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Pretreatment with extrusion before biogas production

Final report

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Contents

Summary

The applicability of extrusion as a pretreatment for biogas production has been evaluated. A pilot-scale extruder with multiple options for configurations and operation monitoring, although not expected to be energetically profitable, has been tested. In addition, a full-scale extruder has been installed at the biogas plant at AU Foulum feeding a 1200 m^3 biogas reactor. Lignocellulose-rich, low-cost farm biomass such as straw, deep litter, meadow grass, clover grass and maize silage has been examined. Extrusion treatments can be performed in various ways, including variations in the pre-cutting methodology, the feeding methodology, the feeding and extrusion treatment velocity, the extrusion operation temperature, and the screw configurations. Of these, the screw configuration of the extruder itself is of major significance; the tests has included configurations of transport elements, reverse elements, kneading elements, angle and length of kneading elements, and number of kneading sections. The effect of extrusion on the biomass, energy consumptions of up-stream stirrer and the extruder, the retention time in the extruder, relaxation time of water in biomass with low frequency NMR, sugar availability to enzymes, free dissolved sugar and total dissolved solids has been evaluated.

An extrusion screw configurations with an extended kneading treatment combined with a reverse combination was most promising. A long kneading section was not energetically favorable; however, cutting it into multiple sections was indicated less energy consumption but causing equal or better biomass effect. Hence, a combination of multiple kneading zones with one or more reverse zones (such as configuration 07 or 09 in table 1) was indicated to be a promising solution. However, whether the possible extra energy consumption could be more than balanced by a methane yield increase is not yet known.

It was observed, that extrusion caused lowered energy consumption of the reactor mixer, indicating sedimentation ability of the biomass. Upon the NMR tests, water was relaxed quicker in biomass after extrusion, indicating a tightened bonding of water to the biomass, and thus likely an increased accessibility of the biomass for the microorganisms. This was also observed with the sugar availability measure, increasing up to 60%. Total dissolved solid and dissolved free sugar was also increased upon extrusion by up to 60% and 30%, respectively, indicating an increased initial chemical degradation. Biogas production velocity was observed to increase, as was for some biomass also an ultimate biogas production observed; well aligned with the other analysis measures.

Cost estimates. Apart from the cost of biomass for the extrusion, which is biomass that otherwise cannot be used as a feed in a slurry-based biogas reactor; there are costs of process energy, maintenance and depreciation. The latter two are estimated to around DKK 140 per ton biomass, and the process energy is estimated to be approximately DKK 60 per ton biomass. The methane yield around 125 m^3 per ton biomass gives an income of DKK 420 per ton biomass. So there is a surplus of around DKK 220 per ton biomass to pay for the biomass, transport and handling.

In slurry-based biogas plants, extrusion of biomass opens up the possibility to use biomass to a larger extent than hitherto has been possible. The extruded biomass mixes better with the slurry, and allows mixing of a broth with high biomass density in a biogas reactor mounted with a vertical central mixer.

1 Introduction

1.1 Conditions

The Danish Government intends to increase the production of renewable energy, including biogas. Introduction of extrusion as a pretreatment to the biogas sector was expected to result in substantial increase in biogas production and profitability. The purpose of the project has therefore been to develop and optimize a pretreatment method based on extrusion technology to increase methane production in biogas plants. Only 40-60% of the organic matter of most un-pretreated biomasses is converted into biogas. A pretreatment technique is therefore of large value, if it causes exploration of the non-degraded organic matter. This may occur by improving the access to otherwise difficult or non-accessible lignocellulosic parts of the biomass and/or depolymerisation causing increased amount of easily degradable monomers. This will result in an increased access for microorganisms and enzymes to the degradable organic material, and hence enhanced hydrolysis. In an initial pilot-scale study on extrusion as a biogas pretreatment, the average increase in methane yields has proved to be 35% and the energy consumption proved relatively low. Extrusion will therefore be examined further in this study.

1.2 Objectives

The project intended to test the hypothesis, that extrusion as a pretreatment to biogas production is advantages. This project therefore aimed at clarifying the physical/chemical impact of the extrusion on fibrous-biomass, optimizing the extruder configuration, testing various biomasses, and validating under continuous, pilot-scale conditions.

1.3 Implementation

Application of extrusion as pretreatment under continuous, full-scale conditions was tested by installing a commercially available Bio-extruder (Lehman, MSZ B 74e, figure 1) ahead of the 1200 m^3 biogas reactor at Research Center Foulum (=project co-funding). This was a co-rotating twin screw extruder, with a capacity of 1-2 ton biomass/hour.

Optimizing of the extruder configuration was tested under batch-wise conditions, by installing a commercially available plastic-extruder (Xinda PSHJ-65, figure 2). This was a counter-rotating twin-screw extruder, with a capacity of 0.01-0.1 ton biomass/hour. The extrusion was not expected to be energetically profitable, but selected because multiple extruder configurations could be easily tested.

Figure 1. Full-scale Lehman extruder up-stream the full-scale biogas reactor

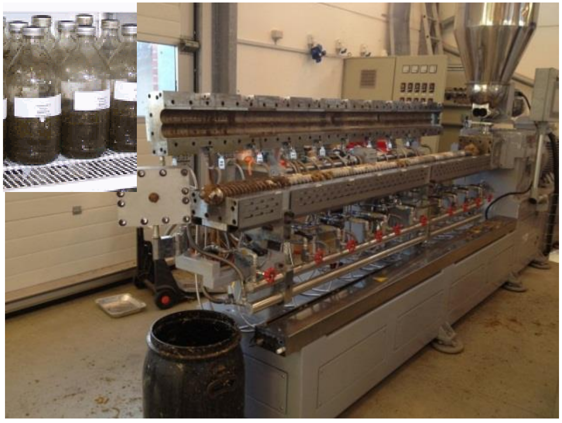

Figure 2. Pilot-scale Xinda extruder

Tests were performed on multiple biomasses (section 5.1), with multiple extrusion configurations (section 5.2) and evaluated by multiple analysis techniques (section 5.3). Various biomass effects were observed (section 5.4). Based on that application of extrusion as pretreatment was evaluated (section 5.5).

Figure 3. Biomasses used for the full-scale extrusion: clover-grass, deep litter, meadow grass and maize silage (from top left to bottom right).

2 Materials: Biomass

The full-scale extruder (Lehman) was fed with clover-grass, deep litter, mixed grass from the meadow, and mays silage (figure 3). The extruded biomass is fed directly into the 1200 m^3 biogas reactor. To the biogas reactor is also fed non-pretreated manure (approximately 50-70% of total biomass volume), and straw pretreated by briquetting (approximately 0-15% of the total biomass volume).

The pilot-scale extruder (Xinda) has batch-wise been fed with primarily wheat straw (figure 4). The dry matter content of the wheat straw was wetted with water from 90% to 40%, and left for saturation in closed bags for 24 hours in order to simulate biomasses with lower dry matter-contents. The selection of straw was done to obtain a relevant and easily repeatable biomass. Experiments have also been performed on an artificial deep litter. This was prepared by mixing wheat straw and manure to 40% dry matter and leaving it for saturation and softening in closed bags for 72 hours. This was done to use an easily repeatable biomass, rather than the very inhomogeneous deep litter. Experiments have also been performed on elephant grass, maize silage and rice straw silage.

Figure 4. Biomasses used in the pilot-scale extruder: wheat straw, deep-litter, elephant grass, maizes silage and rice straw silage.

3 Methods: Extrusion

Multiple parts of the extruder configuration can be varied. The extrusion of biomass requires three separated processes: (i) an initial cutting of the biomass into reduced length (figure 5), (ii) a continuous, stable feeding of the biomass into the extruder, and (iii) the extrusion itself. In the extrusion process, the controllable variables include screw rotation, and temperature. The screw configuration can also be changed.

Figure 5. Extrusion processes

3.1 Cutting

The extrusion process only allows the input to be maximum 50% of the extruder screw diameter, to avoid biomass bridging above the extrusion screws. Thus to e.g. extrude 2 meter long elephant grass in the pilot extruder with 65 mm wide screws, it must be cut into pieces of maximum 30 mm length. And for the full-scale extruder with 250 mm wide screws, it must be cut into 100-150 mm pieces.

For the full-scale extruder, a Bio-mix'er (Strautmann, GE) was employed. It is a 200 m^3 container barrel with three rotating towers of knives on at the bottom. The biomass exits the container through a hole at the bottom, and the removal velocity is controlled by a screw.

The pilot extruder employs a shredder (rs30, Untha, GE). At the bottom of a 1 m^3 hopper, four aligned horizontal axes with intervening, rotating knives are placed. Below the knives is a mesh at 10, 20 or 40 mm.

The material was cut once when passing the knives, but if the biomass was larger than the pores, the knives circulated the biomass back into the hopper.

