EUDP Project: J.nr.: 64015-0080
Project coordinator: Anette Alsted Rasmussen
Project coordinating organisation: IPU, Diplomvej 376, 2800 Kgs. Lyngby, Denmark (www.ipu.dk)

FINAL REPORT

EUDP 64015-0080

Increased biogas yield from straw, straw bedding, and other agricultural waste

[BioYield]

Foto: Torben Skøtt/BioPress

Partners:

IPU, TK Energy, Lunds Universitet,

Kærgård and Lemvig Biogas.

1. Project details

Project title	Increased biogas yield from straw, straw bedding, and other agricultural waste [BioYield]
Project identification (program abbrev. and file)	64015-0080
Name of the programme which has funded the project	EUDP
Project managing company/institution (name and address)	IPU, Diplomvej 376, 2800 Kgs. Lyngby Anette Alsted Rasmussen, aar@ipu.dk
Project partners	IPU, TK Energy, Lunds Universitet, Kærgård and Lemvig Biogas.
CVR (central business register)	IPU: 11 88 89 33
Date for submission	14. november 2018

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Content

2. Short description of project objective and results

2.1 English

The project has been focusing on developing a cost-effective methodology for increasing reactivity and handling properties for agricultural biomass particularly of low quality.

The project has developed a pilot system and test methods. Based on a combined experimental and theoretical study the feasible process window for pre-treatment of wheat straw by roll pressing is presented. The project demonstrate possibilities of how to avoid floating layers and improves knowledge on how to perform calibration and comparison between different measurement methods for biogas potential. Knowledge on how to lower the costs of production of biogas has been improved.

2.2 Dansk

Projektet har fokuseret på at udvikle en omkostningseffektiv metode til at forbedre reaktivitet og håndteringsegenskaber af landbrugsbiomasse, især lavkvalitets biomasse.

Projektet har udviklet et pilot anlæg og testmetoder. Baseret på kombinerede eksperimentelle og teoretiske undersøgelser præsenteres det mulige procesvindue til forbehandling af hvedestrå vha. valsning. Projektet har demonstreret muligheder for at undgå flydelag og udviklet kompetencer vedr. kalibrering og sammenligning af forskellige metoder til at bestemme biogas potentiale. Viden om hvordan omkostninger ved biogas produktion kan reduceres er forbedret.

3. Executive summary

The overall purpose of the activities was to develop a cost-effective methodology for increasing reactivity and handling properties for agricultural biomass particularly of low quality, in order to improve the yield and operating economy of the biogas production by 25 % as compared to presently used techniques.

In the production of biogas from straw, straw bedding and other agricultural waste, it is considered important to pre-treat the straw in order to open the outer shell of the straw to allow an efficient reaction between microorganisms and the interior straw material, which mainly consists of cellulose and hemi-cellulose. Opening of the outer shell is expected to improve the biogas yield and prevent development of floating layer in the biogas reactors.

The present project activities were initiated by developing a pilot plant for rolling of straw as a pre-treatment process to meet the above-mentioned requirements. As compared to other mechanical pre-treatment processes, rolling is considered an energy efficient process. Non-mechanical methods are considered too slow (microbial) and too costly (acid, alkali, hot water) [1]. In this project, literature studies have been performed. Both with respect to pre-treatment principles, mechanical processing of biomaterials and characterization of material from pre-treatment processes.

The project has successfully developed a pilot system for rolling of straw with a capacity of 100-2000 kg / hour. Based on a combined experimental and theoretical study the feasible process window for pre-treatment of wheat straw by roll pressing varying the feed, the roll gap, the roll speed and the moisture content of the bulk straw is presented.

Work on validation of quality biomass after pre-treatment has been carried out. A challenge associated with evaluating biogas production is that the process is very slow and the experiments then become very time consuming. Biogas potential measurement (BMP) is usually performed for 30-90 days in order to deplete the material of any digestible components. In BioYield development of a less time consuming enzymatic hydrolysis (EH) method has been carried out and compared to state of the art methods.

The ongoing technological achievements in the project and changed economic conditions in the industry have had the result that some of the prerequisites for improving the yield and operating economy of biogas production by 25% compared to current techniques are no longer applicable. Among other things, thorough studies have shown that some of the State-of-the-art information existing at project start was stated based on a too weak foundation.

BioYield has shown practical solutions to problems of adding straw into biogas reactors, such as demonstrating possibilities of how to avoid floating layers have been shown and the project has improved knowledge on how to perform calibration and comparison between different measurement methods for biogas potential. Hereby it becomes possible to compare different plants, and to have common references when discussing performance of different technology. BioYield has improved the knowledge on possibilities on how to lower the costs of production of biogas, as it offers the possibility to increase the production by adding of straw, and also the production rate, leading to a larger yield from the same plant.

There is a strong political focus on using biomass resources because they are left-overs from a primary production of grain products. Further is it is a way to create additional income for farmers in a tough market. The sharp fall in oil prices over the last 2-4 years has increased the demands for subsidiaries for biomass-based technologies. Subsidiaries based on political decisions usually make the industry more reluctant to invest.

The currently technology has a good societal and environmental potential. Due to the current structure of the market, business potential for private investors is low.

4. Project objectives

The project objective was to develop a cost-effective methodology for increasing reactivity and handling properties for agricultural biomass particularly of low quality, in order improve the yield and operating economy of the biogas production by 25 % as compared to presently used techniques.

There is an increasing market for processing and handling of straw and other cellulosic residues such as grass and bagasse and there is a strong political focus to use these biomass resources because they are available anyway, and it is a source of extra income the farmers.

In the production of biogas from straw, straw bedding and other agricultural waste, it is considered important to pre-treat the straw in order to open the outer shell of the straw to allow an efficient reaction between microorganisms and the interior straw material. The interior of the straw consist mainly of cellulose and hemi-cellulose. Opening of the outer shell is expected to improve the biogas yield and prevent development of floating layer in the biogas reactors.

The present project activities were according to the project plans initiated by developing a pilot plant for rolling of straw as a pre-treatment process to meet the above-mentioned requirements. As compared to other mechanical pre-treatment processes, rolling is considered as an energy efficient process. Non-mechanical methods are considered too slow (microbial) and too costly (acid, alkali, hot water) [1].

The project has met the original formulated milestones. The project has successfully developed a pilot system for rolling of straw with a capacity of 100-2000 kg / hour and conducted the sub-milestones leading up to this results (see sections 5.8 and 5.10), as a part of this work also a report on evaluation of different pre-treatment principles (see section 5.1).

Work on formulation of scientific evaluation methods and verification of existing data is described in section 5.3. Validation of quality of biomass after pre-treatment is described in sections 5.13. A challenge associated with evaluating biogas production is that the process is very slow and the experiments then become very time consuming. Biogas potential measurement (BMP) is usually performed for 30-90 days in order to deplete the material of any digestible components. In the project development of a less time consuming enzymatic hydrolysis (EH) method has been carried out and described in section 5.4.

The project partners each have different strengths and skills. During the project formulation these versatile skills and possibilities was found to be an advantage. During the project execution this has been demonstrated several times to solve challenges and ensure progress.

It has turned out to be invaluable to have a large floor space for experiments with craftsmen and engineering capabilities with a close insight to the energy sector which was provided by TK Energy. The academic and systematic approach for in depth analysis of biochemical details and documentation where provided by Lund University. IPU provided a systematic and practical examination of several details with respect to straw properties and parameter experiments with rolling of straw, as well as overall project management. Kærgård have had a small role in the project providing input from the farmer’s point of view and also more practical, providing straw to the experiments. Lemvig Biogas has provided input and details from a practical point from a running biogas plant, and a suggestion for an alternative design for a biogas plant.

In total the joined forces from the project partners/project group has shown a good cooperation and strong will to overcome the challenges in the project by providing each of their expertise’s to the project execution.

The ongoing technological achievements in the project and changed economic conditions in the industry have had the result that some of the prerequisites for improving the yield and operating economy of biogas production by 25% compared to current techniques are no longer applicable. Among other things, thorough studies have shown that some of the State-of-the-art information existing at project start was stated based on a too weak foundation.

Due to the above mentioned challenges it was not possible to meet the original formulated overall objective, by conductance of the original formulated milestones and deliverables. By initiating collaboration with another EUDP project: Segrabio, it was possible also to evaluate other pre-treatment methods, as originally planned.

The main findings and perspectives of the project are treated in chapter 6 and 7, and has been disseminated as described in section 5.15.

5. Project results and dissemination of results

In the present chapter the most important project results is presented followed by an overview of the dissemination activities.

The chapter is initiated with results of literature studies of pre-treatment principles, mechanical processing of biomaterials, and characterisation material from pre-treatment methods, and evaluation of theses (section 5.1 to 5.3), followed by methodology development for evaluation of biogas productions in section 5.4.

In section 5.5 to 5.7 the work on design and constructions of the rolling equipment is described. Experimental results with this rolling system are described in section 5.8 and supplementary materials test with wet straw are described in section 5.9. The summary of the findings of the combined experimental and theoretical study of the rolling system is given in section 5.10.

In section 5.11 and 5.12 work with feeding experiments of straw under water/innoculum is presented and evaluated.

Validation of the quality of the biomass after pre-treatment is presented in section 5.13.

The chapter ends with the business plan and an overview of the dissemination activities.

5.1 Pre-treatment principles

In the project different pre-treatment techniques for biogas production have been studied and documented in the report 'Literature Study on Mechanical Processing of BioMaterials' [1]. The report evaluates different pre-treatment principles that can act as alternatives to the pre-treatment technique developed in the BioYield project. Initially the report gives a short overview over a broad range of pre-treatment methods. The work was focused, on mechanical pre-treatment methods as other methods are considered too slow (microbial) and too costly (acid, alkali, hot water) [1]. Based on literature studies the report gives a description of the most common mechanical pre-treatment methods, such as ball milling, knife milling, hammer milling, rolling / compression milling, chopping, shredding, pelletizing and extrusion. The different techniques are evaluated with respect to energy consumption and additional biogas yield. The report also briefly describes some methods applied at Danish biogas facilities. The report gives an overview of relevant assessment criteria. However, it is a difficult task to evaluate and compare different pre-treatment methods as they often influence the up- and down-stream processing costs and therefore cannot be seen as an isolated part of the biogas production. Furthermore there are no agreed standard on how to asses and evaluate the different pre-treatment methods with respect to energy consumption, biogas yield and other factors to a complicated non trivial task. Thus the effects on the economy of a biogas facility are difficult to evaluate. The pre-treatment methods presented in the report all have certain limitations when it comes to moisture content or the opposite equivalent; the dry matter content. Figure 1 shows the ranges in which different pre-treatment methods can be applied. Ball mills and extruders are diverse in operation, but lack handling of dry biomass such as e.g. straw.

Figure 2 couples the specific energy consumption to the application range for the pre-treatment methods. This serves as a tool to determine if a method is better or worse than the ones reported in open literature. The energy consumption is shown in the figure pr. ton of biomass. A better comparison would be if the energy consumption in studies were calculated pr. dry matter content. This will make it easier and more accurate to compare different types of biomasses.

