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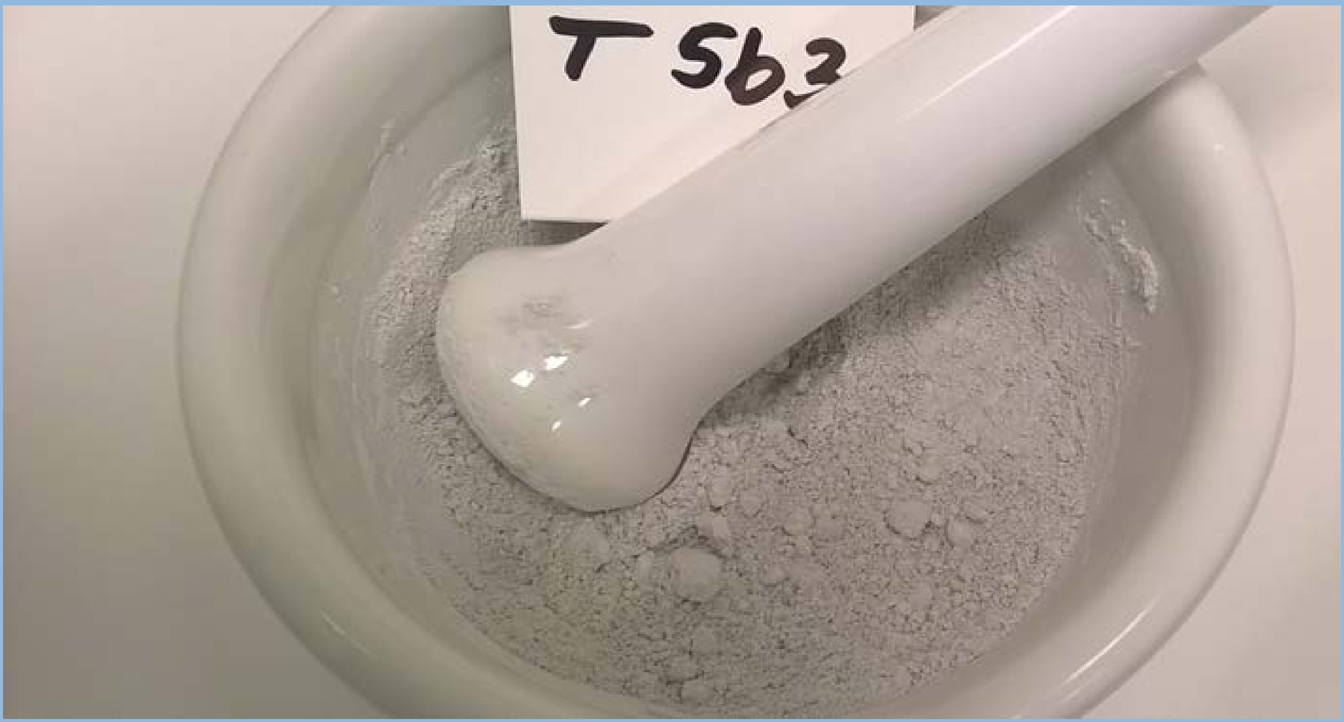
EFFECTS OF MARBLE APPLICATION TO MANURE AND ANAEROBIC DIGESTATES

Final project report



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TITTEL/TITLE

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SAMMENDRAG

Tidligere forsøk og praktiske erfaringer i Norge har vist positive effekter av å tilsette litt finmalt kalk til bløtgjødsel før spredning. Det hevdes at slik tilsetning gir mindre lukt, mindre flueplager og bedre grasavlinger. Effekten av å tilsette finmalt marmor til husdyrgjødsel og råtnerest (etter biogassbehandling av organisk avfall) ble undersøkt i feltforsøk og i kontrollerte forsøk innendørs i perioden 2016-2017. I feltforsøk på Tingvoll gard i 2016 fikk vi 6% høyere tørrstoffavlinger av eng ved andre slått når det ble tilsatt kalk til fersk kugjødsel. Vi fikk samme avlingsøkning med vanlig bløtgjødsel, og bløtgjødsel som var utråtnet i et biogassanlegg. Ved første slått var det ikke sikker avlingsøkning, men dette kan skyldes at gjødsla hadde vært lagret over lengre tid.

I åpne beholdere ble bløtgjødsel fra gris og ku, utråtnet bløtgjødsel fra ku og råtnerest fra Ecopro, med og uten marmorpulver, sammenliknet over 6 uker ved ca. 12 °C, med ukentlig omrøring. For råtnerest fra Ecopro, basert på kloakkslam, husholdningsavfall og fiskegjødsel, ble det en senkning av pH med tilsetning av marmor. For andre gjødselslag var denne effekten liten og usikker. Det ble ikke lavere tap av total-nitrogen (N) eller ammonium fra beholderne ved tilsetning av marmor. Dette undersøkte vi ved å måle innholdet og konsentrasjonen (i % av tørrstoff) av total-N og NH_4-N ved start og slutt av forsøket. Nitrogentapet totalt sett var betydelig i dette forsøket. Det ville være interessant å gjenta forsøket med

betingelser som er mer sammenliknbare med forholdene i praksis. Kalkpulveret var ikke tilsatt dispergeringsmiddel i våre forsøk.

Selv om vi ikke kunne vise noen effekt av reduserte N-utslipp etter marmortilsetning til råtnerest og gjødsel, er finknust marmor et verdifullt jordforbedringsmiddel som kan tilføre jorda verdifulle næringsstoff, og som gjerne kan tilføres gjennom innblanding i husdyrgjødsel.

SUMMARY

In controlled indoor experiments, and a field experiment at Tingvoll farm in 2016, we studied the effect of mixing finely ground calcium carbonate made from marble into animal manure slurries, and digestates. In field, 6% higher yields of dry matter were achieved at the second harvest by such addition to the manure. For the first harvest, no effects were found of marble application. This may be due to the manure being stored for a longer time for the application in spring, whereas manure applied after the first harvest was freshly collected.

In open containers, slurry from pigs and dairy cows, digested dairy cow slurry and digestate from a biogas plant (Ecopro) treating sewage sludge, household waste and fish waste was studied. Substrates with or without addition of marble were analyzed before and after 6 weeks of storage at about 12 °C with weekly stirring. In the digestate from Ecopro, pH decreased by marble addition. For the other substrates, the effect on pH was small and inconsistent. The reduction of the ammonium and total nitrogen (N) contents or concentrations (% of dry matter) in containers during storage did not differ between with and without marble addition. Hence, marble application did not reduce N emissions in this study. The N loss was significant. It could be interesting to conduct a further study with conditions more comparable to a practical situation. The marble was not mixed with dispersion agent in the present study.