3.2 Feeding

For a uniform extrusion treatment, a continuous, stable feeding of the biomass is required.

The full-scale extruder was fed from a conveyer belt directly attached to the screw below the Biomixer. The pilot extruder was fed by a volumetric feeder.

3.3 Extrusion

In the extrusion process, the capacity, screw configuration and temperature were controlled. Other variables include die end, pressure, chemical addition, liquid extraction, and gaseous extraction, but none of these has been examined in this project.

3.3.1 Feeding velocity and retention time

The m3 biomass fed in per hour could be expected to have an impact on the efficiency of the treatment, as this would influence the biomass compaction in the extruder and/or the treatment time would be affected. In the pilot extruder, the retention time within the extruder was tested using blue-colored biomass. The biomass was inside the extruder for 30-90 sec. It was observed that increasing feeding velocity did not increase the retention time with the extruder, thus instead must the compaction with the extruder be affected. A changed compaction could cause changed efficiency of the biomass destruction.

Figure 6. Compression screw element followed by a reverse element (picture of elements for the full-scale extruder)

Figure 7. Screw element order is transport, compression, kneading with different angles and kneader lengths, transport, reverse and transport (picture of elements for the pilot extruder)

3.3.2 Screw configuration

The most essential part of extrusion is the screw configurations. The primary impact of the screw configurations is the on the mechanical tearing of the biomass. The two most important types of biomass treating screw elements for twin screw extruders are reverse elements and kneading elements. The reverse element (figure 6) causes the flow of biomass to be lowered, thereby causing compression downstream and thus biomass tear. Reverse elements exists with different angle of the thread. The kneading elements (figure 7) mold the biomass comparable to the treatment of dough for bread. The possible variations is of number of kneads, the knead lengths and angle between the successive kneads. The biomass kneading will expectedly cause defribilation, and the biomass will be slight reduced in straw length by the transfer between kneads. Reverse kneading elements also exists; the larger angle between kneads will expectedly cause more severe kneading, and the reverse angle will additionally cause compression of the material by causing decreased flow. Additionally, feeding screws, transport and compression screws are of importance,

Table 1. Tested screw configurations, pilot extruder

Screw ID	Zone 1	Zone 2	Zone 3	Zone 4	Zone 5	Zone 6	Zone 7	Zone 8	Zone 9	Zone 10
01 Reverse	Feeding	Transport	Transport	Transport	Transport	Transport	Transport	Reverse	Transport	Transport (cooling)
02 Mild kneading, Angle decrease	Feeding	Transport	Transport	Transport	Transport	Transport	Transport	Kneading *	Transport	Transport (cooling)
03 Mild kneading, angle increase	Feeding	Transport	Transport	Transport	Transport	Transport	Transport	Kneading **	Transport	Transport (cooling)
04 Long kneading	Feeding	Transport	Transport	Transport	Transport	Transport	Kneading *	Kneading *	Transport	Transport (cooling)
05 Kneading with reverse	Feeding	Transport	Transport	Transport	Transport	Transport	Kneading *	Reverse kneading	Transport	Transport (cooling)
06 Kneading and reverse	Feeding	Transport	Transport	Transport	Transport	Kneading *	Transport	Reverse	Transport	Transport (cooling)
07 Long kneading, section	Feeding	Kneading **	Transport	Kneading **	Transport	Kneading **	Transport	Reverse	Transport	Transport (cooling)
08 Multiple reverse	Feeding	Transport	Transport	Reverse	Transport	Reverse	Transport	Reverse	Transport	Transport (cooling)
09 Multiple kneading and reverse	Feeding	Kneading **	Transport	Reverse	Transport Kneading **	Tran Reverse	Tran Kneading **	Transport Reverse	Transport	Transport (cooling)

*Angle between kneas decreasing, ** Angle between kneas increasing but have no effect on the destruction of the biomass. A die end may be added at the screw exist, causing an additional compression of the biomass.

The screw in the full-scale extruder (Lehman) consisted of a feeding zone, a compression zone and reverse zone and a die end. The size of the die end opening was the only possible variable. The pilot extruder screw could be varied. Multiple kneading element types and reverse elements were available. Thus various screw configurations were tested, see Table 1. In general, all configurations consisted of a feeding zone, transport zone, and no die end.

3.3.3 Temperature

The temperature within the extruder may have an impact on the extrusion, primarily on the chemical degradation of the biomass such as polymer degradation. The full-scale extruder could be frictionally heated by the biomass itself, causing the temperature to be typically between 60 and 80 °C. The pilot extruder could be frictionally heated by the biomass. But it could also be heated using attached electric heating elements or cooled using flushing water, thus measurements on the extruder itself indicated temperatures from 30 to 220 °C.

4 Methods: Analyses

4.1 Extrusion

During the extrusion, the performance of the extrusion was evaluated. The temperature of the extruder was logged continuously at 10 positions along the extruder barrel. And the operating capacity was continuously logged by using a balance at the biomass outlet. Further, the momentary energy consumption was logged continuously. Thus the energy consumption in kWh/ton could be calculated.

4.2 Samples

The settlebility of the biomass in liquid was of interest due to the practical performance of the biogas reactor. In full-scale, the energy consumption of the mixing propeller within the biogas reactor was logged. In lab scale, the water retention value was evaluated. This was done by adding water to the biomass, and 24 hours later measure the water absorbed within the biomass. Results from this test were, however, not indicated to be successful to monitor the solubilization of the biomass within the reactor. An alternative methodology, not yet tested quantitatively, could be to place sample on a water surface, and spectroscopically monitor the time of settling.

The surface area of the biomass was expected to increase by the extrusion, and thereby cause increased access for the microorganisms to the biomass. Methodologies for quantifying the surface area was N_2 adsorption, ethylene glycol monomethyl ether adsorption, and adsorption of color molecules in two sizes (Simons Stain), but none of the methodologies were successful; likely due to the inhomogeneous character of the organic biomass. The location of the water within the biomass was also expected to indicate the ability of microorganisms to access the microorganism, and this was measure using low field NMR.

The initial availability of the degradable organic material was expected to impact the velocity of the biogas production. The total dissolved solid (DTS) was determined, as the dry matter content not possible to remove by filtration. The sugar availability was measured as the amount of sugar liberated after enzymatic hydrolysis by addition of cellulase and hemicellulase.

The biogas production in the full-scale reactor was logged continuously; however due to full-scale operation various factors affecting the biogas production simultaneously was unavoidable. In lab scale, the biogas production was determined as the gas volume produced after mixing of the biomass with inoculum from a biogas plant. The initial production was applied to indicate the velocity of biogas production, while the ultimate yield was used to evaluate alterations in the biomass causing access to intially non-degradable carbon-sources.

5 Results: Extrusion effect

Extrusion of the tested biomasses was feasible in the full-scale and pilot-scale extruder (Figure 1). The pilot extruder provides the most variables, thus the majority of the results are obtained on this extruder.

Figure 8. Extrusion 07, inlet (left) and outlet (right) wheat straw at 40% dry matter

5.1 Pre-cutting and feeding

The Biomixer successfully cut the biomass into suitable sizes. The rotating knives may cut the biomass more times than necessary. And the knife tower initially has to operate under a large, heavy load of biomass. Thus the observation is, that the Bio-mixer may use more energy than expected necessary. Rather ridged biomasses were easily treated using the shredder, resulting in fewer energy requirering cuts, than the Bio-mixer. However, soft biomasses tend to be difficult to shred or bridge on the mesh, and is therefore re-circulated excessively. The observation is therefore, that soft biomasses are excessively cut, maybe even crushed and requires high energy consumption. A shredder treatment may thus be a solution for stiff biomasses, while a conveyer belt after a hopper with knives passing frequently could be a promising option for soft biomasses.

The conveyer belt feeding of the full-scale extruder was successful, and the extruder was 100% full. The only problematic observation was that the biomass is not truly continuous due to the dropping of biomass into the screw at the bottom of the Biomixer. The volumetric feeder for the pilot extruder caused bridging above the extrusion screws was troublesome. And the result was, that the extruders transport screws were only filled by 25% (see figure, front page). Ideas to solve this includes feeding screws with teethes to allow biomass to drop into the teethes and force it forward, feeding screws with bendable spikes to disturb the bridging, feeding screw with larger distance between the treads increasing the likelihood for biomass to drop into the screw, a feeding compartment covering a larger area of the screws to increase the likelihood of biomass dropping into the screws, pulsing air above the screws to destroy the bridge, or a force-feeding system with a hopper screw ending directly above the extruder screws. In general, feeding of biomass intro large extruder screws was more successful.

5.2 Extrusion retention time

In the pilot-extruder, most extrusion configurations caused equal retention times at approximately 50 sec (table 1). However, at the long kneading (04) the retention time within the extruder was doubled. This could be explained by the absence of a forward movement within the kneading elements. Splitting the kneading into multiple sections, with transport elements in-between (07) solved this.

