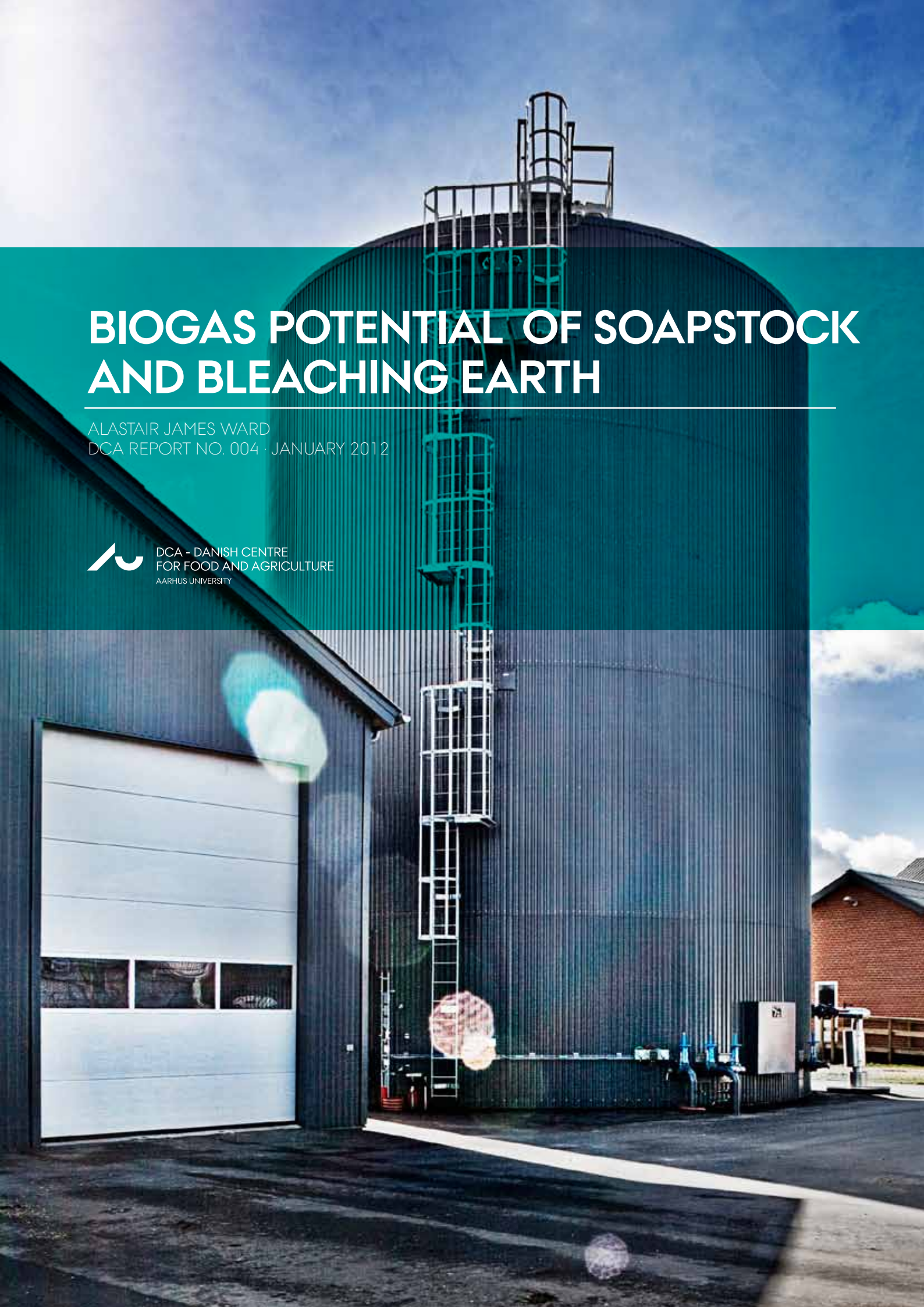


The background image shows a large, dark-colored industrial silo with a spiral staircase leading up to a platform at the top. To the left of the silo is a building with a large white garage door and a small window. The sky is blue with some clouds. The title text is overlaid on the upper part of the image.

BIOGAS POTENTIAL OF SOAPSTOCK AND BLEACHING EARTH

ALASTAIR JAMES WARD
DCA REPORT NO. 004 · JANUARY 2012



DCA - DANISH CENTRE
FOR FOOD AND AGRICULTURE
AARHUS UNIVERSITY

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Scientific report

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Biogas potential of soapstock and bleaching earth

Preface

Recently, a system of regional public funding for research has been established in Norway, to support cooperation between local actors within research and local industries. The regional funding council of Mid-Norway provided funding for a project to study how the energy output from anaerobic digestion of animal manure may be increased by utilizing fish oil residues, rich in fat, as a co-substrate. The project, in Norwegian called “Biogasskunnskap” (Knowledge about biogas), lasted for one year (2011) and was a co-operation between the Norwegian Institute for Agronomical and Environmental Research -Bioforsk, the Norwegian University of Life Sciences, various local industry partners in Mid-Norway and Research Centre Foulum at Aarhus University.

Dr. Alastair Ward and his staff at Foulum have conducted laboratory tests to study how much fish oil by-products that may be appropriate to use as co-substrate in anaerobic digestion of animal manure. Laboratory tests cannot be directly transferred to practical scale, but such results provide a useful point of departure for optimizing the actual biogas plant and process.

The project leader, Dr. Anne-Kristin Løes at Bioforsk Organic Food and Farming division, Tingvoll has discussed the study and the current report with the author. Bioforsk acknowledges the work conducted by Aarhus University, and appreciates the opportunity to use the report series of the University to publish results from the “Biogasskunnskap” project.

Unless otherwise stated, the photographs in the report were taken by Dr. Alastair Ward.

Tingvoll December 2011, Anne-Kristin Løes.