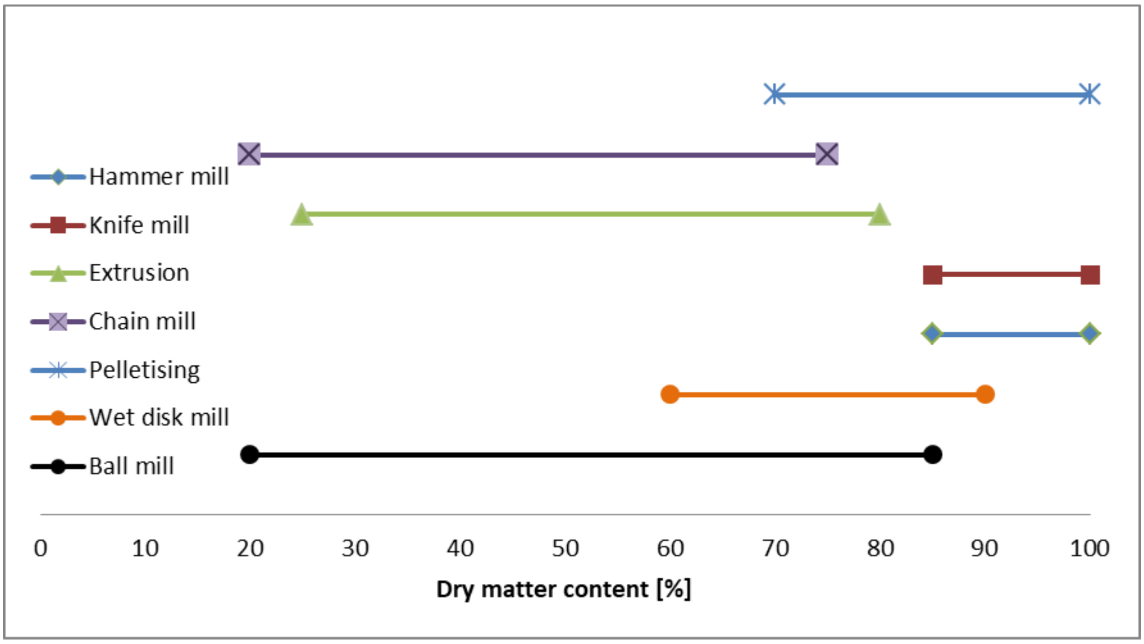


Figure 1: *Application range for pre-treatment methods with respect to dry matter content of the biomass. Sources: [3 - 13].*

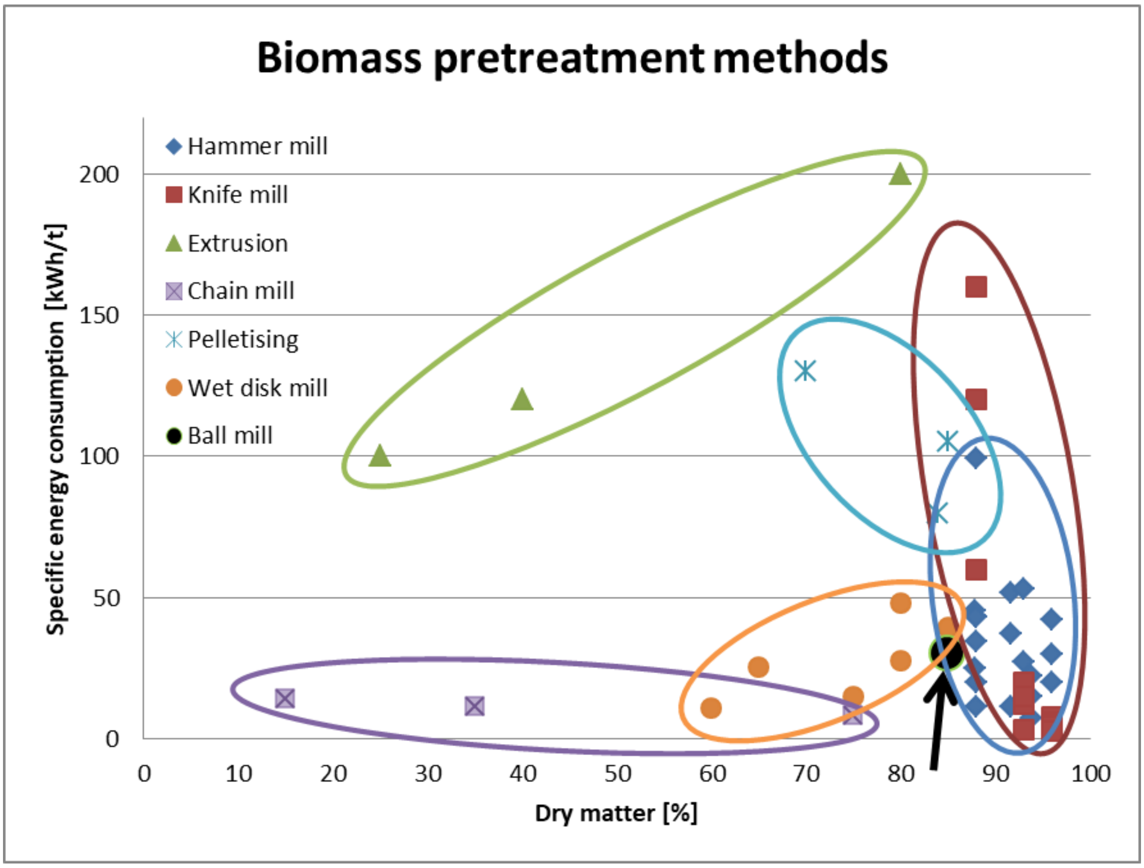


Figure 2: *Specific energy consumption for pre-treatment methods as function of the dry matter content of the biomass. Sources: [3 - 13].*

The tendency is that hammer milling, knife milling and pelletizing works best with biomass with high dry matter content, whereas chain milling and extrusion requires at least 20 % moisture in the biomass. Methods such as wet disk milling, knife milling, hammer milling and pelletizing have relatively low energy consumption and extrusion is more energy demanding. The factors that had most influence on the energy consumption were the mill screen size and dry matter content. Decreasing the mill screen size causes the energy consumption to increase and a higher dry matter content also increases the energy consumption.

5.2 Literature study on Mechanical Processing of Bio Materials

During the project a literature study on mechanical processing of bio materials was performed in order to document the State-of-the-art [1]. Parts of this work are also published in [14]. A resume of the most important findings are given below.

Industrial biomass machines for briquetting and agglomeration

Roll presses applied for briquetting or agglomerating and pressing biomass typically consist of a feeding mechanism and compacting rollers. The feeding can be either passive using gravity, or active using screws for continuous feeding. The performance of the feeding and rolling mechanism depends on the mechanical material properties, which must be considered when choosing and designing the mechanisms [15]. Gravity feed systems rely on flood-loaded chutes to feed roll presses. They are limited in force and require a large roll diameter, and they are most suitable for easy flowing and permeable solids [16]. The primary advantages are low energy consumption, use of wide rolls, and the ability to handle lumpy solids.

In order to obtain higher feed pressures and more consistent flow through the press, screws can be used for feeding. Since screws are capable of reaching considerably higher pressures than gravitational feeding, a smaller roll diameter is needed, but higher feed pressure makes the process more susceptible to material changes and pressure build-ups [17], which increases the complexity of control. The screw is superior to the gravitational feed when requiring a uniform quality of for example briquettes or agglomerated material, as it offers more flexibility and possibilities in controlling the feeding. The screw however feeds in a circle, whereas the cross section of the rolling inlet has a rectangular interface, leading to a non-uniform processing of the material, which can be accommodated for by multiple parallel feeding screws [17]. When using a single screw, two common ways of designing the system, is by feeding a chute, in which the screw is placed, or placing the screw at the outlet of the chute. In order to ensure a more homogeneous material feed, two screws can be used, although it increases the complexity of control.

Mineral comminution industry

In the mineral industry, machines are applied for comminution by breaking material into smaller pieces. Machines designed for the mineral crushing industry are intended for operating conditions requiring high pressures exerted onto materials of high hardness resulting in extensive wear. These machines are applied for crushing cement, clinker and ore minerals as a pre-treatment prior to ball milling. The roll is used for creating inter-particle breakage. Energy consumption in subsequent ball milling is thereby greatly reduced [18].
Commercial machines from FL Smidth, Köppern, Outec, KHD Humboldt Wedag and other manufacturers are all based on the same basic principles. The feeding is in commonly gravitational and controlled by feed gates, but can also be screw fed.

Theoretical study of rolling of porous material

Johanson [19] was among the first to propose a theoretical analysis of rolling of granular solids. The analysis adopts the flow criterion by Jennike and Shield [20] for granular materials, which is based on intergranular Coulomb friction in an isotropic, compressible and cohesive material obeying the effective yield function. Johanson [19] describes the rolling of granular material by defining an *"angle of nip"* α_{nip} such that for any angular position $\theta \geq \alpha_{nip}$

Figure 3, the rolls move faster than the material, so that slip occurs along the roll surface; for any $\theta \leq \alpha_{nip}$, no relative motion occurs between the granular solid and the rolls. When the nip angle α_{nip} $\alpha$and the bulk density at the position $\theta = \alpha_{nip}$ $\theta = \alpha$are known, the maximum density and pressure extended on the material at $\theta = 0$ can be calculated, provided the pressure-density relation is known. The pressure-density relation can be determined by close die compaction tests.

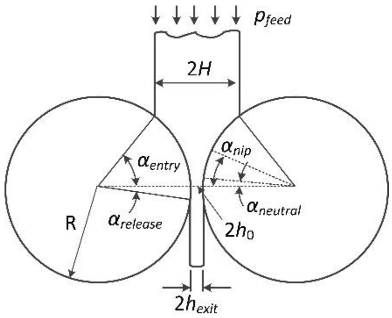


Figure 3: *Rolling model by Johanson [19]*

Dec et al. [21] compares various modelling methods for analysis of powder compaction in rolling and describes the slab method analysis developed by Kuhn and Downey [22]. They find good agreement between calculated and measured pressure distribution in the roll bite when rolling lignite.

Cunningham et al. [23] have established a 2D FE model for analysis of powder compaction in rolling using ABAQUS finite element code. Their material model for microcrystalline cellulose powder is based on Drucker-Prager's spherical cap model and a series of tests using diametrical compression, simple upsetting and closed die compression. Their rolling analysis identifies two slip zones namely one at the entry of the roll gap, another at the exit. In between these two zones a major zone of sticking friction is located. This is an agreement with analysis on skinpass rolling of steel by Kijima and Bay [24], where high friction prevails as in the case investigated by Cunningham. Their results on calculated pressure and friction distribution are in good agreement with experimental measurements by themselves as well as others [25]. Cunningham et al. [23] calculates the increase of relative density with increasing reduction and feed pressure. Maximum density appears at the centre line where $\theta = 0$. Contact with the rolls exceeds beyond this point due to expansion of the bulk powder caused by stress relaxation in the relieve zone.

Guigon and Simon [26] have designed a laboratory roll press with screw feeder and studied the influence of a forced feed system on compaction when rolling monohydrate lactose with 0.5% magnesium stearate. Fixing the screw feeder speed at seven different levels from 10.4 rpm to 45 rpm experiments were carried out with varying roller speed to the upper and lower limits allowing compaction. The lower limit of roller speed appeared, when over-compaction occurred, whereas the upper limit was set by no throughput. Their results indicate that the throughput is only determined by the screw feeder, no matter the roller speed.

5.3 Literature study on characterization of material from pre-treatment methods

During the project a literature review of research reporting alternative methods for evaluating different pre-treatments of lignocellulosic substrates in anaerobic digestion has been performed [27]. Physical properties of lignocelluloses, how they are commonly analysed and whether those methods are suitable for anaerobic digestion feedstocks (as compared to ethanol fermentation feedstocks) was the main focus of this work. The abstract is given below.

Lignocellulosic substrates like food and agricultural wastes are one of the most abundant renewable carbon sources, but its rigid structure makes it resistant to anaerobic digestion (AD). A pre-treatment that disrupts the rigid lignocellulosic structure and increases the bioaccessibility of the cellulose and hemicellulose to the microorganisms, is therefore necessary in order to obtain an efficient process. Depending on whether the pre-treatment is mechanical, thermal, biological or chemical, different chemical or physical substrate properties are targeted.

The aim of the review was to investigate how and when certain chemical and physical properties of lignocellulosic materials, that previous studies have shown to be rate limiting for the methane production in AD processes, can be analysed in order to evaluate the effect of a pre-treatment method. Since the commonly used biochemical methane potential (BMP) tests are both very time consuming (30 – 90 days) and dependent on the activity of the utilized inoculum, there is a demand for a quicker method with higher comparability.

It has been shown that the substrate particle size has a significant effect on the hydrolysis constant of the digestion process [28]. Mechanical pre-treatment, where particle size reduction is achieved, might thus be evaluated according to this correlation, as an alternative to BMP tests.

Steam explosion on the other hand results in cellulose fibre swelling, and if combined with a chemical, lignin and hemicellulose solubilisation can also be achieved. This will lead to an increased surface area of the remaining solids which can be estimated by Simon's staining, Brunauer–Emmett–Teller (BET) analysis, water retention capacity and/or enzyme adsorption analysis. However, BET-analysis requires high density materials for accurate results and the water retention capacity also depends on the crystallinity index (CrI) of the cellulose since the water molecules form hydrogen bonds with the amorphous parts. The choice of measurement method will hence affect the quality of the data. Depending on what method is employed to measure the CrI of cellulose, the results can differ dramatically. M. Hall et al. [29] compared solid state C-NMR to X-ray diffraction analysis, which measured the CrI of Avicel to 61 and 92 %, respectively.

In a study of Feng et al. [30] it was observed that KOH treatment had a significant effect on the methane yield of wheat straw due to lignin removal, but not on grass meadow. In conclusion, the effect of the pre-treatment will largely depend on the kind of feedstock that is being processed.

5.4 Methodology development for evaluation of biogas production

As mentioned above a challenge associated with evaluating biogas production is that the process is very slow and the experiments then become very time consuming. BMP is usually performed for 30-90 days in order to deplete the material of any digestible components. In investigations where BMP experiments are used as a means to quantify the outcome of another process, the pre-treatment in the case of this project, the time between the pre-treatment experiment and the analysis output becomes very long. At the same time it is well known that the rate-limiting step in the BMP experiment is the hydrolysis of the material into shorter hydrocarbons which is used by the bacteria consortia to produce acetate and subsequently methane and carbon dioxide. The aim of this method development was to create a method to describe the quality of the pre-treatment in a less time-consuming way. As straw material mostly contain polymeric carbohydrates (cellulose and hemicellulose) which acts as substrate for the biogas process and that there exists hydrolytic enzymes available in commercial enzyme cocktails the notion was that a shorter (than the BMP test) enzymatic hydrolysis (EH) of the pre-treated material and subsequent carbohydrate analysis would speed up the analysis response time in order to be able to perform an extended analysis of the pre-treatment process.

