

1 **Anaerobic digestion of 30–100-year-old boreal lake sedimented fibre**
2 **from the pulp industry: extrapolating methane production potential**
3 **to a practical scale**

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13

14 **Abstract**

15

16 Since the 1980s, the pulp and paper industry in Finland has resulted in the
17 accumulation of fibres in lake sediments. One such site in Lake Näsijärvi contains
18 approximately 1.5 million m³ sedimented fibres. In this study, the methane production
19 potential of the sedimented fibres (on average 13% total solids (TS)) was determined
20 in batch assays. Furthermore, the methane production from solid (on average 20% TS)
21 and liquid fractions of sedimented fibres after solid-liquid separation was studied. The
22 sedimented fibres resulted in fast methane production and high methane yields of
23 250±80 L CH₄/kg volatile solids (VS). The main part (ca. 90%) of the methane potential
24 was obtained from the solid fraction of the sedimented fibres. In addition, the VS
25 removal from the total and solid sedimented fibres was high, 61–65% and 63–78%,
26 respectively. The liquid fraction also contained a large amount of organics (on average
27 8.8 g COD/L), treatment of which also has to be considered. The estimations of the
28 methane production potentials in the case area showed potential up to 40 million m³ of
29 methane from sedimented fibres.

30

31 Keywords: Pulp and paper industry, sedimented fibre, anaerobic digestion, methane

32

33 **1. Introduction**

34

35 Paper production is a globally growing industry with an annual global production that
36 has increased from ca. 240 to 409 million tons of paper and board from 1990 to 2016
37 (Finnish Forest Industries, 2017). In Finland pulp and papermaking started in the 1870s,
38 when a sulfite pulp process was introduced enabling the use of different raw materials

39 and the production of different paper grades. In the beginning of the 20th century
40 (1890–1913) the production of pulp and paper further increased due to the exports to
41 Russia. (Kuisma, 1993) Currently, the pulp and paper industry in Finland is the 5th
42 largest pulp and paper making country in the world with investments in pulp and
43 biorefineries (e.g. biofuels) with decreasing paper production.

44

45 The pulp and paper industry utilizes vast amounts of water, as much as 200-1000
46 m³/paper ton in the beginning of 20th century (Kamali et al., 2016)) and thus, pulp and
47 paper mills are often situated next to water bodies, such as lakes or seas. In the 1970s
48 the wastewater treatment of the pulp and paper industry in Finland was comprised of
49 some mechanical treatment and aerated lagoons (Junna and Ruonala, 1991), while in
50 the 1980s activated sludge plants were introduced drastically decreasing the wastewater
51 discharges (Rintala et al., 1988) along with intensified research presented e.g. in IWA
52 Forest industry wastewaters conferences (e.g. Water Science and Technology
53 1985;17(1)). Thus, for decades most of the wastewaters were discharged to the near-by
54 water bodies without any treatment.

55

56 During the decades of wastewater discharge, various compounds accumulated in the
57 sediments near pulp and paper mills, including pulp fibres as the major material. In
58 addition, heavy metals, organic chlorine compounds and resin acids were accumulating
59 (Kähkönen et al., 1998; Leppänen and Oikari, 1999; Poole et al., 1977) depending on
60 the pulp and paper manufacturing processes used in the mills and later on the applied
61 wastewater treatment process as well. In the recipient waters, the pulp fibres settle down
62 rapidly (Poole et al., 1977) and in time can be broken down by microbial activity into
63 organics, such as sugars or organic acids, resulting in oxygen depletion and gas

64 generation (e.g. CH₄, H₂S) in sediments (Pearson, 1980). Today, the fibre-rich
65 sediments originating from the activities of the pulp and paper industry can be found
66 from various locations worldwide, including Nordic countries, Canada and China (Guo
67 et al., 2016; Jackson, 2016; Ratia et al., 2013). These polluted sediments are often
68 located near cities and prevent the recreational use of water areas. In addition, they can
69 cause long-term environmental effects on the water bodies, such as oxygen depletion,
70 eutrophication, the release of detrimental compounds from the sediment and toxicity
71 towards aquatic organisms (Lindholm-Lehto et al., 2015; Meriläinen et al., 2000).

72

73 One option initially considered for treating fibre sediments is anaerobic digestion (AD)
74 as it may provide potential for both energy recovery and further use of the digestate.
75 For example, primary sludge in thermophilic conditions resulted in methane yields of
76 230 mL-CH₄/g-VS in batch assays and 190–240 mL-CH₄/g-VS in continuously stirred
77 tank reactor (CSTR) with hydraulic retention times (HRTs) of 16–30 d (Bayr and
78 Rintala, 2012). The anaerobic digestion of WAS from mechanical or chemical pulp
79 mills in batch assays resulted in methane yields of 43–155 mL-CH₄/g-VS (Karlsson et
80 al., 2011). The relatively low methane yields from pulp and paper mill sludges in many
81 studies are due to the incomplete hydrolysis of lignocellulosic constituents present in
82 the sludge, the low nutrient content and the presence of detrimental compounds (Kamali
83 et al., 2016; Meyer and Edwards, 2014).

84

85 To accommodate the increasing population the city of Tampere, Finland, is building a
86 new district of 90 ha along the banks of Lake Näsijärvi on the site of an old pulp mill.
87 The bay area near the old pulp mill received effluents from a sulfite pulp mill from the
88 1910s to the 1980s and has approximately 1.5 million m³ of sedimented fibre in the bay

89 that forms a layer up to 10 m height. In this study, the objective was to assess the
90 methane production potential of the sedimented fibers in the bay area and to initially
91 evaluate the potential role of AD in the treatment and utilization of the fibres. For this
92 purpose, sedimented fibres were collected from the bottom of the lake and their
93 anaerobic degradability was determined. Altogether, nine samples from three different
94 sampling points and depths were collected. The solid and liquid separation efficiency
95 of the sedimented fibres were determined and the methane production potential from
96 the solid and liquid fractions as well as from the original (total) sedimented fibres were
97 determined. In addition, the anaerobic degradation of the sedimented fibres were
98 examined. Finally, the practical scale methane potential of the sedimented fibres in the
99 studied bay in lake Näsijärvi was assessed based on the laboratory batch results.

100

101

102 **2. Materials and methods**

103

104 ***2.1. Sedimented fibres and inoculum***

105

106 Sedimented fibre samples were collected from three different sampling points (A,B,C)
107 at three different depths of the fibre sediment-containing area (ca. 20 ha) in Lake
108 Näsijärvi (Tampere, Finland) near the old pulp and paper mill. The sampling points
109 were chosen to give a representative understanding of the sedimented fibres due to
110 heterogeneous nature of the sedimented fibres, especially at different depths. Sampling
111 depths (Table 1) were chosen based on the estimated total depth of the sedimented fibre
112 layer (ranging between 0 and 10 m) so that they would represent the top, middle and
113 bottom sections of the layer. Samples from each sampling point were taken with an

114 excavator bucket from a sampling ferry (Autiola and Holopainen, 2016). The samples
115 were transported to the laboratory and stored at 6 °C until used. Before the experiments,
116 each sample was homogenized by mixing for 1 min using a concrete mixer attached to
117 a power drill. After mixing, samples for total (TS) and volatile solids (VS) analysis
118 were taken. Different samples for solid-liquid separation and total samples for
119 determining the biomethane potential were used. Digested mesophilic municipal
120 sewage sludge from Viinikanlahti sewage treatment plant (Tampere, Finland) was used
121 as the inoculum for the experiments determining biomethane potential (BMP).

