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5
6 **Effect of Sawdust Addition and Composting of Feedstock on Renewable Energy**
7 **and Biochar Production from Pyrolysis of Anaerobically Digested Pig Manure**

8
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17
18 **A B S T R A C T**

19 Pyrolysis experiments were conducted on the separated solid fraction of anaerobically
20 digested pig manure (SADPM). The aim of these experiments was to investigate the
21 influence of (1) sawdust addition and (2) composting the feedstock, on the products of
22 pyrolysis and on the net energy yield from the pyrolysis process. Mixtures of SADPM
23 and sawdust were made to give the following treatments; manure only, 4:1(w/w) and
24 3:2(w/w). These mixtures were pyrolyzed at 600°C both before and after aerobic
25 composting. The yields of the biochar, bio-liquid and gas were influenced by the

26 addition of sawdust to the SADPM and by composting of the feedstock. With the
27 addition of sawdust, biochar and gas higher heating values (HHV) increased, while bio-
28 liquid HHV decreased. More than 70% of the original energy in the feedstock remained
29 in the biochar, bio-liquid and gas after pyrolysis, increasing as the proportion of sawdust
30 increased. The HHV of the biochar decreased, while the HHV of the bio-liquid
31 increased, after the feedstocks were composted. The energy balance showed that
32 increasing the rate of sawdust addition to SADPM resulted in an increased net energy
33 yield. The addition of a composting stage increased the net energy yield for the manure
34 only feedstock only. However, with increasing sawdust addition, composting of the
35 feedstock reduced the net energy yield.

36

37 *Keywords:* pyrolysis; pig manure; energy; sawdust; biochar.

38

39 **Abbreviations**

40 SADPM: separated anaerobically digested pig manure

41 NACP: nutra ammonium citrate soluble P

42 WSP: water soluble P

43 ABC: acid binding capacity

44 BUF: buffering capacity

45 TGA: thermo gravimetric analysis

46

47

48

49

50 **1 Introduction**

51 In the European Union (EU), thirty percent of sows are located in a major pig
52 production basin which stretches from Denmark, through north western Germany and
53 the Netherlands to Vlaams Gewest in northern Belgium [1]. Pig manure in these
54 concentrated pig farming areas has to be transported to less pig-dense areas for
55 landspreading, thereby increasing the cost of manure handling. Landspreading
56 legislation (Nitrates Directive, 91/676/EEC) [2] has reduced the amount of organic
57 fertilizers which can be spread on land, increasing the cost of manure disposal. There is
58 much interest in anaerobic digestion (AD) as a method of generating renewable energy
59 from manures. However, AD does not reduce the nutrient content of pig manure which
60 still needs to be recycled. One alternative to the problem of landspreading of these
61 digested manures may be to use pyrolysis to produce renewable energy and biochar.

62 Pyrolysis is a process whereby a biomass feedstock, such as woodchips, crop
63 residues or manure, is heated to high temperatures in an oxygen-free atmosphere to
64 produce a solid (biochar), a liquid (bio-liquid) and a gaseous fraction. The proportion of
65 biochar, bio-liquid and gas produced is dependant on the heating rate, the reactor
66 temperature and the residence time of the reactor [3,4]. Gases and bio-liquids produced
67 may be used to provide the energy needs for the pyrolysis process, or converted to
68 electricity and heat. The biochar can be used as a fuel or, alternatively, applied to land
69 as a low-grade fertilizer, soil addendum [5-8] or for carbon sequestration [9-12].
70 Biochar addition to soil may mitigate the negative effects of removing crop residues
71 from soil [13]. Studies in tropical soils of low organic matter (OM) have shown
72 increased biomass yield and plant growth using biochar additions in conjunction with
73 another nutrient source [5,14]. Biochar has been shown to reduce nutrient leaching in

74 soils [15,16], increase nutrient availability for plants and enhance the efficiency of
75 fertilizers [14]. Biochar has also been shown to influence the soil physico-chemical
76 properties, such as pH, porosity, bulk density and water holding capacity [8,14,17].

77 The use of manure biochars may have additional benefits to farmers, as it can be
78 used to alleviate the problems of nitrogen (N) leaching from soils which is a
79 characteristic of the use of manure as a fertilizer [9]. Research has shown an increase in
80 N availability in soil with the addition of biochar from chicken manure [18]. The
81 phosphorus (P) and potassium (K) contents of manure are almost completely recovered
82 in the biochar, leading to higher concentrations in the biochar than in the original
83 manure [19]. Due to its higher N, P and K concentrations, biochar from manure may
84 offer additional benefits as a low-grade fertilizer, even when used without other forms
85 of fertilisation.

86 Pyrolysis could be used as a tool to combat global warming which, according to
87 the International Panel on Climate Change, is very likely due to the combined
88 influences of greenhouse gas (GHG) increases and stratospheric ozone depletion [20].
89 The generation of renewable energy through pyrolysis has been shown to result in net
90 reductions in GHG emissions compared to fossil fuel combustion [21]. However, the
91 net energy generation from the drying and pyrolysis of manure has been shown to be
92 negative due to the high water content (WC) of manures [19]. The addition of sawdust
93 to separated manure reduces the WC and may result in a positive net energy yield.
94 Composting also reduces the WC of the feedstock [22] and may also be incorporated
95 into the treatment process to alter the net energy yield. Some research has been
96 performed on biochar production from manures [19,23,24]; however, no study has yet
97 characterized the biochar and renewable energy production from pig manure after AD.

98 The effect of composting the feedstocks before pyrolysis has also never been previously
99 studied. The objectives of this study were to: (1) investigate the effect of sawdust
100 addition on the products of pyrolysis of separated anaerobically digested pig manure (2)
101 analyze the effect of composting the feedstocks before pyrolysis, and (3) investigate the
102 effect of composting and sawdust addition to the energy balance.