5.3 Extrusion temperature

The temperature increase caused by friction within the extruder was 80-105 °C (measured as the extruders temperature). The temperature depended on the work intensity of the screw configuration, with mild kneading (02) causing the lowest frictional temperature increase, and longer kneading and reverse combinations (e.g. 04, 05 and 06) causing the largest frictional temperature increase.

The effect of the duration of the exposure of the biomass to heating was examined by heating the extruder to 180 °C on one or four of the extrusion zones. In reality, the temperature profile along the extruder was approximately equal, as the limit of the electrical heating capacity of the extruder was reached. Hence, no conclusions, based on a sugar availability and biogas batch test, were indicated.

The effect of the extruder’s temperature was examined by fixing the temperature at 30-180 °C, in steps of 25 °C. Based on a sugar availability test, surprisingly, no significant effect was indicated. In fact, at 30 °C the effect was indicated to be relatively high.

5.4 Energy consumption

A positive energy consumption balance by the extrusion in the pilot-extruder was not expected, as the extruder was purchased to provide multiple extrusion variables, not optimally designed energetically. In general, observation on the pilot extruder indicated the energy consumption for operating the extruder, measured upon idle operation, to be approximately 50% of the total energy consumption. Despite the high energy consumption of the pilot extruder, observations on the relative energy consumption were expected indicative of promising extrusions.

The energy consumption was equal upon treatment of wheat straw at 40% dry matter (figure 9). The exception was the long kneading (04) treatment which likely is related to the observed long retention time within the extruder. The artificial deep litter (straw+manure+time) and the straw and manure mixture (with minimum storage time) indicated lowered energy consumption compared with straw+water. This could be linked to the presence in the manure of particles, which would roll, and of lipids, which would smoothen the biomass. In fact, the storage time (one versus three days for the two biomasses) did not indicate any significant difference, but the tendency was lowered energy consumption after longer storage time, and thus likely increase pre-hydrolysis time. Doubling the extrusion speed, i.e. feeding screw and extruder screw velocity, did not increase the energy consumption.

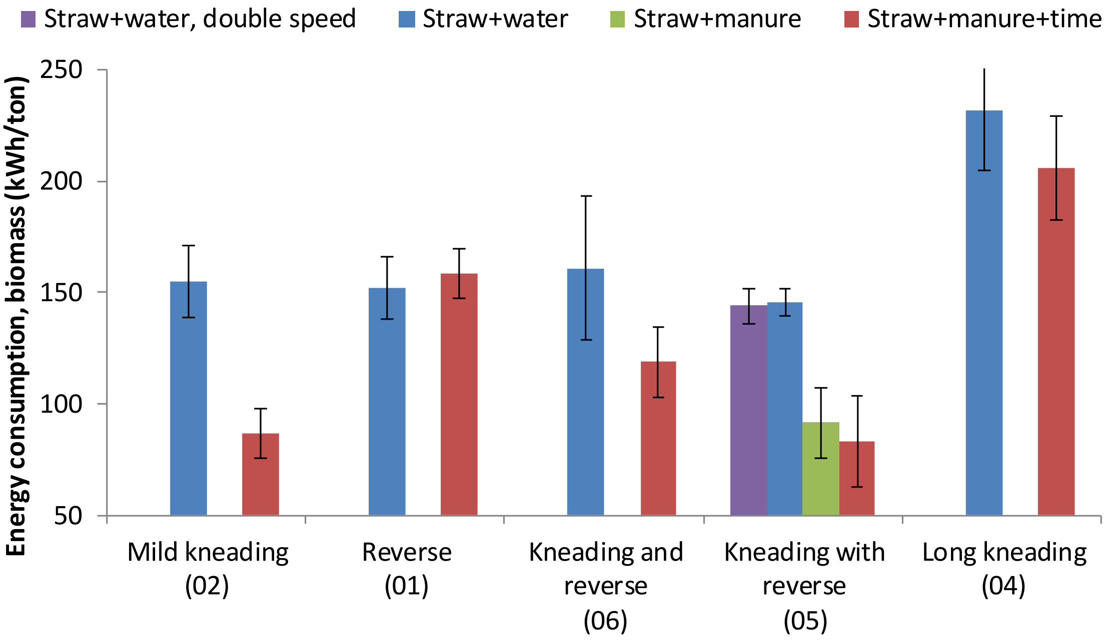

Figure 9. Energy consumption depending on extrusion configuration and biomass. Energy consumption expressed as consumption for biomass treatment, i.e. energy consumption for idle operation of the extruder is subtracted.

Experiments on the long kneading screw configuration divided into one or several sections (04 versus 07) indicated that splitting the kneading into multiple sections consumed the least energy.

5.5 Biomass settling

The energy consumption of the mixing propeller in the full-scale biogas production before and after the initiation of the full-scale extruder showed decreased energy consumption after introduction of the extruder. In fact, the operation of the biogas plant was no longer limited by the mixer, thus the dry matter content within the reactor could be increased from previously 5-6% to 8-9% without mixing problems. This was expectedly because the biomass became dissolvable with the reactor slurry, rather than floating at the surface. This indicates eased dispersion of liquid within the biomass, and thus likely physical changes to a more porous structure.

To obtain an equivalent measure on the biomass from the pilot scale extruder, water retention value was determined on the biomass, but it proved not to be applicable for extruded biomass. An alternative could be to spectroscopically log the settling time of the biomass, after placing it carefully on a water surface. This is expected successful, as non-quantitative visual observations indicates this to differ.

5.6 Biomass availability

An increased biomass access was indicated by the full-scale biogas reactor's eased propeller mixing.

Three methodologies proved not useful for quantifying the internal surface area of the biomass because the particles size also varies and because it is non-ridged upon drying: N_2 adsorption, ethylene glycol monomethyl ether adsorption, and adsorption of color molecules in two sizes (Simons Stain).

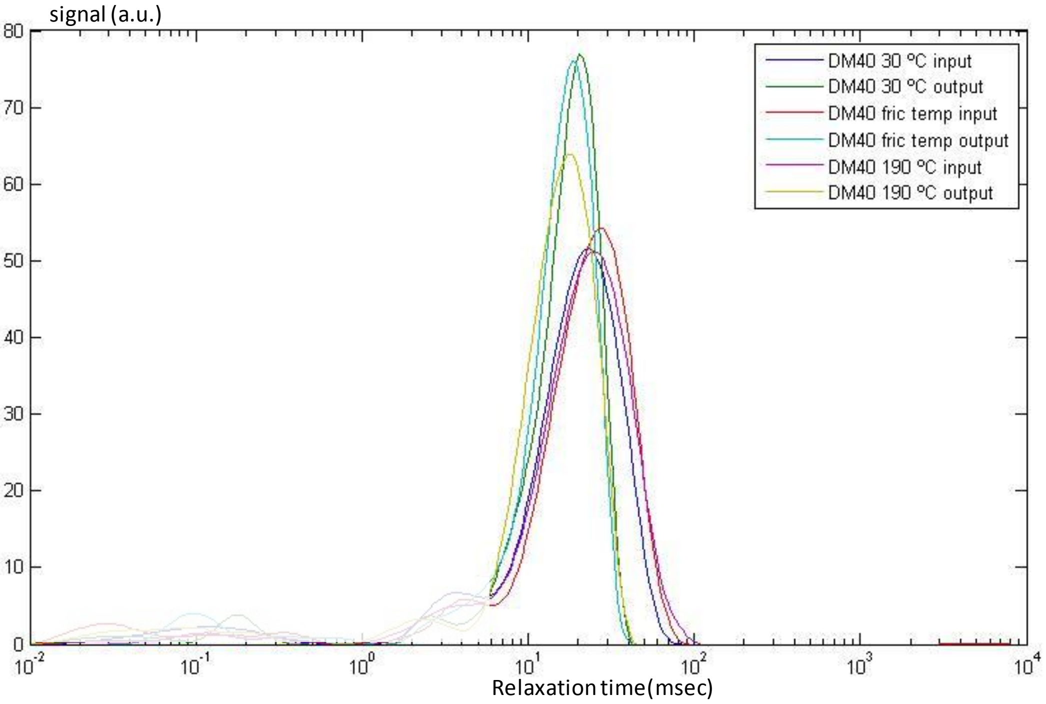

Figure 10. NMR signal from water after extrusion (configuration 06) depending on extrusion temperature and biomass dry matter.