Finely ground marble is still a valuable soil amendment, to maintain or increase soil pH and apply valuable nutrients to the soil. Mixing the marble into animal manure or digestate is a convenient way of applying it to agricultural soil.

LAND/COUNTRY: NORGE/NORWAY	NORGE
FYLKE/COUNTY: MØRE AND ROMSDAL	MØRE OG ROMSDAL
KOMMUNE/MUNICIPALITY: TINGVOLL	TINGVOLL
STED/LOKALITET: TINGVOLL GARD	TINGVOLL GARD

GODKJENT/APPROVED	**PROSJEKTLEDER /PROJECT LEADER**
TURID STRØM	ANNE-KRISTIN LØES

Preface

Anaerobic digestion of various organic materials to produce biogas and fertilizers is a growing technology in Norway. The treatment increases the concentrations of mineral nitrogen (N) in the final slurry (digestate). Possibly, ground calcium carbonate may be added to the digestate to increase the fertilizer quality and reduce N emissions. A project to study this question, “Marmorfiks”, was funded by the Regional Research Fund of Mid-Norway, with significant co-funding from Møre and Romsdal county council and the project participants. This report is the main deliverable from the project “Reduced nitrogen emissions from digestate with application of marble”, with the acronym “Marmorfiks” (2016-2017). In Norwegian, “marble” is “marmor”.

Norwegian Centre for Organic Agriculture (NORSØK) coordinated the project, with SINTEF Energy Research as a scientific partner. Assisting experts have been Dr. John Morken, NMBU and Dr. Lena Rodhe, RISE, Sweden. Industry partners have been:

- Omya Hustadmarmor AS in Fræna, Møre and Romsdal, a large producer of ground calcium carbonate for paper industry
- Ecopro AS, a well-established biogas plant in Verdal, North Trøndelag county, and
- Aukra næringsforum AS in Aukra, Møre and Romsdal, who has an interest in development of regional industry including biogas.

Maryna Zinchenko from Omya Hustadmarmor AS, Tore Fløan from Ecopro AS, Roger Khalil from SINTEF Energy Research AS and Turid Strøm from NORSØK composed the project’s governing board, and met four times during the project with physical meetings at Ecopro and NORSØK, and two web-based meetings.

In close cooperation, the whole consortium developed experimental plans to measure if addition of ground calcium carbonate from marble to various types of organic slurries could reduce N emissions, and possibly increase the yields where animal slurry with marble was applied as fertilizer.

The project comprised four main topics: A literature review of former Norwegian studies, a field experiment to study the effect on perennial ley yields if applied manure was mixed with 2% ground calcium carbonate from marble (= marble), an indoor pot experiment with four manure substrates mixed with two types and two amounts of marble, and an experiment with closed containers of Ecopro digestate mixed with marble where gas emissions were analyzed.

The results of these studies are presented in this report. On behalf of the authors, I am grateful for valuable comments from Lena Rodhe, John Morken and Omya Hustadmarmor AS.

Tingvoll – July 2017

Anne-Kristin Løes, project leader
NORSØK

Contents

Background and former studies

Industrial partners

A small company, "Hustad Kalk og Marmor AS", was established in Elnesvågen, Fræna in Møre and Romsdal county in 1948. The aim was to increase the soil quality of local peatlands by offering lime from local calcium carbonate (marble) to farmers. Access to lime supported the cultivation of significant amounts of peatland along this part of the Norwegian shore. Over time, the company grew big and started cooperation with the international company Omya AG and was renamed to Omya Hustadmarmor AS in 2007. The main product of the company today is pigments for additives in paper production. Raw materials to the plant are delivered from Northern Norway and partly from the local quarries. High degree of whiteness is significant for the paper industry, and hence the stone is purified at the plant before further processing. Fillers and pigments for paper are extremely valuable products derived from calcium carbonate, but there are also many other areas where this high-quality mineral may be useful. After bypassing the purification steps, by-products are especially suitable for agriculture and forestry, lake- and river liming, where whiteness is not important. Omya Hustadmarmor AS is interested in development of new products for such applications.

Ecopro AS is a biogas plant located in Verdal, North Trøndelag. It was established in 2002 as a cooperation between several municipalities, to convert organic waste from Mid-Norway into energy and digestate useful for fertilization purpose. As one of the oldest biogas plants of this type in Norway, stable production has occurred since 2008, and the company is currently investing in upgrading the gas for transport purpose. The utilization of Ecopro digestate in agriculture is expanding, and addition of ground calcium carbonate could possibly be a way to reduce nitrogen (N) emissions from the digestate, which is rich in mineral N**.** Such N is easily lost as nitrous gases during field application.

Effects on grass yields

About 20 years ago (Amundsen 1999, Mo 2008), attention was paid to a possible N conserving effect when ground marble was added to liquid animal manure (slurry) before field application. Mixing the marble into the slurry was originally done to simplify the work of liming agricultural soil. However, the extension service for farmers located near to Omya Hustadmarmor AS noticed that addition of about 2% of ground calcium carbonate to slurry not only contributed to enrich the soil with calcium and slightly increase, or maintain, soil, pH. It also decreased the viscosity and odor of slurry, and gave better plant growth on old meadows (Mo 2008). However, studies in controlled environment could not reveal any significant effects of marble addition when dairy cow slurry was used to fertilize Italian ryegrass, Lolium multiflorum cv. Ajax (Tveitnes 2001). Two levels of marble addition were used, 2.5 and 5% by weight, to manure with on average 7% dry matter. Manure and marble mixtures were prepared by weekly addition of fresh slurry from dairy cows to storage containers during the indoor season. Marble was applied in four different ways:

- Applied in the container before manure addition

- Mixed into the slurry with the weekly addition

- Applied 14-20 days before start of the pot experiment

- Applied 2-6 days before start of the pot experiment

Total yields of four cuts of ryegrass gave 26 g DM per pot in the control treatment (no manure), 30 g for two treatments with mineral N, and 32-38 g for treatments with manure, or manure and marble. No significant differences were found between treatments with and without marble, or the level or timing of marble additions, or one treatment where traditional lime was used instead of marble, 2-6 days before applying manure to the ryegrass pots. No dispersion agent was used in this study. During 1997 and 1998, field experiments in perennial grass ley were conducted in Fræna, Møre and Romsdal county by the local extension service, in cooperation with the Agricultural University of Norway. Statistically significant differences were not found in any case between manure treatments with or without marble. In the field experiments, marble slurry Biokalk 75 was applied. This product is composed of 75% dry calcium carbonate, 25% water, and a dispersion agent.