It is recommended that different enzyme dosages are tested on the substrate so that the hydrolysis does not result in lower yields due to a lack of enzymes. To capture the effect of a pre-treatment on a lignocellulosic material, the properties of that material should be the limiting factor. The commercial enzyme Cellic CTec2 (Novozymes) was used at loadings ranging from 2 – 35 FPU/g dry straw and the resulting sugar yields after 25 hours of hydrolysis are presented in Figure 2. All EH tests were performed in 500 mL bottles with continuous stirring, a liquid volume of 400 mL 0.1M sodium acetate buffer and a dry matter content of 2% (w/w). The temperature was set to 50°C and the pH was kept between 5.0 - 5.5. To prevent any loss of sugars via infection of other bacteria, an antibiotic was used.

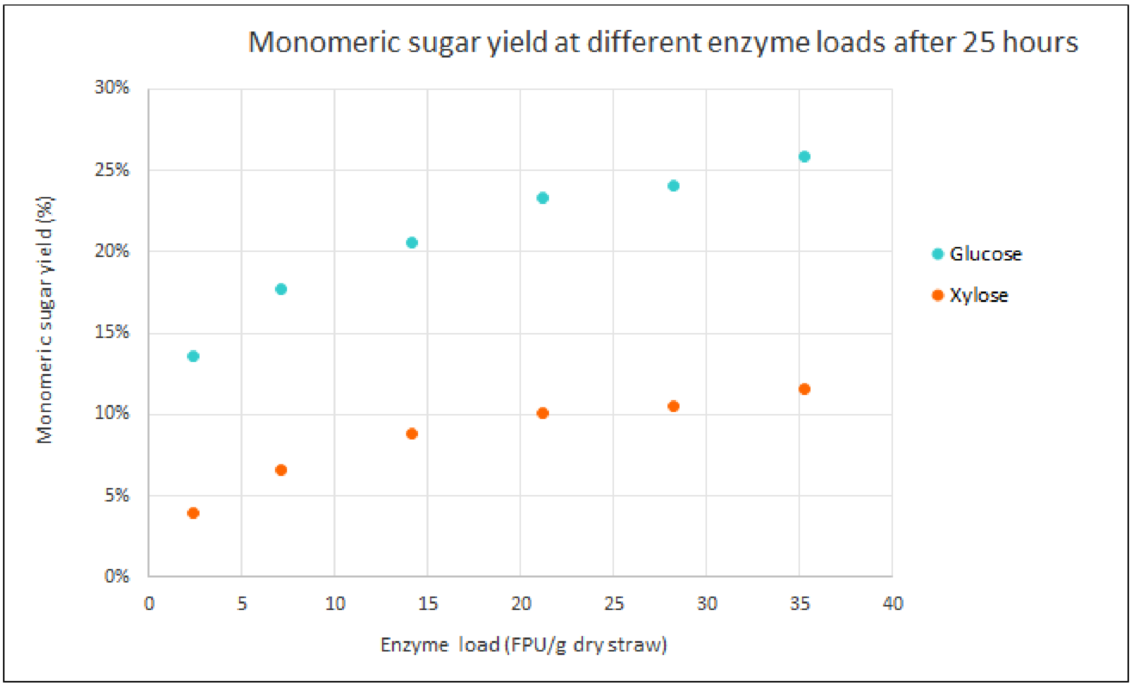


Figure 4: *Sugar yield (Glucose and Xylose) at different enzyme loads*

It was seen that at higher enzyme loads, the effect on the monomeric sugar yields decreased and further experiments were therefore executed at an enzyme dosage of 35 FPU/g DM.

As the result and objective of many mechanical pre-treatments is to achieve size reduction of the material, rolled straw was fractioned into different particle sizes by sieving. Enzymatic hydrolysis experiments as well as BMP tests were performed on the different straw samples and a comparison is presented in Figure 5. The cumulative methane yield, meaning the BMP, did not seem to depend on the particle size of the straw. The methane production rate on the other hand generally increased at a decreased particle size and a similar trend was observed in the enzymatic hydrolysis tests between the sugar yield and the straw particle size. A linear correlation between the glucose yield and the methane production rate could thus be identified (R^2 = 0.890). Additional EH tests were also performed on untreated and rolled straw (non-size fractioned). The results fitted well with the linear model.

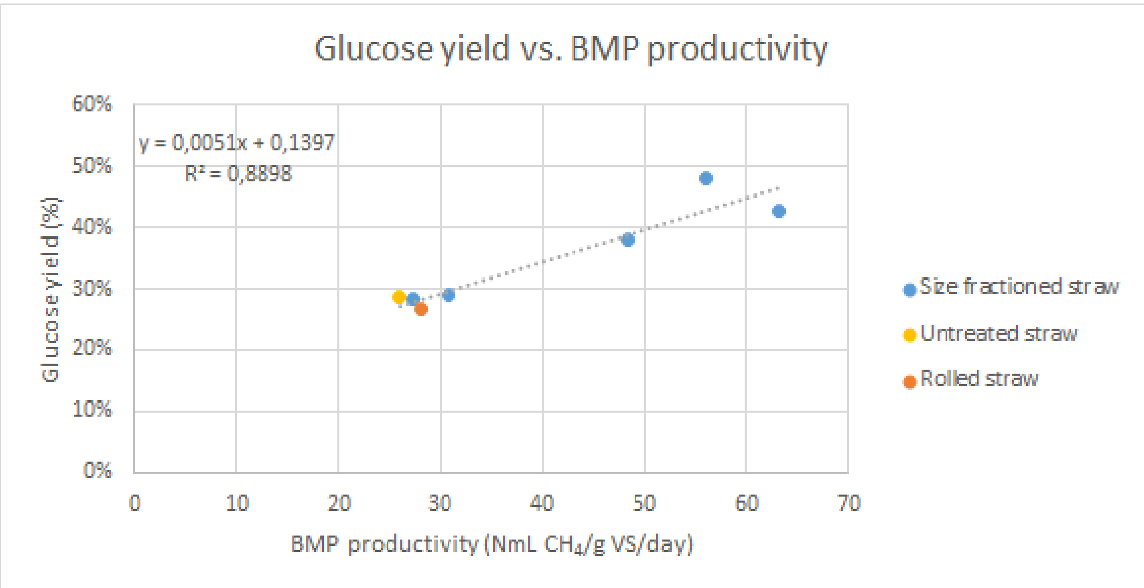


Figure 5: *Glucose yield vs BMP productivity*

Correlation between the glucose yield obtained by enzymatic hydrolysis tests and the methane production rate obtained by biochemical methane potential test is seen for mechanically pre-treated wheat straw. This shows that enzymatic hydrolysis could be an alternative faster evaluation method to the BMP method for this kind of substrate.

However, it should be noticed that this correlation has only been tested for mechanically pre-treated wheat straw. The hydrolysis bacteria in anaerobic digestion and the commercial hydrolytic enzymes act according to different mechanisms on both a structural and chemical level. It is therefore possible that this method will not be suitable for other kinds of materials or more severe pre-treatments.

5.5 Design and Construction of the biomass pre-conditioner

As discussed in section 5.1 and elaborated in figure Figure 2 there is several ways in which straw can be pre-treated before injected to a biogas reactor.

In the BioYield project rolling was chosen as pre-conditioner as it was expected to provide several advantages compared to hammer milling, knife milling, extrusion and other pre-treatments techniques. Below are listed some of the main advantages:

1. Lower specific energy consumption
2. Rather low wear of the equipment
3. Rollers are highly scalable
4. High hydrostatic pressure, which could facilitate pressing of water/inoculum into the straw material
5. Can work with wet material

1. The expected lower energy consumption compared to hammermilling and knifemilling is explained by the lack of energy requirement for breaking the straw into smaller particles . Compared to extrusion the energy requirement in rolling is lower due to lower stresses and strains as well as friction against the tools.

2 Low wear is expected as the material is mainly compressed by the rollers, with limited relative sliding during the process.

3. Rolling is already used in many applications including rolling of porous materials, which means that considerable experience in construction and maintenance of rollers exists even at large scale production.

4. It is expected that any free water/inoculum will be pressed into pores and thereby replace/remove trapped air. This should limit the tendency to formation of floating layers in the reactor. Furthermore, the liquid could include bacteria’s, which would thus be provided to the internal of the straw package. This would extend the active surface area and thus speed up the process.

5. Rolling is expected to work with a larger span of moisture than e.g. hammermilling, which has difficulties to break more ductile materials, like wet straw. Rolling should be relative unaffected of the presence of moisture, as long as the density of the material is increased sufficiently and friction against the rollers is high enough to provide the required work of the straw package.

In the project a rolling mill system that serves as a test set-up is developed. It has a capacity of 100-2000 kg/hour. Some of the findings of the work on pre-conditioning by rolling have been published in [14]. Parts of this work is described below and supplemented with additional findings.

A combined experimental and theoretical study has been carried out. A mechanical testing procedure of the bulk straw material including closed die compaction testing as well as simple upsetting of pre-compacted billets of straw has been carried out, based on which a mathematical model for the yield surface of the straw package is determined fitting to a geological cap model for porous material similar to the Drucker-Prager spherical cap model. The objective of determining this material model is to establish a numerical model of the rolling process in order to determine the influence of material and process parameters on the process window.

An experimental test campaign is carried out, in order to determine the feasible process window for pre-treatment of wheat straw by roll pressing varying the feed, the roll gap, the roll speed and the moisture content of the bulk straw.

Design of Rolling Equipment
Figure 6 shows the custom built, pilot rolling mill situated at TK Energy, one of the industrial partners in the project. Figure 6 shows a schematic drawing of the equipment. It is a 2-high mill with roll diameter D_{roll} = 695 mm and roll width w = 95mm. It is fed by two horizontal screws, an Ø300 mm dosing screw located above and before the injection screw, which is Ø180×500 mm on its cylindrical part but with a conical end, which fits into a conical housing of length 400 mm with end opening Ø95 mm. The end of the conical housing is mounted close to the roll gap, which is variable in size ($2 \le h_1 \le 12$ mm). The speed of rotation of the dosing screw can be varied within the range $2 \le n_{dosing} \le 30$ rpm whereas the injection screw speed can be varied in the range $15 \le n_{inject} \le 150$ rpm. The roll speed is variable in the range $3{,}5 \le n_{roll} \le 30$ rpm.

The dosing screw controls the feed rate, whereas the injection screw compresses the material and controls the feed pressure.

Figure 6: *Custom built rolling mill for crushing of straw*

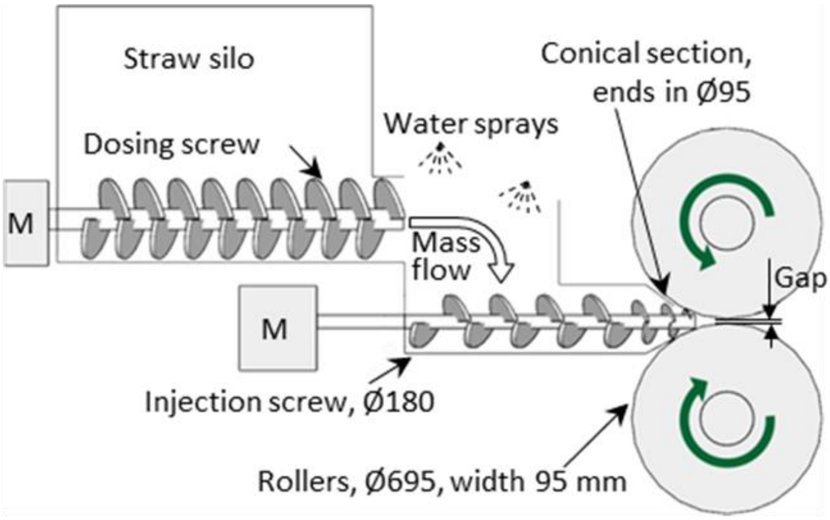


Figure 7: Schematic of equipment

5.6 Mechanics of rolling porous materials

The rolling process is based on friction between workpiece material and rolls delivering the required energy to deform the straw billet and drag it through the roll gap. As mentioned in section 5.2 two slip zones appear in the roll gap, namely one at the entry of the roll gap, another at the exit. In between these two zones a major zone of sticking friction is located. Friction on the billet in the inlet zone and in the sticking region is directed forward and thus facilitating the process, whereas friction in the outlet zone is opposing the billet movement. In order to ensure sufficient friction for the process to run the straw billet needs to be compacted to a certain density in front of the roll gap. This is the objective of the injection screw. If the density becomes too high at the inlet to the roll gap, the rolls may skid towards the billet, which creates excessive frictional heat and eventually make the rollers stop due to limitation of the roll torque.