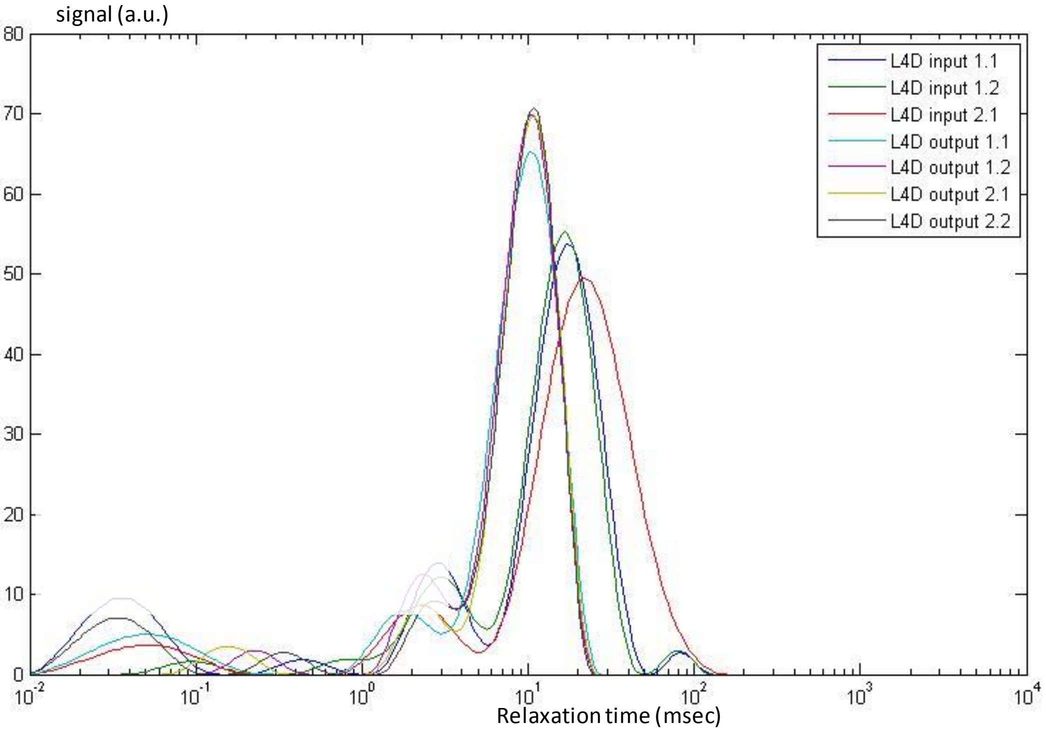

Figure 11. NMR signal from water after extrusion (configuration 07) depending on extrusion temperature and biomass dry matter.

The water position was evaluated using low field NMR. The relaxation times were shortened after extrusion (figure 10 and 11), indicating stonger attachment of water to the carboxylic hydroxyl groups in the biomass. Hence, more carbohydrates were accessible, and the biomass was more hydrophilic. This indicates removal of the covering hydrophobic lignin layer from the carbohydrates, thus defibrillation. As water showed increased access within the biomass, the availability of the biomass for the enzymes and microorganisms may also be expected.

The extrusion with multiple kneading section (07) showed larger effect than kneading and reverse extrusion (06) (Figure 10 and 11). Thus, efficient kneading appears to cause a larger availability of the biomass.

Another measure of the availability of the biomass is to allow enzymatic degradation of the accessible carbohydrates, cellulose and hemicellulose, into sugar monomers, and then quantify the monomers; i.e. measure the sugar availability. Extrusion is indicated to increase the sugar availability (figure 12 and 13). The efficiency of the screw configurations depend on the biomass. Kneading indicated largest increase for the softer artificial deep litter (figure 12), i.e. 'gentle' defibrillation in the kneading element. Reverse treatment was indicated to cause the largest increase upon treatment of the more frictional 40% dry matter straw, i.e. likely one biomass particle may create larger frictional destruction on another biomass particle in the biomass clog in the reverse element.

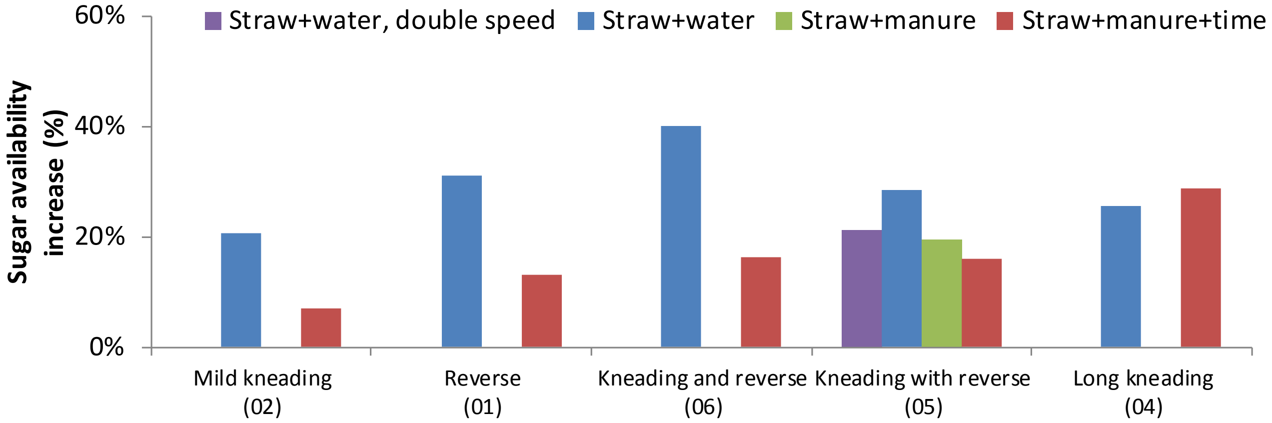

Figure 12. Sugar availability, relative increase, upon extrusion depending on extruder screw configuration and biomass type

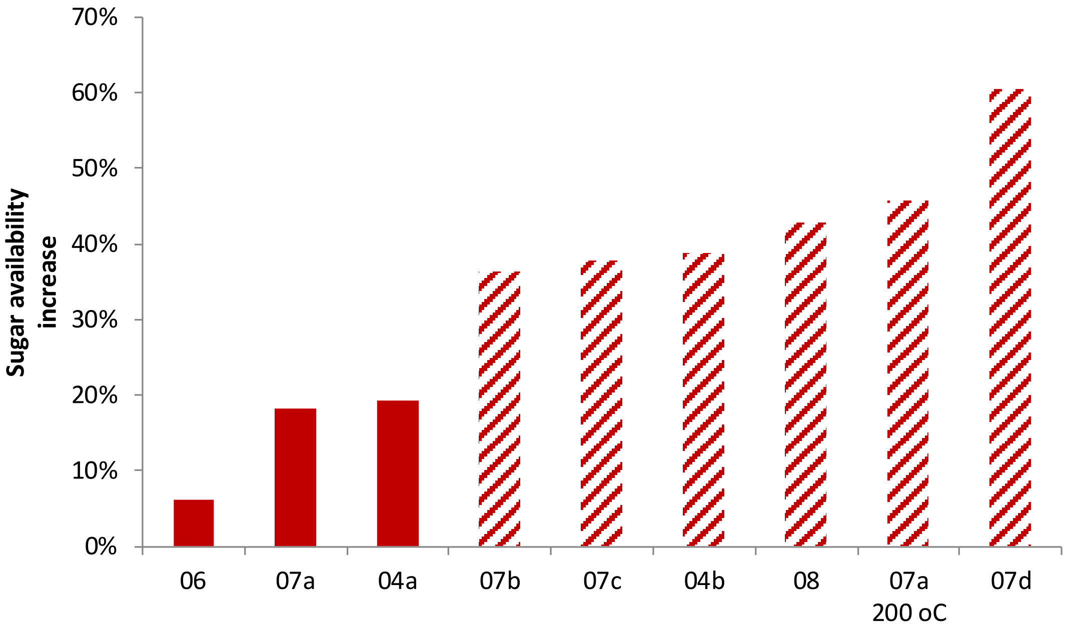

Figure 13. Sugar availability, relative increase, upon extrusion of straw+water depending on extruder screw configuration (significantly different groups marked with different shadings)

The more vigorous screw configurations were more successful (figure 13). A variation within the kneading zone of angles and lengths of kneads or in the reverse position has been tested, but in fact labeled equally in the figure. 04a had a longer kneading zone than 04b; 07a had only two kneads and a reverse zone, 07b had three kneads but no reverse, 07c had three short kneads and a reverse zone, while 07d had three slightly longer kneads and a reverse zone. In general though, the more vigorous the kneading or reverse treatment, the more successful. Very long kneading zones (04a) was less successful, likely due to the previously mentioned transportation difficulties, causing large amounts of biomass within the kneads, and thus less possible work performed by the kneads on the individual part of biomass. The largest relative increase was at 60% (07b), and was obtained with three relatively short kneading sections and ending with a reverse zone (07d).