103

104 **2 Materials and Methods**

105 ***2.1 Feedstock***

106 The pig manure was taken from an 800-sow pig farm in Roughty Valley Pig
107 Unit, Kilgarven, County Kerry, Ireland. This farm contained pigs from all stages of
108 production, fed with a dry feeding system and housed in slatted sheds above
109 underground manure storage tanks. The liquid manure was transferred from the
110 underground storage tanks to a 45m³ overground storage tank prior to anaerobic
111 digestion. The anaerobic digested manure was generated in a mesophilic anaerobic
112 digester with a residence time of 20 to 30 days. The anaerobically digested pig manure
113 was separated by decanter centrifuge (GEA Westfallia Separator UCD 205, Bönen,
114 Germany). The separated solids of AD pig manure (SADPM) had an average dry matter
115 content of 310 g kg⁻¹. This SADPM was mixed with Sitka Spruce sawdust, produced
116 without bark (WC = 144 g kg⁻¹ ±27 g kg⁻¹, bulk density = 40 kg m⁻³ ±2 kg m⁻³), at
117 different rates (4:1 and 3:2) based on their wet weights. These feedstocks were then
118 pyrolysed both before and after composting. The feedstocks were: SADPM only (MO);
119 SADPM and sawdust mixed at a 4:1 ratio (4:1); SADPM and sawdust mixed at a 3:2
120 ratio (3:2); composted SADPM (MO(CP)); SADPM and sawdust mixed at a 4:1 ratio
121 and composted (4:1(CP)), and SADPM and sawdust mixed at a 3:2 ratio, and

122 composted (3:2(CP)). The composting process consisted of 56 day aerobic composting
123 in small-scale (40 - 50 kg of compost was produced for each mixture of SADPM and
124 sawdust) composting tumblers similar to the methods described by Nolan et al. [25]. All
125 materials were dried at 60°C for 48 hours and milled before storage in a refrigerator
126 (~4°C) prior to pyrolysis and analysis.

127

128 **2.2 Pyrolysis**

129 A laboratory-scale pyrolysis reactor (5cm i.d.) was used to produce the biochar,
130 bio-liquid and gases. A temperature of 600±10 °C and a residence time of 15 minutes
131 were used for all feedstocks. Approximately 50g of feedstock was used for each
132 pyrolysis run. The vapor exhaust end of the pyrolysis reactor was connected to a
133 condensing zone which was maintained at a temperature of -7±1°C. A flow of 50
134 cm³·min⁻¹ N₂ for 15 minutes before pyrolysis commenced, ensured that the atmosphere
135 inside the reactor was oxygen free. The vapours produced by the pyrolysis process were
136 carried through to the condensing zone, where the condensable fractions were converted
137 to bio-liquid. The bio-liquid was collected in a round bottomed flask at the end of the
138 condensing zone and the gases were collected in Tedlar gas sampling bags.

139 After pyrolysis, the biochar was moved to the cooling zone of the pyrolysis
140 reactor and allowed to cool in the N atmosphere before being removed from the reactor.
141 The mass of the biochar and bio-liquid was determined after pyrolysis to allow
142 calculation of the percentage yield of biochar and bio-liquid. Gas yield was calculated
143 from the difference after biochar and bio-liquid yield calculation.

144

145 **2.3 Chemical and Physical Properties of Biochar**

146 Feedstock and biochars were subjected to proximate analysis for WC (B/EN
147 14774:3:2009), ash content (BS/EN 14775:2009) and volatile matter content (VM)
148 (BS/EN 15148:2009).
149 Ultimate analyses of duplicate samples of feedstock and the biochars were performed.
150 Samples were analyzed for total carbon (C), nitrogen (N), sulphur (S) and hydrogen (H)
151 contents in accordance with CEN/TS 15104:2005. Elemental analysis was undertaken
152 using a “CHNOS Elemental Analyser Vario EL Cube” (Elemental Analysensysteme
153 GmbH, Hanau, Germany) operated at a combustion temperature of 1100 – 1200°C. All
154 analysis was performed on a dry basis (db). H/C ratio was calculated from the ultimate
155 analyses as the mol ratio on a dry basis. Higher heating value (HHV) (MJ kg^{-1}) was
156 calculated from the ultimate analyses using the equation (1) [26]:

157

$$158 \text{ HHV} = 0.3491 \text{ C} + 1.1783 \text{ H} + 0.1005 \text{ S} - 0.1034 \text{ O} - 0.0151 \text{ N} - 0.0211 \text{ Ash} \quad (1)$$

159

160 Biochar pH measurements were performed in water solution using a bench top
161 meter (SevenEasy, Mettler-Toledo, Switzerland) at a biochar/distilled water ratio of
162 1:10 (w/v). Acid binding capacity (ABC) and buffering capacity (BUF) were measured
163 after Lawlor et al. [27] to final pH values of 6.5 and 7. In this method, 0.5g of biochar
164 was suspended in 50cm^3 de-ionized water and continuously stirred. Titrations of 100
165 mol m^{-3} hydrochloric acid were performed using increments from 0.01cm^3 to 0.1cm^3
166 until a pH of 6.5 and a pH of 7 were attained. Acid binding capacity was calculated as
167 the amount of acid in milli-equivalents required to reach the target pH. Buffering
168 capacity was calculated by dividing the ABC by the change in pH units. This test was
169 repeated using lime to compare the potential of the biochar as a replacement for lime.

170 The fertilizer value of the biochar was investigated by determining the
171 concentrations of P and K. The amount of P available to plants is given by the nutra
172 ammonium citrate soluble P (NACP) concentration [28]. This test is used to denote the
173 P content of common fertilizers. Formic acid soluble P relates to less available P [29],
174 and is often found in high concentrations in slow release fertilizers, often utilised in
175 forest plantations. Total P was analyzed by acid digestion according to Standard Method
176 4500-P B&E [30]. Water soluble P, nutra ammonium citrate soluble P, and formic acid
177 soluble P were determined after Byrne [31]. Total K was also determined after Byrne
178 [31]. Brunauer-Emmet-Teller (BET) surface area of the biochars was measured by
179 nitrogen gas sorption analysis at 77K using a surface area analyser (Micromeritics
180 Gemini 2375 V5.01). Samples were pre-treated by degassing at 300°C for 4 hours in
181 nitrogen gas.