5.7 Modelling and testing of Mechanical Properties of Bulk Straw

Theoretical model for porous material

A single straw can be considered as a hollow tube. Bulk straw is made up of these tubes, and thus has porosities in between straws, but also inside each straw. The research on mechanical properties is limited and confined to the areas of bale and pellet compression. No attention has thus been payed to the mechanical characterization and modelling of straw for applications different than bale and pellet compression. This means that high density compaction and material failure has been given very little attention.

Bulk straw behaves very non-linear upon loading similar to very ductile materials, and is therefore challenging to model. Mechanical properties of straw are by nature stochastic. The tensile strength, compressive strength, flexural strength and shear strength all vary from straw to straw.

Straw behaves orthotropically as the strength of the material varies with the direction of loading similar to composite materials e.g. fibre reinforced polymer. The handling of straw additionally makes the bulk straw behaviour stochastic, as straws are aligned differently in each bulk.

Upon loading bulk straw, it exhibits a combination of elastic strain, compaction and possibly no or very little plastic strain, as straw is brittle. The magnitude of each straw varies with loading condition. Upon compacting and thus densifying bulk straw, the strength of the material changes similar to porous materials e.g. powders and soil as a result of increasing pressure. This behaviour can depend on the straw or particle size, be attributed to internal friction, interlocking of straws, and dilation of the straw.

No single material model can cover all the above mentioned phenomena. The more complex and nonlinear the material behaviour becomes, the more difficult it is to model, and thus a compromise must be chosen from presently available mathematical material formulations. A material model typically consists of three parts namely: 1) a volumetric response describing the volumetric change of elements, 2) a deviatoric response defining the changes in shape of elements and 3) a failure model. The volumetric response represents the compaction and elastic deflection of the porous material, whereas the deviatoric response describes the strength of the material. The failure model concerns the criterion for material fracture.

A mathematical model of the mechanical properties of bulk straw is required in order to model the rolling pre-treatment process. Since this is chosen to be performed in the program LS-DYNA a material model available in this code has been selected. As such the geologic cap model named MAT_025 in LS-DYNA [14] has been chosen as one of the most flexible models able to model isotropic, porous materials, which means that anisotropy of the bulk straw is neglected for reasons of simplicity. The yield surface according to the model is plotted in the $I_1-\sqrt{J_2}$ space in Figure 8 where:

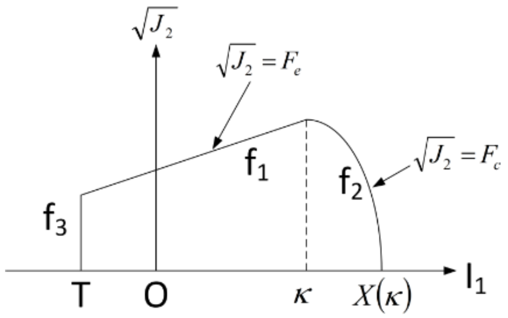


Figure 8: *Geological cap yield surface, LS_DYNA MAT_025.*

(1)

is the first invariant of the stress tensor, *p* is the hydrostatic pressure and:

$$\sqrt{J_2} = \sqrt{\frac{1}{2}s_{ij}s_{ij}} = \frac{\sigma_{eq}}{\sqrt{3}} \tag{2}$$

where s_{ij} is the deviatoric stress tensor. It should be noted that all normal stresses σ_i, σ_{ii} are defined as positive in compression. The failure envelope is given by:

$$F_e = \alpha - \gamma \exp(-\beta \times I_1) + \theta \times I_1 \tag{3}$$

The cap surface is given by:

$$F_c = \frac{1}{R}\sqrt{[X(\kappa) - L(\kappa)]^2 - [I_1 - L(\kappa)]^2} \tag{4}$$

$X(\kappa)$ is the intersection between the cap f_2 surface and the I_1 axis:

$$X(\kappa) = \kappa + RF_e \tag{5}$$

$L(\kappa)$ is defined by:

$$L(\kappa) = \begin{Bmatrix} \kappa & if\ \kappa > 0 \\ 0 & if\ \kappa \le 0 \end{Bmatrix} \tag{6}$$

The volumetric plastic strain:

$$\varepsilon_V^p = \ln\frac{\rho}{\rho_0} \tag{7}$$

where ρ_0 and ρ are the initial and current straw densities, is given by the following hardening law:

$$\varepsilon_V^p = W\{1 - \exp[-D(X(\kappa) - X_0)]\} \tag{8}$$

The tension cut-off surface f_3 is described by:

$$f_3 = T - I_1 \tag{9}$$

where T is the maximum hydrostatic tension the material can sustain.

Testing of mechanical properties

Testing of the mechanical properties of bulk straw were performed by Closed Die Compaction (CDC) tests as well as simple upsetting (SU) between plane, overhanging tool plates of pre-compacted straw billets at different densities made by pre-compaction in closed die, see Figure 9. The test billets were all prepared by pre-compacting the material to one of the following five densities: ρ = 800; 900; 1000; 1100; 1200 kg/m^3 in the closed die. The closed die was a steel tube of inner diameter Ø51 mm, and the billets were made by pouring a certain amount of straw at a time into the tube, compacting this to a relative low density (<200 kg/ m^3) then pouring additional material in, until a final height of app. 70 mm with required density could be achieved.

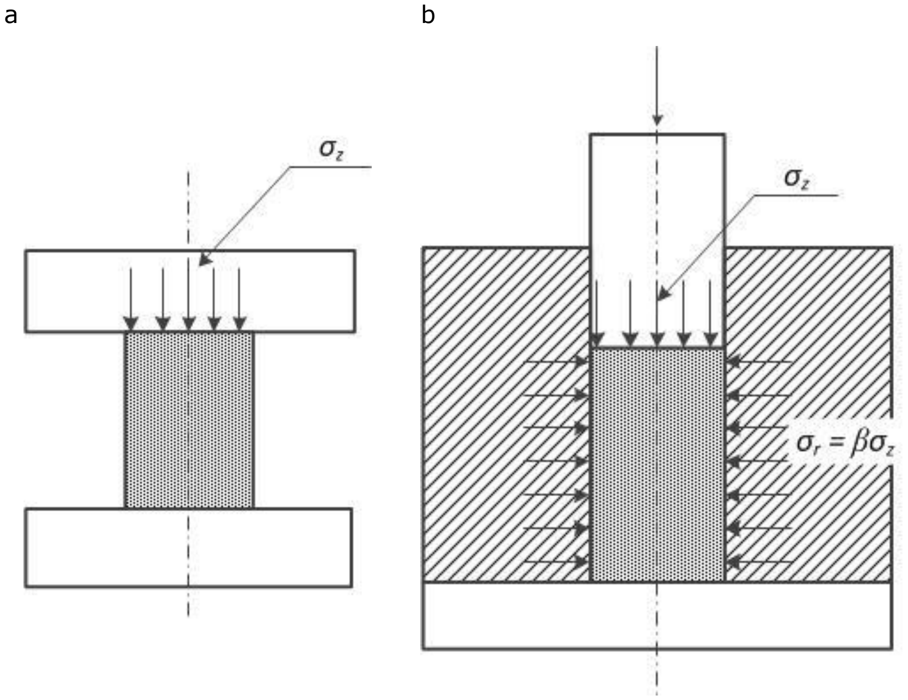


Figure 9: Schematic of a) Simple upsetting test, b) CDC test.

The radial stress in the simple upsetting test is zero. As regards the CDC test the radial stress can be calculated by multiplying the axial stress with a factor β:

$$\sigma_r = \beta\sigma_z$$

where $0.3 \leq \beta \leq 0.7$ [31]. It is then possible to express I_1 as well as $\sqrt{J_2}$ for the two tests as indicated in Table 1.

Table 1: Radial pressure, I_1 and $\sqrt{J_2}$ for simple upsetting and closed die compression.

	Freeform upsetting	Closed die compaction
σ_r	0	$\sigma_r = \beta\sigma_z$
I_1	σ_z	$(1+2\beta)\sigma_z$
$\sqrt{J_2}$	$\frac{p_z}{\sqrt{3}}$	$\frac{1-\beta}{\sqrt{3}}\sigma_z$

The initial density of the bulk straw was approximately $\rho_0 = 400$ kg/m^3 and the first significant load measurement was obtained after a compaction to 800 kg/m^3. This corresponds to a pre-compaction:

$$\varepsilon_V^p = \ln\frac{800}{400} = 0.69$$

The stress ratio $\beta = 0.5$ was assumed and the constants *α, β*, ɣ,* X_0 in the material model were all assumed equal to zero, which corresponds to no material strength in hydrostatic tension. Based on these assumptions the resulting material parameters in the LS-DYNA material model were calculated as shown in Table 2.

Table 2: Parameters in material model

α	β*	ɣ	θ	W	D	R	X_0
0	0	0	0.58	1.06	0.04	2.58	0

Figure 10 shows the density versus I_1, i.e. versus the hydrostatic pressure *p* see Eqn. (1). Figure 11 shows the yield surface versus I_1, i.e. the hydrostatic pressure, with the density as a parameter. Referring to Eqn. (1) the ordinate $\sqrt{J_2} = \sigma_{eq}/\sqrt{3}$, which for simple upsetting without friction implies that $\sqrt{J_2} = \sigma_0/\sqrt{3} = k$ (see Table 1), where σ_0 is the flow stress and *k* is the shear flow stress of the material. Figure 11 represents the required material model for numerical modelling of rolling of the porous straw material. It is noticed that the flow stress increases exponentially with the hydrostatic pressure as well as with the density.

In a more popular description Figure 11 shows when a material (here straw at different densities) will break (flow) if it is subjected to various hydrostatic stresses.

The abscissa axis with the hydrostatic pressure (I_1) can in a simplified way be understood as the material being compressed from all directions at the same time, e.g. wrapped in a film and exposed to a pressure from surrounding. An analogy could be to lower the film-wrapped straw in a liquid, which is then pressurized.

The ordinate axis with $\sqrt{J_2} = \sigma_0/\sqrt{3} = k$ tells when the material (straw) will float/break.

Above tells if a material (straw) is subjected to a hydrostatic compression (pressure), at a given density (see the different legends), then it will break if it is subjected to a load (tension/compression) in any direction larger than $\sqrt{J_2} = \sigma_0/\sqrt{3} = k$. If the load is smaller the material (straw) will not float / break. This means that any load/pressure situation *inside* the "cone"/ curves for the various densities will cause no damage to the material.

It also means that the material (straw) newer can be subjected to loads *outside* the "cone"/curves, since the material (straw) will break before that load situation will occur (it will always break when the load situation reaches the curve, which then either will change material properties (meaning the curve will change) or change the load situation (moving the load away from the curve).

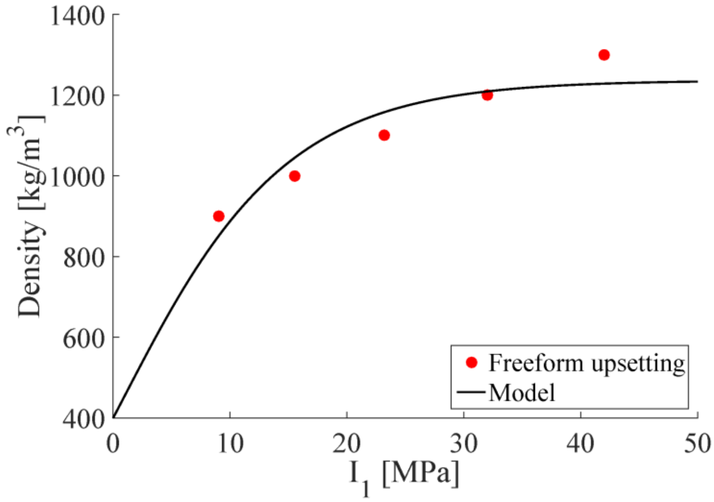


Figure 10: *Density versus the first invariant of the stress tensor.*

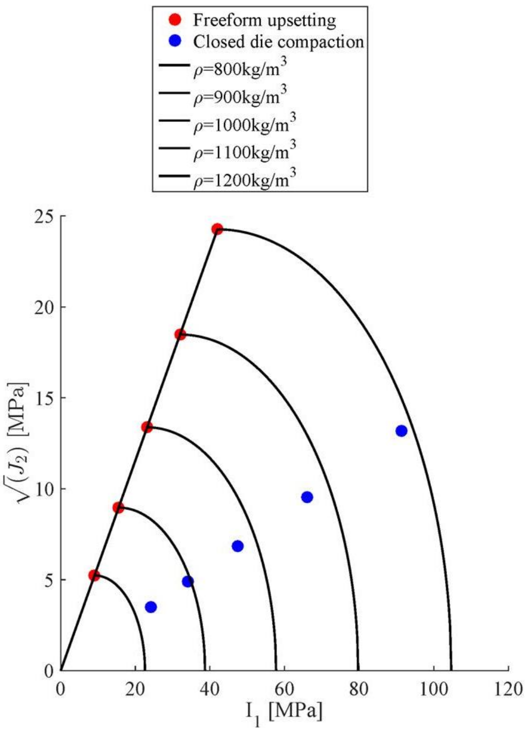


Figure 11: *Experimentally determined yield surface*

5.8 Experimental results with the developed rolling system

Control of mass flow

A number of rolling experiments with chopped wheat straw were performed. During the individual experiments, variables such as dosage rate, injection screw speed, roller speed, moisture content in raw material and addition of water between dosage and feeding screw have been varied.