Extruding mays cob and rice straw with configuration 06 also indicated increased sugar availability, with 40% and 100% increases.

Upon extrusion, large carbohydrates were observed to become increasingly available upon extrusion. It could also be relevant to observe, if in fact the sugar monomers has become available directly by the extrusion, i.e. likely a chemical destruction of the carbohydrates upon the treatment. Indeed this occurs (figure 14). The order of impact effect of the screw configuration appeared approximately equal for the tested biomasses. The effect appeared more pronounced for the straw rather than the deep litter, perhaps indicating high friction to be of significance.

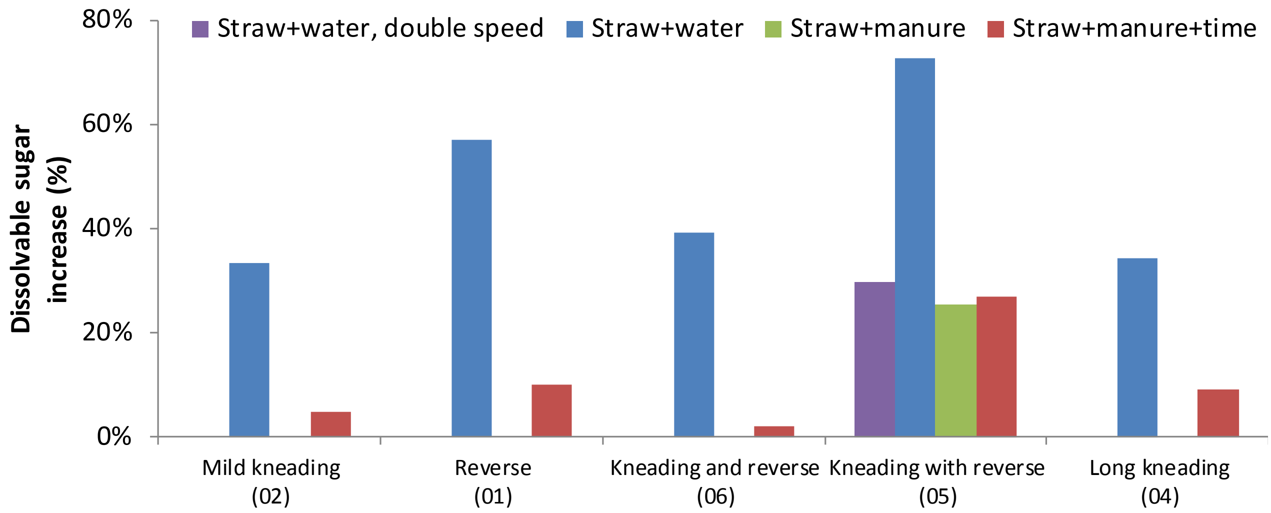

Figure 14. Relative increase in amount of dissolvable sugar directly upon extrusion depending on screw configuration and biomass type.

Of the organic matter in the biomass, not only the sugar may become increasingly dissolvable directly upon extrusion. To examine the chemical and physical degradation of non-dissolvable organic material upon extrusion, the amount of dissolvable dry matter was determined (figure 15). Indeed also the dry matter did in general increase upon extrusion. The biomass with the lowest dry matter content (40% versus 70%) appeared to increase the most; this could merely be because the increased water content causes the dissolved dry matter to become more easily be squeezed out of the biomass. The effect was observed upon operation under multiple extruder temperatures, with the absence of heating being superior; this could be because lignin would not melt and re-attach the small particles.

Hence, increased availability of water, for cellulose/hemicelluloses, of sugar and of dry matter is indeed indicated, thus increased access of the enzymes and microorganisms for the anaerobic digestion is likely.

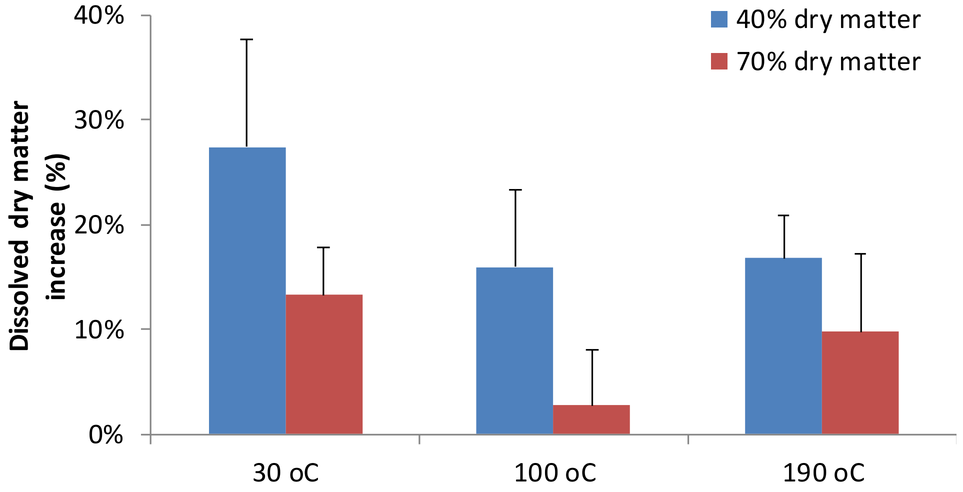

Figure 15. Dissolvable dry matter after extrusion of straw (configuration 06) depending on extrusion temperature and biomass dry matter content.

5.7 Biogas potential

The intent is to increase the methane production on the biogas plant by extrusion of the biomass used. Tests has been performed typically under non-continuous conditions in contrast to normal operation at a biogas plant, thus only indications of full scale effect can be obtained. The velocity of the production is of importance, as the biomass is typically not kept within the biogas reactor until fully degraded of economic reasons. Thus high initial production is essential. And the production in batch within the initial 30 days can be used as indication of the effect on the degradation velocity. The CH_4 production velocity was indicated to increase (figure 16, 17, 19 and 20), varying from -10% to +70%. In one study on straw, the increased production of CH_4 declined rapidly within the first month (figure 16).

The pattern of increasing velocity was comparable for the different varieties of straw dry matter content and extrusion temperature (figure 16), but with low dry matter content of the biomass (40%) appearing to be more affected upon extrusion and thus cause a more rapid gas production compared to high dry matter content (70%).

In a study on various biomasses and screw configurations, the smoother artificial deep litter was less affected than the straw at equal dry matter (figure 17). Thus it appears that on optimum in terms of internal biomass friction exist; with high dry matter content (figure 16) and straw (figure 17) being most frictional.

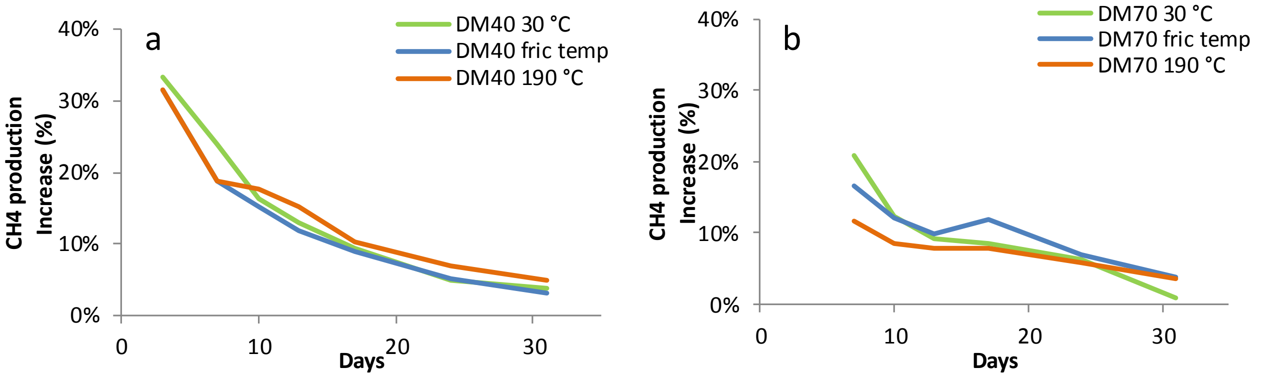

Figure 16. Increase in CH4 production after extrusion with configuration 06 on straw with 40% dry matter depending on extruder operation temperature, a: straw with 40% dry matter, b: straw with 70% dry matter