122

123 Table 1

124

125

126 *2.2. Solid-liquid separation*

127

128 For the BMP determination, liquid and solid fractions from 5 L of sedimented fibres
129 were separated by removing liquid from the total samples with a juice press (simulating
130 a screw press in a smaller scale) that had a volume of 12 L (diameter 360 mm, height
131 600 mm) and where the pressure was realized with a lever arm. The amount of water
132 to be removed was determined based on the TS content (9.3–21.6%) of the total sample
133 so that for the resulting dry residue (TS) was approximately 20%. For sample A(0-1
134 m), the TS was already above 20 % and thus, no water was removed. TS and VS were
135 determined for the dry and liquid fractions. In addition, the total and soluble chemical
136 oxygen demand (COD_{tot} and COD_s) were determined for liquid fractions.

137

138

139 **2.3. BMP batch assays**

140

141 Liquid and solid fractions of the sedimented fibres were both analysed for their BMP
142 separately. In addition, the BMP of the total sedimented fibre samples (without solid-
143 liquid separation) from sampling point B was determined.

144

145 For solid and total samples, BMP was determined in duplicates in 1 L glass bottles
146 containing 350 mL inoculum and sedimented fibre samples at a ratio of 2.0 g-
147 $VS_{\text{substrate}}/\text{g}-VS_{\text{inoculum}}$. 67 mL of 42 g/L NaHCO_3 (final concentration 4 g/L) was added
148 to each bottle as a buffer and the liquid volume was adjusted to 700 mL with distilled
149 water. For liquid fractions of sedimented fibres, the BMP was determined in triplicates
150 in 120 mL serum glass bottles containing 30 mL of inoculum, liquid fibre samples (the
151 final total COD concentration was 5.4–9.6 g/L and was dependent on the sample), and
152 6 mL of 42 g/L NaHCO_3 to have a final concentration of 4 g/L. Distilled water was
153 added to reach a total liquid volume of 60 mL. Control samples (inoculum only) were
154 prepared by replacing the substrate with distilled water both in 1 L and 120 mL glass
155 bottles. The cumulative methane production of the inoculum was excluded from the
156 cumulative methane production of the samples. The methane yields are given as the
157 average value of the parallel samples.

158

159 The pH of the batch bottle contents was between 7.0 and 8.1. Headspaces were flushed
160 with N_2 gas for 3 min after sealing to ensure anaerobic conditions. The 1 L glass bottles
161 were placed in a water bath at 35 °C and connected to aluminum gas bags (SupelTM
162 Inert Foil Gas Sampling Bags, Supelco, USA) for collection of the produced gas. The
163 120 mL serum bottles were placed in a static incubator at 35 °C. The content and

164 volume of the gas were analysed 1–3 times a week. At the end of the assay pH, TS and
165 VS, COD_s and volatile fatty acids (VFAs) were determined.

166

167 ***2.4. Analyses and calculations***

168

169 The methane yields in solid and liquid fibre fractions in sampling point B were also
170 calculated against the g VS of the total sedimented fibre sample (L CH₄/kg VS_{total}). The
171 methane yield of the total samples was calculated by dividing the cumulative methane
172 production (mL CH₄) with the mass of VS added to the bottle (g VS). The methane
173 yields of the liquid and solid samples were calculated with equations 1 and 2,
174 respectively.

175

$$176 \text{ Methane yield} = \frac{\text{cumulative methane production}}{m_{VS}/[(V_l * VS_l)/(V_t * VS_t)]} \quad (1)$$

177

$$178 \text{ Methane yield} = \frac{\text{cumulative methane production}}{m_{VS}/[(V_t * VS_t - V_l * VS_l)/(V_t * VS_t)]} \quad (2)$$

179

180 , where m_{VS} is the mass of VS added to the bottle (g VS), V_t and V_l the volumes of the
181 total and liquid samples before and after the solid-liquid separation (L), respectively,
182 and VS_t and VS_l the percentage of VS in the total and liquid samples before and after
183 the solid-liquid separation, respectively. The total nitrogen and soluble nitrogen after
184 filtration (0.45 μ m) were analysed with Kjeldahl nitrogen analysis, where nitrate and
185 nitrite were reduced and organic carbon degraded in sulphuric acid combustion with a
186 catalyst. Ammonia was released from the formed ammonium sulphate with NaOH and
187 ammonia was distilled to a boric acid containing an indicator. The concentration of
188 ammonia was determined from the distillate by titrating with sulphuric acid. Total

189 phosphorous and soluble phosphorous after filtration (0.45 μm) were analysed with
190 inductively coupled plasma mass spectrometry (ICP-MS). Before ICP-MS, the sample
191 was degraded with microwaves in nitrohydrochloric acid. (Ramboll Analytics, Finland)

192

193 TS and VS were analyzed according to standards SFS-EN 14346 and SFS-EN 15169,
194 respectively. For liquid samples, COD_{tot} and COD_{s} were analysed according to standard
195 SFS 5504. For COD_{s} analysis, samples were filtered (0.45 μm , Chromafil Xtra PET).
196 pH was measured with WTW ProfiLine pH 3210 and WTW pH 330i meters.

197

198 The methane content of the produced biogas was measured with a Perkin Elmer Clarus
199 500 GC-FID gas chromatograph with a Mol-Sieve 5A PLOT column. Column, detector
200 and injector temperatures were 100 $^{\circ}\text{C}$, 250 $^{\circ}\text{C}$ and 230 $^{\circ}\text{C}$, respectively. The carrier
201 gas was helium at a flow rate of 14 mL/min. For the 1 L bottles, the volume of produced
202 biogas was measured from the gas bags using the principal of water displacement. All
203 measurements were performed at room temperature ($\sim 20^{\circ}\text{C}$) and atmospheric pressure
204 (~ 1 atm). The gas bag was connected with tubing to an air-tight water column that was
205 opened from the bottom. The gas from the gas bags replaced a certain mass of water
206 from the water column and the mass of the replaced water was weighed and converted
207 to a volume at STP. For the 120 mL serum bottles, the volume of methane was
208 calculated based on the percentage of CH_4 in the headspace, where the overpressure
209 was accounted for and released when the CH_4 content increased above 90% (Angelidaki
210 et al., 2009). Air temperature and pressure as well as water bath temperature were
211 monitored throughout the experiment. The methane production results were converted
212 to STP conditions (0 $^{\circ}\text{C}$, 1 bar). The methane yield was calculated per VS of substrate

213 added (L CH₄/g VS) for solid and total fibres and per COD_s added (L CH₄/g COD_s) for
214 liquid fibre fractions.

215

216 VFAs (acetate, propionate, butyrate, isobutyrate and valerate) were analysed with a
217 Shimadzu GC-2010 Plus chromatograph with an Zebran ZB-WAX Plus column and a
218 flame ionization (FID) detector. Helium was the carrier gas with a flow rate of 82
219 mL/min and the injector and detector temperatures were 250°C. The oven temperature
220 programme was as follows: 40°C for 2 min, 20°C/min increase until 160°C, 40°C/min
221 increase until 220°C, and 220°C for 2 min. Before VFA analysis, the samples were
222 filtered (0.45 µm, Chromafil Xtra PET).