As seen in Figure 7 the dosing screw is located above the injection screw and initially with a horizontal overlap of approximately 400 mm. The purpose is that the straw should fall from last part of the dosing screw into the injection screw. The capacity of the injection screw must exceed the mass flow of the dosing screw as otherwise the material will accumulate between the two screws. This means that increasing dosage rate requires increasing injection screw speed to prevent material from accumulating in the funnel.

The rotational speed of the different parts (dosage, feed and roller) is regulated by frequency converters. It was found that even at low speeds on the rollers it was necessary to apply a high speed of the injection screw to transport enough material and build up an appropriate pressure in front of the rollers so that the bulk of straw was sufficiently compacted for subsequent crushing by the rolling process instead of just being transported through the rollers without any crushing. In practice, it was found appropriate to run with a velocity of the injection screw corresponding to an electrical frequency between 90 and 150 Hz. Unfortunately, the motors are power-limited in the frequency range 50 - 150 Hz. This means that a higher feed rate provides a lower available torque. As an example a reduction of the injection speed (Frequency converter setting) from 100 Hz to 90 Hz gives rise to an increase in mass flow from 0.08 kg/s to 0.11 kg/s at a fixed rolling peripheral speed of 163 mm/sec.

If material flow is increased a higher pressure will be built up in front of the rollers. As this pressure rises, the injection screw requires a larger torque. If the injection screw is able to deliver this torque, the operating conditions will remain stable. If the torque limit of the motor, however, is exceeded, the frequency converter will lower the speed to protect it from overload. If dosing is not stopped in time, the high pressure in front of the rollers will stop rotation of the injection screw and / or the rollers. This situation is named *clogging* in the following.

If friction between straw and rolls is small, it is usually the injection screw that stops, when too much straw is dosed. This is due to the pressure build up in front of the roll gap to an extent where the torque limit of the injection screw is exceeded. If friction is larger, the rollers will stop first due to the roller torque limit. Subsequently the injection screw will stop as pressure builds up in front of the rollers. Both situations are referred to as clogging.

Determination of process window

The objective of the experimental plan is to determine the window in which the rolling process could operate without clogging. The process parameters investigated are the roll gap, the mass flow and the speed of rotation of the rollers, the latter expressed as the peripheral speed of the rollers. Two roll gaps were investigated, namely 6 and 8 mm corresponding to a reduction of 94% and 92% respectively. Larger roll gap resulted in little if any crushing and smaller roll gap caused clogging.

Preliminary experiments on rolling of moist straw showed it possible to run with larger mass flow than with dry straw. It was decided to investigate this further by filling the dosage system with dry straw and then in selected experiments spray the straw with a water mist between the dosing screw and the injection screw, see Figure 7. In these experiments, three nozzles were applied and a water supply of approximately 1 kg/min was provided. Referring to Figure 12 and

Figure 13 the limits of dosing dry straw and wet straw, respectively, were found in this way. The mass flow indicated in the figures is based on dry straw from the dosing system containing 15% water (by mass). The figures shows the successful experiments for the dry as well as the wet straw with green markers whereas clogging is shown with red markers. The added water corresponds to between 12% and 26% mass for 6 mm gap and 9% to 13% mass for the 8 mm gap implying a total water content of 24-35% for 6 mm gap and 22-25% for 8 mm gap. The percentages are for maximum mass flow achieved at the different roller speeds.

Figure 12

Figure 13 shows that higher mass flow is possible with wet than dry straw and that increased roller speed increases the maximum mass flow in case of wet straw, whereas it decreases in case of dry straw. The latter is explained by the above mentioned limit in roller torque.

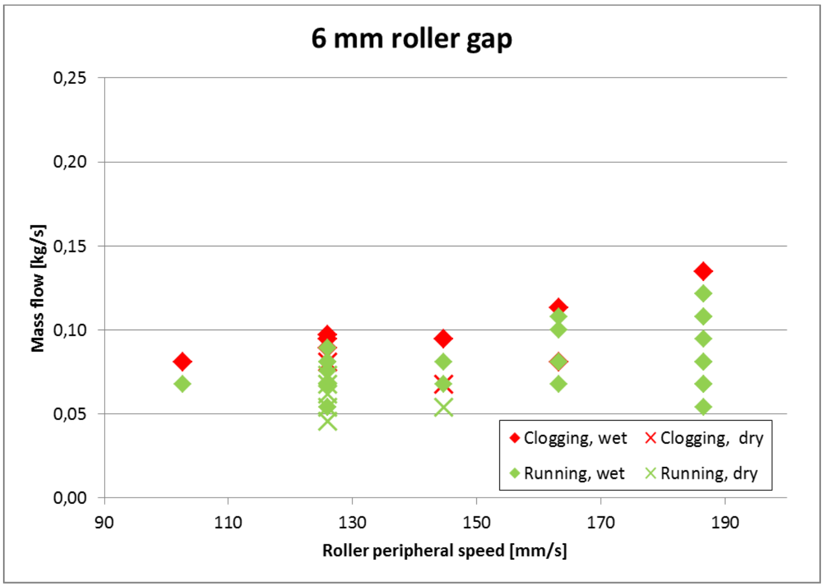


Figure 12: *Mass flow vs. roller speed for experiments with 6 mm roller gap.*

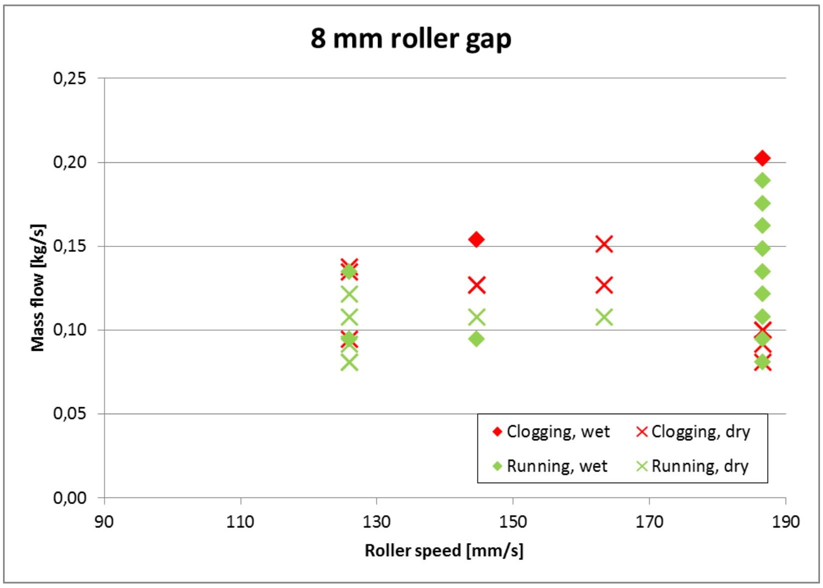


Figure 13: *Mass flow vs. roller speed for experiments with 8 mm roller gap*

For the case of 8 mm roll gap a special experiment was carried out rolling first wet straw with high mass flow / density by water spraying the dosed material. After running through a stable period, water spraying was stopped to investigate how the process developed. It turned out that clogging occurred stopping the rolls and injection screw when dry straw entered the rollers.

When comparing the mass flow with the roller speed and the area of the roll gap the characteristic maximum density of the material in the roll gap can be determined, see Figure 14 and Figure 15. It is noticed that the density may rise up to 1200-1400 kg/m^3 and that higher densities are obtained with 8 mm roll gap than 6 mm in the high end range of roller speed (145-190 mm/s). Aiming at larger densities by increasing the dosage further is not possible due to clogging.

Maximum theoretical density of wheat straw can be calculated from the weighted densities of the relative mass percentages of the chemical components. For wheat straw with 15% water content the maximum theoretical density becomes approximately 1550 kg/m^3. In this calculation the effect of compacting mixed molecules is not taken into account. Nor is the compressibility of solids taken into account [32].

In Figure 12 representing the high reduction with 6 mm roll gap the limiting mass flow before clogging in case of wet straw is seen to be almost proportional to the rolling speed. The added mass of water by spraying is not included in the calculated mass flow. According to

Figure 14 the maximum density is approximately 1200kg/m^3 (without the contribution from the added mass from the water spray). If rolling is performed close to this limit, the straw output is crumbled. If the mass flow and the density are reduced the straw becomes more coherent and the output is plate formed. This occurs at densities between 750 and 1000 kg/m^3. At even lower densities the straw becomes looser again. The reason that the straw crumbles at the highest densities is that it breaks down into smaller pieces by fracture and crushing of the straw stalks, which results in less coherence. Running with large mass flow results in large pre-compaction of the bulk straw before rolling. As long as clogging is avoided this will lead to improved crushing of the slab during rolling and thereby finer particles and crumbling, which is the objective of the roll pressing operation. The issue is therefore to run with as high density as possible but still ensuring that clogging does not occur.

Reduced mass flow and density diminishes the decomposition and the cohesion of the output increases. This may possibly be due to moist lignin that has been heated above the glass transitions temperature, Tg (similar to wood pellets). Tg_{lignin} = 53 – 63 °C [33], which makes the straws stick together in form of a plate. The plate, however, has only little strength. Even lower mass flow/density causes decreasing cohesion as the straw does not heat up as much and the pressure is less (compare pellets and lignin that bind). After the straw plate has left the roll gap, it expands to a thickness of about 5 times the height of the roll gap.

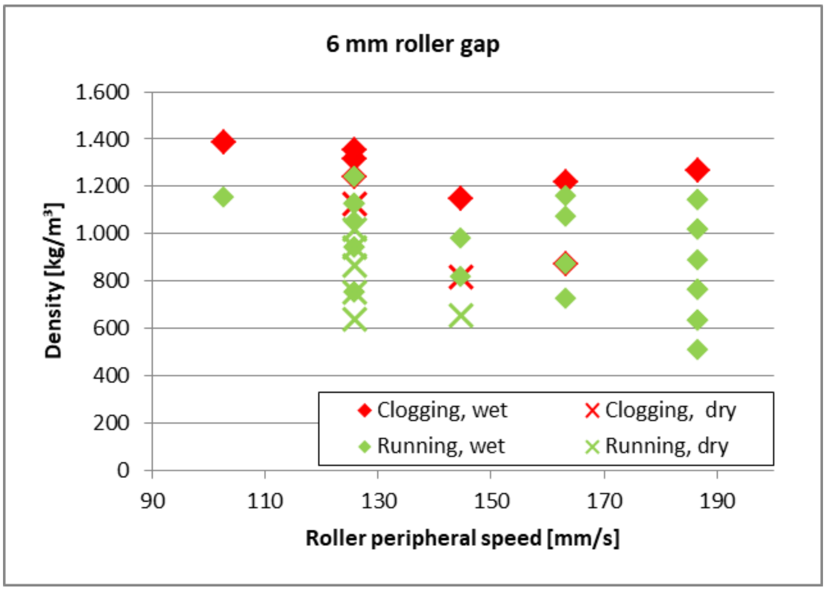


Figure 14: *Density vs. roller speed with 6 mm roller gap.*

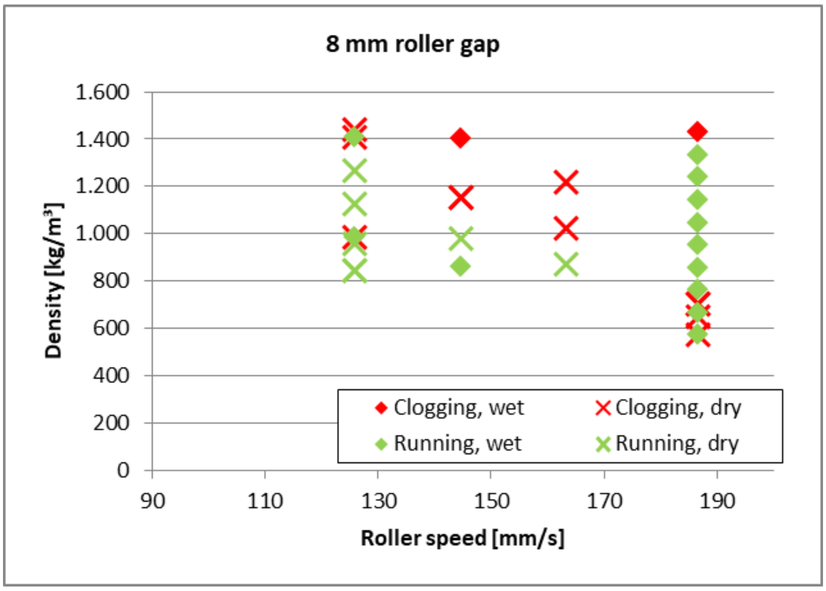


Figure 15: *Density vs. roller speed with 8 mm roller gap.*

5.9 Supplementary materials tests with wet straw

The significant difference observed in maximum mass flow and density, when rolling dry and wet straw, caused a decision to carry out new material tests by closed die compaction and simple upsetting comparing the data for dry and wet straw. Figure 16, representing the closed die compaction tests, shows that the stress-density curves are almost coinciding. This is probably due to the fact that the relative location between the individual straws is not changed during CDC testing. It is anticipated that this fact implies mechanical interlocking and limited effect of interior lubrication between straws. According to Figure 16 the density for dry straw reaches a maximum of approximately 1600 kg/m^3. This is slightly above the maximum theoretical density. Furthermore, the two curves intersect each other. Both of these observations may be explained by uncertainties in the measurements and difficult calibration of the zero position for the measurements. Offsetting the curve for dry straw lateral with 3% removes the intersection and the "too high density" calculation.

