PRELIMINARY INVESTIGATION OF PHYSICAL, CHEMICAL AND TECHNOLOGICAL PARAMETERS OF BIOGASIFICATION AND BRIQUETTING OF FRACTIONATED SILAGE

Andres Menind, Liis Oper, Liina Nurk
Estonian University of Life Sciences
andres.menind@emu.ee, liis.oper@emu.ee, liina.nurk@emu.ee

Abstract. The duration of the cycle of fermentation of pressed liquids may be several times shorter than the duration of the cycle of ordinary fermentation process which uses silage as regular substrate for biogas reactors. Also the fermentation of pressed liquids has approximately 1.5 times higher biogas yield per dry-matter (DM). Therefore, the volume of reactor decreases as well as the total cost of establishment. Dewatering the silage and drying the press cake are additional operations. Drying is a good possibility for utilizing the remaining heat from CHP. The quality and chemical composition of such silage press cake briquettes substantially approves due to their approximately twice lower content of ash and harmful elements. These briquettes have high volume weight (about 940 $kg \cdot m^{-3}$) and high heating value. Experiments of biogasification process and yields both for press liquid and press cake were conducted.

Keywords: briquetting, dewatering, press cake, press liquids, silage, biogas.

Introduction

By the renewable energy act, Estonia has a strategic plan to increase the renewable energy share up to 25 % of final consumption; in 2005 this figure was 18 % [1]. One possible source for additional renewable energy supply is hay which is traditional fodder for animals in Estonia, but today as the usage of hay in agriculture is decreased and therefore it could be used in local energy systems.

There are around 200 thousand hectares of permanent grasslands (grassland occupation over 5 years) in agricultural production in Estonia. The active sown area had changes of 11 % over the years 2006 – 2008 and production of green fodder in tons was from 1.5 to 1.9 Mt in a year [2]. About 283,000 ha of agricultural land are abandoned and 123,000 ha are no longer in agricultural registers. Compared to activities of animal husbandry in regions we may assume that 40 – 50 % of grasslands are not used for fodder production, but have been cut for land maintenance once a year [3]. Biomass production in meadows is in range from 1.7 to 5.7 $t \cdot ha^{-1}$ per year. The total production from semi natural meadows is approximately 182,000 t per year of dry matter [4].

It is difficult to say what is the energy potential of unused land in Estonia. There is not enough information about the current state of abandoned fields and cultivation plans for future of Estonian farmers is not well known. By rough estimation the potential for bioenergy from natural grasslands, unused fodder from grasslands, and abandoned agricultural land is 6.66, 2.3 and 6.93 PJ respectively, total potential therefore being about 16 PJ annually [5]. Kask has estimated that renewable energy potential of biogas production based on biomass from abandoned agricultural land and came out with 5 PJ in a year [6].

The development of the integrated generation of solid fuel and biogas from biomass (IFBB) is aimed at increasing the efficiency of converting biomass into energy [7]. Biomass, e.g., from semi-natural grasslands which is difficult to exploit in conventional bioenergy-converting systems, as the chemical composition is detrimental both for conventional anaerobic digestion as a result of high fibre concentrations [8] and for direct combustion because of high concentrations of elements that cause corrosion inside the combustion chamber, ash softening and hazardous emissions [9].

Due to the availability of resources but problematic aspects using such materials IFBB technology might be a promising solution for Estonia. In this study methods and results for samples from meadows which are under environmental protection and compared to the silages produced as cattle feed implementing IFBB technology are described. Energy input, output and other lucrative changes compared to classical silage digestion or biomass briquetting are under focus as well.