A pot experiment with oats fertilized with pig manure was conducted as a part of a master thesis (Hegstad 2001). No significant differences were found between pots fertilized with manure + Biokalk 75, and manure only.

Effects on soil pH and manure viscosity

The liming effects in soil of field experiments conducted in 1997 and 1998 were studied in a master thesis by Øygard (1999). Soil pH increased with increasing amounts of marble slurry (Biokalk 75); to 6.55 with 5 % marble addition by weight, and 6.48 with 2.5% addition. Soil pH with application of manure without marble was 6.31.

Lower viscosity has been reported as one positive effect of marble application (Mo 2008). This was studied by Hanssen et al. (1999), see Figure 1. It is interesting to note that addition of limestone increased the viscosity (= less rapid floating), whereas Biokalk decreased the viscosity. This is most likely related to the dispersion agent.

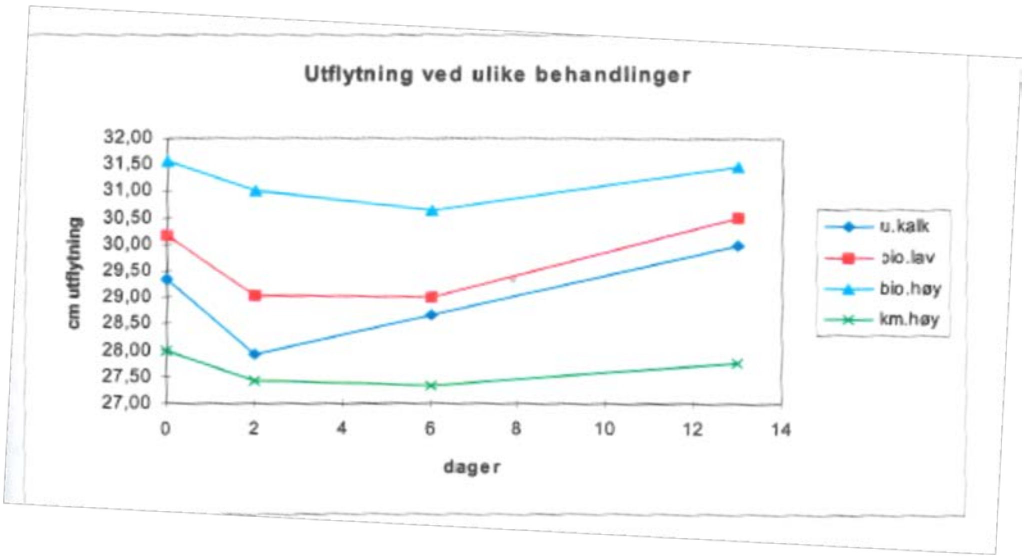


Figure 1. Ability of floating (in Norwegian, "utflytning"; the opposite of viscosity) of manure with no addition of marble ("u. kalk", dark blue line), with addition of dry, ground limestone ("km", green line) and with addition of two levels of Biokalk ("bio", marble slurry with dispersion agent, red and light blue lines). From Hanssen et al., 1999.

As far as we are informed, studies of how dispersion agents affect animal manure have not yet been conducted in Norway, and are hard to find in scientific literature.

N emissions from manure

During storage, N emissions from manure are mainly by ammonia gas, NH_3. Ammonium ions (NH_4^+) are present in urine and liquid manure, and the dynamic equilibrium between these species is affected by temperature, aeration, and the pH in the substrate (Figure 2). By increased pH, the concentration of ammonia in solution (liquid form) increases. When ammonia is lost, e.g. by ventilation, more ammonia will be formed. The ammonia at the surface of a solution is in dynamic equilibrium with ammonia gas in the air above the surface, the balance being determined by the temperature (Sommer & Husted 1995).

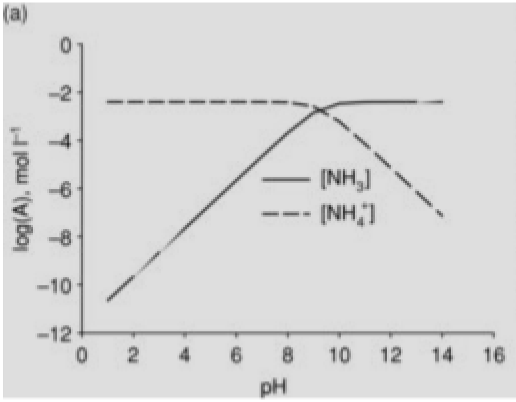


Figure 2. Relationship between NH_3 concentration in solution and NH_4^+ concentration in solution as a function of manure pH. From Sommer et al., 2013.

Hanssen et al. (1999) proposed pH decrease and binding of N in lignin as possible explanations for reduced N emissions from manure mixed with finely ground calcium carbonate. The main chemical species controlling pH in slurry are NH_4^+/NH_3, CO_2/HCO_3-/CO_3^{2-} and CH_3COOH/CH_3COO^- (Sommer & Husted 1995). When hydrogen ions are released, pH in the solution decreases. When fresh urine is mixed with solid manure and water in a slurry, urea - $CO(NH_2)_2$ - is hydrolyzed to form di-ammonium carbonate, $(NH_4)_2CO_3$, which easily emits ammonia. Application of calcium ions, e.g. as $CaCl_2$, may cause a precipitation of $CaCO_3$, reducing the risk of ammonia losses. However, when calcium is applied as calcium carbonate, this reaction is not likely. More likely, the significant increase in specific surface by addition of finely ground calcium carbonate may offer an explanation. When >50% of the marble particles are <8-10 µm, the specific surface is about 2 m^2 per g (Tveitnes 2001).

Increased specific surface will increase the reactivity for both chemical and microbiological processes, which may lead to production of CO_2. CO_2 production is acidifying, due to the equilibrium between CO_2 and $H_2CO_3/HCO_3^-/CO_3^{2-}$. Such acidification may reduce loss of ammonia, especially if ammonium ions can be adsorbed on the surfaces of the applied material. It is also possible that hydrogen ions are attached to the surfaces of the applied material, and are released in the manure because of surface reactions, reducing manure pH. Acidification will support dissolution of $CaCO_3$. Dissolution of $CaCO_3$ leads to formation of $Ca(HCO_3)_2$, which is easily soluble and increases concentrations of Ca^{2+} and bicarbonate (HCO_3^-). Increased concentration of Ca^{2+} may enhance the amount of hydrogen ions being released from material surfaces. Increased concentration of bicarbonate, which is the dominating carbonate species at pH 7, in combination with acetate from degradation of organic matter, may produce organic acids, decreasing manure pH.