Purpose

The purpose of the study described in this report was to examine the co-digestion of soapstock and bleaching earth by-products of the Omega 3 oil refining industry with cattle manure to produce biogas. Ratios of cattle manure to by-product were based on previous experience (e.g. Ward 2010) and physical handling limitations.

Introduction

Anaerobic digestion of animal manure to produce biogas is currently underdeveloped in Norway. This can be attributed to small farm sizes and low energy costs which reduce the profitability of farm scale biogas plants. An effective method of increasing the productivity of small scale agricultural biogas plants is the addition of wastes from industry, particularly the fish processing industry which plays an important role in the Norwegian economy. This study investigates the biogas potential of two currently non-utilised by-products of the Omega 3 fish oil refining process, soapstock and bleaching earth. Other by-products such as stearin are also suitable for biogas production (Ward 2010) but may be better utilised for direct combustion at

the processing plant or for conversion to biodiesel in the future if economically viable. However, fish oil soapstock and spent bleaching earth both contain large amounts of oils and fats which have a high biogas potential. The annual quantity of these by-products from the Norwegian Omega 3 processing industry has been estimated at 20,000 tons of soapstock and 30,000 tons of bleaching earth (Ward and Løes 2011). Currently, much of this material (soapstock) is shipped to Denmark for utilization in biogas plants, whereas the bleaching earth is sometimes deposited as landfill.

The refining and concentration of Omega 3 oils (a valuable product for human consumption) from oily fish is a multi-step process (Figure 1). The first step is neutralization where an alkaline solution removes unstable fatty acids into a liquid by-product called soapstock. The next step is a bleaching process using montmorillonite clay, commonly referred to as bleaching earth. The spent bleaching earth is an oil-rich by-product produced at this stage. Winterization is a filtering step to remove any remaining unstable fatty acids. The by-product here is stearin, a waxy substance. The deodorization step is a distillation procedure to remove most of the Omega 3 oils. Following deodorization is an esterification of the remaining material to produce ethyl esters which are again distilled to concentrate any remaining Omega 3 oils. The non-Omega 3 oil at this stage is mostly used as a fuel for the processing plant.

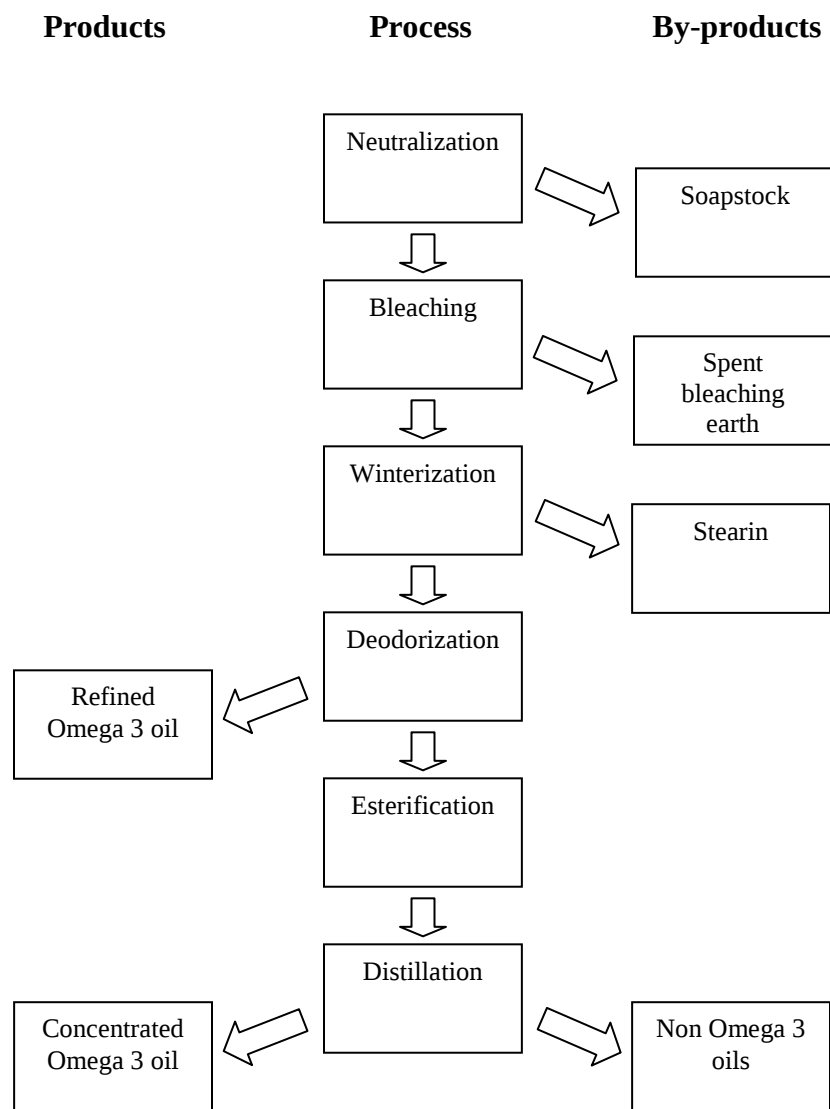


Figure 1. Schematic diagram of the refining process of fish material to Omega 3 oil for medicinal use, kindly made available from the Norwegian company GC Rieber Oils (Ward and Løes 2011).

Spent bleaching earth (Figure 2) from animal and vegetable oil refining is an oil rich resource that is available in large quantities throughout the world. It has been used as a feedstock for biogas plants for many years in Europe and is still used in such a way, e.g. in Germany. However, the minerals in bleaching earth can cause abrasive damage to equipment leading to increased wear. The particles will also sink to the bottom of a biogas plant that does not have sufficient mixing to maintain them in suspension, meaning that the effective volume of

the digester tanks will decrease over time and will thus eventually need emptying and subsequent re-starting of the plant. There may also be some concern regarding the presence of heavy metals and organic pollutants in bleaching earth, particularly when the digested material from a biogas plant is applied to agricultural land as a fertilizer. This topic is analyzed as a separate part of the “Biogasskunnskap” project. Another issue with bleaching earth is the very high dry matter content. Such a dry material has to be mixed with a very fluid material to ensure pumping and digester mixing is possible, and the physical addition of the material into a biogas plant requires consideration.