223

224

225 **3. Results**

226

227 *3.1. Characterization of the sedimented fibre samples*

228

229 The pH of the total sedimented fibre samples was between 4.1 and 5.0, except for A(2-
230 3m) that had a pH of 6.5 (Autiola and Holopainen, 2016). The mass balances of the
231 solid and liquid fractions after solid-liquid separation (Fig. 1) show that most of the TS
232 and VS end up in the solid fraction that comprises of 46–70% of the volume of the total
233 sample.

234

235 Figure 1

236

237 The TS and VS for the total and solid fractions of sedimented fibres and of the
238 inoculum, and COD_s for the liquid fractions of sedimented fibres are presented in Table
239 2. TS for the total samples was between 9.3 and 13.9%, except for A(0-1m) that had a
240 TS of 21.6%. After solid-liquid separation the TS of the solid fractions was in the range
241 of 17.4–21.1%, which is close to the aimed TS value of 20%. Both for total and solid
242 samples, the VS/TS ratio was over 93%, except for sample (A(2-3m)) that had a VS/TS
243 ratio of 87%. The liquid fractions contained CODs in the range of 5.5–13.9 g/L, of
244 which the soluble COD was 42–92 % (2.3–12.7 g/L) (Table 2). The total (7.1–9.0 g/L)
245 and soluble (5.4–6.2 g/L) COD in the liquid fractions at different depths from sampling
246 points A and C were similar, while there were large variations in the total (5.5–13.9
247 g/L) and soluble (2.3–12.7 g/L) COD for sampling point C (Table 2).

248

249 Table 2

250

251 The nitrogen and phosphorous in four of the total samples and in two of the pore water
252 samples as well as the organic acids in the pore water samples of two of the samples
253 were determined. The total nitrogen in the four total samples (A(2-3m), B(3-4m), B(5-
254 6m) and C(4-5m)) varied between 2.9 and 4.7 g/kg TS and the phosphorous between
255 0.27 and 0.31 g/kg TS. In the pore water, the total VFA content varied between 2.4 and
256 6.4 g/L (Table 3). The concentrations of organic acids increased, while the
257 concentrations of total nitrogen decreased with sampling depth (Autiola and
258 Holopainen, 2016).

259

260 Table 3

261

262 **3.2. Methane production potential**

263

264 *3.2.1 Solid fractions of sedimented fibres*

265

266 Methane production from the solid fractions of sedimented fibres started in less than
267 one week, and 80% of the methane produced in 30 d was produced in the first two
268 weeks (Fig. 2). For two of the sampling points (A and C), higher methane yields were
269 obtained from the deeper sediments, e.g. 250% more methane was produced from
270 sample A(2-3m) than from A(0-1m) and 23% more methane was produced from sample
271 C(4-5m) than from C(0-1m). However, from sampling point B, 35% higher methane
272 yields were obtained.

273

274 The assays were continued until day 56, but 94–97% of the methane was produced in
275 the first 30 d except for sample A(0-1m), where 89% of the methane was produced in
276 the first 30 d. The highest methane yield, 320 L CH₄/kg VS, on day 30 was obtained
277 from the solid fractions of sedimented fibres originating from the deepest samples (4–6
278 m, Fig. 2). While in other sampling points the methane yields were higher (180–320 L
279 CH₄/kg VS), in sampling point A(0-1m) the methane yield was only 80 L CH₄/kg VS.
280 Thus, there is a large variation in the methane yields between individual samples
281 originating from different sampling points and depths (Fig. 2). However, the
282 sedimented fibre from sampling point A(0-1m) differed from the others, as it had a
283 higher TS (21.6%) in the beginning and consisted mainly of woody pieces, while the
284 other samples had a felt-like structure and had, based on visual observations, been
285 subjected more to biological degradation.

286

287 Figure 2
288

289 The digestates were characterized in the end of the assays (Table 4). The pH of the solid
290 fractions of sedimented fibre samples did not change much during the assays (from
291 initial 7.1–7.5 to final 7.5–7.6). The measured TS and VS removals of the solid fibre
292 samples (with the inoculum's TS and VS subtracted) on day 56 were 63–78% and
293 63–78%, respectively. The TS and VS removal was not dependent on the depth of the
294 sedimented fibres. No VFAs were detected at the end of any of the assays.

295

296 Table 4

297

298 *3.2.2 Liquid fractions of sedimented fibres*

299

300 When studying the methane production from the liquid fractions of sedimented fibres,
301 the trends were similar to that of the solid fractions. There was no clear trend between
302 methane yields (L CH₄/kg COD_{added}) and the depths of the samples. Again, more
303 methane was produced from deeper samples of the sampling point B, while from
304 sampling point C the highest methane yields were obtained from the middle layer (1-2
305 m) (Fig. 2). In addition, methane production started fast and >80% of the methane
306 produced in 30 d was produced in the first five days. From the liquid fractions methane
307 yields were the highest (280±20 L CH₄/kg COD) for sample B(5-6m), while the average
308 was 240±40 L CH₄/kg COD. The experiments were continued for 68 days, but over
309 87% of the methane was produced in the first 30 d.

310

311 The digestates of the liquid fractions were characterized in the end of the assays (Table
312 4). The pH decreased during the experiments (68 d) from 7.2–8.1 to 6.9–7.0. The COD_s

313 decrease was in the range of 76–84%, except for the sample A(2-3m) that had COD_s
314 decrease of 66%. The final COD_s was 0.41–0.53 g/L (Table 4). No VFAs were detected
315 at the end of the experiments, indicating that not all the soluble COD was anaerobically
316 biodegradable.

317

318 *3.2.3 Total sedimented fibre samples*

319

320 As with solid and liquid fractions of sedimented fibres from sampling point B, the
321 methane yields from the total sedimented fibre samples increased with the sample
322 collection depth. As with solid sedimented fibre fractions, over 80% of the methane
323 produced in 30 d was produced during the first two weeks of the experiment (Fig. 2).
324 The differences in the methane yields were considerable; an average methane yield of
325 340 L CH₄/kg VS was obtained from total sample at the depth of 5–6 m, while from
326 the depth of 0-1m 210 L CH₄/kg VS was produced (Fig. 2). The digestates of the total
327 samples were characterized in the end of the assays (Table 4). The pH changed from
328 7.0–7.4 to 7.5. The TS and VS removals on day 56 were 59–62% and 61–65%,
329 respectively, and no VFAs were detected at the end of the experiment. The experiment
330 was continued until day 56, but ≥95% of the methane was produced in the first 30 d.