Since marble is used as a liming agent in soils, it might be expected that marble application to manure will increase manure pH and hence the losses of ammonia. However, in a short time perspective (weeks), a pH *decrease* in manure pH has been demonstrated as an effect of marble addition, summarized by Mo (2008) to be expected to be about 0.3 pH units. During longer storage, manure pH may increase both in general and with application of marble, as shown by Hegstad (2001) for pig slurry stored for 2 ½ months in 40 liter containers.

Here, manure pH changed from initially 6.8 to 7.1 without application of Biokalk, 7.8 with 3% Biokalk, and 7.3 with 12 % Biokalk. In the treatment with 6% Biokalk, the pH was 7.0. This shows that a possible pH decrease is temporarily. Morken (2005) found very small differences in manure pH for pig and cow slurry mixed with 3% Biokalk: 7.99 vs. 7.89 with Biokalk for pig slurry, 8.08 vs. 8.11 for cow slurry; the storage time with marble applied was not mentioned.

Hanssen et al. (1999) found slightly lower manure pH for cow slurry amended with marble or limestone after varying period of storage. Manure without marble or limestone had pH 7.49, whereas the treatments with marble varied between 7.38 and 7.46. Hanssen et al. (1999) also measured pH during 350 hours after addition of finely ground calcium carbonate from marble (no dispersion agent) or limestone from another industry than Omya Hustadmarmor AS (Figure 3). A pH drop was found for all treatments, even the control, during the first 25 hours. pH dropped from 7.7 to 7.6 in the control, and from about 7.6 to 7.5 in other treatments. Thereafter pH steadily increased, but kept somewhat lower in the treatments with marble. For ground limestone, there was hardly any difference from the control. The maximum difference between control and limed treatment was 0.3 pH units. This is likely what has caused Mo (2008) to conclude that pH is "typically" reduced by 0.3 pH units, as referred above. However, the results seem not to justify such a conclusion in general.

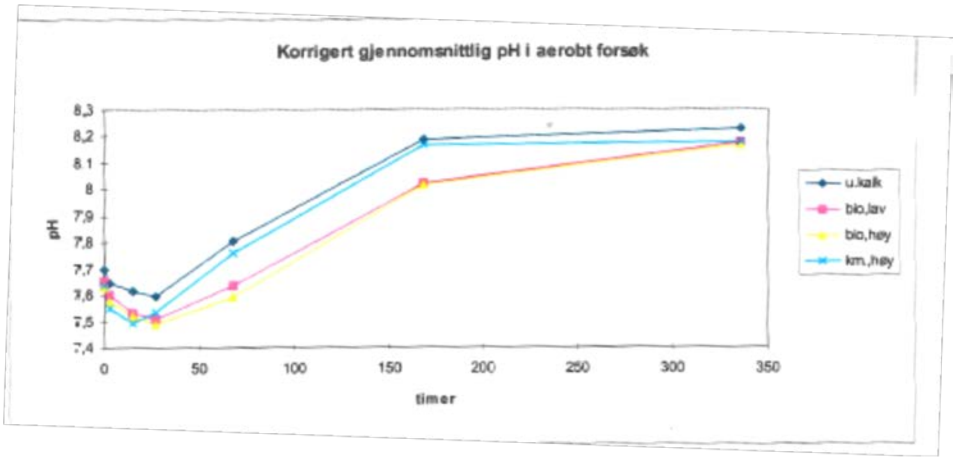


Figure 3. pH over time (totally 350 hours) in dairy cow slurry (control: "u. kalk", dark blue line), and slurry amended with 2.5 or 5% Biokalk ("bio, lav", pink line and "bio, høy" yellow line), or 4% limestone ("km", light blue line). From Hanssen et al., 1999.

Lignin can bind significant amounts of N, as shown by the traditional practice of covering compost windrows with peat. Ramirez et al. (1997) produced an ammonium-ligno-sulphonate, acting as a slow-releasing N fertilizer, with 13% N. Lignin also has a high affinity for calcium (Torre et al. 1992). This mechanism is more difficult to study than manure pH, and has so far only been proposed as a mechanism to explain better plant growth in old grassland, where soil fungi are likely present to digest manure lignin and release the fixed N (Mo 2008).

Studies of N emissions in field (Morken 2005) were only successful for pig slurry, and showed slightly *higher* N emissions from pig slurry with 3% Biokalk over 7 days, 25.5% compared with 24.5 % NH_3 emitted from manure with no marble (in proportion of NH_3 applied). 4.4 kg manure per m^2 was applied in this study, in wind tunnels with passive filters to record the NH_3.

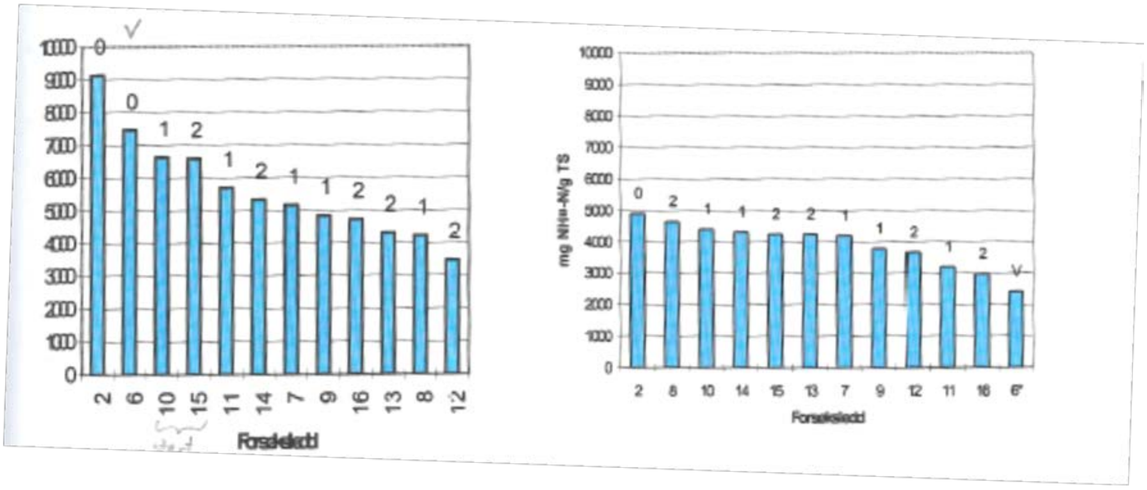


Figure 4. Ammonia emissions recorded by trapping NH_3 in acid, measured as mg NH_3-N per g dry matter of manure, in a continuously ventilated system (left) and periodically ventilated system (right). Treatment 2 = cow slurry without addition of marble, 6 = control + water, other treatments with different amounts and timing of marble addition. From Hanssen et al., 1999.