Soapstock is a much easier material to physically handle in a biogas plant due to it being a liquid of low viscosity at room temperature (Figure 2). Chemically, the substance may also be a challenge due to the sodium hydroxide which raises sodium concentrations to levels which are inhibitory to the biogas process, although once co-digested with manure this ceases to be a problem. The addition of sodium hydroxide can in some cases increase the pH significantly, with a value of pH 9.8 having been reported previously (Ward and Løes, 2011) although normally the oil refining industry aims for a more conservative use of NaOH. Typical pH-values for soapstock from G C Rieber are 4-5.



Figure 2. Samples of soapstock (left) and bleaching earth (right) showing the fluid nature of soapstock at room temperature (about 20°C), and the clay-like appearance of bleaching earth.

Methods

The work was carried out as batch experiments (Møller et al., 2004) for a period of 92 days. All samples were measured in triplicate.

Batch experiments were carried out in glass infusion bottles of 500 mL total volume. To each bottle 200 g of inoculum material was added, the inoculum was sourced from the post digestion tank (temperature *ca.* 35°C) at Aarhus University biogas plant, Research Centre Foulum, Denmark. Prior to use, the collected inoculum was incubated at 35°C for two weeks to remove a large portion of the residual biogas potential, then filtered to remove larger particles. The filtering process provides a less heterogeneous inoculum which reduces experimental error in the batch assay.

Bleaching earth and soapstock for the experiment were provided by the G C Rieber factory, Kristiansund, Norway, and were sampled in February 2011. The experimental substrates consisted of bleaching earth and soapstock mixed with cattle manure at ratios of 2.5, 5, 7.5, 10 and 12.5 % w/w and 2.5, 5, 7.5 and 10 % w/w respectively. The upper limits were based on previous experience of cattle manure co-digestion with fish stearin (Ward 2010).

Twenty five grams of each experimental substrate was added to each inoculated infusion bottle, which were then sealed with a butyl rubber stopper and flushed with nitrogen for two minutes to remove oxygen in the headspace (Figure 3). Finally, the bottle was shaken thoroughly by hand to mix the inoculum and substrate.

In addition to the experimental assays, one set of triplicate bottles had inoculum alone added as a blind control.



Figure 3. Flushing a batch test bottle with nitrogen to remove air in the headspace.

The bottles were then incubated at 35°C in total darkness (Figure 4) and the gas produced was measured when the pressure in the bottles was sufficient to cause a noticeable bulge in the rubber tops. The measuring dates were at approximately four day intervals at the start when gas production is usually highest (except when there is a lag period due to inhibition which will delay initial gas production by days or even weeks) and less frequently towards the end of the experiment, with the final two measurements being 32 days apart. The batch assay bottles were shaken vigorously following each gas measurement. The gas volume measurement was by the acidified (*ca.* pH 2) water displacement method (Figure 5). The acidification prevents the carbon dioxide in the biogas dissolving in the water and giving a false reading. The apparatus consists of plastic cylinders (nine in total) which are sealed at the top and open at the bottom and stand in containers filled with the acidified water. Biogas enters each cylinder *via* a hypodermic needle connected to a tube which in turn runs through the open bottom of the cylinder and up inside, terminating at the top. In addition there is a second tube running next to the first tube inside each cylinder. This tube is connected to a vacuum pump. The pump removes gas from the cylinder headspaces, thus drawing the acidified water up into the cylinders until all the gas has been removed, at which point the pump is stopped. During measurement, the hypodermic needle is pushed through the butyl rubber caps on the batch bottles allowing biogas to enter the cylinders. The weight of the water column in the cylinders and the pressure in the bottles allows the water level in the cylinder to fall until the pressures equalise with atmospheric pressure, at which point the water levels (and therefore the gas volumes) can be read from calibrated graduations marked on the cylinders. During volumetric measurement, a small gas sample of 30 mL was collected for compositional analysis by gas chromatography (GC) to determine relative proportions of both methane and carbon dioxide. However, the biogas produced is contaminated with nitrogen from the initial flushing of the bottles so the final percentage of methane and carbon dioxide was calculated by assuming that these two gases together are equivalent to 100 % of the biogas volume, calculated as shown below:

$$\% \text{CH}_4 (\text{actual}) = \frac{\% \text{CH}_4 (\text{measured})}{\% \text{CH}_4 (\text{measured}) + \% \text{CO}_2 (\text{measured})} \times 100$$

The gas yields were calculated by measuring the gas production of the tested samples, then subtracting the gas production of the inoculum-only blind control. In the results, only methane yields are presented.



Figure 4. Batch assay bottles undergoing incubation.