331

332 *3.3. Comparison of methane yields from different sedimented fibre fractions*

333

334 Methane production from the total, solid and liquid fractions of sampling point B were
335 compared by projecting the methane yields against the VS of the original (total) sample
336 before solid-liquid separation and against VS removal (Fig. 3). Most of the methane
337 was produced from the solid fraction (95.9–98.4 %) of the sedimented fibres. The liquid

338 fractions resulted only in less than 4% of the methane obtained from the total samples
339 (Fig. 3), which corresponds to the VS-content of the liquid fraction. Comparing the
340 methane yields calculated against the VS removed (Fig. 3.B), the sum of the solid and
341 liquid fractions resulted in 79–97% of the methane yield of the total sample. The
342 differences between the methane yields of total samples and the sum of solid and liquid
343 fraction can be explained by the i) heterogenous sample, ii) small sample volume (<
344 0.1 L) used for the incubations compared to the large original sample volume of the
345 sedimented fibres (10 L) and iii) the high VS content of the total sedimented fibres (ca.
346 12% VS). This is also supported by the large variation between the different sampling
347 points and on average the methane yields for total, solid and liquid fractions were
348 400 ± 110 , 340 ± 100 and 7 ± 5 L CH₄/kg VS_{removed}, respectively.

349

350 Figure 3

351

352 **4. Discussion**

353

354 **4.1. Characteristics of sedimented fibre**

355

356 The studied fibres have been accumulating over a period of 60 to 100 years from pulp
357 mill with different pulping processes (sulfite, chemi-thermomechanical pulping), raw
358 water intake systems, and different wastewater treatment methods. Various processes
359 have apparently occurred at the studied sediments in boreal conditions, e.g. annual ice
360 cover, water flows and temperatures. Previous research on the on-site degradation of
361 sedimented fibres has suggested that more rapid hydrolysis of fibres occurs in anaerobic
362 sediments that already contain high volumes of deposited fibres due to the enrichment

363 of hydrolytic bacteria in the anaerobic sediments (Pearson, 1980). Furthermore, it has
364 also been proposed that the long-term exposure of fibre discharges to sediments with
365 restricted water exchange will eventually lead to the the deoxygenation of the bottom
366 waters as well as the elimination of the fauna (Pearson, 1980). In the studied bay area
367 of Lake Näsijärvi, large volumes of fibres (1.5 million m³) have sedimented over a long
368 period of time (60 to 100 years) in a relatively small area (ca. 20 ha) with apparently
369 low water exchange. During the sedimentation period, the hydrolysis of the fibres has
370 likely occurred in the sediments followed by a decrease in the activity of the fauna. In
371 addition, the oxygen in the sediments is likely consumed in the beginning of the
372 sedimentation period leading to anaerobic conditions.

373

374 The organic content of the sediment was high (95% VS/TS, 12.4% TS and 11.7% VS),
375 while VS/TS ratios of 51-80% and 65-97% have been reported for pulp and paper mill
376 primary sludge (1.5–6.5% TS) and biosludge (1.0–2.0% TS), respectively (Meyer and
377 Edwards, 2014). Thus, the sedimented fibres have considerably higher VS/TS content
378 than the present pulp and paper mill sludge, indicating the higher degradation potential
379 of the sedimented fibres.

380

381 The sedimented fibres contained negligible concentrations of nutrients, i.e. 2.9–4.7 mg
382 N/kg TS and 0.27–0.31 mg P/kg TS. The low nutrient concentrations indicate that a
383 lack of nutrients may slow down the anaerobic treatment of sedimented fibres,
384 especially in continuous processes. Bayr and Rintala (Bayr and Rintala, 2012) reported
385 nitrogen concentrations of 0.1 and 1.9–2.0 g/L for primary sludge and biosludge,
386 respectively, from pulp and paper mills. In addition, Kinnunen et al. (2015) reported
387 phosphorous and nitrogen contents of 1.2-8.6 g P/kg TS and 41-81 g N/kg TS,

388 respectively, for pulp and paper mill biosludge. The low nitrogen content has been
389 reported to limit methane production from primary sludge and biosludge of pulp and
390 paper mills (Bayr and Rintala, 2012), and the low nitrogen content of the sedimented
391 fibres will likely also affect continuous methane production from sedimented fibres.

392

393 ***4.2. Solid-liquid separation of sedimented fibres***

394

395 The applied simple solid-liquid separation of the sedimented fibres simulated
396 mechanical dewatering, e.g. using a screw press. Screw and filter presses are often used
397 for dewatering sludge in municipal wastewater treatment plants as well as in the pulp
398 and paper industry (Ojanen, 2001; Saunamäki, 1997). Mechanical dewatering in the
399 pulp and paper industry is often enough to increase the solid content up to 30–40% TS
400 before, e.g. combustion, of the sludge but usually requires addition of polymers and/or
401 thermal treatment before dewatering (Ojanen, 2001). In this study, some of the water
402 was easily removed from the sedimented fibres without the addition of polymers and
403 >40% of the water could be removed to obtain a TS and VS content of an average
404 19.5% VS and 18.4% VS (94% VS/TS), respectively, while the potential for higher
405 solid fraction TS% was not attempted. Thus, using a screw press to separate water from
406 sedimented fibres should also be feasible at a larger scale and results in a solid fraction
407 with a high VS/TS content. The total and soluble COD content of the separated liquid
408 fractions was high, on average 8.8 ± 2.5 and 6.7 ± 3.1 g/L, respectively, and shows that
409 the liquid fraction requires treatment before discharge. The liquid fraction of
410 sedimented fibres was biodegradable with a soluble COD removal of 66–84%,
411 indicating the potential for biological treatment processes.

412

413 *4.3. Methane production potential from the sedimented fibres*

414

415 The results in this study show that by adding an inoculum and adjusting the pH near
416 neutral, which is optimal for methanogens, significant anaerobic degradation of
417 sedimented fibres (61-65% VS removal in total sedimented fibres) takes place in the
418 batch assays resulting in the production of methane. Batch incubations with sedimented
419 fibres only (without inoculum) at a neutral pH indicated that there are no indigenous
420 microorganisms in sedimented fibres that could convert fibres into methane (results not
421 shown). From solid fractions and total samples, the highest (and average) methane
422 yields were 320 L CH₄/kg VS (270±40 L CH₄/kg VS) and 340 L CH₄/kg VS (250±80
423 L CH₄/kg VS), respectively. From the liquid fractions, the methane yields were at the
424 highest (and in average) 280 L CH₄/kg COD (240±40 L CH₄/kg COD). Compared to
425 typical methane yields from pulp and paper industry primary sludge, biosludge or their
426 mixture (Table 5), the results from this study were higher. In addition, the VS removal
427 was considerably higher for total and solid fractions of sedimented fibres compared to
428 primary sludge and/or biosludge from pulp and paper industry (Table 5). The present
429 methane yields are in the same range as obtained in the typical sewage sludge digesters,
430 e.g. 260 L CH₄/kg VS (Luostarinen et al., 2009).

431

432 Table 5

433

434 In the batch assays of this study, the methane production started fast and ≥80% of the
435 methane was produced during the first two weeks. For comparison, in similar type of
436 batch assays it took 55 days (Bayr et al., 2013) and 40 days (Karlsson et al., 2011) to
437 reach ca. 80% of the methane yield from pulp and paper mill (ca. 110 mL CH₄/g VS)

438 and Kraft pulp mill (ca. 190 mL CH₄/g VS) biosludge, respectively. The fast methane
439 production in batch assays can be attributed to optimized conditions, i.e. a pH
440 adjustment to neutral (7–8) and the addition of microorganisms as well as nutrients
441 with inoculum (digestate from anaerobic treatment of municipal sewage sludge). The
442 high methane yields are surprising compared to the methane yields of primary sludge
443 and biosludge from the pulp and paper industry (Table 4) suggesting that the long
444 storage of the fibres in the sediments has likely resulted in the microbial hydrolysis of
445 the fibres (Pearson, 1980), enabling faster anaerobic degradation in the batch assays of
446 this study. It has been reported, for example, that lignin derived from hardwood can be
447 partly biodegraded in anoxic sediments in the long term (almost 300 d) studies (Benner
448 et al., 1984). Furthermore, Meriläinen et al. (2001) suggested that resin acids, wood
449 components that are inhibitory for anaerobic digestion (Meyer and Edwards, 2014), can
450 be microbially degraded in the long term and/or may be discharged to the receiving
451 water bodies. These factors, among others, may have resulted in better degradation and
452 biomethane production from sedimented fibres.