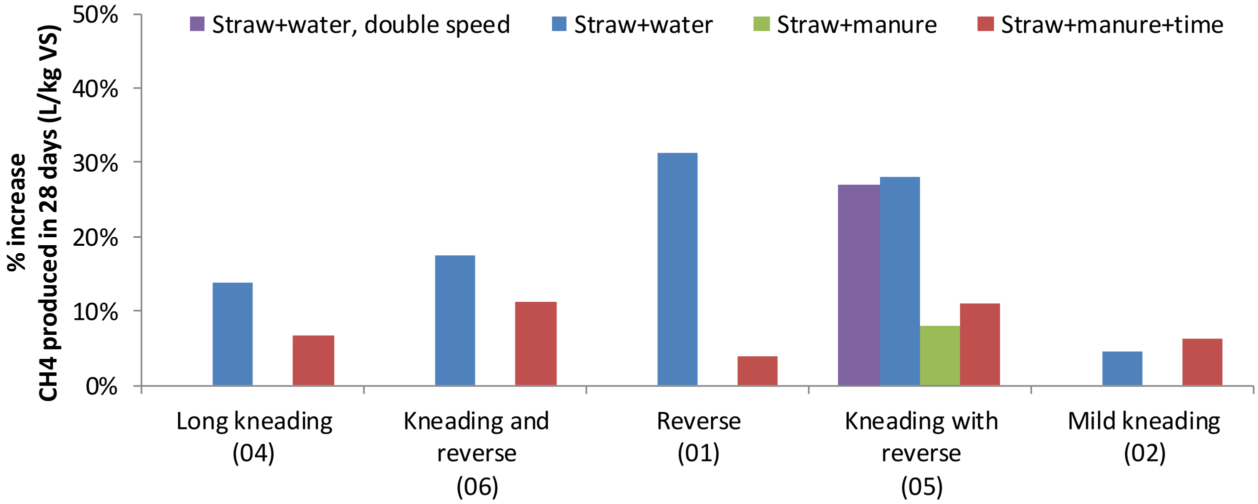

Figure 17. Increase in CH_4 production within the first 28 days depending on screw configuration and biomass

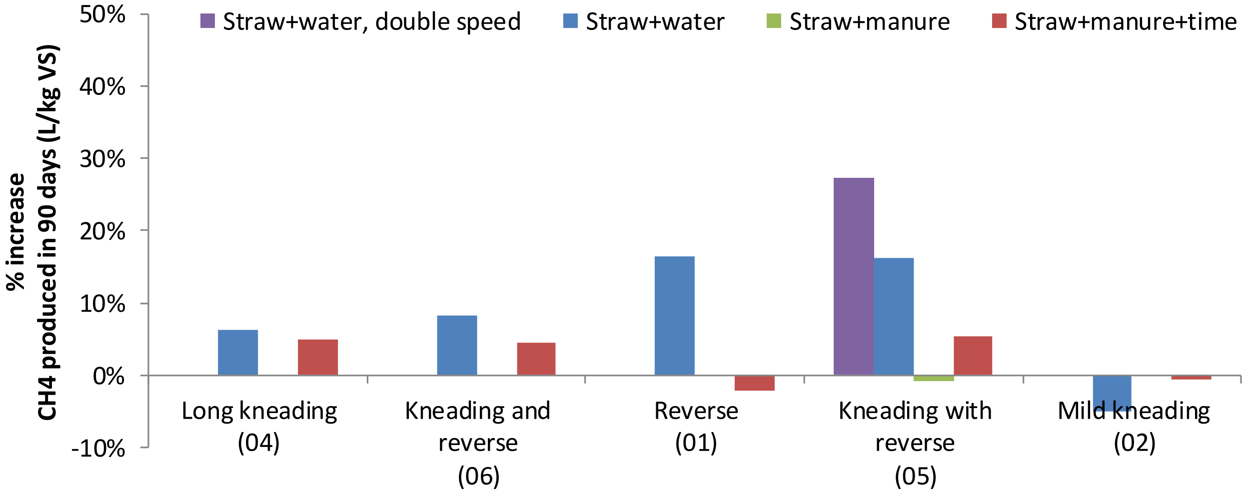

Figure 18. Increase in CH4 production within the first 90 days depending on screw configuration and biomass

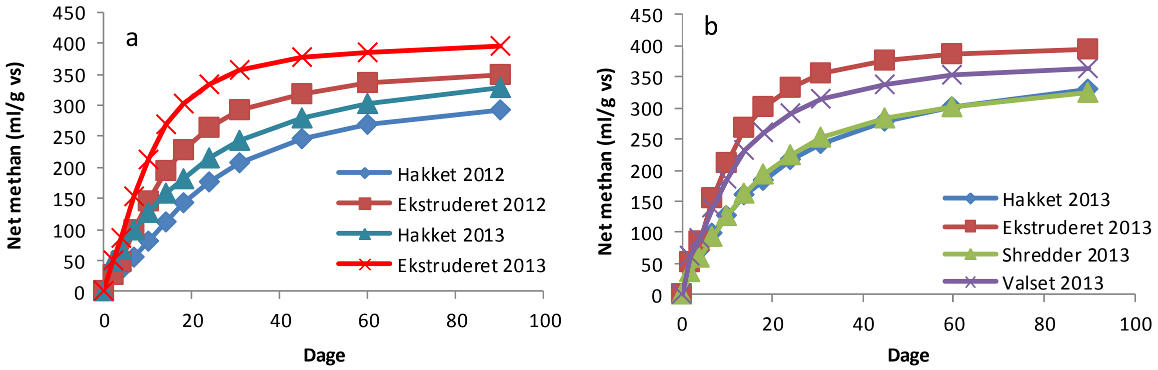

Figure 19. Biogas production timeline from Miscanthus, a: two harvests were treated in full-scale extruder and batch tested, b: miscanthus was shredded and subsequently extruded in pilot extruder (configuration 06) or rollar milled and batch-tested.

The screw configuration was indicated to have an impact on the degradation velocity (figure 17), differently affected for different biomasses, but with reverse elements tending to be effective for the frictional straw, and kneading effective for the softer deep litter. Thus agreeing with the observations on sugar availability.

The ultimate yields in biogas batch tests were obtained when biogas production was stopped, typically after 90 days. The ultimate production indicated the full degradability of the organic biomass. Some combinations of biomass and extrusion treatment conditions caused no ultimate effect (figure 18, 19 and 20). But most show an increased degradability, some up to 20%, indicating that extrusion has caused otherwise non-degradable organic matter to become degradable, e.g. by defibrillation/un-entanglement of the lignocellulose.

Miscanthus was extruded in full-scale reactor and also pilot-scale extruder, and both treatments caused a 40% increase in methane production after one month in batch (figure 19). Straw has also been extruded in both extruders, but results have not always been as equal as the miscanthus. It was also observed that shredding (cutting) of the biomass did not increase the biogas production, as intended. Rollar milling also increased the biogas potential, but only 50% the increase of the extrusion.

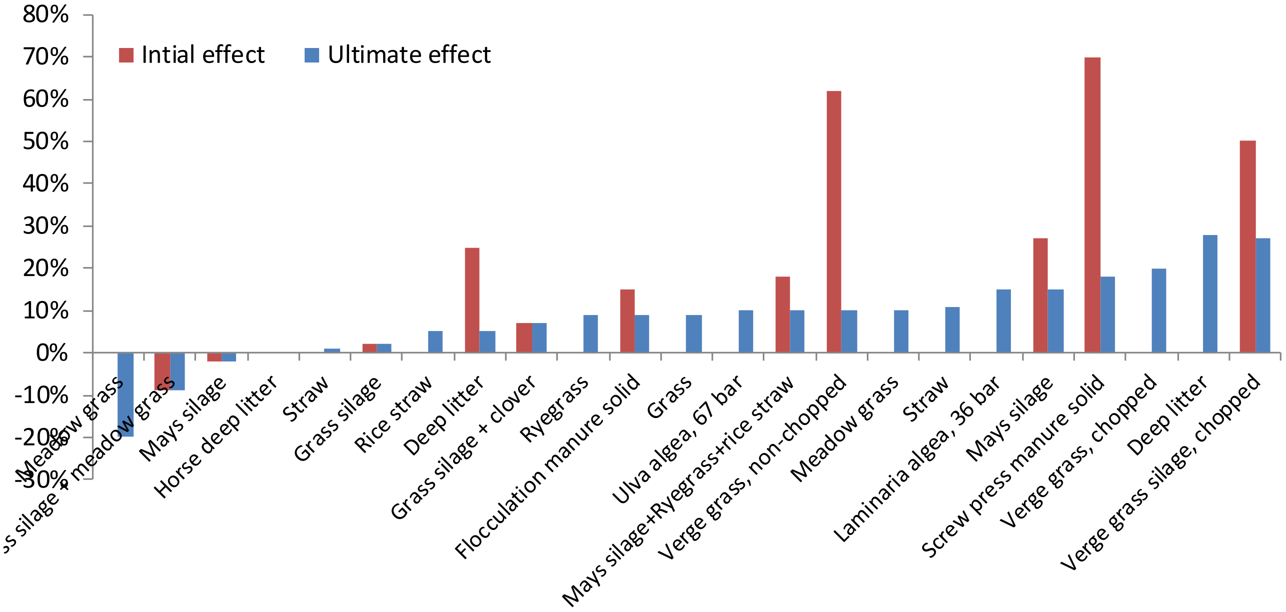

Figure 20. Methane production increases depending on biomass type. The majority has been performed on the full-scale Lehman extruder, or extruded under comparable conditions. Initial effect (red) should be comparable to approximately 30 days in figure 16-18, and the ultimate effect (red) comparable to the 90 days in figure 17.