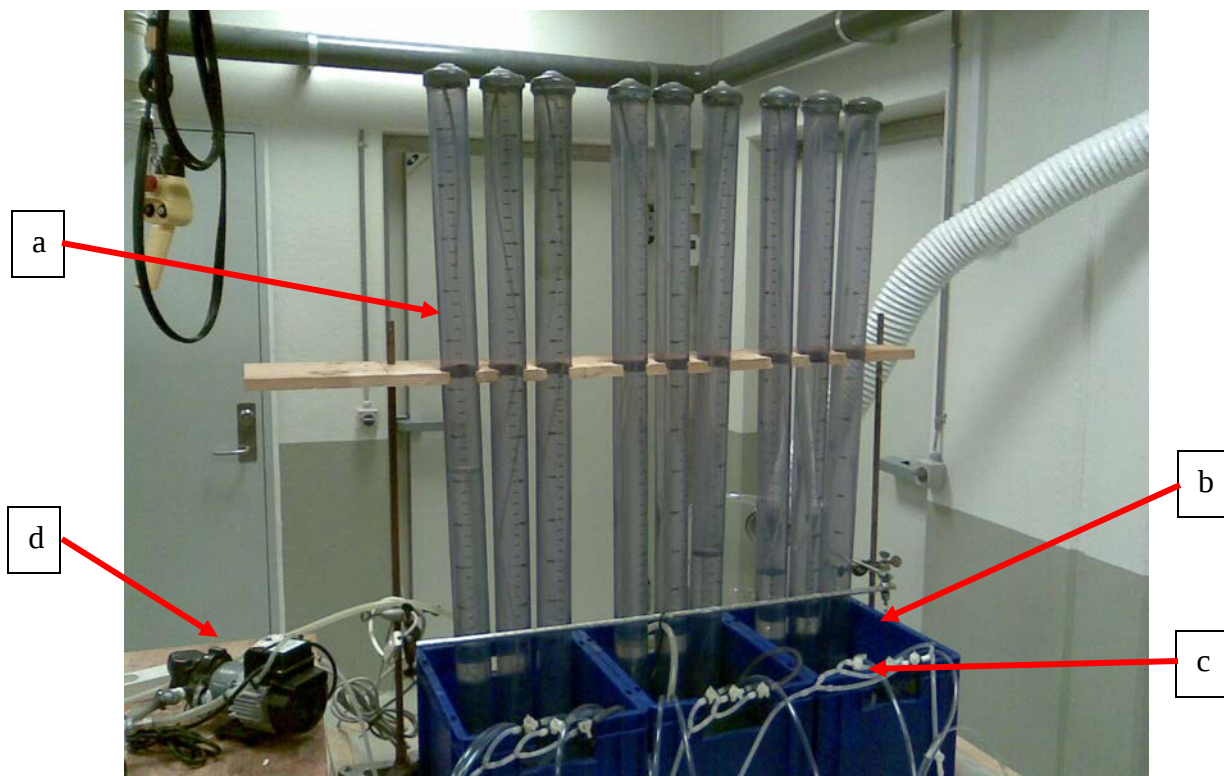


Figure 5. Acidified water displacement method for gas volume measurement, showing (a) cylinders, (b) acidified water containers, (c) biogas tubing and (d) vacuum pump.

The accumulated methane yields were recorded in terms of both litres per kg organic matter and litres per kg wet weight. Methane yield per kilogram of organic matter (referred to as volatile solids or VS) gives an indication of the degradability of a material and the overall efficiency of the process whereas methane yield per kilogram of wet weight is of interest in estimating methane production in relation to the size of the digester. After 90 days of batch digestion the methane production is expected to be practically zero. Thus the final yield figures are considered to be the Ultimate Methane Yield, the maximum methane produced by anaerobic digestion.

Volatile solids were measured by first determining the dry matter by drying at 105°C for 24 hours then incineration at 550°C for 2 hours to leave an ash residue. Subtracting the dried weight from the wet weight allowed the percentage of dry matter to be determined, whereas subtracting the ash weight from the dry weight allowed determination of volatile solids.

To examine the pH effect of the soapstock, the pH was measured in raw soapstock and cattle manure and also immediately following addition to cattle manure in the ratios used in the batch assays and again following two days incubation at 35°C.

Results

Dry matter and volatile solids

The dry matter and volatile solids values of the inoculum and the individual and mixed substrates used are shown in Table 1. The choice of a maximum 12.5 % bleaching earth was based on the dry matter of the mixed material. At nearly 18 % dry matter this was considered to be the upper limit of what would be feasible for pumping and mixing. Even after digestion the dry matter will still be high because 58 % of the dry matter is inorganic clay and therefore indigestible. The high dry matter value of soapstock was not considered to be a problem affecting handling of the material as the term “dry” here refers to material that is not water, and although soapstock contains very little water it has a similar viscosity at room temperature. However, the viscosity of soapstock at lower temperatures increases considerably: a small sample was observed visually as the temperature increased from -8°C to room temperature. Below about +5°C the material appears to be too viscous to pump effectively, and below about -5°C it forms a solid. This could be a storage issue for Norwegian biogas plants during the winter, although biogas plants which treat materials with a melting point within normally expected ambient temperatures should use a heated storage container. For example, the full scale biogas plant at Aarhus University Research Centre Foulum, Denmark, uses a fat storage tank maintained at 30°C.

Table 1. Dry matter and volatile solids values of individual and mixed materials. Mixed materials are described by the percentage wet weight of bleaching earth or soapstock in cattle manure.

Material	Dry matter (%)	Volatile solids (g/kg)
Inoculum	2.52	16.7
Cattle manure	8.36	68.7
Bleaching earth	84.16	355.0
Soapstock	98.90	993.5
Cattle manure and 2.5 % bleaching earth	10.26	75.8
Cattle manure and 5 % bleaching earth	12.15	83.0
Cattle manure and 7.5 % bleaching earth	14.05	90.2
Cattle manure and 10 % bleaching earth	15.94	97.3
Cattle manure and 12.5 % bleaching earth	17.84	104.5
Cattle manure and 2.5 % soapstock	10.62	91.7
Cattle manure and 5 % soapstock	12.89	114.7
Cattle manure and 7.5 % soapstock	15.15	137.7
Cattle manure and 10 % soapstock	17.41	160.7

Soapstock

The pH measurements for soapstock, cattle manure and soapstock added to cattle manure are shown in Table 2.

Table 2. pH measurements in soapstock, cattle manure and the experimental ratios of the two substrates after immediate mixing and 48 hours incubation at 35°C.

Substrate	Immediate pH measurement	pH after 48 hours incubation at 35°C
Soapstock	4.12	NA
Cattle manure	7.26	7.12
Cattle manure + 2.5% soapstock	7.11	6.99
Cattle manure + 5% soapstock	6.92	6.82
Cattle manure + 7.5% soapstock	6.80	6.83
Cattle manure + 10% soapstock	6.87	6.78

As can be seen, in this sample of soapstock NaOH was not in excess and therefore the pH was low because of the free fatty acids removed from the fish oil during the neutralisation process. Adding the soapstock to cattle manure slightly lowered the manure pH, but the optimum range for methane production is around pH 6.8 – 7.2 (Ward et al. 2008) so pH was not inhibitory. Incubating at 35°C for 48 hours reduced the pH of most soapstock / manure mixtures by *ca.* 0.1 unit, which was attributed to volatile fatty acid (VFA) production following fermentation of the cattle manure as the manure alone also had a similarly decreased pH after the incubation period.