Materials and methods

Four different silage samples, chosen from various areas, different in nutrition value, fibre and content of organic matter, were used for the study. Two samples of silage of biomass grown on nature preserve, embarrassed with commitment to make one late harvest once a year in July and two typical samples grown as cattle feed of Estonia were investigated. Dewatering screw press Vincent CP4 was used to separate the silage into two fractions: liquid fraction for biogasification and solid part for biogasification and for briquetting. Biomethane potential tests (BMP) were made using plasma bottles with a volume of 550 ml. Incubated (for 48 hours on 36 ºC) and sifted (sieve 1 mm) inoculum (150 ml) were used 3 times for each test material (0.3 g TS (Total Solids) per bottle)) and for the blank. Batch raw data was measured during a 68 day period. Silage on this study had no thermo-chemical pre-treatment but grinding using equipment RS06 with side screen 45 mm. Briquetting (briquetting press Weima C150) and energy consumption measurement for possessing were performed in the Biofuel Laboratory.

In the Laboratory of Plant Biochemistry, the press fluid and press cake were analyzed for Ca, P, Mg and K by using the following methods: Determination of Phosphorus in Kjeldahl Digest by Fiastar 5000. AN 5242. Stannous Chloride method, ISO/FDIS 15681; Determination of Calcium in Kjeldahl Digest by Fiastar 5000. AN 5260. o-Cresolphthalein Complexone method ISO 3696; Determination of Magnesium by Fiastar 5000. ASTN90/92. Titan Yellow method; Potassium and/or Sodium in Plants (Flame Photometric Method). (956.01). Official Methods of Analysis. 1990. Association of Official Analytical Chemists. 15th Edition. (AOAC). Other organic constituents: crude protein (CP), neutral detergent fiber (NDF), acid detergent fiber (ADF) and total dry matter (DM) where used following methods: Protein (Crude) Determination in Animal Feed: Copper Catalyst Kjeldahl Method. (984.13) Official Methods of Analysis. 1990. Association of Official Analytical Chemists. 15th Edition. (AOAC); The Determination of Neutral Detergent Fibre in Feed. Tecator ASN 3434. (Foss Tecator Fibertec 1020); The Determination of Acid Detergent Fibre in Feed. Tecator ASN 3436. (Foss Tecator Fibertec 1020) and Total Dry Matter by Oven Drying for 2 hr at 135 ºC. (920.15) Official Methods of Analysis. 1990. AOAC 15th Edition. Ash behaviour characteristics were determined by the standard CEN/TS 15370-1. Ash content was measured at 550 ºC and the calorific value of samples using equipment IKA C 5000 ISO 1928.

Results and discussion

1. *Laboratory analyses*

The ash quantity in press cake (PC) and ash behavior characteristics are in relation of distribution of elements between fractions. Potassium (K) has the biggest (88.6) massflow in percent of the described elements to press fluid (PF) Fig.1. The DM content in the silage varied from 19.8 – 30.5 %. Agro and Lauri silos have similar organic constituents and content of elements as Palupõhja and Siimusti respectively. Notable is that Siimusti cake content of K (0.066 %) is lower than for other samples. Relative importance 46.8 – 63.6 % by mass for PF is a good result for separation without thermo- chemical pretreatment. The laboratory results for organic constituents and elements are presented in Table 1 and Table 3.

Table 1

Content of chemical elements of various silage and press cake samples

Material	Ca %	P %	Mg %	K %	DM %	% of cake
1. Agro cake	1.099	0.187	0.36	0.278	47.05	36.4
2. Agro silage	0.98	0.311	0.265	1.448	19.75	–
3. Palupõhja cake	1.007	0.144	0.306	0.253	49.94	53.2
4. Palupõhja silage	1.061	0.181	0.264	0.599	30.48	–
5. Lauri cake	0.953	0.216	0.296	0.695	42.91	36.4
6. Lauri silage	0.878	0.264	0.215	1.835	21.25	–
7. Siimusti cake	0.993	0.122	0.3	0.066	49.34	45.2
8. Siimusti silage	1.022	0.2	0.256	1.277	22.7	–

The elemental distribution between the fractions is presented in Fig 1. On the average about 65 % of the elements: P, K, Mg and Ca are pressed out to PF and are possible to return back to the fields. The transfer of potassium, magnesium and phosphorus to the liquid phase ranged from 0.52 to 0.85 of the amount in fresh matter, calcium transformation was lower, at the transfer ratio 0.44 – 0.48 [7]. The test results about transfer of K and P were even higher 57.7 – 97.7 % and for magnesium were measured 38.4 – 50.6 % which are lower. Ca transfer to liquid phase on average 56.3 % is higher compared to the above mentioned author results.