The sugar availability test also indicated increased degradability of the biomass after extrusion. Testing the correlation between the sugar availability and the biogas production velocity, significant correlation is observed (figure 21). A significant correlation is also observed with the ultimate biogas production, however, less significant. This indicates that increased sugar availability causes an increased velocity of the entire anaerobic digestion pattern.

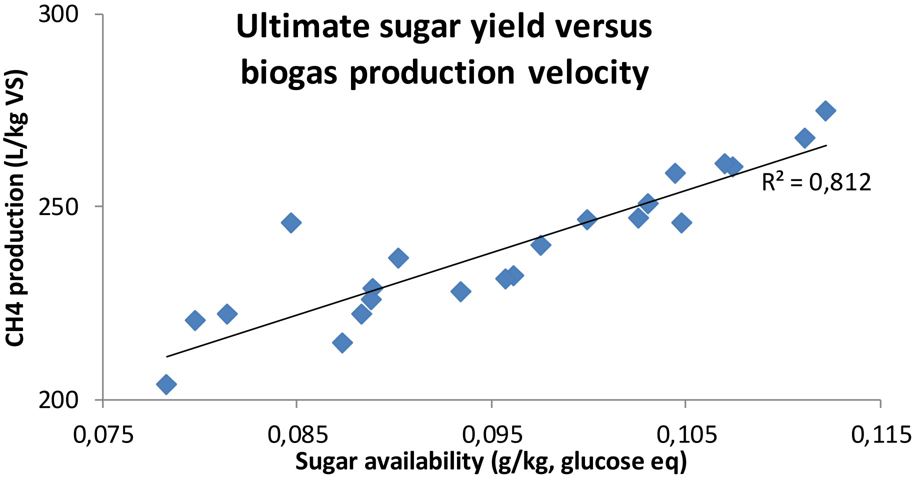

Figure 21. Correlation between sugar availability measure and the biogas production within 28 days. Tested on data from experiment in figure 12 and 17.

The increase in biogas production due to extrusion has been indicated to decrease over the course of experiments for the project years (figure 22). The inoculums for all batch tests were collected from the biogas reactor, which has been fed with the biomass from the full-scale extruder for two years, thus the dry matter content has been increased from 4-5% to 8-9%, and the share of lignocellulose has increased.

Hence, it is possible, that the pool of microorganisms within the biogas reactor has adapted to the new lignocellulose rich biomass in the reactor, causing an increasing degradation efficiency of the non-extruded lignocellulose. This was indeed indicated by a 26% (+/- 7%) higher CH_4 production from straw (figure 22). To avoid this unintended batch experiment development, inoculum could be collected from a different biogas reactor. It also means, that the absolute estimates of increases in biogas production velocities (figure 16-19) could be underestimating the effect observed upon initiation of an extrusion pre-treatment.

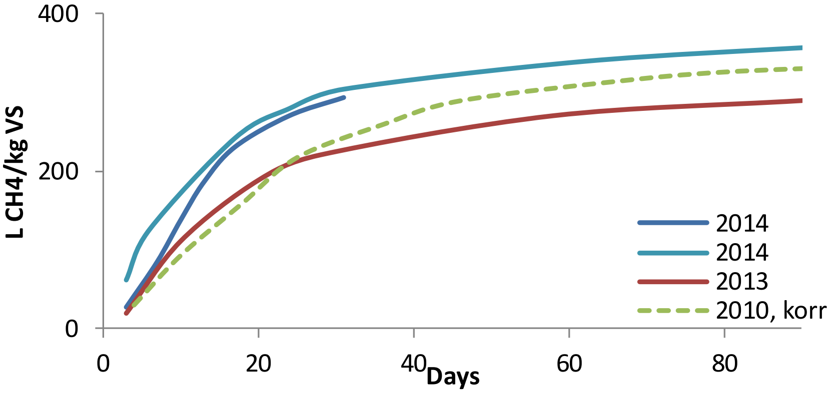

Figure 22. Development in CH4 production in batch tests on equal biomasses (straw) and from the same biogas reactor, from mid 2012 fed with extruded lignocellulose rich biomasses

Table 2. Performance of the full-scale extruder and biogas reactor depending on the operation time of the extruder.

Periode, quarter in 2012 (90 days)	Extruded biomass (ton)	Retention time, biogas reactor (days)	Gas production CH_4+CO_2+more (1000 m^3)
1	0	15,9	206
2	55	16,3	291
3	930	13,5	338
4	1500	13,2	391
Increase 1→ 4		-17%	+90%

Under full-scale conditions, it was not possible to estimate the biogas production increase solely due to the extrusion treatment. This is because no control scenario is feasible, as the biomass was not to possible feed into the reactor previously. What could be compared is the gas production before and after initiation of the extrusion pre-treatment (table 2). However several interferences causes this to be linked with error; the dry matter content increased from 4-5% to 8-9% (would increase gas production), the biomass' average retention time within the biogas reactor was 17% decreased (would decrease gas production), the general variation in the fed biomasses (could increase or decrease gas production), and extrusion of the lignocellulose rich biomass. Hence, the effect of the extrusion pretreatment on a biomass's biogas production cannot be isolated. It is observed that the biogas production was increased by 90%. The observed 90% increase in gas production may primarily be ascribed to the dry matter increase from 4-5% to 8-9%, indicating that the source of organic matter has approximately doubled in equivalence to the gas production.

In general, it does indeed appear beneficial for the gas production to extrude the biomass.

6 Discussion: Extrusion as pretreatment before biogas production

The project intended to test the hypothesis, that extrusion as a pretreatment before biogas production is advantages. This project therefore aimed at clarifying the mechanism of the extrusion, optimizing the extruder configuration, testing various biomasses, and validating under continuous, pilot-scale conditions.

6.1 Extrusion mechanism

An increased biogas production upon extrusion could be expected due to two mechanisms. An increased physical access for the microorganisms and enzymes, could cause an increased success of organic degradation, and thus improve the biogas production. Splitting of chemical structures in the biomass, would simplify the rate limiting initial degradation of the chemical structures, and thus also improve the biogas production.

An increased ultimate biogas production could be relevant, which could be tested in batches by quantifying the biogas production until production has stopped. This could be increased, if organic compounds have become available upon extrusion, which would otherwise not be available. Lignocellulose structure is rigid, causing typically 30-50% of the organic content to be degraded upon biogas production, thus this could potentially be improved upon extrusion. Most importantly, an increased degradation velocity could be caused by extrusion. Both the biogas velocity and ultimate biogas production could be increased by an increased structural accessibility and by increased initial chemical degradation.

An increased surface area could be indicated by a lowered energy consumption of the mixer in the biogas reactor, by increased settling ability in a water column, a lowered relaxation time in a the low frequency NMR spectrum of the biomass' water, by increased dissolved dry matter, and by increased sugar availability.

Organic polymer destruction could be indicated by an increased content of dissolved sugar and an increased amount of dissolved dry matter.

The mechanism of the extruder appeared from the results to be a combination of the two, thus physical wearing causing disintegration of the lignin-hemicellulose-cellulose structures, and an increased surface area. The result was an increased microbial accessible surface area, and increased access to low molecular weight energy rich carbon sources. Both could lead to an increasing velocity of the biogas production, while an increased ultimate biogas production would likely primarily be caused by increased organic polymer destruction.

6.2 Extrusion operation

Four categories of pre-treatments exist: mechanical, thermal, chemical and biological. The mechanical pretreatments tear or rip the biomass into small particles and thereby increase the accesses to the organic compounds. The thermal pretreatment is primarily carried out wet, and the treatment causes lignocelluloses structure changes, because some of the molecules breaks apart and some hemicelluloses dissolves and is relocated. The result is a higher access to the organic compounds. The chemical pretreatment with e.g. alkali typically removes compounds like hemicelluloses and lignin and thereby raise accesses to the intended compounds. The biological pre-treatment typically initiates hydrolysis, thereby minimizing the rate-limiting degradation step in biogas production.

Extrusion is a mechanical pretreatment. But the friction causes the temperature of the extruder to increase to 100 oC, thus low-thermal pre-treatment is also performed. The study, though, does not indicate any advantage in the heating, thus merely the mechanical pretreatment itself appears preferable.