In laboratory, Hanssen et al. (1999) in initially found significant reductions in emissions of ammonia from cow slurry treated with marble or limestone, when the system was continuously ventilated. This ventilation made the manure dry out completely, which is a quite artificial situation, and which increases ammonia loss. When the system was ventilated for 15 minutes per 3 hours, the losses were reduced by about 50%. In this system, manure mixed with water had less ammonia loss than manure treatments with marble or limestone. In a follow-up experiment with Biokalk, dispersion agent included, losses were higher from slurry with Biokalk compared with no addition of marble. Hence, it is not possible to conclude from studies conducted until now, that addition of finely ground calcium carbonate to animal manure reduces N emissions during storage or spreading. Some studies have demonstrated such an effect, whereas other studies have not shown any effect.

Aim of the present study

The experiments in the present project were undertaken to study whether addition of finely ground marble (calcium carbonate) to digestate and manure could reduce N-emissions from digestate, and from animal manure in general. Specific objectives were 1) to measure the yields of perennial ley with digested and undigested slurry from dairy cows, with and without addition of marble. Increased yields with marble application would indicate reduced N-emissions. 2) to assess methods to measure NH_3 emissions in field. 3) to measure N-emissions from various digestates and animal manures with and without marble addition, and analyze pH, nutrient concentrations, dry matter, carbon contents etc. before and after addition of marble. 4) to compare calcium carbonate granules with slurry when mixed into digestate and manure, and assess benefits and drawbacks of mixing the granules with water before application. 5) to review existing literature, establish networks and provide a scientific basis for further proposal(s), and write such proposal(s). 6) to report the outcome of the experiments and disseminate the knowledge to relevant users.

Out of the specific aims, no. 4 was replaced by a more thorough study of objective 3, by agreement with the governing board. The remaining objectives have been achieved by the results presented in the present report. Initiatives to apply for further project funding should come from the industry partners.

Methods

Field experiment

A biogas plant was established at Tingvoll research farm in 2010, to produce biogas by anaerobic digestion of the slurry from the herd of 25 organically managed dairy cows. Concurrently, a long-term field experiment with four replicate blocks was established to study yields and soil characteristics by different manure application in perennial grass-clover ley. Low (L) and high (H) application levels of digested manure (D) and untreated manure (U) were compared, with a control treatment with no manure application. The fertilization comprises 11 (L) or 22 (H) kg total N per daa and year, equal to about 3 or 6 tons of slurry, applied with 2/3 in spring and 1/3 after the first cut of grass-clover. Two cuts per year have been recorded since 2011, with a season of re-establishing the ley in 2014. Within four replicate blocks, treatments were randomly distributed on experimental plots sized 3 m x 8 m. During 2011-14, two systems were compared; arable crops vs. perennial ley. The slurry was diluted by water to < 5 % dry matter (DM) and applied by hand, using 10-liter cans. The experimental soil is a loamy sand with high content of organic matter, pH 6.04 (2013), low status (< 4 mg 100 g^{-1} dry soil) of phosphorus (P) and very high status (>120 mg 100 g^{-1} dry soil) of acid-soluble potassium (K).

Since 2015, the arable system has been used for perennial ley as well, offering 8 replicate plots of each manure treatment DL, DH, UL, UH and Control. This allowed for testing addition of ground calcium carbonate, here called marble, to the slurry in half the replicate plots in 2016 (except the Control treatment). 2% (by weight) of ground calcium carbonate type **T509** (97% $CaCO_3$, about 150 mg sulphur (S) per kg DM, 90% <20 µm, d_{50}= 4 µm) was added to digested and undigested slurry which was stored in 1000 litre containers, on May 9 before application in field on May 10 and 11, 2016. Stirring was performed thoroughly after addition, and before and during application in field.

The same procedure was repeated on June 15 (adding marble to manure) and June 16 (application in field), after the first cut of grass-clover, which occurred on June 9. The second cut occurred on August 4. By harvest of the experimental plots, harvest plots sized 1.2 m x about 7 m were cut within each experimental plot, and the actual lengths recorded. Fresh yields of ley were recorded, and samples taken for determination of DM and botanical composition (grass, clover, weeds).

The manure application in spring 2016 occurred about two weeks later than normal, because of delayed transport of marble. The grass was already well growing at manure application, and we feared that the trampling during spreading might harm the plants. However, the weather during sampling was dry, which favoured N emissions, but protected the grass sward and plants from being smeared. In June, the weather conditions were again sunny during manure application. This might have increased the possibility to reveal effects on reduced N emissions from manure with marble.

The manure applied in May had been stored since spring 2014, in 1000 litre white plastic containers which were stored on the north side of a farm building and covered by an aluminium roof to avoid heating from sunlight. For application in June, fresh manure and fresh digested manure were collected from the cow house at Tingvoll farm. Manure nutrient concentrations were analysed at Eurofins, Sweden. Analyses from 2014 vs. 2016 showed a slight decrease in dry matter (DM); for undigested manure from 5.4 to 4.9%, for digested manure from 5.0 to 4.2%. The total N concentration was increased in the undigested manure, from 2.6 to 3.2 kg tot-N per ton manure. In the digested manure, the total N concentration had decreased from 3.1 to 2.2 kg per ton. This can be explained by a higher proportion of mineral N (NH_4^+) in digested manure in 2014; 68 vs. 61%. This had changed in 2016; and the proportion of mineral N was then 75% in undigested manure, and only 63% in digested manure.

In fresh manure from June 2016, corresponding values were 5.7 % DM for undigested manure and 3.5 for digested manure. Based on regression equations from 14 analyses of undigested, and 16 of digested manure from Tingvoll farm, all made by Eurofins, the total N content was estimated to 2.6 kg per ton for undigested and 2.3 for digested manure.

Statistically significant differences of yields between treatments were assessed by variance analysis and Tukey's test to compare average values, Minitab software.

Open container experiment

Four types of manure were studied in open 5 litre containers between October 17 and November 22, 2016, at Tingvoll. A room with one window and electric heating in the floor was used for the study. We aimed for temperatures relevant for storage of manure during Scandinavian conditions in spring and early summer. The temperature was recorded and was relatively stable. It varied between 9 and 15°C, with one drop to 4°C one weekend. The average temperature during the study was 12.6 °C.