Figure 6 shows the methane yields in terms of VS of the tested ratios of cattle manure and soapstock. The curves clearly assume a sigmoid shape due to an initial lag in methane production. This lag phase is more pronounced at higher proportions of soapstock. This lag is typically the result of inhibition of the biogas process. In a batch assay, there is a sudden addition of substrate to a microbial population that is in the stationary phase (due to the inoculum being collected from a post digestion tank and the additional two week digestion prior to the batch assay to remove the major part of substrate). The substrate load is sufficiently high to cause an overproduction of intermediate products such as VFA which are inhibitory to further degradation of the VFAs themselves and also to methane production. The intermediate products are slowly metabolised until their levels are no longer inhibitory at which point methane production increases. When oil-based feedstocks are digested, it is also likely that long chain fatty acids (LCFA) are causing inhibition. These can chemically inhibit key microbial processes and also physically inhibit them by creating an impermeable oily coating on the microbial cell membranes. The inhibition is further shown in the biogas composition data, which was fairly stable at 65-70% methane for soapstock additions up to 5 % w/w, but was considerably lower during the early stages of digestion of higher shares of soapstock; as low as 28 % methane after 4 days digestion of 12.5 % w/w soapstock.

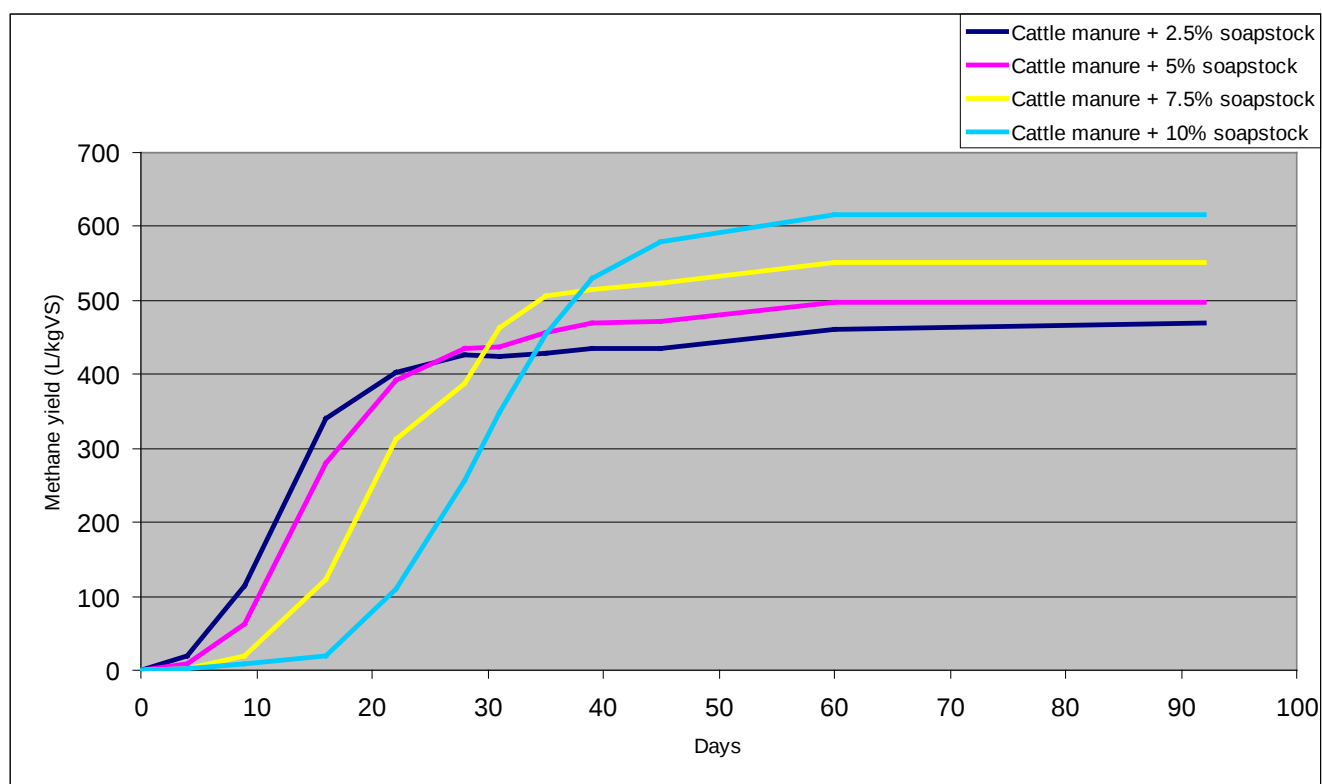


Figure 6. Methane yield of cattle manure and soapstock mix, expressed as litres of gas per kg volatile solids.

Table 3 shows the final methane yields in terms of both VS and wet weight. From this data a methane yield for soapstock can be calculated. This calculation requires an estimate of the methane potential of the cattle manure alone, which can be assumed to be very similar to the 318 L/kgVS found following 2.5 % addition of bleaching earth. From this, the methane potential of soapstock alone can be calculated at between 660 and 760 L/kgVS. Such a methane potential is relatively high, and compares quite favourably to more concentrated lipid materials such as stearin from fish processing (methane yield = 919 L/kgVS) as reported by Ward (2010). This also compares well with the maximum theoretical yield of palmitoleic acid, a typical LCFA found in fish oil, of 992 L/kgVS. The theoretical yield of palmitoleic acid was calculated using the formula proposed by Buswell (1936) which uses C, H and O composition of the substrate to estimate methane yield and therefore does not take into account inhibition and thus is often much higher than the ultimate methane yield determined by batch assay.