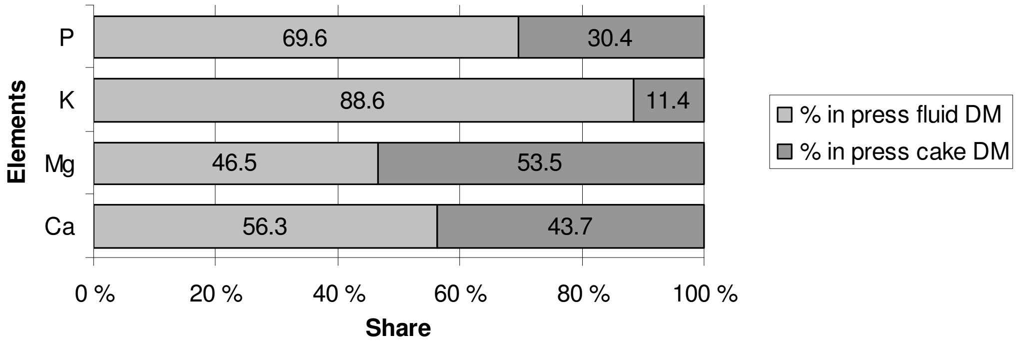


Fig. 1. **Deviation of elements in press cake and in press fluid**

2. *Ash content and ash behaviour characteristics*

The chemical composition data are also used for investigation of the ash behaviour characteristics. The chemical elements on which the ash melting characteristics mostly depend (for example, Ca, Mg, K) are decreased in press cake. The total content of those elements in row silage is possible to calculate summarizing the contents in the fractions ($g \cdot kg^{-1}$). Based on the concentrations of K, Ca and Mg ($g \cdot kg^{-1}$ DM), the ash softening temperature (AST), an important indicator to describe the ash melting behaviour during combustion, was calculated according to equation 1.1 [10].

$$AST = 1172 - 5.39 \times K + 25.27 \times Ca - 78.84 \times Mg, \text{°C}. \quad (1)$$

AST for average cake was 1171 ºC. The calculated temperatures of AST for PC approaches to the level of the corresponding value of common firewood. The laboratory test results for ash behaviour characteristics of the briquettes obtained on the described methods on the average are presented in Tab. 2. On comparison of the laboratory test results and AST received on using calculation according to [10] appears that theoretically softening temperature is higher.

Table 2

Average ash behaviour parameters by laboratory test results

Ash behavior characteristics	Temperature	Standard
Softening	1080	CEN/TS 15370-1
Hemisphere	1130	CEN/TS 15370-1
Flowpoint	1150	CEN/TS 15370-1

The ash content of press cakes varied from 4.87 (Lauri) to 8.56 % (Palupõhja). The average ash content of cake samples was 6.44 %. The briquettes made from tried and proportionally mixed cake gave the ash content of 6.59 % as a test result, which corresponds practically to the average. The ash content of silage was 7.8 – 9.8 % (Fig. 2).

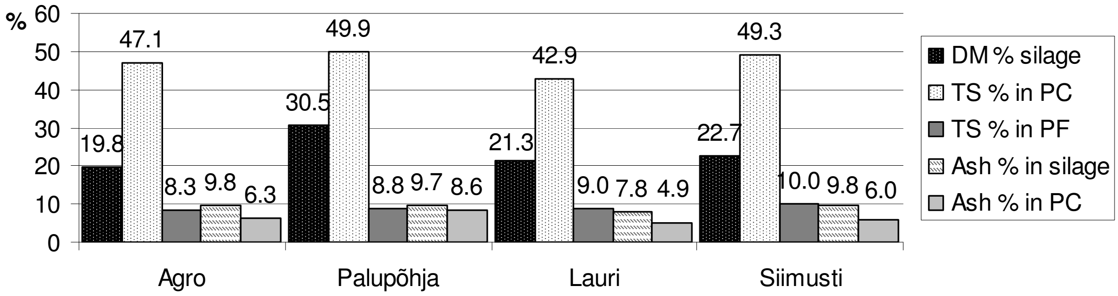


Fig. 2. **Dry-matter and ash contents in silage and press cake:**
TS – total solids; PC – press cake; PF – press fluid