The curve for wet straw represents the density of 15% wet straw without taking into account the influence of the additional 11% of water for wetting.

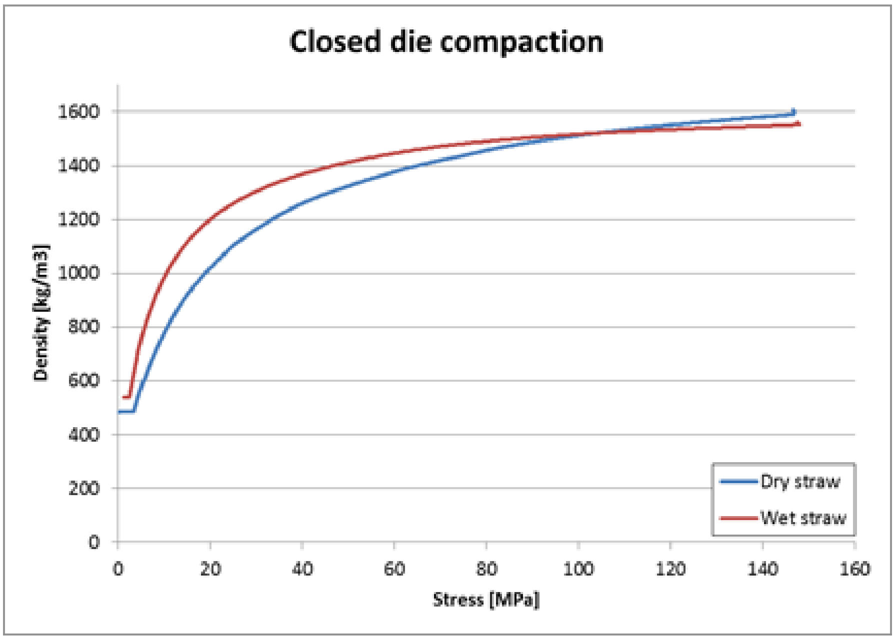


Figure 16: *Density vs. stress for closed die compaction test.*

Figure 17, representing the simple upsetting tests, shows a large difference between wet and dry straw. This is probably due to the less confined deformation in simple upsetting compared to the closed die compaction test, which allows relocation of individual straws during deformation. In this case the lubricating effect of the water is significant. In good correspondence with this observation it is noticed in Figure 12 and

Figure ***13*** that the maximum possible mass flow increases with water lubrication. Figure 17 furthermore shows that the wet straw collapses at much lower stresses than the dry straw.

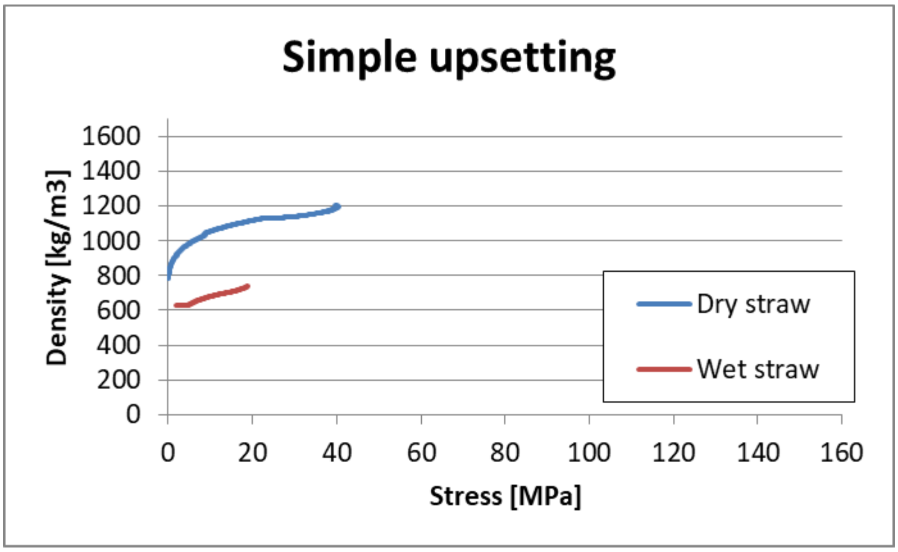


Figure 17: *Density vs. Stress for simple upsetting test.*

Figure 18:

Top: Loose straw and a billet with Ø51 mm made of same amount (80 g).

Middle: left: 2 billets of wet straw. Right 2 billets of dry straw. All billets are compacted with a punch pressure of 146 MPa.

Bottom: left wet straw after simple upsetting. Right: Dry straw after simple upsetting.

During handling of the straw for both CDC tests and simple upsetting tests significant differences in behaviour between wet and dry straw were observed. Figure 18 shows the amount of straw compacted to a single billet, the individual billets before and after upsetting. All billets and the pile of loose straw have the same mass of 60 gram. The added water is 6 gram, which is not added to the total mass in the density calculations. The wet billets have clearly expanded significantly more than the dry billets. During simple upsetting large difference was observed with respect to the load the collapse occurred and the behaviour in the collapse. In case of wet straw the collapse was continuously progressing with a significant diameter increase, whereas the collapse in case of dry straw was almost instantaneously, with an ejection of some straw to the side. Besides this small amount of straw ejected at collapse there was hardly any diameter increase on the billet.

5.10 Summary of the findings of the combined experimental and theoretical study of the rolling system.

A pilot rolling mill with a double screw feeder is designed and constructed for crushing of bulk straw. Experiments show that the roll speed and the roll reduction should be chosen within a specific range depending on the injection screw speed to avoid blocking or insufficient compaction. A mechanical testing procedure of the bulk straw material including closed die compaction testing as well as simple upsetting of pre-compacted billets of straw is carried out based on which a mathematical model for the yield surface is determined fitting to a geological cap model for porous material similar to the Drucker-Prager spherical cap model.

Inspired from the above mentioned investigations, an experimental test campaign has been carried out, in order to be able to determine the feasible process window for pre-treatment of wheat straw by roll pressing varying the feed, the roll gap, the roll speed and the moisture content of the bulk straw.

Wet straw is significantly easier to roll than dry straw. Rolling wet straw densities up to 1400 kg/m^3 can be achieved whereas the maximum density for dry straw was significantly lower with the available torque in the system.

In closed die compaction of straw a density close to the theoretical maximum of 1550 kg/m^3 can be achieved at a pressure of 100-150 MPa if the billet is low (25 mm). Higher billets will probably not be possible to compact to such densities due to die friction towards the die walls, unless compaction is performed stepwise.

Whereas the load displacement curves for wet and dry straw in closed die compaction are very similar, this is not the case in simple upsetting, where wet straw appears to have lower density than dry straw at same stresses. This is most likely due to the fact that the closed die compaction does not allow free movement and relocation of the straw, which implies that adding of water does not show any lubricating effect, whereas the water in wet straw acts as a lubricant in the more free movement appearing in simple upsetting lowering internal friction as well as friction towards the tools.

An important objective of the two material tests was to determine a mathematical model of the yield surface versus hydrostatic pressure and density as shown in Figure 11. The purpose of this was to establish a Finite Element Model the rolling process in order to predict the process window numerically. Preliminary attempts on this have been done, a fully useful model would require a significant longer research project, probably at least a 3 years PhD project, since the material is not only porous but also brittle and has very large elastic spring back.

5.11 Feeding experiments of straw under water

A challenge in the biogas sector is how to avoid build-up of floating layers in the biogas reactors at the top of the tank, as this is affecting the biogas reaction as well as the stirring mechanism. Experiments in the project with rolled straw, have showed that even though the rolled straw is initially compressed during the rolling process, the bulk density decreases again as the pressure is released when the straw exits the rollers. Apparently the straw is, after rolling, not damaged enough to avoid that it draws air into its microgeometries, and problems with floating layer development have not been avoided by rolling to a sufficient extent (se section 1.5.8). Therefore the possibility of feeding straw into the tanks in a way that would make it sink to the bottom was investigated during the project.

The idea of removing the air from the straw and let the straw expand under water was initially investigated in a simple hydraulic press setup. A small amount of straw was placed in a cylinder and compressed under water. Even though most of the air was chased, some air bubbles remained trapped in the straw structure and got compressed. When releasing the pressure, this air expanded again and caused the straw to come up to the surface. Pictures from this are shown in Figure 19 and Figure 20.

The results indicate that it is now enough just to compress straw to a density above water to make it sink in the biogas reactors. It is also necessary to remove air/gas trapped and compressed inside the straw.

Figure 19: *Testing in a simple hydraulic press setup*

Figure 20: *Straw after testing in a simple hydraulic press setup*

The principle was then tested with a screw plug feeder. It was observed that with enough compression applied, the straw was clearly sinking to the bottom of the tank. It is believed that the movement of the screw helps the air to leave the straw's structure.

A correlation between compression applied to the straw plug and sinking/floating was also observed.

Figure 21: *Testing by use of a screw plug feeder*

Following this successful test, it was decided to carry on another experiment where straw was fed into a liquid similar to the content of a biogas reactor. The purpose of the experiment was to assess the effect of the digestion process on the sinking properties of the straw and whether the production of biogas would lift the straw after some time. (the theory was that the bacteria would produce gas inside the micro channels of the straw, thus reducing the density and make it float)

Setup and procedure

A gas tight tank with glass walls was built for the experiment:

- Dimensions: 450 mm x 300 mm x 1000 m
- Plexiglas lid equipped with valves for filling water and inoculum and taking biogas out
- Discharge valve at the bottom for emptying
- Inlet on the side for mounting of the feeder

Figure 22: Picture of the gas tight tank

Before being mounted on the tank, the feeder was run aside in order to build up a tight straw plug. The tank was then filled with about 120L of water and 5L of inoculum before 500 grams of straw was fed into this mixture. 20L of liquid was then removed from the tank in order to dismount the feeder and close the inlet with a blind flange.

The inoculum was provided by Lund University which was also participating in the experiment.

Figure 23: *Picture of the container with inoculum*

The tank was placed in a chamber designed for the experiment:

- Chamber volume: 6 m^3
- Isolated with 220 mm thick polystyrene walls.
- Temperature measured by Pt1000 Danfoss ESM-11 and regulated by a temperature controller Danfoss ERC-211 at 37°C +/- 0.7°C
- Heat generated by a 1 kW electric fan heater placed inside the chamber
- Tank lit up by LED light on the back side and filmed by time-lapse camera Brinno BCC200 taking a picture every 30 seconds.

The setup is shown in the images below

Figure 24: *Pictures of the tank in the constructed chamber*

The production of biogas was measured by a mechanical gas-meter GMT BK-G4M placed outside the chamber and monitored daily.