182

183 ***2.4 Thermo Gravimetric Analysis***

184 The six feedstocks were subject to thermo gravimetric analysis runs (TGA)
185 using a TGA analyser (Q600 TGA/DTA Thermal Analyser System, TA Instruments).
186 Samples were placed in open top alumina crucibles and were pyrolised with N as a
187 carrier gas at a flow rate of 100cm³ min⁻¹. A temperature ramp of 10K min⁻¹ from
188 ambient temperature up to 600°C was used to create a temperature-based mass profile.

189

190 ***2.5 Gas and Bio-liquid Analyses***

191 Elemental analyses of duplicate samples of the bio-liquids were preformed in
192 accordance with CEN/TS 15104:2005 as above. Samples were analyzed on the same
193 day as production to ensure there was no reduction in the quality of the bio-liquids

194 associated with storage. Higher heating values (MJ kg^{-1}) were calculated from the
195 ultimate analyses using equation (2) [32]:

196

$$197 \quad \text{HHV} = 3.55 \text{ C}^2 - 232 \text{ C} - 2230 \text{ H} + (51.2 \text{ C} \cdot \text{H}) + 131 \text{ N} + 20600 \quad (2)$$

198

199 Analysis of the major non-condensable gases produced by pyrolysis was
200 undertaken using a gas chromatograph (Agilent Micro GC, Agilent Technologies). The
201 yields were quantified by calibration with a standard gas mixture consisting of N_2 , H_2 ,
202 CO , CO_2 , CH_4 , C_2H_4 , C_2H_6 and C_2H_2 in helium. Higher heating values ($\text{MJ} \cdot \text{m}^{-3}$) under
203 standard conditions were calculated from the gas composition results using the equation
204 (3) [33]. Standard conditions were taken as a temperature of 20°C and a pressure of 100
205 kPa.

206

$$207 \quad \text{HHV} = 39.82 \text{ CH}_4 + 12.63 \text{ CO} + 12.74 \text{ H}_2 + 7.29 \text{ C}_2\text{H}_6 + 63.42 \text{ C}_2\text{H}_4 + 58.06 \text{ C}_2\text{H}_2. \quad (3)$$

208

209 **2.6 Energy Balance**

210 The energy required to treat digested manures, through pyrolysis, can be split
211 into four processes: separation, composting, drying, and pyrolysis (Figure 1). The
212 separation energy requirements of anaerobically digested pig manure using a decanter
213 centrifuge was taken as 7.92 MJ t^{-1} manure input [34] and the quantity of manure solids
214 produced per tonne of anaerobically digested pig manure input was taken as 0.07 t [35].
215 The energy use during composting was estimated to be 18.4 MJ t^{-1} input material [36].
216 The overall mass reduction of the composting pile from the beginning to end of the
217 composting process was taken to be 50% [37], including a reduction of 15% in the

218 water content. The drying energy requirement was estimated by adding the heat
219 required to increase the temperature of the wet feedstock from ambient (10°C) to 100
220 °C, plus the latent heat required to evaporate the water from the feedstock [19]. The heat
221 capacity of the wet manure was estimated to be 3.292 kJ kg⁻¹ K⁻¹ [38], while the heat
222 capacity of sawdust with a WC of 120 g kg⁻¹ was estimated to be 1.5 kJ kg⁻¹ K⁻¹ [39].
223 The efficiency of the drying process was estimated at 80% [19]. The energy requirement
224 for pyrolysis was the energy required to increase the temperature of the dried feedstock
225 from 100 °C to 600 °C. The heat capacity of the dried manure was estimated to be 0.92
226 kJ kg⁻¹ K⁻¹ [38], while the heat capacity of dry sawdust was estimated to be 2.51 kJ kg⁻¹
227 K⁻¹ [39]. The energy loss during pyrolysis is estimated to be 5% [19].

228 There are two output options investigated (Figure 1) with regards to the end use
229 of the products (bio-liquid, biochar and gases). For option one, all three pyrolysis
230 products are used as a fuel. Option two uses the bio-liquid and gases as a fuel, while the
231 biochar is used as a soil addendum. When calculating the energy production from
232 combustion of the biochar, bio-liquid and gases, a combined heat and power (CHP)
233 efficiency of 80% was assumed.

234 “(Figure 1 here)”

235

236 **2.7 Statistical Analysis**

237 Data was analyzed using the Statistical Analyses System (SAS, V9.1.3, 2002-
238 2003). Comparison of yield, elemental analyses, proximate analyses and HHV were
239 performed using the Proc Mixed SAS procedure. Treatment was included as a fixed
240 effect. For all analyses, significance was at $p < 0.05$.

241

242 **3 Results and Discussion**

243 **3.1 Feedstock Characteristics**

244 Proximate and ultimate analyses of the biomass feedstock are given in Table 1.
245 The addition of sawdust to the SADPM resulted in higher VM and FC contents ($p <$
246 0.001 and $p < 0.01$, respectively), while the ash and N contents decreased with sawdust
247 addition ($p < 0.01$ for both). Feedstock MO(CP) was an exception to this, where the N
248 content was unusually low. The low N content was most likely caused by leaching
249 which occurred from this feedstock during the composting process. There was an
250 increase in C contents when sawdust was added to the SADPM, although this increase
251 was not significant ($p = 0.26$). There was no significant difference in feedstock HHV
252 with increasing sawdust addition ($p > 0.05$). Composting of the feedstocks resulted in
253 lower VM contents ($p < 0.001$). Ash contents were also higher, although the increase
254 was not significant ($p > 0.05$). Organic matter is decomposed by microorganisms during
255 composting, resulting in higher ash contents, while VM is lost as gases during the
256 composting process [22]. There was a reduction in C contents after composting,
257 although the decrease was only significant in the 4:1 treatment ($p < 0.05$). Carbon is lost
258 during the composting process, primarily as carbon dioxide [22]. The decrease in C
259 contents and the increase in ash contents, following composting, resulted in a decrease
260 in HHV in the composted feedstock ($p < 0.001$).