The harvest time, soil type and species divergence are supposed as reasons for differences of the ash content of both silage and press cake. The samples with higher moisture content (Lauri, Siimusti and Agro) gave better results to reduce the ash content in the press cake briquettes. The firewood ash content is roughly 1 %, in spite of pressing out liquids from silage and decreasing the mineral content in press cake, it is still hard to reach the firewood ash content level.

3. *Biogasification tests*

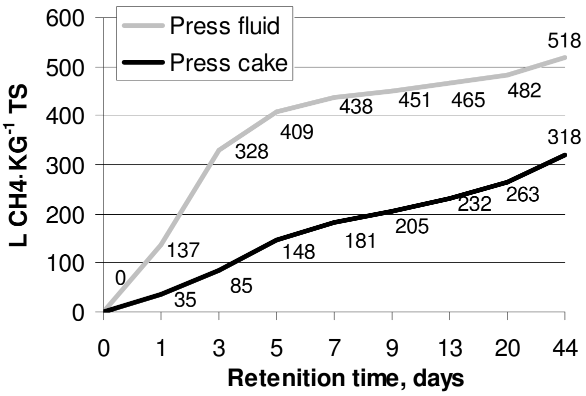
Biomethanization tests (methane yield and process parameters) are based on the plant chemical composition data Table 1 and Table 3. The DM content of silage varied 19.8…30.5 % and the total solid (TS) content of PF 8.3 – 10.0 %. Proportionately, 0.09 – 0.23 of the DM contained in the silage was transferred into the press fluid during mechanical dehydration without hydrothermal conditioning [11]. As a result of dewatering there was obtained a press cake DM content of 42.9…49.9 % which corresponds to [10]. As the result of dewatering, most of the organic constituents, like NDF, ADF and CP contents in press cake increase.

Table 3

Laboratory analyses for organic constituents

Sample	pH	DM, %	DM, % (lab.)	CP, %	NDF, %	ADF, %	DDM, %	ME, $MJ \cdot kg^{-1}$
Agro cake	4.41	47.05	96.97	9.9	66.06	45.34	53.58	8.105
Agro silage	4.82	19.75	94.14	16.64	54.01	36.95	60.12	9.324
Palupõhja cake	4.73	49.94	96.67	8.55	67.56	45.69	53.31	8.055
Palupõhja silage	4.77	30.48	95.92	10.16	65.81	43.6	54.94	8.359
Lauri cake	4.52	42.91	96.49	9.56	63.94	42.34	55.92	8.541
Lauri silage	4.52	21.25	92.63	13.23	51.05	33.19	63.04	9.87
Siimauti cake	4.54	49.34	96.82	7.13	70.93	47.23	52.11	7.831
Siimusti silage	4.29	22.7	92.4	9.09	56.78	36.97	60.1	9.321

Summarized methane yields are presented in Fig 3. The press fluid methanization process results present higher process activity and total yield of methane (l $CH4 \cdot kg^{-1}$ TS). 80 % of total methane potential of substrates was measured during a period 68 days, it is achievable by about 7, 28 and 25 days for press fluid, press cake and silage respectively. The results show that the retention time is possible to be shortened 3.5 – 4 times in order to receive optimal methane yield using press fluid for digestion. A higher rate of hybridization and absence of recalcitrant organic constituents like lignin are the supposed reasons which give advantages to the press fluid digestion.