The substrates studied were digested (CD) and non-digested dairy cow slurry (C) from Tingvoll farm, undigested pig slurry (P) from the farm of Stein Erik Meisingset, Tingvoll and liquid fraction of digestate (D) from Ecopro biogas plant, where main substrates are sewage sludge, source-separated organic household waste and fish sludge. The liquid fraction is produced by decanting the digestate, dividing it into two fractions with highly different DM. Solid digestate has about 26 % DM, whereas the liquid fraction has 0.5-5 % DM. Characteristics of the substrates are shown in Table 1. The cow manure contained only small amounts of bedding material, which was wood shavings. The pig slurry contained a significant amount of sawdust. The liquid fraction, further called digestate, was low in DM, but high in N.

Table 1. Characteristics and chemical composition of substrates used for studying N emissions from open containers. IL= Ignition loss, % of DM. NH_4-N and concentrations of total elements = g per kg fresh sample. pH measured at Tingvoll; other data from Agrilab, Sweden.

Substrate	DM, %	IL, %	pH	Tot-N	NH_4-N	Tot-C	Tot-Ca	Tot-S	Tot-P
Cow slurry (C)	3.8	78	7.3	1.85	0.90	18	0.9	0.17	0.28
Digested cow slurry (CD)	3.0	70	8.9	2.36	1.49	12	0.9	0.20	0.31
Pig slurry (P)	6.6	85	7.3	3.10	1.90	31	1.7	0.30	0.70
Digestate (D)	0.7	66	8.5	2.60	2.14	3	0.1	0.03	0.03

2500 g substrate was added to each bucket, followed by addition of dry ground calcium carbonate for treatments with marble. Two types of ground calcium carbonate (= marble) were compared; **T509 and T563**. T563 has the same chemical composition as T509, but a finer texture with 90% of particles < 2 µm (= clay fraction), d_{50} = 1 µm. For abbreviation of treatments, T509 was called "c" (coarse), and T563 was called "f" (fine).

For each substrate, we had two replicate buckets. The control was substrate with no addition of marble. For each type of marble, c and f, we compared 2 and 4 % addition (by weight), which comprised 50 or 100 g of ground carbonate. The five treatments were abbreviated as 0 (control), 2c, 4c, 2f and 4f, in addition to the substrate indication C, CD, P or D. As an example, P0, Pc2, Pc4, Pf2 and Pf4 comprise the five treatments for pig manure. Altogether, 40 buckets with substrate and two buckets with de-ionized water were included. The substrate buckets were numbered 1-42, and randomly arranged on a table. The buckets had a diameter of 21 cm. Mixing of marble and substrate occurred by stirring the mixture 100 rounds in each direction. Controls were also stirred. A separate stirring stick was placed in each bucket.

Samples for chemical analyses were prepared at the start of the experiment, by weighing 400 g substrate into separate bottles and adding 8 or 16 g of marble where relevant, and shaking the bottles well. All buckets had one corresponding sample. At the end of the experiment, samples for analysis were taken from each bucket after stirring. Samples were frozen immediately at 18°C, and transported over night to the laboratory (Agrilab, Uppsala, Sweden). Substrate samples were analysed for DM, ignition loss (= volatile solids, VS), total N, C, S, K, Mg, Ca, Na and P, and NH_4-N.

Substrate pH and redox potential was measured regularly (Picture 1), with hand-held electrodes from VWR. Before measurement, the bucket was well stirred (ca. 100 rounds in each direction). On the same dates, bucket weights were recorded to estimate evaporation. The weight loss varied between 18.5 % and 29 % of the initial weight of the substrate. No systematic variation between treatments or substrates occurred. The loss from buckets containing only water was 25%, which indicates that weight losses from decomposition of organic matter were small. It seemed to be somewhat higher losses from buckets placed in the corner positions of the table, and along the inner wall which the table stood up against.

Picture 1: Open containers filled with pig slurry, Ecopro digestate, cow slurry or digested cow slurry (left), to measure pH and red-ox potential over time (right) and to measure losses of N during six weeks with repeated stirring. Photos by Reidun Pommeresche.

Closed container experiment

Four 60 litre containers were filled with 50 litres of un-decanted digestate (about 5% DM) at Ecopro on January 11 2017. Thereafter, 1 kg of dry T509 (2%), 1 kg of dry T563 (2%) or 3 kg of dry T563 (6%) were added to the three containers marked T2, T3 and T4, and well mixed with the substrate. T1 was the control with no marble applied. The containers were closed, wrapped in isolating material and transported to SINTEF Energy Research, Trondheim. The digester temperature at Ecopro is 39-41 °C.

Picture 2. Containers with Ecopro digestate ready for collection of gas at SINTEF Energy Research, Trondheim. Photo by Roger Khalil.

The containers were attached to 10 litre gas collection bags (Picture 2), placed on heating pads and then isolated. Room temperature was about 21°C.

Once the gas bags were fully inflated, they were put inside a heating chamber which maintained a temperature of 60 °C, for at least one hour. This was done to prevent ammonia from condensing inside the gas bag. During gas measurement, the gas bag was attached to a heated hose while still inside the heating chamber and the FTIR (Fourier Transform Infrared Spectroscopy) was commanded to start. This triggered the FTIR pump to start taking the sample to the gas cell. All components of the FTIR which consist of a heated hose, a gas conditioning chamber and the FTIR were kept at a constant temperature of 180 °C. It was possible to take 2 – 3 consecutive measurements before the gas bag was fully emptied. An average concentration for each gas bag is presented in this report. After the first measurements, it was discovered that the methane concentration was well above the measurement range of the FTIR.

A gas chromatographer (GC) was employed to better quantify the major species. The GC has its own pump and was placed just after the FTIR. The FTIR is of type Gasmet DX-4000 and was pre-calibrated for the quantification of H_2O, CO_2, CO, NO, N_2O, NO_2, SO_2, NH_3, HCl, HF, CH_4, C_2H_6, C_2H_4, C_3H_8, C_6H_{14}, CHOH and HCN. The GC is of type Varian CP-4900, has two separate columns, each attached to a thermal conductivity detector. The GC can measure O_2, N_2, H_2, CO, CO_2 and CH_4 in addition to many other compounds that are not relevant for this study.

Results and discussion

Field experiment

Significant yield differences were achieved in the field experiment at both harvest dates, as shown in Picture 3.