261 “(Table 1 here)”

262

263 **3.2 Biochar Characteristics**

264 The biochar yields obtained from the pyrolysis process are shown in Figure 2.
265 Biochar yield was influenced by feedstock composition. However, composting of the

266 feedstock had no significant effect of biochar yield ($p > 0.05$). The addition of sawdust
267 to SADPM reduced biochar yield significantly ($p < 0.05$). In a study using poultry
268 manure [24], the increasing addition of wood also resulted in decreasing biochar yield.
269 The higher biochar yield for the manure only feedstock was due to changes in the high
270 ash content of the manure, which decreased with sawdust addition (Table 1).

271 “(Figure 2 here)”

272 The results of proximate and ultimate analyses of the biochars are shown in
273 Table 2. The C content of the biochar increased as the proportion of sawdust in the
274 feedstock was increased ($p < 0.01$ for non-composted feedstock and $p < 0.05$ for
275 composted feedstock). This resulted in an increase in HHV and a reduction in the H/C
276 ratio ($p < 0.05$ for both). This was a result of the higher C content and lower ash
277 contents of the feedstocks with added sawdust. The FC was also increased ($p < 0.05$),
278 while ash and N contents decreased with sawdust addition ($p < 0.05$ and $p < 0.001$,
279 respectively). Composting of the feedstock increased VM and N contents in the sawdust
280 amended treatments only ($p < 0.05$ and $p < 0.001$, respectively). In the MO feedstock,
281 there was no significant increase in VM and N contents when the feedstocks were
282 composted ($p = 0.83$ and 0.13 , respectively). Carbon and FC contents in the biochars
283 were not significantly changed when the feedstocks were composted ($p > 0.05$ for both).
284 With composting, biochar HHV was not significantly changed ($p > 0.05$). Biochar
285 surface area increased with sawdust addition to the SADPM, but decreased when the
286 feedstocks were composted.

287 “(Table 2 here)”

288 The MO biochars without sawdust addition had a HHV lower than a low-rank
289 coal (lignite, 16 MJ.kg^{-1}). The 3:2 biochars with the higher rate of sawdust addition had

290 a HHV in the range of bituminous coal (17-23 MJ kg⁻¹), and above that of peat (14-21
291 MJ kg⁻¹). On the basis of yield and HHVs of the feedstock and biochar, 26-38% of the
292 non-composted feedstocks energy was retained in the biochar. However, when the
293 feedstock was composted, this proportion increased to 35-44%.

294 The H/C ratios decreased from 1.28 and greater for the feedstocks to less than
295 0.50 in the biochars. This reduction indicates an increase in aromaticity in the biochars
296 when compared to the feedstocks. Charring of lignin and cellulose has been reported to
297 cause loss of aliphatic components along with the conversion of ring structures into
298 aromatic compounds [40]. This increase in aromaticity can also be seen in the increased
299 FC contents of the biochars when compared to their feedstock. Increases of 181-243%
300 were shown in the FC contents of biochars from non-composted feedstock, while
301 increases of 117-222% were found in the FC contents of biochars from composted
302 feedstock.

303 The concentration of total P, the soluble fractions of P and total K in the biochar
304 are given in Table 2. Composting of the feedstock resulted in no major change in
305 biochar total K concentrations and small increases in total P concentrations. Biochar
306 total P and total K decreased with increasing sawdust addition. The amount of P leached
307 from soil is dependant on the amount of water soluble P (WSP) available [41].
308 Concentrations of WSP are generally very high (150-500 g kg⁻¹) in super phosphate
309 fertilizers. The WSP concentrations in the biochars studied are very low (< 0.16 g kg⁻¹),
310 indicating unsuitability as a fast release fertilizer. However, it also indicates that P
311 leaching from the biochar would probably be very small and that biochars might be
312 suitable as a slow release P fertilizer.

313 The concentration of NACP decreased when feedstocks were composted and
314 when sawdust was added, reducing the fertilizer value of the biochar. The percentage of
315 total P which is available to plants also decreases when feedstocks are composted and
316 amended with sawdust. Plant-available P decreased from 97% of total P for the MO
317 biochar to 32% of the total P for the 3:2(CP) biochar. Formic acid P accounted for
318 between 14% and 34% of the total P concentration. There was a decrease in formic acid
319 P concentrations with sawdust addition and after composting of the feedstock.

320 Acid binding capacities and BUF to achieve pH values of 6.5 and 7.0 are given
321 in Table 3. The optimum pH for grass, wheat, barley and maize is reported to be 6.5,
322 while a soil pH of 7 is recommended for white clover, beet, beans, peas and oilseed rape
323 [42]. The biochar from SADPM alone had the highest BUF for both pH 6.5 and 7.0.
324 The addition of sawdust to the SADPM resulted in a lower ABC and BUF in the
325 biochar for both pH values. Composting of the feedstock also reduced ABC and BUF.
326 In comparison, when this test was repeated using lime in place of biochar, values of 161
327 and 307 were recorded for BUF-6.5 and BUF-7, respectively. This demonstrates that
328 lime has a significantly greater buffering capacity, indicating that while biochars offer
329 some ability to increase soil pH when added to acid soils, they are not an adequate
330 replacement for lime.

331 “(Table 3 here)”

332

333 ***3.3 Thermo Gravimetric Analyses***

334 Figure 3 (a) and (b) show the mass (Thermo gravimetric (TG)) and derivative of
335 TG curves (DTG) of the feedstocks, respectively. The TG analyses show the loss of
336 mass of the feedstock as the temperature increases from ambient temperature to 600°C.

