g COD	[64]
Food waste	CSTR	40	65 mL/g VS	546 mL/g VS	[7]
Cassava stillage	CSTR	55	56.6 mL/g VS	249 mL/g VS	[14]
Water hyacinth	Batch	35	51.7 mL/g VS	143.4 mL/g VS	[15]
Food residue biomass	CSTR	40 H_2, 35 CH_4	102.6 mL/g	519 mL/g	[65]
Wheat straw	UASB	70 H_2, 55 CH_4	89 mL/g VS	307 mL/g VS	[66]
Sedimented fibre	Batch	37	10.8 mL/gVS	95 mL/gVS	Current study
Sedimented fibre	Batch	55	42.1 mL/gVS	334 mL/gVS	Current study

CSTR: continuously stirred tank reactor,
UASB: upflow anaerobic sludge blanket

Table 6

Characteristics of the influents and effluents of the one- and two-stage anaerobic digestion at 37 and 55 °C.

One-stage anaerobic digestion					
Parameter	Influent of CH_4 production	Effluent of CH_4 production at 37 °C	Removal (%)	Effluent of CH_4 production at 55 °C	Removal (%)
TS %	4.8±0.35	2.4 ± 0.2	50.0 ± 0.2	2.5 ± 1.9	47.7 ± 1.9
VS %	2.7±0.2	1.1 ± 0.1	59.0 ± 0.1	1.1 ± 0.8	59.0 ± 0.8
COD_{sol} (g/l)	NM*	5.2 ± 0.2	---	6.1 ± 0.2	---

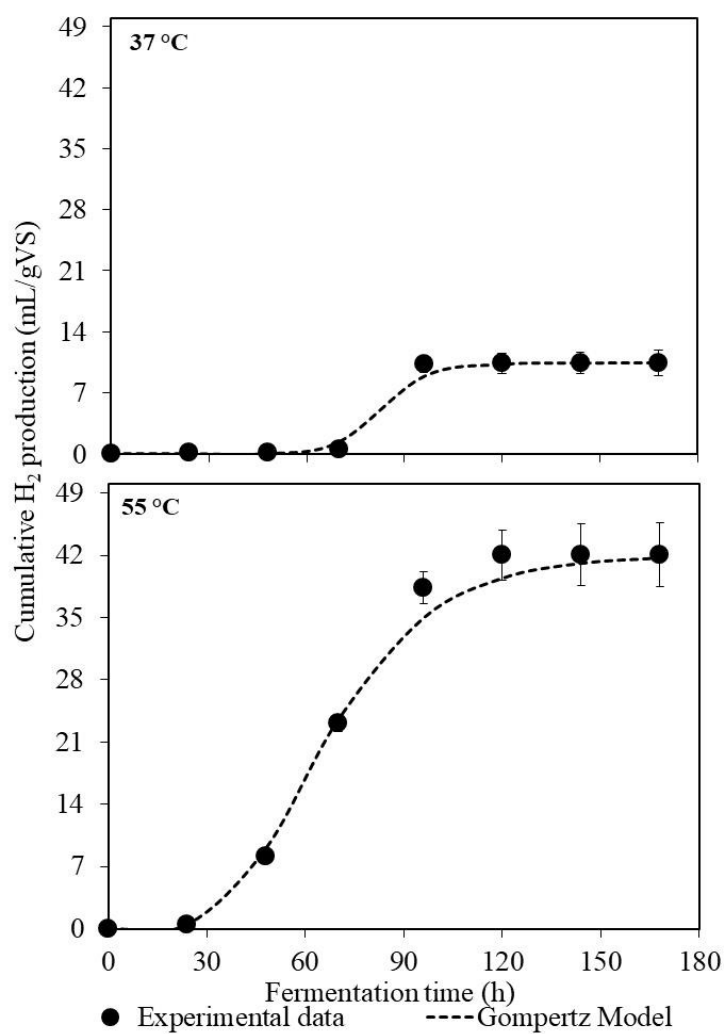| Two-stage anaerobic digestion						
Parameter	37 °C		Removal (%)	55 °C		Removal (%)
	Influent of CH_4 production	Effluent of CH_4 production		Influent of CH_4 production	Effluent of CH_4 production	
TS %	4.6±0.35	2.5 ± 0.6	45.7±0.14	4.2±0.8	2.4±0.1	42.9±0.25
VS %	3.7±0.1	1.3 ± 0.5	64.9±1.4	2.9±0.4	1.2±0.1	58.2±0.21
COD_{sol} (g/l)	8.4±0.8	6.7 ± 0.9	20.2±10.5	8.9±1.5	6.3±0.2	29.2±1.7