Picture 3. The "SoilEffects" field experiment in Tingvoll during the first cut of experimental plots with perennial ley, June 9 2016. The experiment was used in 2016 to study the effect of mixing finely ground calcium carbonate into the manure about 24 hours before field application. Photo by Anne de Boer, NIBIO.

The yield levels were increased by application of slurry, as shown for the second cut in Figure 5. More slurry gave higher yields. As was also found in other years of this field experiment since 2011, there was no significant difference in ley yields between digested and undigested slurry. Addition of marble to the manure increased the yields significantly for this cut (p= 0.035), but not for the first cut. The average yields at second cut with marble applied were 344 kg DM per daa, vs. 324 for treatments without marble. For the first cut, the corresponding values were 393 and 381 kg DM per daa. Even if the general increase with addition of marble was statistically significant, the difference between treatments with and without marble added for each type of manure were not (interval plot lines overlapping in Figure 5).

As far as we are informed, this is the first time that increased yields by marble addition to the manure have been demonstrated in a field experiment in Norway. The fact that yields were higher with marble additions both for digested and undigested manure in the second cut, strengthens the hypothesis that this result is related to the application of marble.

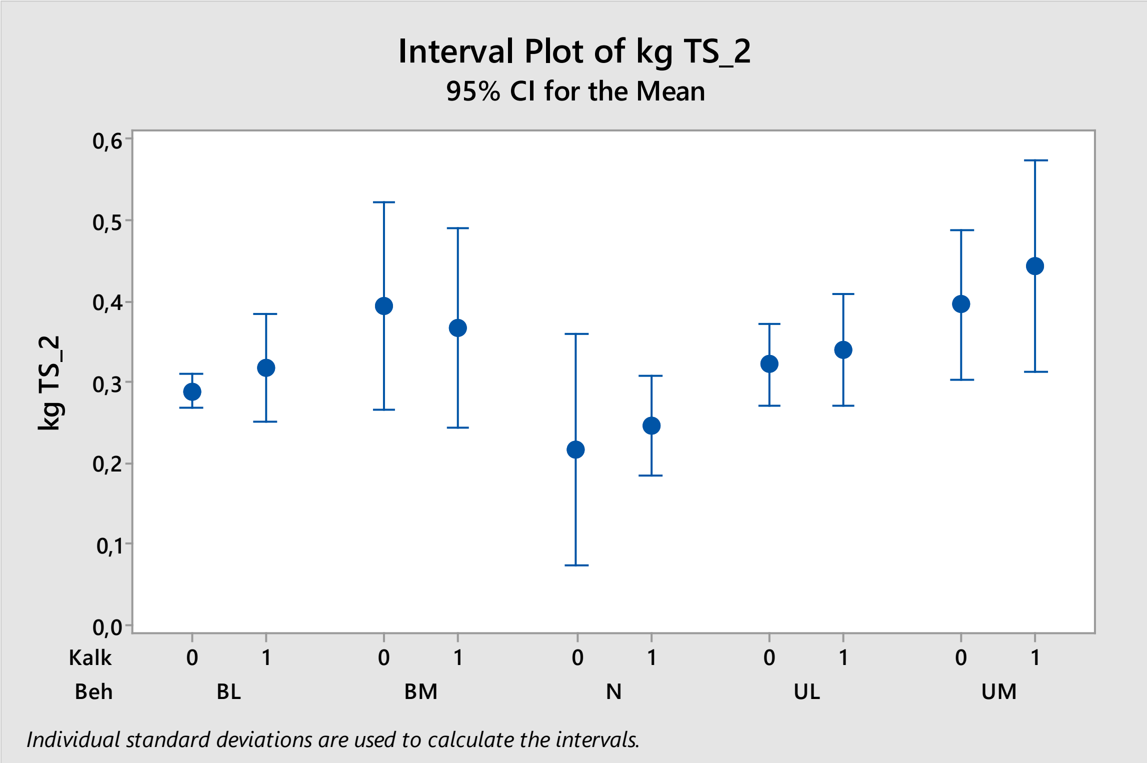


Figure 5. Second cut yields of perennial grass-clover ley established 2014 (kg DM per m^2), with application of undigested (U) or digested (B) dairy cow slurry in low (L) or high (M) amounts (3 or 6 tons per daa and year). Treatments where finely ground calcium carbonate was applied are shown by Kalk = 0 or 1. N=Control (no manure); please observe that no marble was applied to this treatment.

One explanation for no significant effects in the first cut may be that the manure was applied quite late, only 32 days before harvest. For the second harvest, 49 days passed between manure application and harvest. Another explanation is that the manure used for application in field in spring had been stored in closed 1000 litre containers since spring 2014. On average for three containers of each manure type (digested and undigested cow slurry) analysed in 2014, and two containers analysed in 2016, the DM decreased from 5.4 to 4.9% for undigested slurry, and from 5.0 to 4.2% for digested slurry. This is to be expected with manure storage over time. The content of total N increased in the undigested slurry, from 2.6 to 3.1 kg per ton, whereas it decreased in the digested slurry, from 3.1 to 2.2 kg per ton. Hence, treatments with undigested slurry were "favoured" compared with digested slurry at spring application in 2016. Another issue related to N was that the proportion of mineral N (ammonium) increased in undigested slurry during storage, from 61 to 75%, whereas it decreased in digested slurry, from 68 to 63 % of total N. Somewhat more or less N applied than foreseen by the experimental plan should not influence differently on the effect of marble additions to the manure. However, the different patterns of changes in total N content and proportion of mineral N indicates that the storage had affected the manure characteristics so that this manure was less representative than the manure used to fertilize the second harvest of grass.

The water used to dilute the slurry during field application has a relatively high pH due to ground marble being used for sanitation. pH in diluted and non-diluted slurry, with and without addition of marble, is shown in Table 2 for manure samples applied in field on June 15-16, 2016. In field, only diluted slurry was applied. Samples for measuring pH in undiluted slurry were taken from the storage containers.

Table 2. Effect on manure pH of addition of marble (2% by weight) and water.

Type of addition	pH digested slurry	pH undigested slurry
No additions	7.84	7.49
Water	7.94	7.63
Marble	7.80	7.54
Water and marble	7.89	7.64

As can be seen, the pH increased by 0.1-0.2 units by water dilution, whereas the effect on manure pH from marble addition was small and inconsistent. The differences in pH by marble addition are not large enough to explain the yield increase. Hence, the reasons for a possible reduction in N emissions, and thereby increased N uptake by plants related to higher yields, remain unexplained from this field experiment.