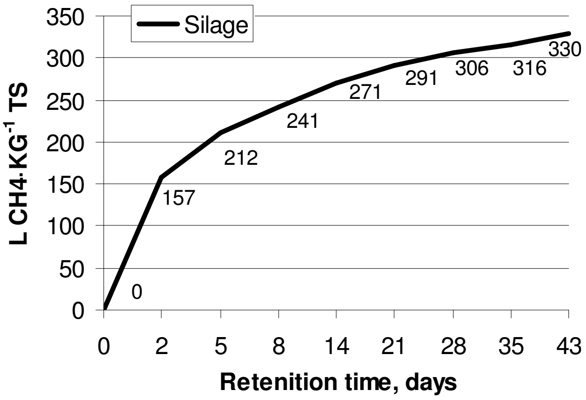


Fig. 3. **Summarized methane yield and process speed of press cake, press fluid and silage**

A quick and efficient process of press fluid makes it possible to reduce the capacity of digesters. The total solid content of the press fluids 83…100 $g \cdot kg^{-1}$ TS enables to produce about 42 m^3 CH4 per ton of press fluid (80 % of potential). According to [11], 0.27 – 0.62 of the DM was transferred when a hydrothermal conditioning (10 – 90 ºC) was applied beforehand. So it makes possible to produce 61 m^3 CH4 per ton of average silage discribed in this paper and with the energy content 622 kWh respectively. Biogas production in case of whole silage digestion (WSD) is in a range of 63 – 71 m^3 CH4 [12]. The whole possible biogas production per ha (2100 kgTS·ha^{-1}) is in case of PF digestion (0.5 of the DM was transferred) 438 m^3 CH4 and WSD about 561 – 632 m^3 CH4 according to [12]. The corresponding value, based on the biomethanization tests of silage Fig. 3, for one hectare (2100 kgTS·ha^{-1}) is 611 m^3 CH4 per hectare.

4. Briquetting

Briquettes produced from silage PC have a high volume weight (938 kg·m^{-3}). The briquette bulk weight is directly influenced by the volume weight of the briquettes and makes about 63 % of it according to the Estonian standard EVS-EN 15103:2010. The bulk weight of the press cake briquettes was about 591 kg·m^{-3}. The volume of the briquettes was 90 cm^3 which is lower than it was received on the briquetting tests of common herbaceous materials. The calorific value is 5.41 MWh·DM·t^{-1} which is slightly higher than common briquettes made from herbaceous materials due to lower mineral content. In Table 4, there are calorific values of the press cake briquette and some other briquette samples presented.

Table 4

Calorific value of press cake briquette compared to other samples

Sample	Moisture, %	Calorific value Q kJ·g^{-1}	Calorific value DM Q kJ·g^{-1}	Ash, %
Meadows	10.61	17.455	19.527	7.2
Rye straw	8.7	17.207	18.85	4.8
Silage cake	8.87	17.72	19.446	6.44
Reed canary grass	8.52	17.343	18.96	5.5
Reed (winter harvested)	11.53	16.864	19.061	–

Conclusions

The reutilization of abandoned land and the potential of unused biomass can be used in the production of energy and heat with the aim of producing bioenergy. Press fluid as biogasification substrate makes it possible to operate in short retention times and higher methane yields. The volume weight of loose hay is low (about 20 – 50 kg·m^{-3}) but as a result of briquetting technology application, the density of the material increases up to 40 times. There is not a big difference in the calorific value (MWh·DM·t^{-1}) comparing herbaceous (PC) briquettes with firewood or wood briquettes. On the average, about 65 % of elements: P, K, Mg and Ca are pressed out to PF and are possible to return back to the fields. The AST for average cake was 1171 ºC. The calculated temperatures of AST for PC approaches to the level of the corresponding value of common firewood. Integrated generation of solid fuel and biogas from biomass enables usage of CHP heat for pre-treatment and thermal drying year around. IFBB has about 60 % higher energy production. A more complicated technological chain using IFBB is partly reduced by simplified biogas plant requirements and a decreasing need for capacity.

Acknowledgements

This project was supported by the Estonian Environmental Investment Centre Project No. 47.