Table 3. Ultimate biogas and methane yields of the tested mixtures of cattle manure and soapstock.

Substrate	Methane yield (L/kgVS)	Methane yield (L/kg wet weight)
Cattle manure and 2.5 % soapstock	470	43.1
Cattle manure and 5 % soapstock	499	57.2
Cattle manure and 7.5 % soapstock	551	75.9
Cattle manure and 10 % soapstock	615	98.8

The yield in terms of wet weight (Figure 7) shows that soapstock addition can increase the methane yield considerably. An addition of 5% soapstock could almost double the methane yield when compared to the estimated yield of cattle manure alone (it must be noted that the methane yield of cattle manure alone was not measured in this study and this estimate is based on 2.5 % bleaching earth mixed with cattle manure data). Increasing the proportion of soapstock increases the yield further, for example at 5 % soapstock addition the 30 day methane yield is about 20 % greater than the comparable yield at 2.5 % soapstock addition, whereas after 92 days the difference is 27 %. This is partly because soapstock has a much higher proportion of VS per kg wet weight than cattle manure (Table 1) and partly because soapstock has a much higher methane yield per kg VS than manure.

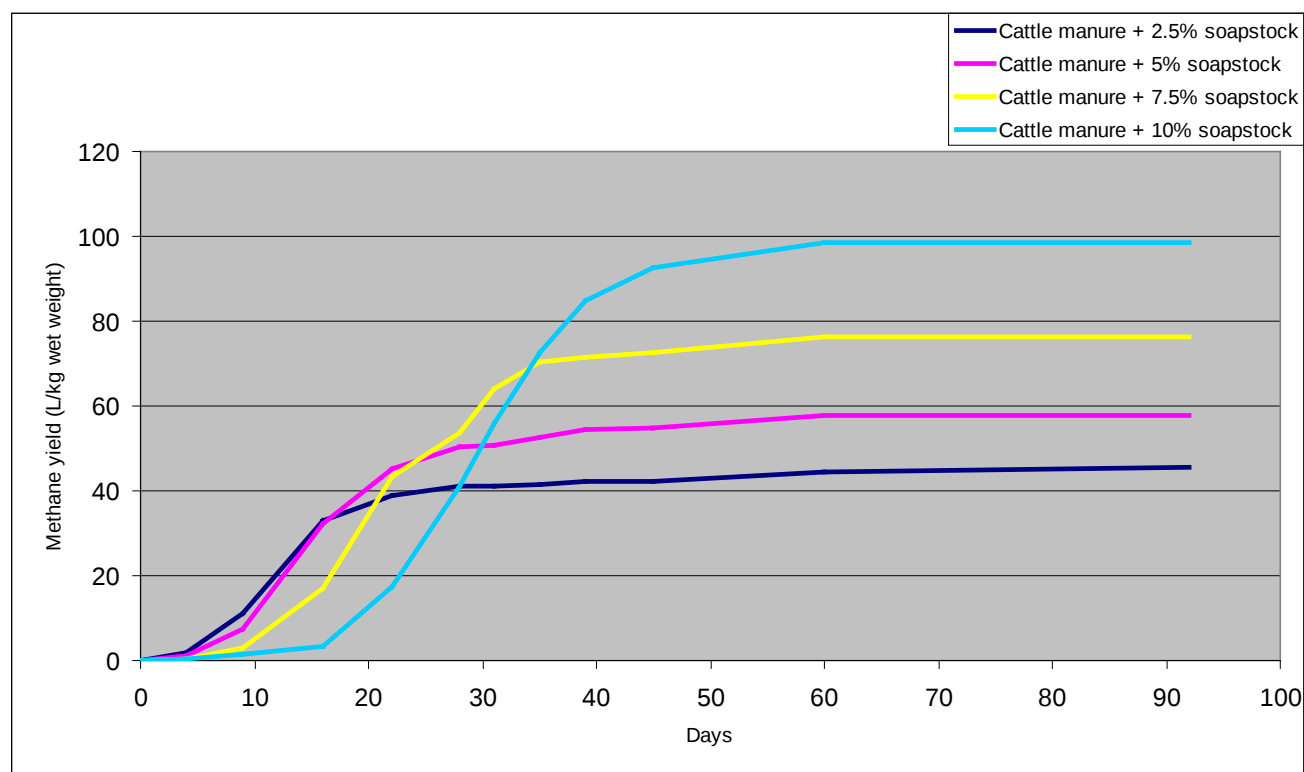


Figure 7. Methane yield of cattle manure and soapstock mix, expressed as litres of gas per kg wet weight.

However, such a result is a warning to biogas plant operators interested in using soapstock as a co-substrate. Although it is not possible to directly compare batch digestion gas production rates with a continuous process, the lag time before methane production commences increases considerably with an increasing share of soapstock. For example, at 30 days, the methane yield for 10 % soapstock addition is about half of the final yield at 92 days, whereas at 30 days the yield for 5 % soapstock addition is more than 90 % of the final 92 day yield. It is recommended that a biogas plant that wishes to add soapstock as a co-substrate should observe the process carefully as the soapstock concentration is increased to avoid potential overload. Such observations should include gas flow, gas composition and frequent analysis of VFA to look for any changes.

Bleaching earth

The results presented in Figure 8 show the accumulated methane yield of the experimental mixtures of bleaching earth in terms of VS (litres methane output per kg organic matter added to the bottle in the substrate). It is interesting that the yields tend to decrease as the proportion of bleaching earth in the substrate increases, and that this is not changed during a prolonged period of digestion up to 92 days. After about 60 days, very little methane was produced, as shown by the curves (Figure 8). The final biogas and methane yields per kg VS and per kg wet weight are shown in Table 4. The biogas composition was quite similar across all bleaching earth ratios tested, being between 65 and 67 % methane. As shown in Figure 8, the optimal addition of bleaching earth appears to be 7.5 % but the differences between the various ratios is small.

Table 4. Ultimate biogas and methane yields of the tested mixtures of cattle manure and bleaching earth.