453

454 The results suggest that the studied sedimented fibres can be biologically treated as
455 such without pretreatment or after mechanical solid liquid separation (dewatering).
456 Both solid and liquid fractions contain biologically anaerobically degradable organics
457 and serve as a good feedstock for anaerobic bioprocessing, e.g. solids in continuously
458 stirred tank reactors and liquids in upflow anaerobic sludge blanket reactors. Different
459 reactor configurations should be considered for treating the different fractions, as solid-
460 liquid separation would considerably decrease the volume of the fraction (solid) that
461 contains most of the methane potential.

462

4.4. *Extrapolation of the methane production to practice*

464

465 There are approximately 1.5 million m³ sedimented fibres originating from the pulp
466 and paper industry in the studied bay area in Lake Näsijärvi, Finland. As the average
467 methane yield for the solid fractions of the sedimented fibres was similar to the methane
468 yield of total samples (250 L CH₄/kg VS), the anaerobic treatment of the solid fractions
469 after solid-liquid separation may be attractive and would result in smaller volumes to
470 be treated. In this case, however, the treatment of liquid fraction also has to be
471 considered.

472

473 In Table 6, the methane production potentials of the total, solid and liquid fractions of
474 the sedimented fibres in the practical scale are given based on the methane yields
475 obtained in the laboratory assays. From the samples analysed in this study, it can be
476 estimated that the organic matter content (VS) of the total samples is around 12%. Thus,
477 there are 0.19 million m³ sedimented fibres as VS. Based on these values (and assuming
478 a density of 1000 kg/m³ for the sedimented fibres), the overall methane production
479 potential of the total sedimented fibres is 44 million m³. As a comparison, the overall
480 methane production potential from the biowaste and the sludge from municipal
481 wastewater treatment plants and septic tanks in the city of Tampere and the surrounding
482 area (Pirkanmaa region; 500 000 inhabitants) is estimated to be ca. 5.6 million m³/year
483 (Mönkäre et al., 2016). Thus, the methane production potential from sedimented fibres
484 is 7 to 8 times higher than the yearly methane production potential of biowaste and
485 municipal sludge in the Pirkanmaa region. Anaerobic treatment of the sedimented fibres
486 in the studied bay area of Lake Näsijärvi (Finland) could generate income which to
487 certain extent, could compensate for the treatment and remediation costs

488

489 Table 6

490

491 In solid-liquid separation, on average 56% of the volume end up in the solid fraction
492 resulting in a volume of solid fraction of 0.84 million m³ corresponding to 0.16 million
493 m³ sedimented fibres as VS. Thus, the methane production potential of the solid
494 sedimented fibres is 39 million m³ (Table 6). In addition, solid-liquid separation of the
495 sedimented fibres would result in a large volume of liquid (0.66 million m³) that has on
496 average a total and soluble COD concentrations of 8.8±2.5 and 6.7±3.1 g/L,
497 respectively. With an average methane yield of 240±40 L CH₄/kg COD, the liquid
498 fraction has a methane production potential of 1.4 million m³ (Table 6). A typical
499 domestic wastewater contains 0.34–1.02 g COD/L depending on the strength of the
500 wastewater (Metcalf and Eddy, 2014). The design load of the new centralized regional
501 sewage treatment plant under construction in Tampere, Finland, is ca. 7,700 t BOD/year
502 in 2020 (Tampere Water, 2010). The liquid fraction of sedimented fibres would result
503 in an overall load of 5,800 t COD (Table 6). Assuming a BOD/COD ratio of 0.5, this
504 would convert to an overall load of ca. 2,900 t BOD, which is a bit over 30% of the
505 annual design load of the regional sewage treatment plant in Tampere. However, it is
506 likely that the remediation of the sedimented fibres will take several years and, thus,
507 the wastewater load would also be generated over many years. The COD concentrations
508 of the different pulp and paper mill wastewaters applicable for anaerobic treatment
509 typically range from 0.7 to 25 g/L (Meyer and Edwards, 2014). Thus, wastewater
510 treatment technologies suitable for pulp and paper mill wastewaters could also be used
511 to treat the liquid fractions of sedimented fibres. Examples of such processes include
512 anaerobic digestion in anaerobic filters, upflow anaerobic sludge bed reactors and

513 anaerobic membrane bioreactors (Kamali et al., 2016). Treating solid and liquid
514 fractions separately would enable the treatment of the solid fraction with longer HRTs,
515 while the liquid fraction could be treated anaerobically in reactors enabling shorter
516 HRTs and offering the possibility to consider, e.g. the combination of a leach bed
517 reactor and an upflow anaerobic sludge bed reactor.

518

519 The sedimented fibres in Lake Näsijärvi originating from the pulp and paper industry
520 are not unique. For example, the pulp and paper industry has resulted in lake pollution
521 and sedimented fibres all over the world, including Nordic countries, Canada and China
522 (Guo et al., 2016; Jackson, 2016; Ratia et al., 2013). Since many of the old industrial
523 sites have been situated close to large cities, problems arise when the water bodies are
524 transformed into recreational grounds. Thus, dredging the sedimented fibres from the
525 lakes and treating them will become increasingly important in the future.

526

527 Further studies on the treatment of the total sedimented fibres using different reactor
528 configurations and process conditions are ongoing with the aim to provide information
529 for the technical feasibility of different systems and to make cost analyses. In addition,
530 the digestates of the reactors will be characterized to develop the use of the digestates.

531

532 **5. Conclusions**

533

534 In this study, it was reported for the first time that the anaerobic degradation of old
535 sedimented fibres result in the production of methane with high methane yields (250 ± 80
536 L CH₄/kg VS). When the sedimented fibres are separated to solid and liquid fractions,
537 the solid fraction (ca. 56% of the original volume) has the highest methane production

538 potential ($230 \pm 60 \text{ CH}_4/\text{kg VS}_{\text{original}}$). However, the liquid fraction still contains a high
539 amount of COD ($8.8 \pm 2.5 \text{ g/L}$) that requires treatment before discharge. At the site
540 under investigation, there is methane production potential up to approximately 40
541 million m^3 .

542

543 **Acknowledgement**

544 This project was funded by the city of Tampere. The authors would like to thank the
545 “lifeguard” of Hiedanranta, Reijo Väliharju, from the city of Tampere for his support
546 during the project and Ramboll Finland Oy for providing the sedimented fibre samples
547 and Leena Ojanen for her help in the laboratory.