References

1. Directive 2009/28/EC of the European Parliament and of the Council of 23 April 2009 on the promotion of the use of energy from renewable sources
2. Agriculture in figures 2009. 2009. Statistics Estonia.
3. Roostalu, H., Astover, A., Kukk, L. & Suuster, E. 2008. Bioenergy production possibilies in agriculture I osa. Maamajandus. 10, 32–35 (in Estonian).

4. Melts, I., Heinsoo, K., Sammul, M. & Pärn, L. 2008. Energetic potential of semi-natural meadows. Vollmer, E. & Normak, A. (eds.): Investigation and usage of renewable energy sources tenth conference proceedings: Ecoprint, Tartu, pp. 88–96 (in Estonian).
5. Roostalu, H., Astover, A., Kukk, L. & Suuster, E. 2008. Bioenergy production possibilies in agriculture: III osa. Maamajandus. 12, 34–36 (in Estonian).
6. Kask Ü. 2008. Potential of biogas production. Eesti Põllumees, 17, 14–16 (in Estonian).
7. Wachendorf M., Richter F., Fricke T., Grass R., Neff R. (2009): Utilization of semi-natural grassland through integrated generation of solid fuel and biogas from biomass. Grass and Forage Science, 64, 132-143.
8. Prochnow, A., Heiermann, M., Plochl, M., Linke, B., Idler, C., Amon, T., Hobbs, P.J., 2009. Bioenergy from permanent grassland – a review: 1. Biogas. Bioresour.Technol. 100, 4931–4944.
9. Obernberger, I., Brunner, T., Barnthaler, G., 2006. Chemical properties of solid biofuels – significance and impact. Biomass Bioenergy 30, 973–982.
10. Hartmann, H., 2001. Brennstoffzusammensetzung und -eigenschaften (Composition and characteristics of fuels). In: Kaltschmitt, M., Hartmann, H. (Eds.), Energie aus Biomasse: Grundlagen, Techniken und Verfahren. Springer-Verlag, Berlin, pp. 248–272.
11. Richter, F., Fricke, T., Wachendorf, M. (2011) Influence of sward maturity and pre-conditioning temperature on the energy production from grass silage through the integrated generation of solid fuel and biogas from biomass (IFBB): 1. The fate of mineral compounds Bioresource Technology 102 (2011) 4855–4865.
12. Handreichung. Biogasgewinnung und-nutzung. 2006.
13. Agriculture in figures 2008. 2008. Statistics Estonia.
14. Astover, A., Roostalu, H., Lauringson, E., Lemetti, I., Selge, A., Talgre, L., Vasiliev, N., Mõtte, M., Tõrra, T. & Penu, P. 2006. Changes in agricultural land use and in plant nutrient balances of arable soils in Estonia. Arch. Agron. Soil Sci. 52(2), 223–231.
15. Heinsoo, K., Hein, K., Melts, I., Holm, B. & Ivask, M. 2011. Reed canary grass yield and fuel quality in Estonian farmers' fields. Biomass and Bioenergy. 35, 617–625.
16. Kers, J., Kulu, P., Aruniit, A., Laurmaa, V., Križan, P., Šooš, L., Kask, Ü. 2010. Determination of physical, mechanical and burning characteristics of polymeric waste material briquettes. Estonian Journal of Engineering, 4, 307–316.
17. Kolář. L, Kužel. S, Peterka. J and Borová-Batt. J. 2010 Agricultural Faculty of the University of South Bohemia in České Budějovice Czech Republic. Utilisation of Waste from Digesters for Biogas Production, Biofuel's Engineering Process Technology,191-220
18. Noormets, M., Raave, H., Viiralt, R., Kuusemets, V., Alaru, M., Kuht, J., Talgre, L. & Makke, A. 2007. Herbaceous energy crops in Estonia. Projekti aruanne. 97. [www.bioenergybaltic.ee/?id=1302]
19. Greenhouse gas emissions and fertilizer production [online][viewed: 21.01.2012.] Available at: http://www.fertilizer.org/ifa/HomePage/SUSTAINABILITY/Climate-change/Emissions-from-production.html