Figure 25: Gas meter mounted on the pipe at from the digestion chamber

5.12 Results of the feeding experiments of straw under water

Biogas production

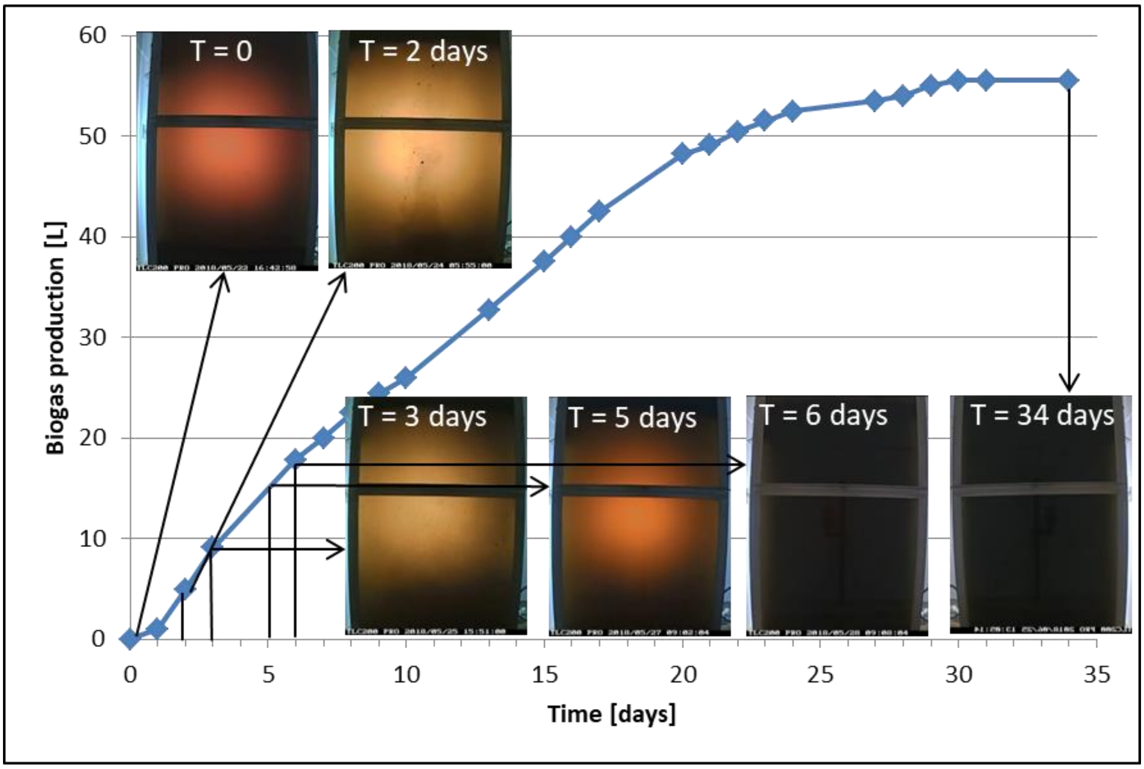


Figure 26 Biogas production during experiment with selected still frames from the time lapse movie [36]

The movie of the experiment showing the behaviour of the straw during biogas production can be seen on https://www.youtube.com/watch?v=J7zyrDmr6Pg [35]

Figure 27 Top of the chamber after 34 days. Floating un-digested straw is on the surface

Observations

- Straw started floating after 1 day
- Tank went completely black after 5 days and 12 hours
- Amount of biogas produced is 20-25% of theoretical value

Discussion of the results of the feeding experiments of straw under water

A floating layer started forming after about 24 hours of operation, matching with the beginning of the biogas production. Presence of gas bubbles under the floating layer of straw could be observed after 48 hours of operation.

There seems to be a correlation between floating of the straw and biogas production. It is assumed that the biogas trapped inside or under straw particles results in lifting of the straw to the surface of the reactor. Figure 28 Shows visible gas bubbles in/under the floating layer.

Figure 28 Gas bubbles under the floating layer after 2½ days

Visibility

After 5 days, the visibility through the tank decreased and it became difficult to observe changes or displacement of the straw. The blackening of the tank was relative fast, and changed significantly in 24 hours.

Biogas production

Theoretically, it is expected to be able to produce 200 to 250 L of biogas from 500 grams of straw, and maybe 10 L from the inoculum itself. However, the experiment shows that only 56 L of biogas were produced.

The lower production of biogas could be related to the floating layer and the presence of gas trapped under it. The floating straw could be difficult to access for the bacteria and it is uncertain whether the straw at the top of the layer has been digested throughout the experiment. The formation of a floating layer decreases the contact surface area between the bacteria or enzymes and the straw. In addition, the contact surface area needs to be wet. Any gas production that might occur inside the straw will therefore probably lead to a decrease in wet contact surface area which slows down the digestion process further. Finally, if the produced methane gas is not removed, it may slow down occurring chemical reactions via product inhibition.

Another reason for lower conversion could be the gas trapped under the floating layer. It could also participate in slowing down the biogas production process.

Conclusions of the Feeding experiments of straw under water

The purpose of the experiment was to analyse the behaviour of straw during biogas production. The straw was injected into the biogas tank by a screw plug feeder in a way that ensures the straw to be sinking to the bottom after feeding. It was observed that part of the straw came up to the surface and formed a floating layer after the biogas production process started.

The straw floating layer remained present throughout the experiment and could be a reason for lower biogas production. This experiment enlightens that the problematic of straw floating layers in biogas reactors is not only related to feeding but can also be affect the process later on, when biogas is produced and straw is lifted to the surface.
The tested screw plug feeder could be of interest for the biogas plants wanting to process straw as it solves the problem with floating layers when feeding straw into the reactor tanks.

Further experiments where a stirring mechanism is added to the experimental setup would be interesting to carry out and be more representative of biogas reactors. Stirring of the straw could potentially free the gas trapped in or between straw pieces.

5.13 Validation of the quality of the biomass after pre-treatment

The aim of pre-treating wheat straw is to improve the handling properties of the material, reduce the risk of floating layer formation in the anaerobic digester, increase the total methane yield and increase the production rate. To evaluate the roller, floating tests as well as biochemical methane potential tests (BMP) were performed on untreated and rolled material. Other mechanical pre-treatments were also included in the study to investigate differences. As the main mechanism of most mechanical pre-treatments is particle size reduction of the biomass, this parameter was studied further.

Experimental procedures

BMP method:
For measurement of the biochemical methane potential (BMP) value of mechanically pre-treated wheat straw, an Automatic Methane Potential Test System (AMPTS II, Bioprocess Engineering) was used, whereby data of the volumetric methane production was obtained. Digested residue from a full-scale digester was added to the straw to inoculate and start up the biogas tests. The tests were in most cases run at 37°C, until the gas production stagnated (appr. 35 days) whereby the test was stopped. After termination, the pH was measured to check for potential pH drop due to accumulation of inhibitory compounds. The pH showed to be around 7 – 7.8 for all samples and no inhibition was therefore suspected.

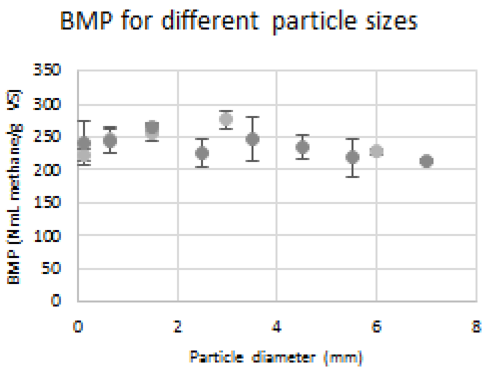
Figure 29: Equipment for BMP measurements

Floating tests:
Wheat straw was mechanically pre-treated by rolling, knife milling, pelletizing, dry extrusion and wet extrusion in order to compare the effect it would have on the floating capacity. The straw samples were added to 500 mL bottles with filled with water, thoroughly mixed and then left to soak for 24 hours before observation of floating layers.

Particle size determination:
Pre-treated wheat straw was fractioned into different particle sizes: 4-8 mm, 2-4 mm, 1-2 mm and 0.250-1 mm by sieving, see Figure 30.

Figure 30: Sieving equipment (left) and resulting fractions of straw (right).

Evaluation of size fractioned wheat straw
BMP tests were performed on a series of different straw particle sizes to assess if that parameter would have any effect on the cumulative methane production. The results from repeated BMP tests showed that no such correlation could be observed, see Figure 31.



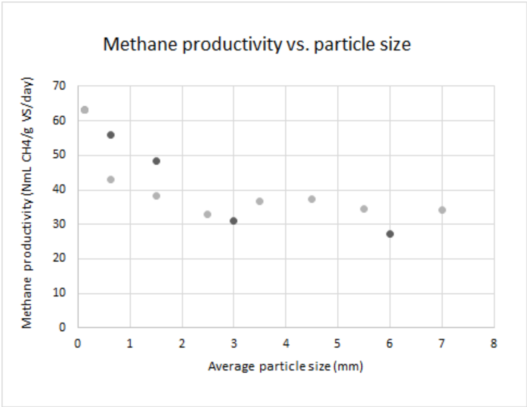


Figure 31: *Cumulative methane production (left) and methane productivity (right) for size fractioned wheat straw.*

The theoretical BMP maximum for the wheat straw was calculated based on the cellulose and hemicellulose content (35% and 26% of dry straw, respectively) to ca. 270 NmL CH_4/g VS. However, this value is underestimated as eventual proteins and other extractives will most probably contribute to the methane production. The particle size fractions generated BMP values around 230 NmL/g VS (85 % of theoretical maximum).

That the methane production rate increases with a decreased particle size is in agreement with literature and is often explained by the increased surface area of the material that occurs via size reduction. Although the results between different test runs vary, similar trends were noticed in 3 repeated set-ups. For particles with an average diameter of 3 mm or above, the methane production rate did not differ. This is most likely due to that those particles consisted of smaller pieces of straw that had formed larger lumps upon compression during the rolling process. When dissolved in water, the actual particle size of the straw fragments in those lumps were below 4 mm.

These results show that the decrease in particle size of wheat straw will have a positive effect on the methane production rate which in turn can be translated to shorter hydraulic retention times in the digester which allows smaller reactor volumes for same production rate.

Evaluation of mechanically pre-treated wheat straw

Wheat straw was mechanically pre-treated by rolling, knife milling, pelletizing, dry extrusion and wet extrusion in order to compare the effect the pre-treatment would have on the floating capacity and the BMP test. Since the biggest issue with digestion of straw is its low bulk density which causes accumulating floating layers that builds up over time. The contact surface area between the biomass and the anaerobic bacteria becomes very limited which results in a prolonged hydraulic retention time. Other studies therefore suggest that the organic loading rate should be set according to the rate of the floating layer formation [34].

Evaluation of floating properties

The results from the floating test showed that the pelletized straw immediately sank to the bottom of the flask and showed minimal floating layer after 24 hours. Both knife milling and extrusion (both dry and wet) resulted in significant initial floating layers, but the majority of the straw had sunk to the bottom of the flask at the end of the test. If comparing the formed floating layers of the pre-treated straw to that of the untreated straw, it is very visible that the total volume of both the initial and final floating layers have been majorly reduced. However, in the case of rolled straw, the difference is not as big.

Figure 32: *Left picture: pre-treated wheat straw, Right picture: the floating layers after 0 and 24 hours for untreated, rolled, knife milled, dry extruded, wet extruded and pelletized wheat straw.*

It is no surprise that the straw pellets did not float as they have a very high density compared to the other samples. Although the rolled straw is initially compressed during the rolling process, the bulk density decreases again as the pressure is released when the straw exits the rollers. In addition, particle size reduction was not achieved to the same degree as for extrusion and knife milling which might explain the poor sinking qualities and less dense characteristics of the floating layer. The wet extruded straw did not sink as well as the dry extruded straw. During wet extrusion, the temperature in the extruder does probably not reach as high values as during dry extrusion since there is water present to capture excess heat from breakage of the fibers. This is hard to evaluate since it was not possible to measure the temperature inside the extruder. In this study, there was limited difference in particle size between the two extruded straw samples.

For a more qualitative analysis of the floating layer formation tendency of wheat straw, the substrate should preferably be studied during BMP tests. However, since the inoculum has such a dark color, it is very difficult to distinguish between floating sludge and floating straw which therefore complicates the formulation of a proper floating test method. Additionally, there might be a risk that straw which initially sinks to the bottom of the reactor, starts rising to the surface when biogas starts to be produced inside of the straw particles. However, only studies on sludge could be found on this topic.

Evaluation of BMP

Based on the results from the BMP tests run on size fractioned straw, the floating tests and what degree of size reduction that was achieved, the pre-treatments were ranked according to the expected methane production rate (high to low); *Dry extruded, Wet extruded, Knife milled, Pelletized, Rolled and Untreated straw.*

The results from the BMP test are shown in Table 3 below, after 35 days of digestion. The BMP rate is defined as the maximum achieved methane production rate which occurred after 3-4 days for all samples. As the straw samples with the highest BMP values did not necessarily generate the highest rate, the time *T80* was calculated and shows the time when the methane yield is 80% of its maximum (final) BMP value. An alternative factor to compare could be the yield at a certain hydraulic retention time. The increase of the BMP and the BMP rate was calculated based on the results of the pretreated samples compared to the untreated sample.

Table 3: *The cumulative methane yield and production rate after 35 days of digestion.*

	BMP [NmL CH_4/g VS]	**Increase**[a]	**Methane production rate** [NmL CH_4/g VS/day]	**Increase**[a]	**T80**[b] [days]
Untreated	237	-	26	-	12
Rolling	287	21%	28	7.7%	14
Knife milling	269	14%	31	19%	12
Pelletizing	239	0.8%	38	46%	7
Dry extrusion	237	0%	41	58%	6
Wet extrusion	246	3.8%	39	50%	7

[a]The increase was calculated with the results for the untreated straw as a reference.
[b]T80 refers to the time it takes for the methane yield to reach a value of 80% of the maximum BMP.