548

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- 639
- 640

641 **Figure captions**

642

643 Figure 1. The separation of the volume of the total sedimented fibre into solid or liquid
644 fractions (shown in arrows, %) and total (TS) and volatile (VS) solids in in the solid
645 and liquid fractions. The results are shown for different sampling points (A,B,C) and
646 for different depths. Solid-liquid separation was not done for sample A(0-1m).

647

648 Figure 2. Average methane production from different fractions of sedimented fibres
649 taken from different sampling points and depths: solid fractions from sampling points
650 A (A), B (B) and C (C) and total fibres from sampling point B (D) with minimum and
651 maximum values, and liquid fractions from sampling points A (E), B (F) and C (G)
652 with standard deviations.

653

654 Figure 3. The methane yields of the total as well as solid and liquid fractions of the
655 sedimented fibres from sampling point B. The methane yields are projected against the
656 VS of the total sample added to the incubation (A) or removed during the incubation
657 (B).

658

659

660 Table 1. Sampling depths at different sedimented fibre sampling points. The samples
661 were named based on this information.

Sampling point	Sampling depths (m)	Sample name
A	0-1	A(0-1 m)
	1-2	A(1-2 m)
	2-3	A(2-3 m)
B	0-1	B(0-1 m)
	3-4	B(3-4 m)
	5-6	B(5-6 m)
C	0-1	C(0-1 m)
	1-2	C(1-2 m)
	4-5	C(4-5 m)

662
663

664 Table 2. Total (TS) and volatile (VS) solids and the ratio of VS/TS for the inoculum
 665 and total and solid fractions of sedimented fibres as well as soluble (COD_s) and total
 666 (COD_t) COD for the liquid fractions of sedimented fibres.

Sample	Total			Solid fraction			Liquid fraction		
	TS (%)	VS (%)	VS/TS (%)	TS (%)	VS (%)	VS/TS (%)	COD _s (g/L)	COD _t (g/L)	COD _s /COD _t (%)
A(0-1 m)	21.6	20.7	95.7	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
A(1-2 m)	11.2	10.43	93.1	17.4	16.2	93.3	5.9	8.7	68
A(2-3 m)	11.1	9.6	87.0	18.2	15.9	87.0	6.2	8.2	76
B(0-1 m)	11.0	10.5	95.8	17.8	17.1	96.0	2.3	5.5	42
B(3-4 m)	13.9	13.5	97.3	20.6	19.8	96.2	9.1	9.9	92
B(5-6 m)	12.7	12.1	94.7	20.4	19.4	95.4	12.7	13.9	92
C(0-1 m)	9.9	9.5	95.3	21.1	20.2	95.6	5.8	9.0	64
C(1-2 m)	9.3	8.9	95.3	19.9	19.02	95.5	5.4	7.1	77
C(4-5 m)	10.9	10.3	94.7	20.7	19.7	95.0	5.9	8.1	73
Inoculum	2.6	1.4	54.5	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.

667 n.a. = not analysed

668

669 Table 3. Nutrients and organic acids present in the pore water samples of two
670 sedimented fibre samples (Autiola and Holopainen, 2016).

Parameter	Pore water	
	B(3-4m)	B(5-6m)
TN (mg/L)	43	57
P (mg/L)	9.5	5.9
NO ₃ ⁻ (mg/L)	< 1.0	< 1.0
Total PO ₄ ²⁻ (mg/L)	16	8.6
Formic acid (mg/L)	< 30	< 30
Acetic acid (mg/L)	1100	2900
Propionic acid (mg/L)	620	820
Butyric acid (mg/L)	640	890
Lactic acid (mg/L)	< 51	1800

671

TN = total nitrogen, P = phosphorous

672

673 Table 4. The characteristics of the digestates in the end of the assays. No VFAs were
 674 detected at the end of any of the assays.

Fraction	Sample	pH	TS (%)	VS (%)	VS/TS (%)	sCOD (mg/L)
Solid	A(0-1 m)	7.5	1.7	1.0	58.0	n.a.
	A(1-2 m)	7.6	1.3	0.8	61.4	n.a.
	A(2-3 m)	7.6	1.3	0.7	53.3	n.a.
	B(0-1 m)	7.5	1.4	0.8	58.8	n.a.
	B(3-4 m)	7.5	1.6	1.0	60.2	n.a.
	B(5-6 m)	7.5	1.7	1.0	57.4	n.a.
	C(0-1 m)	7.5	1.7	1.0	60.1	n.a.
	C(1-2 m)	7.5	1.5	0.9	58.6	n.a.
	C(4-5 m)	7.5	1.7	1.0	56.0	n.a.
Liquid	A(1-2 m)	7.0	1.3	0.6	46.8	490
	A(2-3 m)	7.0	1.4	0.7	46.4	530
	B(0-1 m)	7.0	1.4	0.7	47.5	460
	B(3-4 m)	7.0	1.4	0.7	46.6	410
	B(5-6 m)	6.9	1.4	0.8	46.5	490
	C(0-1 m)	7.0	1.4	0.7	46.8	460
	C(1-2 m)	6.9	1.3	0.6	47.9	500
	C(4-5 m)	7.0	1.5	0.7	46.1	500
Total	B(0-1 m)	7.5	1.8	1.0	57.6	n.a.
	B(3-4 m)	7.5	1.8	1.1	59.1	n.a.
	B(5-6 m)	7.5	1.8	1.0	55.7	n.a.

675 n.a. = not analysed

676

677 Table 5. Methane yields from primary sludge of the pulp and paper industry and from
 678 sedimented fibres used as substrate in this study.

Substrate	Batch/reactor	Methane yield (L CH₄/kg VS)	VS removal (%)	Reference
Primary sludge	CSTR	190-240	25-40	(Bayr and Rintala, 2012)
Mixture of primary and biosludge	CSTR	150-170	29-32	(Bayr and Rintala, 2012)
Mixture of primary and biosludge	CSTR	230	59	(Ekstrand et al., 2016)
Biosludge	Batch	100-200	n.g.	(Karlsson et al., 2011)
Biosludge	Batch	50-100	n.g.	(Bayr et al., 2013)
Biosludge	Batch	85-102	n.g.	(Kinnunen et al., 2015)
Total sedimented fibre	Batch	250 ± 80	61-65	This study
Solid fraction of sedimented fibre	Batch	270 ± 40	63-78	This study