337 The primary devolatilisation stage is indicated by the highest peaks on the DTG curve
338 (Figure 3b). The temperatures of primary devolatilisation are shown in Table 4. The
339 onset temperature (T_{on}) was determined as the point of 5% loss of weight (db). The
340 temperature at maximum devolatilisation (T_{max}) was determined from the peaks on the
341 DTG curves. The final temperature of primary devolatilisation (T_f) was determined
342 from the DTG curve as the temperature corresponding to the intersection of the tangent
343 lines in the primary and secondary devolatilisation stages. The final temperature of
344 primary devolatilisation was similar for all feedstock at $370\text{ }^{\circ}\text{C} \pm 6\text{ }^{\circ}\text{C}$. The temperature
345 range (T_r) of devolatilisation is the difference between T_f and T_{on} . The primary
346 devolatilisation stage is followed by the secondary devolatilisation stage (Figure 3b)
347 when heavier chemical structures are degraded [43].

348 Similarly to the results seen in Section 3.2, sawdust addition and composting of
349 the feedstocks reduced biochar yield. The MO feedstocks had a greater secondary
350 devolatilisation stage, indicating the presence of heavier chemical structures in the
351 manure [43]. This stage was reduced with sawdust addition.

352 The addition of sawdust to SADPM increased T_{on} due to the presence of more
353 complex compounds in the sawdust. Composting of the feedstock caused a reduction in
354 T_{on} . This is due to the degradation of complex compounds into simpler molecules
355 during the composting process [22]. These simpler compounds are more easily
356 volatilised at lower temperatures. The temperature at maximum devolatilisation
357 increased with sawdust addition and with composting of the feedstocks. The
358 temperature range reduced with sawdust addition and increased with composting of the
359 feedstock due to the changes in T_{on} .

360 “(Table 4 here)”

361 “(Figure 3 here)”

362

363 **3.4 Characteristics of Bio-liquid and Gas**

364 Bio-liquid yield ranged from 110 to 300 g kg⁻¹ (Figure 2). Sawdust addition to
365 SADPM increased bio-liquid yield ($p < 0.01$), however, composting of the feedstock did
366 not significantly alter the yield ($p > 0.05$). Between 21 and 46% of the volatiles were
367 found to be condensable. Similar to the overall bio-liquid yield, sawdust addition and
368 composting increased the proportion of volatiles which were condensable. Elemental
369 analyses of the bio-liquids are shown in Table 5. Carbon contents of the bio-liquids
370 decreased as the proportion of sawdust in the feedstock increased ($p < 0.01$). This
371 results in a decrease in HHV ($p < 0.01$). Mante and Agblevor [24] also found increased
372 bio-liquid yields, decreased C and N contents and HHV values by adding wood to
373 poultry manure. Composting of the feedstock resulted in increased C and N contents (p
374 < 0.01 and $p < 0.05$, respectively) and HHV of the bio-liquids ($p < 0.01$).

375 “(Table 5 here)”

376 Gas yield ranged from 340 to 510 g kg⁻¹ (Figure 2). Yields decreased
377 significantly when the feedstocks were composted ($p < 0.05$). This is because gases,
378 primarily carbon dioxide (CO₂) and ammonia (NH₃), but also nitrous oxide (N₂O) and
379 methane (CH₄), are lost during the composting process [22]. The compositions of the
380 non-condensable gases are given in Table 6. As N₂ was used as a carrier gas during
381 pyrolysis, the proportion found in the produced gas was artificially high. Therefore, the
382 proportion of N₂ in the manure only feedstock was estimated to be 10%, as found in Ro
383 et al. [19] and this was revised downwards for all other feedstocks based on the
384 proportion of manure to sawdust in the feedstock. The largest component gas in the

385 mixtures was CO₂ (35-48%) followed by CO (15-37%). CO₂ decreases the overall
386 quality of the gas produced, therefore, pyrolysis conditions which reduce the production
387 of CO₂ are desirable [13,23]. Increasing the rate of sawdust addition to SADPM
388 decreased the CO₂ concentration and increased the CO concentrations in the gas
389 produced, similar to the finding of a similar study by Mante and Agblevor [24] using
390 poultry manure. The addition of sawdust to the SADPM also increased the
391 concentrations of CH₄ and H₂, while there was a reduction in C₂H₄ concentration. There
392 was little difference in the overall gas composition or HHV when the feedstock was
393 composted.

394 “(Table 6 here)”

395 More than 54% of the original energy in the feedstock remained in the biochar,
396 bio-liquid and gas after pyrolysis. This figure ranged from 54-81% for the non-
397 composted feedstock, increasing as the proportion of sawdust increased. For the
398 composted feedstock, the figure was higher (70-94%) and rose with increasing sawdust
399 addition.

400

401 **3.5 Energy Balance**

402 The inputs required and the outputs obtained from each feedstock are given in
403 Table 7. All values are calculated on the basis of one tonne of liquid pig manure after
404 anaerobic digestion.

405

406 **3.5.1 Inputs**

407 There are no differences between feedstocks for the separation energy
408 requirement. However, following separation, different quantities of sawdust were mixed

409 with the separated manure solids to form the various feedstocks (Table 7). The addition
410 of sawdust to SADPM increased the energy requirements for composting, drying and
411 pyrolysis, due to the increased mass of the feedstock. The quantity of bio-liquid, gases
412 and biochar produced were increased, resulting in greater energy generation.

413 The input energy required for the composting process was relatively small in
414 comparison to the other processes. However, the inclusion of a composting step had a
415 large influence on the energy requirements of the drying and pyrolysis processes. The
416 energy required for drying showed a decrease of approximately 58% when a
417 composting stage was included. The energy required for pyrolysis also showed a
418 reduction of between 31 and 43% with the addition of a composting stage. These
419 reductions were due to a mass reduction of 50%, including a 15% decrease in water
420 content, which occurs during the composting process [37].