*Not measured

Table 7

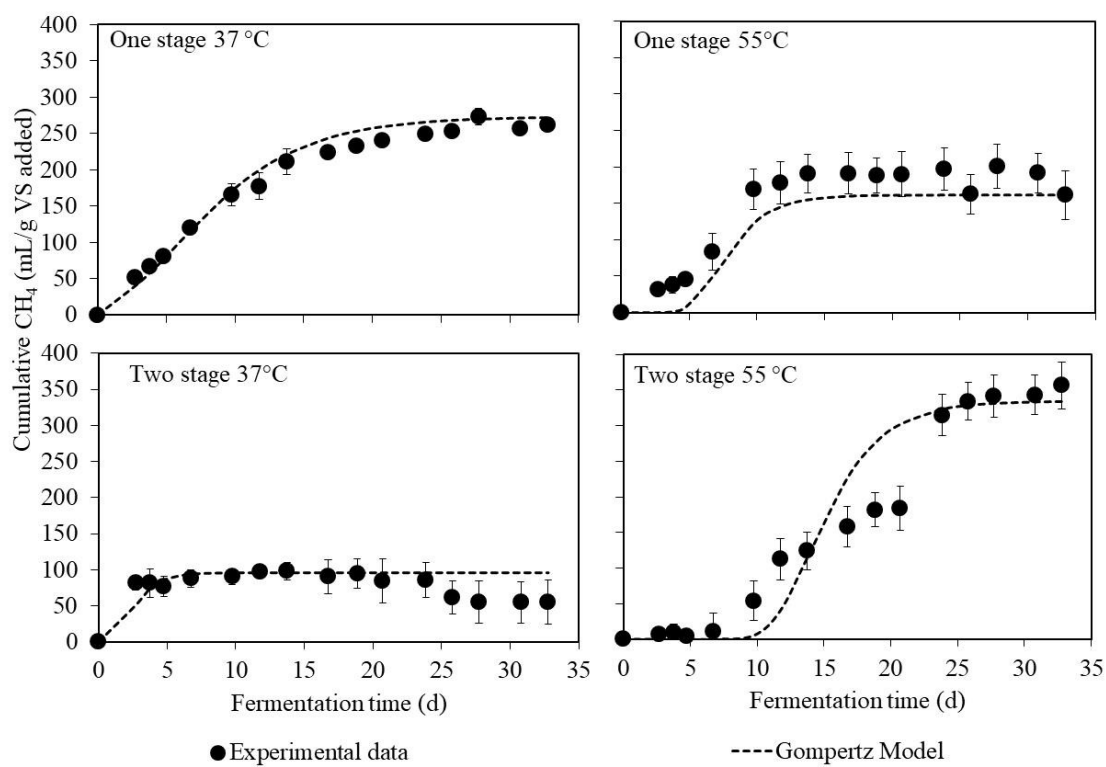Net energy recovery from sedimented fibres in one- and two-stage anaerobic digestion at 37 and 55 °C.

Energy yield	Two stage digestion		One stage digestion	
	37 °C	55 °C	37 °C	55 °C
Hydrogen yield (mL/g VS)	10.8	42.1	--	--
Methane yield (mL/g VS)	95	334	273	161
Energy yield (kJ/g VS)	3.73	13.2	10.3	6.1
Energy yield (kWh/L)	1.0	3.7	2.9	1.7
Total Energy (kWh) x10^6	180	660	520	310

kWh/L refers to the energy contained in 1 L of biomass, assuming its density of 1 kg/L
The biodegradable fraction used for net energy calculations is 180 000 m^3 of total SF volume of 1.5 million m^3

Fig. 1. Hydrogen yield from sedimented fibres at 37 and 55 °C, error bars represent standard deviations.

Fig. 2. Methane yield of one- vs. two-stage anaerobic digestion at 37 °C and 55°C, error bars represent standard deviations.