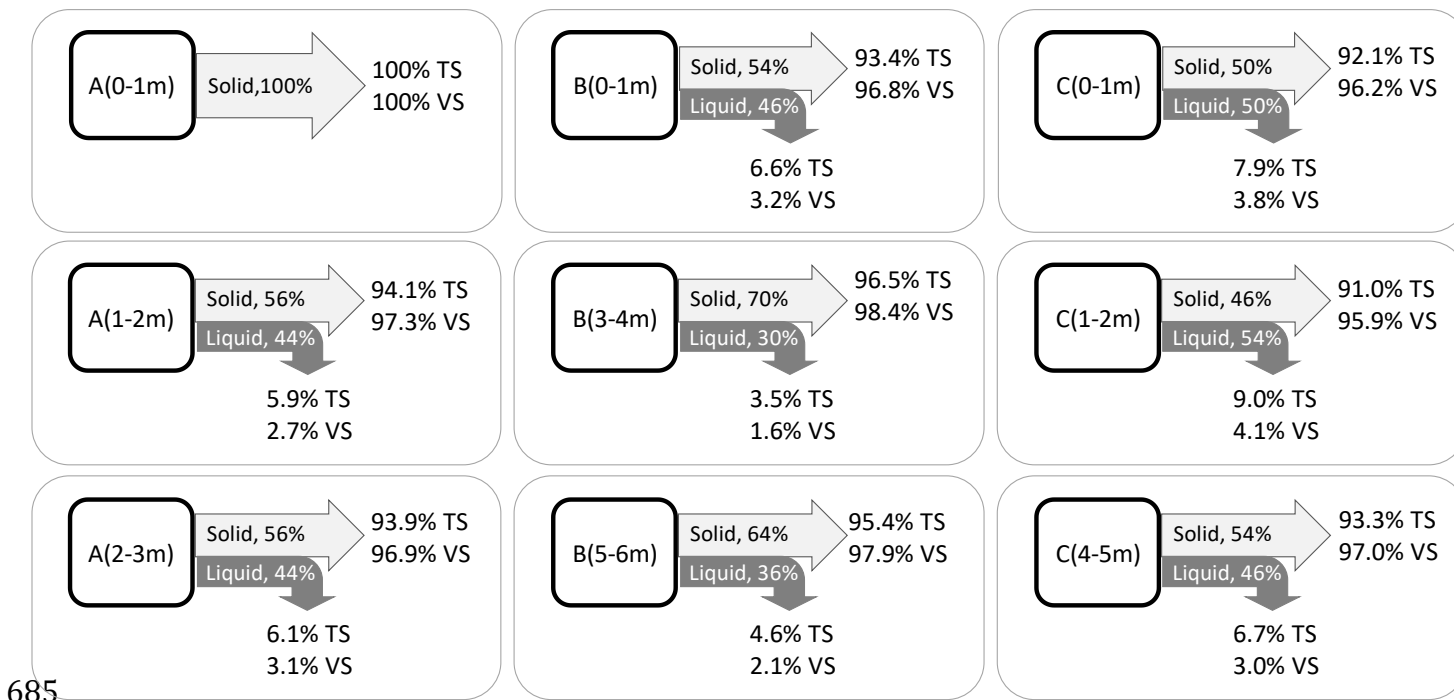
679 CSTR = completely stirred tank reactor, n.g. = not given

680

681 Table 6. Extrapolation of the methane production potential from total and solid
 682 fractions of sedimented fibres at a practical scale.

	Total sedimented fibre	Solid fraction of sedimented fibre	Liquid fraction of sedimented fibre
Volume (m³)	1 500 000	840 000	660 000
TS (%)	12.4	19.5	1.6
Volume (m³ TS)	186 000	164 t 000	11 000
VS (%)	11.7	18.4	0.7
Volume (m³ VS)	176 000	155 000	4 600
tCOD (g/L)	n.a.	n.a.	8.8
Total (t COD)	n.a.	n.a.	5 808
CH₄ production potential (million m³)	44	39	1.4