A screw configuration with reverse treatment was indicated to be good for frictional biomass, while kneading treatment was indicated to be good for biomass causing less friction. This is conceivable, as reverse elements primarily relies on friction between biomass particles within the compressed zone, while kneading primarily relies on the screw causing impact on the biomass and is thus not depended on the biomass' frictional ability. An extended kneading treatment combined with a reverse combination was indicated promising. The long kneading was not indicated energetically promising; however, dividing it into multiple sections was indicated less energy consumption but causing equal or better biomass effect. Hence, a combination of multiple kneading zone with one or more reverse zones (such as configuration 07 or 09 in table 1) was indicated to be a promising solution. However, whether the possible extra energy consumption could be more than balanced by a methane yield increase is not yet known.

The Lehman extruder is set-up with one reverse zone (figure 6) without external temperature control (configuration 01). It could be relevant to replace this screw configuration with kneading, possibly multiple kneading zones before the reverse element.

No clear trend has been shown in the superior characteristics of the biomass, but e.g. straw appear superior to the softer deep litter, and 40% dry matter appears superior to 70% dry matter. In order for the operation of an extruder in a biogas plant to be practical, the extruder must treat the non-settling biomasses available on-plant; only very few negative biogas effects of the extrusion has been observed, thus it is not indicated problematic to treat all lignocellulose rich biomasses on the biogas plant.

6.3 Biogas production increase

The entire purpose is an increased biogas production under full scale operation.

The production rate was in batch test indicated to increase by -10% to 70%, with most treatments being in range of +10% to +20%. This measure may though be an underestimation, due to inoculum issues at experiments, when the extruder is initiated on the biogas plant. Some treatments also indicated an increased ultimate gas production ranging from -20% to +25%, with the majority being between 0% and 10%. Thus, an increased gas production from lignocellulose rich biomass is likely under full-scale continuous conditions. At full-scale, no biogas production increase was possible to demonstrate, but it is not unlikely that it did increase.

6.4 Pretreatment economy

The economy of the pretreatment was estimated based on the energy consumption and produced biogas during 2012, where in the first quarter no extruded biomass was used, and in the last quarter 1,500 tons of biomass was extruded (table 3). In the same period, the gas production increased with 90% (table 2), which corresponds to around 975 MWh energy. However, this is an effect of increased the doubling of dry matter load in the biogas reactor and combined with the effect of the extrusion. However, it clearly indicates that extrusion do have a positive impact on the biogas production.

Table 3. The effect of using extruded biomass on gas production in 2012.

Period, quarter in 2012 (90 days)	Extruded biomass (ton)	Extrusion+biomixer, Energy consumption (MWh)	Energy produced* (MWh)
1	0	0.02	1,060
2	55	1.3	1,500
3	930	52	1,950
4	1,500	68	2,035
Increase 1→ 4	1,500	68	975

*The energy produced is assuming 52% methane in the gas and 10 kWh/m^3 methane.

Cost of pretreatment. Apart from the cost of biomass for the extrusion, which is biomass that otherwise cannot be used as a feed in a slurry-based biogas reactor, there are costs of electricity (covered in table 3), maintenance and depreciation. The latter two are based on data from 2014 estimated to around DKK 140 per ton biomass. The methane yield around 125 m^3 per ton biomass gives an income of DKK 420 per ton biomass. The expenses to electricity, maintenance and depreciation are estimated to roughly DKK 200 per ton biomass, so there is a surplus of around DKK 220 per ton biomass to pay for the biomass, transport and handling. It should be emphasized that the calculation is tentative and an exact calculation should be based on two or more years in order to get better estimates of income and costs of repair and maintenance.

In slurry-based biogas plants, extrusion of biomass opens up the possibility to use biomass to a larger extent than hitherto has been possible. The extruded biomass mixes better with the slurry, and allows mixing of a broth with high biomass density in a biogas reactor mounted with a vertical central mixer.

7 Dissemination activities

7.1 Demonstrations

The Biogas Plant at Research Centre Foulum has more than 500 visitors a year, from primary school, to high school classes, to internationally highly recognized scientists, and to advisory services and the industry. Both extruders are typical parts of the tour.

7.2 Teaching

High school classes are offered a one-day class in pretreatment of lignocellulose rich biomasses before biogas production (see offered program at http://agro.au.dk/videnudveksling/for-gymnasier/klassebesoeg/fra-graes-til-gas/). Extrusion is included in parts of the environmental courses at our master education at the department of Engineering, including as the performed treatment in a large laboratory exercise at a course in Biorefinery.

7.3 Popular papers

Sørensen KL ”Ekstrudering af enggræs giver mere gas” in Effektivt Landbrug, 11-09-2012

Tobberup S ” Millioninvestering i forbehandlingsanlæg til gavn for økologisk biogas”, in international and national newsletter from Aarhus University/Foulum, 11-2012

Hjorth M/Skøtt T ”Extrusion as a pretreatment to biogas production”, in FIB – forskning i bioenergi, brint og brændselsceller, 09-2014.

7.4 National workshop

Møller HB and Hansen MM ”Driftserfaringer med ekstruder” at the seminar ’Grøn Energi dag’, arranged by INBIOM at AU/Foulum, 05-09-2012 (presentation)

Hansen MM and Møller HB ”Driftserfaringer med forbehandling af tungtomsættelig biomasse i ekstruder – effekt på biogaspotentialer, kapacitet, elforbrug og økonomi” at seminar ’Biogas og ekstrudering af fast plantebiomasse’ arranged by INBIOM, held at AU/Foulum, 07-12-2012 (presentation)

Møller, HB ”Halm til biogas” at the seminar ’Økonomiseminar for biogas’, 10-12-2012 (presentation)

Møller HB and Hansen MM ”Driftserfaringer med ekstruder” Grøn Energi dag, arrangement på Foulum, 05-09-2012 (poster)

7.5 International conference

Wahid R, Hjorth M, Møller HB, Kristensen SB, Gamot LF “Extrusion as pretreatment of lignocellulosic materials for boosting methane production” at Biogas Science 2014 in Vienna, 26/30-10-2014 (oral presentation)

Frydendahl S, Hjorth M, Cacciatore V, Wahid R, Mortensen R “Extrusion methodology for biogas and bioethanol pretreatment of lignocellulosic biomasses” at 11th International Conference on Renewable Resources and Biorefineries, in York, UK, June 3 – 5, 2015 (Oral presentation)

7.6 Student reports

Kristensen SBS and Tang Z ”The effect of mechanical pretreatment on the biogas potential of wheat straw” Open project Report/Aarhus University, 06-2011

Kristensen SBS “The effect of mechanical pretreatment on the biogas potential of annual lignocellulosic biomass” Master thesis report/Aarhus University, 01-2012

Paz JM “Reducing sugars availability in wheat straw, ryegrass and meadow grass samples after mechanical pretreatment by means of roller mill” Open project report/Aarhus University, 24-05-2012

Paz JM “Laboratory analyses to evaluate extrusion of lignocellulosic biomass as a pretreatment for biogas production” Master thesis report/Aarhus University, 04-04-2012

Markfoged M, Flecks M, Melvad P, Elefsen S, Juul T “Analyse of slidprisme” Projekt rapport/Ingeniørskolen Aarhus, 31-05-2013.

Jensen MH, Tojoga V, Mikkelsen HH, Andersen D "Analyse af slid på snekke" Projekt rapport/Ingeniørskolen Aarhus, 31-05-2013.

Gamot LF "Extrusion of biomasses at Aarhus University, Denmark" Engineering internship project report/Ecole National Supérieuere de Mines de Douai, 09-2013

Mortensen R "The effect of high temperature during extrusion of wheat straw" 03-03-2014

Mortensen R "Optimizing the biogas potential of wheat straw via extrusion pretreatment" 01-10-2014

7.7 Scientific articles

Hjorth M, Gränitz K, Adamsen APS and Møller HB "Extrusion as a pretreatment to increase biogas production" Bioresource Technology 102, 4989-4994. 2011; published initially in project

Wahid R, Hjorth M, Kristensen S, Ward AJ, Møller HB "Extrusion as pretreatment for boosting methane production: Effect of screw configurations and feeding velocity"; Energy Fuel 29, 4030-4037, 2015

Frydendahl S, Jørgensen U, Hjorth M, Felby C, Gislum R "Comparing methods for measuring the digestibility of miscanthus in bioethanol or biogas processing" GCB Bioenergy (in print) 2016

Frydendahl S, Hjorth M, Gislum R, Jørgensen U "Miscanthus pretreated by extrusion or roller milling before biogas production"; under revision, May 2016, Bioressource Technology

Mortensen R, Hjorth M, Kristensen S, Felby C "Effect of dry matter content and treatment temperature on extrusion upon pretreatment before biogas production"; to be submitted autumn 2016

Cacciatore V, Hjorth M "Effect of extrusion screw configurations as pretreatment to biogas production on mays, rice silage and wheat straw"; to be submitted autumn 2016