Substrate	Methane yield (L/kgVS)	Methane yield (L/kg wet weight)
Cattle manure and 2.5 % bleaching earth	318	24.3
Cattle manure and 5 % bleaching earth	313	26.0
Cattle manure and 7.5 % bleaching earth	327	29.4
Cattle manure and 10 % bleaching earth	310	30.1
Cattle manure and 12.5 % bleaching earth	278	29.4

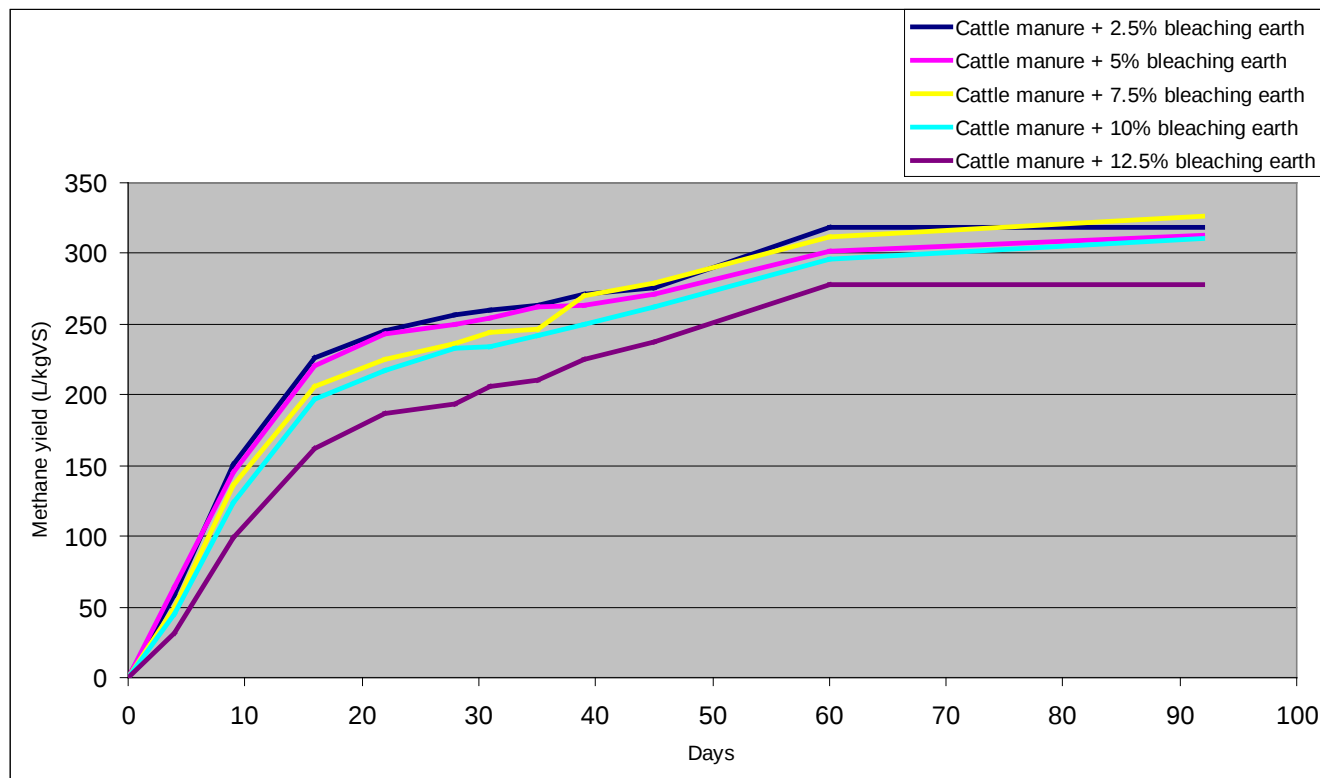


Figure 8. Accumulated methane yield of cattle manure mixed with bleaching earth, expressed as litres of gas per kg volatile solids. Yields were measured as required: approximately every four days at the start of the experiment and up to 30 days at the end of the experiment.

Figure 9 and Table 4 show that the methane yield per kg wet weight increases as the proportion of bleaching earth increases up to a maximum of 10 % w/w. In this study, adding 12.5 % bleaching earth reduced the yield slightly, which suggests inhibition. However, it has previously been shown that the clay minerals present in bleaching earth have a beneficial effect on biogas production by reducing inhibition by lipids in continuously fed stirred reactors (Angelidaki et al., 1990). Inhibition by intermediary compounds such as VFA, as discussed above, would normally be overcome before the end of the batch assay. However, it has been shown that LCFA inhibition of anaerobic digestion is permanent (Angelidaki and Ahring 1992), which could explain the relatively low yields of bleaching earth. It has previously been shown that co-digestion of olive oil bleaching earth and pig slurry gave a methane yield of 340 L/kgVS (Campos et al., 2000) which compares well with the findings of this study, although the previous study showed greater inhibition at a 12.5 % mix of bleaching earth, which was attributed to LCFA inhibition.