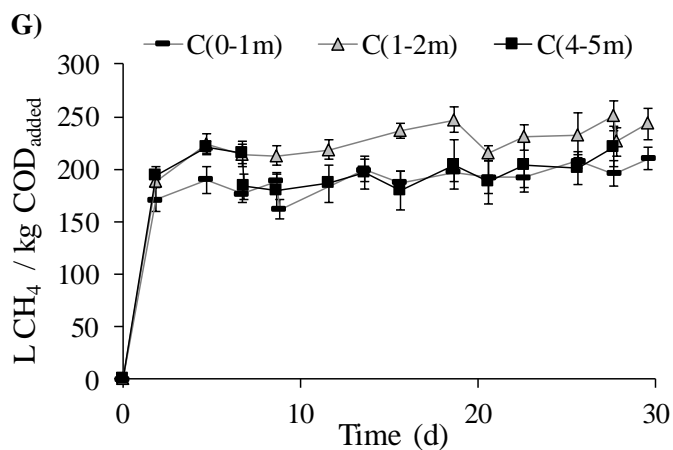
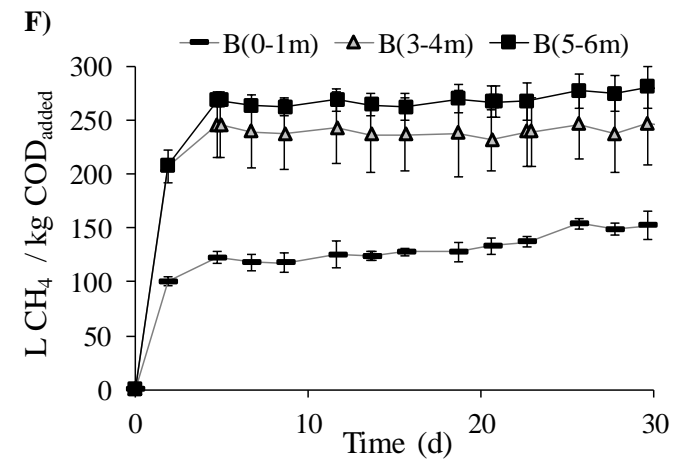
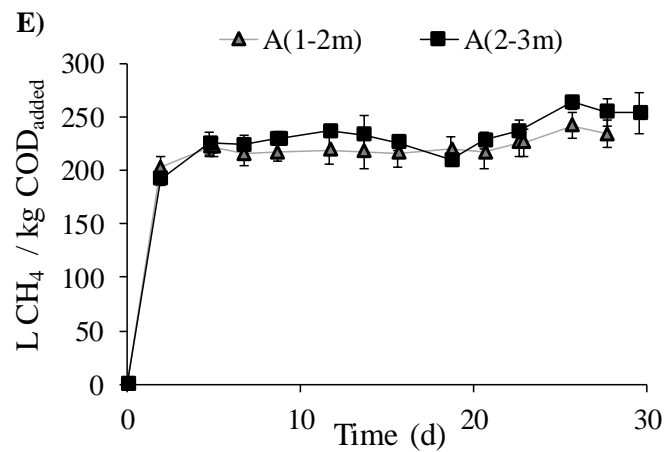
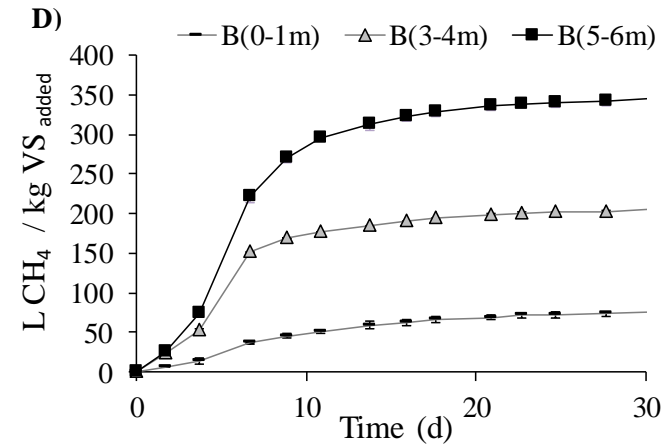
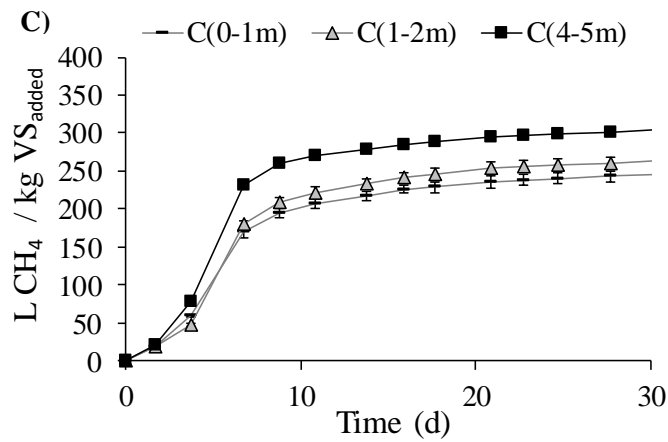
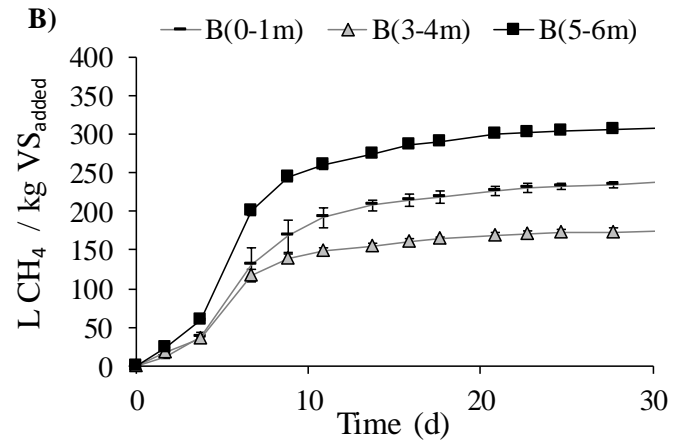
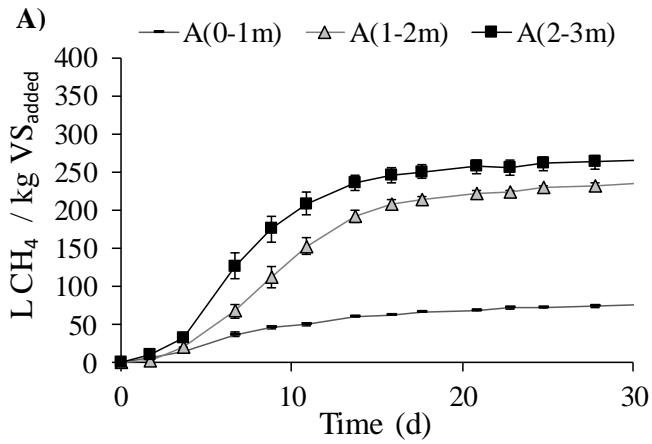
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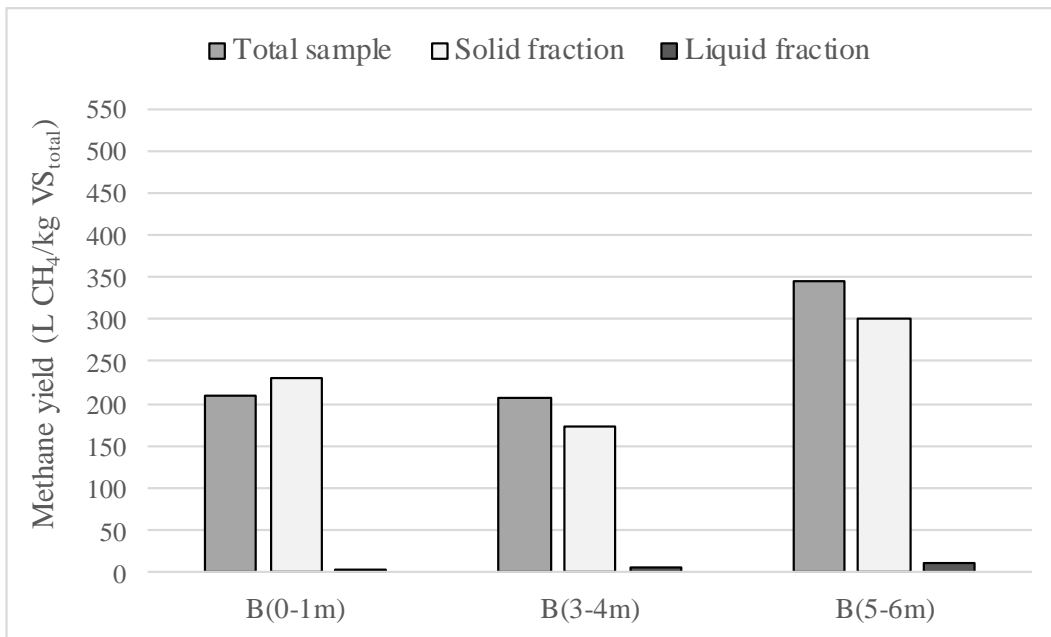
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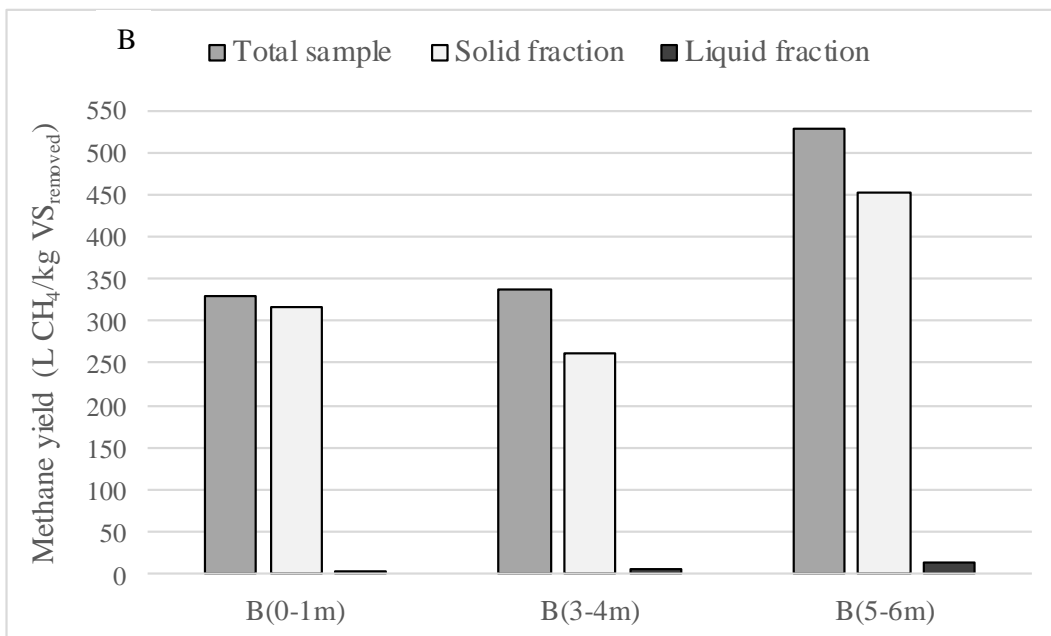
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696 with standard deviations.
697



698



699

700 Figure 3. The methane yields of the total as well as solid and liquid fractions of the

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702 the VS of the total sample added to the incubation (A) or removed during the

703 incubation (B).

704