421

422 ***3.5.2 Outputs***

423 The amount of biochar, bio-liquids and gases is dependant on whether a
424 composting stage is applied. The quantity of biochar produced decreased by between 20
425 and 38% when a composting stage was incorporated. Similarly, bio-liquid and gas
426 production decreased by between 36 and 47% with the addition of a composting stage.

427 The severity of these reductions increased with sawdust addition. This resulted in
428 reduced energy generation when a composting step was applied. Therefore, although the
429 inclusion of a composting stage reduced the energy requirements for drying and
430 pyrolysis, there was a decrease in energy production.

431

432 ***3.5.2.1 Output 1: Using Biochar as a Fuel***

433 The results of using the biochar produced through pyrolysis as a fuel are shown
434 in Table 7. There was a positive net energy yield for all feedstocks, except for the MO
435 treatment. However, the addition of sawdust to SADPM substantially increased the net
436 energy yield. The addition of a composting stage resulted in a reduced net energy yield
437 with the exception of the MO treatment, where the addition of a composting stage
438 changed a negative net energy yield to a positive net energy yield.

439

440 *3.5.2 Output 2: Using Biochar as a Soil Addendum*

441 The results of using the biochar produced through pyrolysis as a soil addendum
442 are shown in Table 7. There was a positive net energy yield only when sawdust was
443 added to SADPM. For the MO treatment, the energy generated from the combustion of
444 the bio-liquid and gas was not sufficient to sustain the treatment processes. The addition
445 of sawdust substantially increased the net energy yield. The addition of a composting
446 stage had a positive effect in treatments MO and 4:1, while for 3:2, the addition of a
447 composting stage reduced the net energy yield. Some feedstocks did show a small
448 increase in the biochar nutrient concentrations (Table 2) when a composting step was
449 incorporated. However, the nutrient concentration of the biochar produced decreased
450 with sawdust addition to the manure (Table 2), decreasing the value of the biochar as a
451 soil addendum.

452 *“(Table 7 here)”*

453

454 **4 Conclusions**

455 The influence of (1) the addition Sitka Spruce sawdust and (2) composting of the
456 feedstock on the products of pyrolysis of anaerobically digested pig manure was

457 significant. The proportion of biochar, bio-liquid and gas produced, and the physical
458 and chemical characteristics of these products were influenced by both sawdust addition
459 and feedstock composting. Increasing the sawdust content in the wood/manure mixture
460 decreased the biochar yield and increased the bio-liquid yield. The biochar showed
461 increased heating values, but reduced nutrient concentrations with increasing sawdust
462 addition. The heating value of the gases produced also increased, while that of the bio-
463 liquid was decreased with sawdust addition. Composting of the feedstock before
464 pyrolysis increased the biochar and bio-liquid yield, but decreased the gas yield. The
465 biochar showed reduced heating values, while the bio-liquid heating values were
466 increased with composting.

467 The net energy yield from biochar and renewable energy production increased
468 with sawdust addition to the manure digestate. The energy yield also increased when the
469 manure only (without sawdust addition) feedstock was composted before pyrolysis.
470 However, with increasing sawdust addition, composting of the feedstock reduces the net
471 energy yield. If the biochar is used as a fuel, all feedstocks except for the non-
472 composted manure only feedstock, produced a positive net energy yield. Should the
473 biochar be used as a soil addendum, then sawdust addition to the manure may be
474 required to produce a positive energy yield.

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596 **Figure Captions**

597 Figure 1: Schematic outlining the process, inputs and outputs for the energy balance.

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599 Figure 2: Pyrolysis product yield: effect of sawdust addition and composting of
600 feedstock.

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602 Figure 3: (a) Mass (TG) curves and (b) derivative mass (DTG) curves during N
603 pyrolysis: ambient – 600°C, 10K min⁻¹; mass (%) is the percentage of actual weight.

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620 **Table 1. Proximate and Ultimate Analysis of Feedstock before Pyrolysis**

Parameters	MO	4:1	3:2	MO (CP)	4:1 (CP)	3:2 (CP)
Water content (g kg ⁻¹)	85	70	74	90	62	58
Volatile matter (g kg ⁻¹ _{db})	697	718	755	628	663	740
Fixed C (g kg ⁻¹ _{db})	81	150	163	108	171	170
Ash (g kg ⁻¹ _{db})	222	132	82	264	166	90
N (g kg ⁻¹ _{db})	45	30	17	19	42	30
C (g kg ⁻¹ _{db})	452	476	490	406	414	442
H (g kg ⁻¹ _{db})	51	53	52	47	47	51
O (g kg ⁻¹ _{db})	219	301	354	258	319	378
H/C (mol ratio _{db})	1.37	1.34	1.28	1.39	1.35	1.37
HHV (MJ kg ⁻¹)	19.1	19.5	19.5	16.5	16.4	17.3

621 db=dry basis; (CP)=composted feedstock; HHV=higher heating value

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Table 2. Yield, Chemical Analysis, Higher Heating Value and Surface Area of Biochar

Parameters	MO	4:1	3:2	MO(CP)	4:1(CP)	3:2(CP)
Yield (g kg ⁻¹)	434	321	306	493	391	345
Water content (g kg ⁻¹)	53	55	77	30	48	58
Volatile matter (g kg ⁻¹ _{db})	226	166	171	230	204	192
Fixed C (g kg ⁻¹ _{db})	262	423	561	233	372	547
Ash (g kg ⁻¹ _{db})	512	412	268	536	424	261
N (g kg ⁻¹ _{db})	38	27	22	40	33	27
C (g kg ⁻¹ _{db})	338	516	669	330	501	624
H (g kg ⁻¹ _{db})	10	11	11	13	11	13
H/C (mol ratio _{db})	0.34	0.26	0.20	0.48	0.26	0.25
HHV (MJ kg ⁻¹)	11.3	18.2	24.0	11.6	17.9	22.1
Total P (g kg ⁻¹ _{db})	31.8	23.4	23.7	32.1	30.2	25.9
Water Sol. P (g kg ⁻¹ _{db})	0.13	0.15	0.15	0.11	0.09	0.09
NACP (g kg ⁻¹ _{db})	30.7	19.6	14.8	27.3	15.1	8.4
Formic Acid P (g kg ⁻¹ _{db})	10.8	7.5	5.5	8.8	5.9	3.6
Total K (g kg ⁻¹ _{db})	16.1	12.3	9.0	16.6	12.3	8.6
S _{BET} (m ² g ⁻¹)	17.0	19.4	24.4	14.2	17.4	23.2