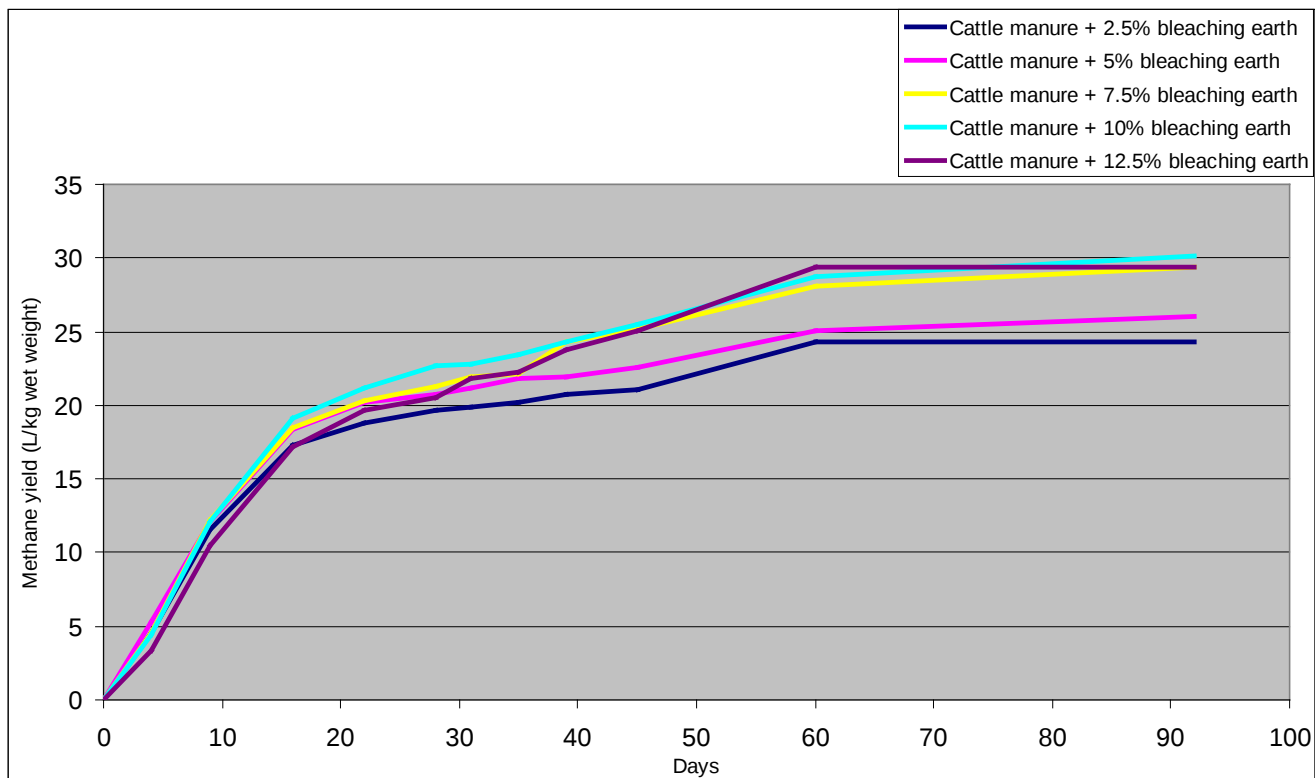


Figure 9. Methane yield of cattle manure and bleaching earth mix, expressed as litres of gas per kg wet weight of substrate added to each bottle.

Additionally, from the data presented in Table 1, it is likely that adding 12.5 % bleaching earth will also create problems with pumping of the digestate.

It was of some concern that the bleaching earth (in particular) would form sediment between measurements, however this is quite normal in batch testing without constant mixing and the long assay period is designed to reduce the effect of inefficiencies in methane production caused by this.

The practical consequence of the results shown in Figures 8 and 9 and Table 4 is that adding bleaching earth as a co-substrate to a biogas plant will decrease the overall yield in terms of VS, but because the bleaching earth used in this study was found to be 35.5 % (w/w) organic matter it is a much more energy-dense substrate and so will increase the methane yield on a wet weight basis when added at up to 10 % w/w. From the results presented it can be calculated that a biogas plant that is solely treating cattle manure can increase its volumetric methane production by approximately 35 % upon addition of 10 % (w/w) bleaching earth.

Conclusion

Using by-products of the Omega 3 production process for biogas production shows great potential, especially for soapstock.

The methane yield of soapstock was calculated to be between 660 and 760 L/kgVS and as the material is more than 99 % VS it is a very concentrated substrate for biogas production. The effect of such a concentrated substrate is more pronounced when the methane yield per kg wet weight is examined, as the methane yield increased from 69.6 L/kg to 144.5 L/kg when comparing 2.5 % soapstock addition with 10 % soapstock addition. However, increasing the proportion of soapstock in cattle manure increased the lag phase during which very little methane was produced in the batch assays. Although this lag phase is difficult to compare with the performance of a full scale continuously fed process, care is advised when adding soapstock above 5 % w/w in cattle manure to avoid inhibition of the process by process intermediates such as LCFA or VFA. It may also be beneficial for the pH of soapstock to be measured prior to digestion as it has been found that this can vary widely from strongly alkaline when excess NaOH is added to fairly acidic when less NaOH is used.

Bleaching earth also increased the methane yield in terms of L/kg wet weight, but did not increase yield in terms of L/kgVS, as no specific pattern was seen. The methane yield of bleaching earth was difficult to determine precisely but was estimated to be relatively low at approximately 320 L/kgVS. This suggests LCFA may have become inhibitory, particularly at higher concentrations of bleaching earth as a noticeably lower methane yield (in terms of L/kgVS) was recorded at 12.5 % bleaching earth addition. Further, addition of high concentrations of bleaching earth could place additional stress on pumping and mixing equipment due to the high dry matter content, as well as abrasive damage and potential long term problems with sedimentation.

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DCA - National Centre for Food and Agriculture is the entrance to research in food and agriculture at Aarhus University (AU). The main tasks of the centre are knowledge exchange, advisory service and interaction with authorities, organisations and businesses. The centre coordinates knowledge exchange and advice with regard to the departments that are heavily involved in food and agricultural science. They are:

Department of Animal Science
Department of Food Science
Department of Agroecology
Department of Engineering
Department of Molecular Biology and Genetics

DCA can also involve other units at AU that carry out research in the relevant areas.

SUMMARY

Fish processing is an important industry in Norway. The refining of fish oils to produce Omega 3 oils for human consumption produces two by-products which are rich in oil but are currently not utilised: soapstock and bleaching earth. On the other hand, biogas production in Norway is underdeveloped due to low energy costs and small farm sizes which reduce the profitability of this otherwise effective method of treating organic wastes and reducing greenhouse gas emissions from agriculture. To increase the profitability of small biogas plants, this report investigates the utilisation of fish soapstock and bleaching earth as co-substrates with cattle manure for biogas production.