According to the results, the rolling pre-treatment showed the highest increase in BMP value of 21% compared to the untreated straw. Despite the yield increase, the methane production rate was not improved much by this pre-treatment. Dry extrusion showed no increase in maximum achieved BMP value, but the production rate was improved with 58%. It is unclear why the rolling and the knife milling pretreatments led to an increased methane yield. A possible reason could be that there is more heat development during the pelletization and extrusion which might lead to chemical reactions that converts degradable compounds to non-degradable. These compounds might counteract the positive effects that the pretreatments have on the decreased recalcitrance of the straw. However, no chemical characterization was performed to confirm the theory.

Surprisingly, knife milling did not improve the methane production rate nearly as much as the pelletizing. It was believed that the smaller particle size of the knife milled straw would improve its digestion rate to a higher degree than the pelletized straw. These results suggest that not only the particle size is determinative for the methane production rate. Since the knife milled straw had a higher floating capacity, it might have prolonged the time for the digesting bacteria to reach the substrate. Another reason could be that the pelletizing process is a more severe pre-treatment than knife milling and could therefore lead to a reduced recalcitrance of the straw by the initial melting and subsequent recondensation of lignin in the form of droplets onto the external surface of the material. This relocation of the lignin has previously been shown to increase the porosity of lignocellulosic biomass, making the digestible substrate more available to hydrolytic enzymes and bacteria.

The time *T80* showed that the digestion time for the untreated, rolled and knife milled straw was almost twice as long as for the other samples. This suggests that the stagnation in methane production happens faster for the latter straw samples. A quicker stagnation would imply that the hydraulic retention time can be shortened. However, methanogens need a hydraulic retention time of minimum 10-15 days due to their slow growth rate which will set a limit to the minimal reactor volume. Faster anaerobic hydrolysis could however permit a higher organic loading rate. Other studies have shown indications of that a too high size reduction (below 200 µm) can lead to a too fast hydrolysis which may cause accumulation of volatile fatty acids and thus decrease the pH in the reactor, thereby inhibiting the methanogenesis [35].

Summary of the validation

The results show that the decrease in particle size of wheat straw from pre-treatment will have a positive effect on the methane production rate which in turn can be translated to shorter hydraulic retention times in the digester which allows smaller reactor volumes. The rolling pre-treatment showed the highest increase in BMP value, 21% higher compared to the untreated straw. Despite the yield increase, the methane production rate was not improved much by this pre-treatment, however.

When comparing the formed floating layers of the pre-treated straw to that of the untreated straw, it was clearly visible that the total volume of both the initial and final floating layers were reduced by pre-treatment.

Although the rolled straw is initially compressed during the rolling process, the bulk density decreases again as the pressure is released when the straw exits the rollers. Experiments in the project with rolled straw, have showed that even though the rolled straw is initially compressed during the rolling process, the bulk density decreases again as the pressure is released when the straw exits the rollers. Apparently the straw is, after rolling, not damaged enough to avoid that it draws air into its microgeometries, and the thereby associated problems with floating layer development have not been avoided by rolling to a sufficient extent. Particle size reduction by rolling, was not achieved to the same degree as for extrusion and knife milling which might explain the poor sinking qualities and less dense characteristics of the floating layer. As mentioned in section 1.5.7 pressing straw directly in liquid reduces the problem, but in this situation there is still too much air in the straw. As also mentioned in section 1.5.7 the same principle has been tested with a screw plug feeder. It was observed that with enough compression applied, the straw sinks to the bottom of the tank. It is believed that the movement and squeezing from the screw helps the air to leave the straw's micro structure.

5.14 Business plan

The activities in the project concerning the business plan have focussed on the possibilities for implementing the developed technology in the biomass industry [37]. Based on our findings there is a growing market for development of technology for processing and handling of straw and other cellulosic residues such as grass and bagasse. Straw and other soft biomasses is an abundant biomass resource with a high potential. [38, 39]

There is a strong political focus to use these biomass resources because they are available anyway, and it is a source of extra income the farmers. Biomass is needed in the energy sector in order to balance energy production from solar and wind power [40]. Thus, there is a need to develop methods for cheap processing and handling of straw in biogas and other energy plants. Although it looks promising from a political point of view, the commercial dimension must not be neglected. The large decreases in oil prices over the last 2-4 years have increased the requirements for green support for biomass based technologies. Such green support based on political decisions often makes industry more reluctant to invest. In BioYield this has been shown by calculating the surplus from production of biogas from straw with and without green subsidies [37]. Here it is clear that the surplus is a deficit if there is no green support as the prices and market currently are. However, this is without taking in the secondary effects of biogas production into consideration.

Political decided businesses can be very unstable. As a consequence of this investors normally require short depreciation periods in order to minimise the consequences of changing political decisions. The price of the feeding systems therefore needs to be low for short time depreciation.

The current technology has a good societal and environmental potential. As it offers the possibility to increase the amount of biogas produced at biogas plants by feeding low cost cellulosic based plants into biogas reactors. It was never the intention of the BioYield project to put a finished physical pre-treatment product on the market, and as described earlier the ongoing technological achievements in the project and changed economic conditions in the industry have had the result that some of the prerequisites for improving the yield and operating economy of biogas production by 25% compared to current techniques are no longer applicable. As described in section 5.15 several other achievements has been met during the project.

During the project TK Energy has been in contact with by several companies to discuss feeding mechanisms of biomaterial into different reactors. None of these has, however, turned into directs orders yet. It is however the goal that one of these contacts will lead to investment in a pilot plant, where additional experience can be gained on construction of the developed feeding mechanisms. Some of the activities of the BioYield project is already continued in other projects (e.g. The EUDP project Segrabio), which is improving the possibilities of commercialize technology developed.

5.15 Dissemination of the results

A M.Sc. project has been completed and presented at Lund University, and a phd-study is currently an on-going activity. An article in the magazine 'Forskning for Bioenergi' have been published. Several workshops among the project partners have been held, at the laboratories / test facilities of the partners. Several meetings with stakeholders, e.g.: with representatives of Danish straw suppliers and biogas plants (including 'brancheforeningen for Biogas') are held. Work developed in the project has been presented at the following conferences:

- 25th European Biomass Conference and Exhibition (EUBCE) in Stockholm,
- the Conference on Biotechnology for fuels and chemicals in San Francisco,
- the 15th IWA World Conference on Anaerobic Digestion, the EUBCE conference in Copenhagen May 2018,
- an abstract is send to IWA World Water Conference in Tokyo, 2018.

6. Utilization of project results

There is an increasing market for processing and handling of straw and other cellulosic residues such as grass and bagasse and there is a strong political focus to use these biomass resources because they are available anyway, and it is a source of extra income the farmers.

The project has improved and collected state-of the art knowledge in literature studies and evaluations of these. These findings has been made available in [1,2,14,27].

BioYield has documented problems with the methods used today for measuring biogas potential, and productions rates, thus it is difficult to compare and trade with resources for biogas production, as the value of the waste products becomes unclear, and can be evaluated different at different plants, resulting in different payment to the suppliers.

The project has successfully developed a pilot system for rolling of straw with a capacity of 100-2000 kg / hour. Based on a combined experimental and theoretical study the feasible process window for pre-treatment of wheat straw by roll pressing varying the feed, the roll gap, the roll speed and the moisture content of the bulk straw is presented.

An objective of the mechanical material tests was to determine a mathematical model of the yield surface versus hydrostatic pressure and density. The purpose of this was to establish a Finite Element Model of the rolling process in order to predict the process window numerically. Preliminary attempts on this have been done, a fully useful model would require a significant longer research project, probably at least a 3 years PhD project, since the material is not only porous, but also brittle and has very large elastic spring back – all properties, which make it difficult to model.

Further BioYield has shown practical solutions to problems of adding straw into biogas reactors.

- Possibilities of how to avoid floating layers have been shown
- Possibilities of how to increase digestion rate of straw in biogas reactors has been shown.
- The project has improved knowledge on how to perform calibration and comparison between different measurement methods for biogas potential. Hereby it becomes possible to compare different plants, and to have common references when discussing performance of different technology.
- BioYield has improved the knowledge on possibilities on how to lower the costs of production of biogas, as it offers the possibility to increase the production by adding straw into biogas reactors, and also the production rate, leading to a larger yield from the same plant.

BioYield has improved knowledge on how to increase supply safety or the independence of fossil fuels as it offers the possibility to store VE-energy (biogas upgraded to natural gas) in the existing gas grid, thus support the more fluctuating energy sources like solar and wind, and in overall contributing to making the energy sector more green.

BioYield contributes to the possibilities of increasing an existing and limited marked within the biogas plants. The production of biogas is already a good idea from an environmental point, as it takes already existing resources and use these for energy production in an existing energy system instead of these resources being either burned (e.g. food waste) with a low yield or spread across fields (e.g. manure and slurry) with the hereby direct emission of methane to the atmosphere without capturing of the potential energy. Further methane released to the atmosphere has a high CO_2-equivalent when relating to greenhouse effect.

It was not the intention of the BioYield project to put a finished physical pre-treatment product on the market, whereby the direct growth within project partners is limited. Several companies within the VE industry have shown interest in the techniques and experience developed.

The results have been disseminated as described in section 5.15.

7. Project conclusion and perspective

The project has successfully developed a pilot system for rolling of straw with a capacity of 100-2000 kg / hour. Based on a combined experimental and theoretical study the feasible process window for pre-treatment of wheat straw by roll pressing varying the feed, the roll gap, the roll speed and the moisture content of the bulk straw is presented.

Work on validation of quality of biomass after pre-treatment has been carried out. A challenge associated with evaluating biogas production is that the process is slow and the experiments then become time consuming. Biochemical methane potential measurement (BMP) is usually performed for 30-90 days in order to deplete the material of any digestible components. In the project development of a less time consuming enzymatic hydrolysis (EH) method has been performed. Also evaluations of the biomass tendency to result in floating layers have been evaluated.

Correlation between the glucose yield obtained by enzymatic hydrolysis tests and the methane production rate obtained by biochemical methane potential test was seen for mechanically pre-treated wheat straw. This shows that enzymatic hydrolysis could be an alternative faster evaluation method to the BMP method for this kind of substrate.

When comparing the formed floating layers of the pre-treated straw to that of the untreated straw, it was clearly visible that the total volume of both the initial and final floating layers was reduced by the pre-treatment.

The results show that the decrease in particle size of wheat straw from pre-treatment will have a positive effect on the methane production rate which in turn can be translated to shorter hydraulic retention times in the digester which allows smaller reactor volumes. The rolling pre-treatment showed the highest increase in BMP value, 21% higher compared to the untreated straw. Despite the yield increase, the methane production rate was not improved much by this pre-treatment,.

The ongoing technological achievements in the project and changed economic conditions in the industry have had the result that some of the prerequisites for improving the yield and operating economy of biogas production by 25% compared to current techniques are no longer applicable. Among other things, thorough studies have shown that, it was not possible to reproduce some of the results presented in literature considered as state-of-the-art at project start.

One of the achievements in the project has to do with challenges of floating layer development of rolled straw (which is also valid for any other state-of-the-art feeding techniques of straw into biogas-reactors). Although the rolled straw is initially compressed during the rolling process, the bulk density decreases again as the pressure is released when the straw exits the rollers. Experiments in the project with rolled straw, have showed that even though the rolled straw is initially compressed during the rolling process, the bulk density decreases again as the pressure is released when the straw exits the rollers. The straw is apparently not damaged enough after rolling to avoid that the straw draws air into its microgeometries, and problems with floating layer development have not been avoided by rolling to a sufficient extent. Particle size reduction was not achieved to the same degree as for extrusion and knife milling which might explain the poor sinking qualities and less dense characteristics of the floating layer. Pressing straw directly in liquid reduces the problem, but in this situation there is still too much air in the straw. The same principle has been tested with a screw plug feeder. It was observed that with enough compression applied, the straw sinks to the bottom of the tank. It is believed that the movement and squeezing from the screw feeder helps the air to leave the straw's structure.

There is an increasing market for processing and handling of straw and other cellulosic residues such as grass and bagasse and there is a strong political focus to use these biomass resources because they are available anyway, and it is a source of extra income the farmers.

The decreased oil price over the last 2-4 years has increased the demands for subsidiaries for green biomass-based technologies. Such subsidiaries are based on political decisions, which often make the industry more reluctant to invest. The currently technology has a good societal and environmental potential, as it offers the possibility to increase the amount of biogas produced at biogas plants by feeding low cost cellulosic based plants into biogas reactors. Due to the current structure of the market, there is a low private economic investment and business potential.

8. Annex

Relevant links:

Papers published at EUBCE conference can be accessed here:
http://www.etaflorence.it/proceedings/ (Full open access after free registration)

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