645 db=dry basis; (CP)=composted feedstock; HHV=higher heating value; S_{BET}=BET surface area,

646 NACP=Nutra Ammonium Citrate Soluble P.

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666 **Table 3. pH, Acid Binding Capacity (ABC) and Buffering Capacity (BUF) of Biochar**

Parameters	MO	4:1	3:2	MO (CP)	4:1 (CP)	3:2 (CP)
pH	9.3	9.0	8.9	9.4	9.5	8.7
ABC-6.5	158	90	42	140	72	27
ABC-7.0	95	55	25	94	51	14
BUF-6.5	41	27	13	39	21	8
BUF-7.0	56	36	18	48	24	12

667 (CP)=composted feedstock

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693 **Table 4. Temperatures of Primary Devolatilisation (°C)**

Parameters	MO	4:1	3:2	MO (CP)	4:1 (CP)	3:2 (CP)
T _{on}	191	202	223	124	172	191
T _{max}	323	342	347	331	349	354
T _r	184	167	149	240	202	184

694 (CP)=composted feedstock; T_{on}=onset temperature; T_{max}=maximum temperature; T_r=temperature range

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728 **Table 5. Yield, Elemental Analysis and Higher Heating Value of Bio-liquid**

Parameters	MO	4:1	3:2	MO(CP)	4:1(CP)	3:2(CP)
Yield (g kg ⁻¹)	119	163	255	169	208	303
N (g kg ⁻¹)	45	36	26	56	48	34
C (g kg ⁻¹)	388	358	354	438	421	394
H (g kg ⁻¹)	92	94	94	90	88	95
HHV (MJ kg ⁻¹)	15.3	13.6	13.2	18.1	17.1	15.4

729 (CP)=composted feedstock; HHV=higher heating value

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761 **Table 6. Yield, Composition (volume fraction) and Higher Heating Value of Non-condensable Gases**

Parameters	MO	4:1	3:2	MO (CP)	4:1 (CP)	3:2 (CP)
Yield (g kg ⁻¹)	447	516	439	338	401	352
N ₂	10.0	5.6	3.2	10.0	5.6	3.2
CH ₄	6.1	11.3	13.5	6.2	11.5	11.7
CO ₂	43.2	34.8	35.2	47.5	38.7	34.5
CO	19.3	31.0	36.2	14.8	25.6	37.2
H ₂	2.4	3.4	3.5	2.5	3.2	3.3
C ₂ H ₆	0.9	1.5	1.4	1.1	1.3	1.3
C ₂ H ₄	8.9	6.7	5.7	10.0	8.0	6.9
C ₂ H ₂	0.3	0.3	0.3	0.3	0.3	0.2
Other C _x H _y *	8.8	5.4	1.1	7.5	5.9	1.6
HHV (MJ m ⁻³)	11.7	14.4	15.1	12.0	14.4	15.2

(CP)=composted feedstock; HHV=higher heating value; *=by difference

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788 **Table 7: Inputs and Outputs from each Feedstock and Process (Values per tonne liquid manure)**

	MO	4:1	3:2	MO(CP)	4:1(CP)	3:2(CP)
Inputs						
Separation (MJ)	-7.9*	-7.9	-7.9	-7.9	-7.9	-7.9
Composting (MJ)	0.0	0.0	0.0	-1.3	-1.6	-2.1
Drying Heat (MJ)	-168.9	-177.8	-192.7	-72.3	-76.2	-81.3
Pyrolysis (MJ)	-9.5	-29.8	-63.7	-6.6	-18.3	-36.3
Sawdust (t)	0.0	0.0175	0.0467	0.0	0.0175	0.0467
Outputs						
Bio-liquid & gases (t)	0.0112	0.0238	0.0425	0.0069	0.0131	0.0224
CHP Generated (MJ)	86.0	217.6	413.2	63.6	133.8	239.3
Biochar (t)	0.0084	0.0112	0.0188	0.0067	0.0084	0.0117
CHP Generated (MJ)	76.2	163.1	361.1	61.8	119.7	207.6
Output 1: Biochar as a Fuel						
Energy Balance (MJ)	-24.1	165.1	509.9	37.3	149.5	319.2
Output 2: Biochar to Soil						
Energy Balance (MJ)	-100.4	1.9	148.9	-24.5	29.8	111.7

789 * Positive values indicate an energy generation, while negative values indicate an energy requirement.

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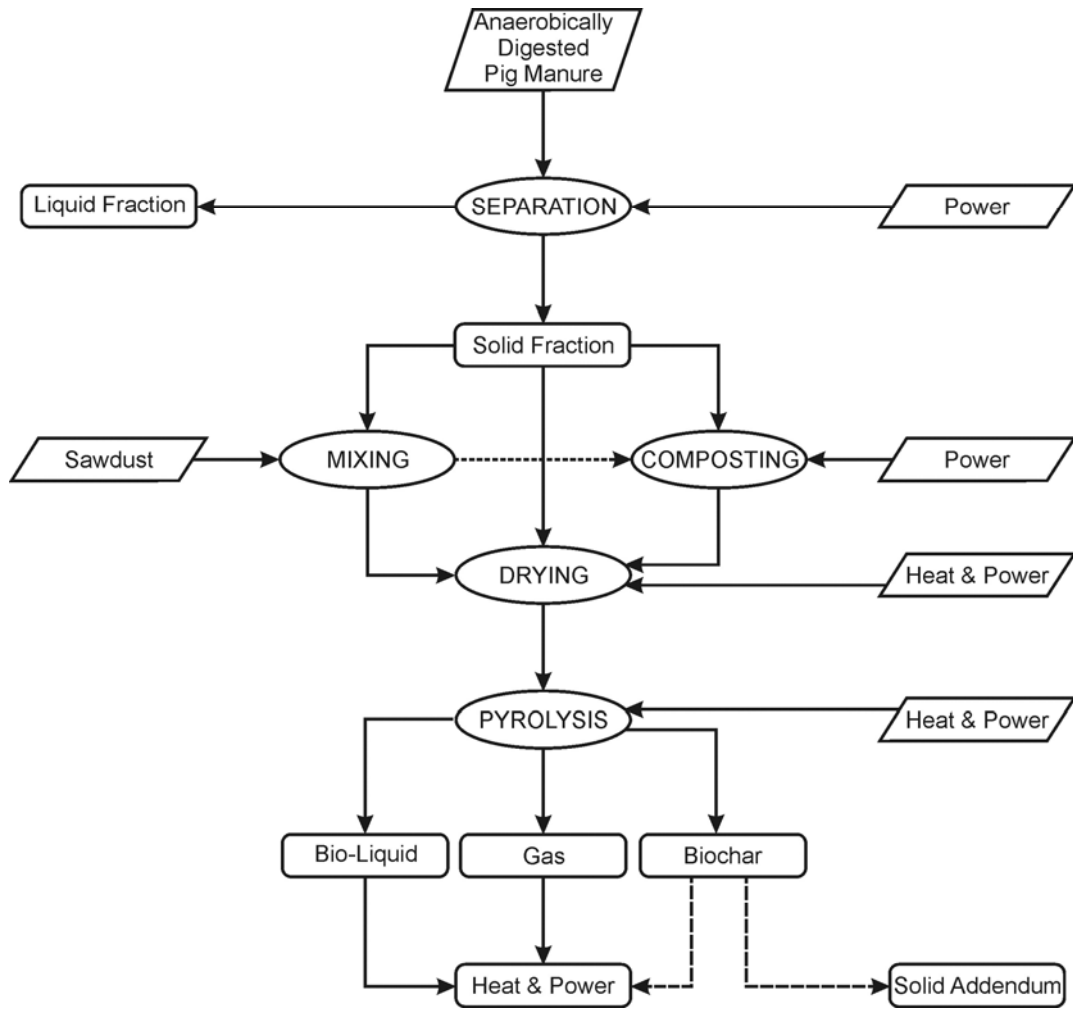
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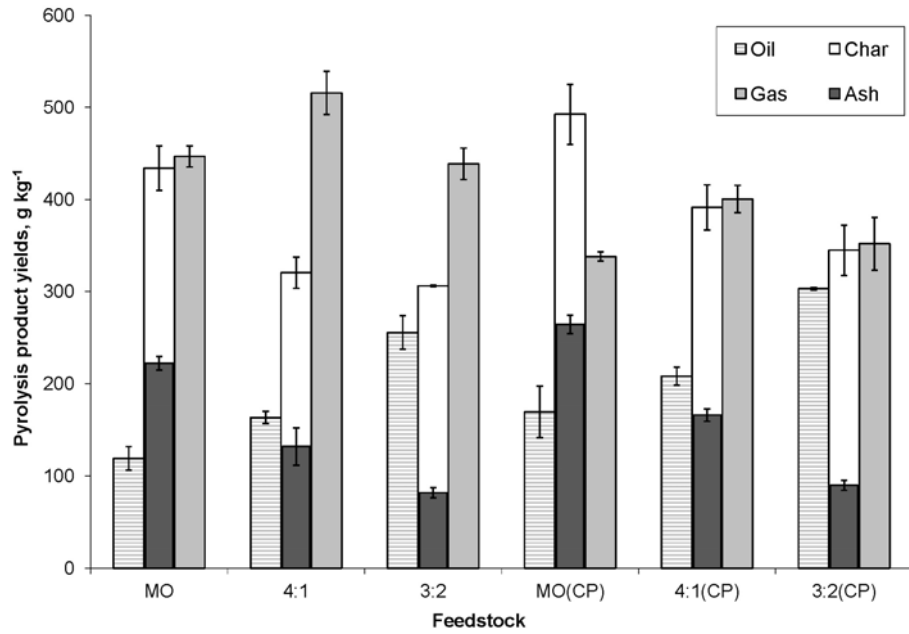
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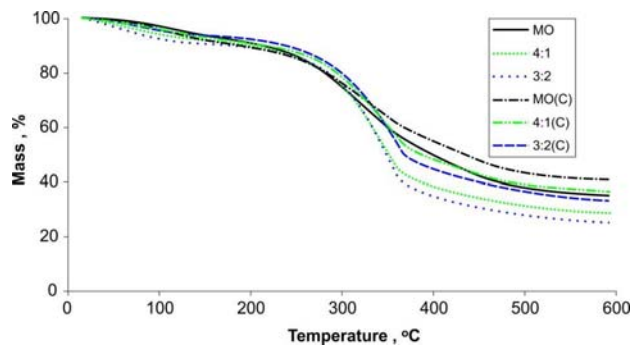
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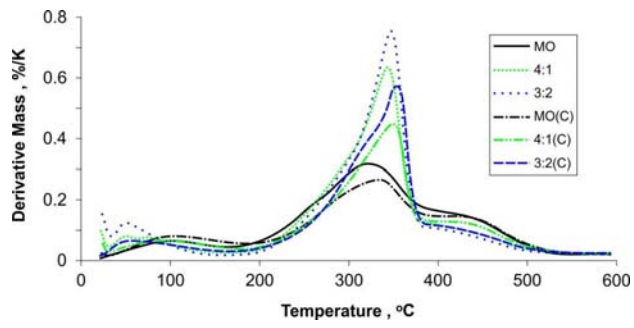
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826 Figure 3



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