Open container experiment

Substrate pH

Effects of pH were generally small. To better reveal the differences between the various applications of calcium carbonate, the y-axis varies between the figures of the substrates.

pH increased steadily in the manure substrates where no marble was applied (Figure 6). For Ecopro digestate, the pH increase was small compared with manure substrates. The increase was largest in digested cow slurry, and pig slurry. In de-ionized water, the pH initially increased, then it decreased to about pH 8.5 (Figure 5). The losses of ammonia lead to a decrease in pH, and do not explain the pH increase.

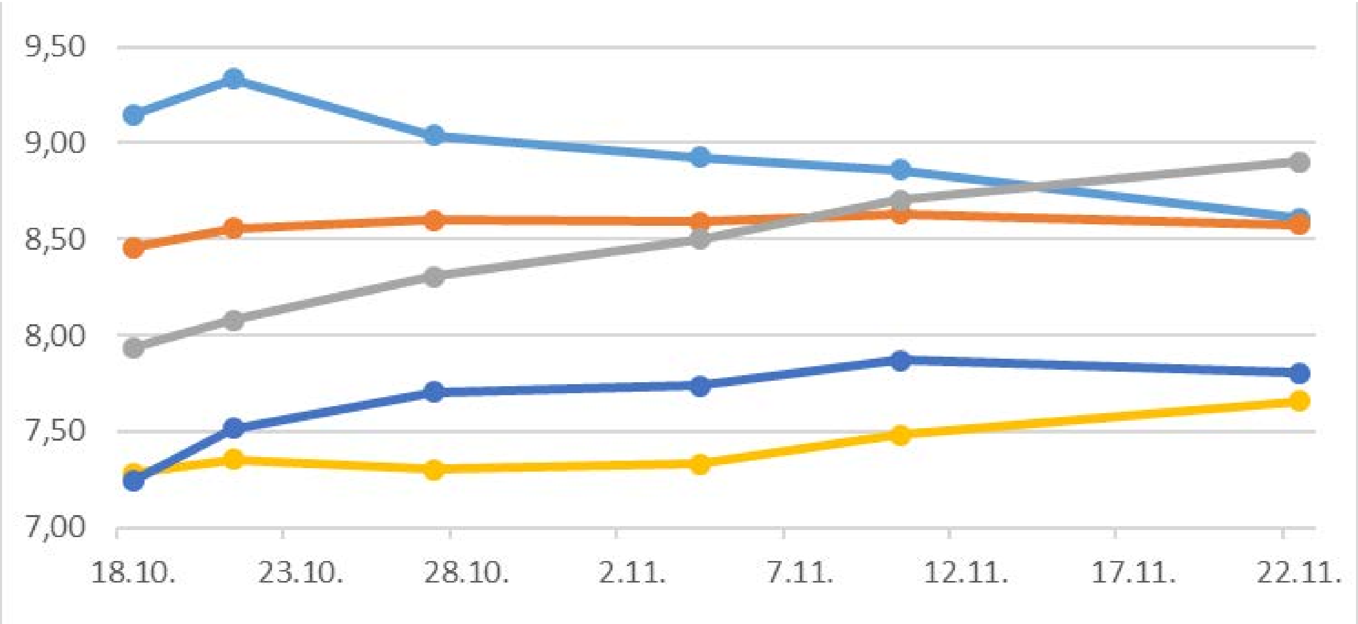


Figure 6. Values of pH during storage of cow slurry (yellow line), pig slurry (dark blue line), digested cow slurry (grey line), digestate from Ecopro (orange line) and de-ionized water (light blue line).

The effect of marble addition on substrate pH varied somewhat between the substrates. For Ecopro digestate, the pH decreased immediately about 0.05 pH units with addition of marble (Figure 7). The pH then increased for some days, but kept lower with marble than in the control. With 4% marble addition, the main trend over six weeks was a pH decrease, whereas with 2% it was quite stable. The pH decreased more with more finely ground calcium carbonate, and with larger addition. This substrate has little organic matter, but high concentrations of ammonium. It would be interesting to study in more detail if pH in Ecopro reject water (digestate) may be regulated by addition of ground calcium carbonate, possibly combined with dispersion agent.

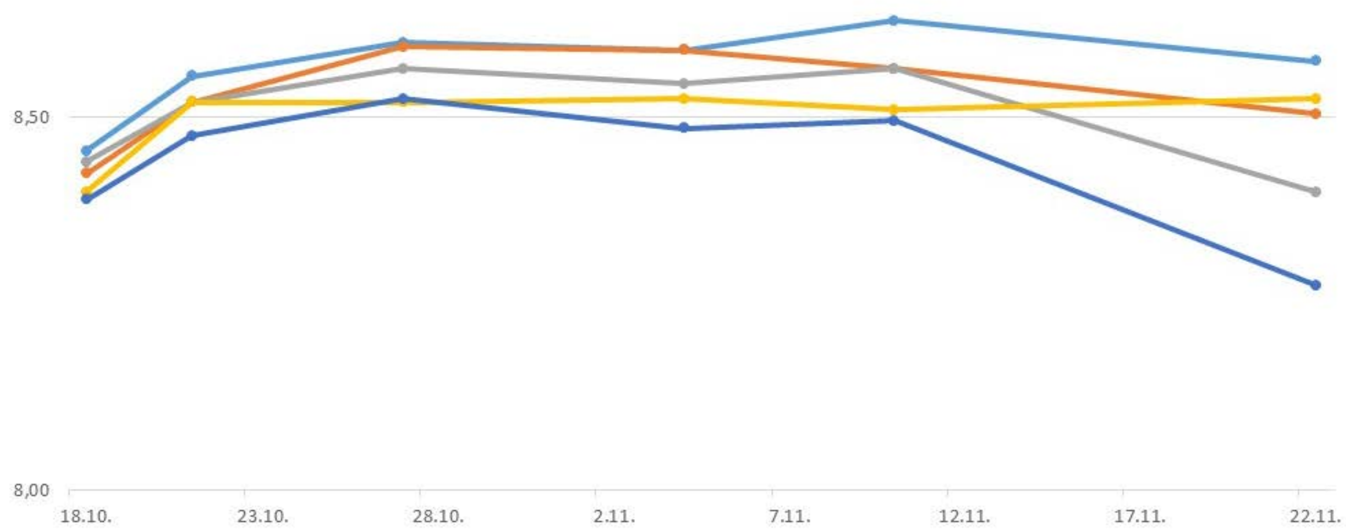


Figure 7. Values of pH during storage of digestate from Ecopro with no marble addition (light blue line), addition of 2% coarser marble T509 (orange line), 4% T509 (grey line), 2% very fine marble T563(yellow line) and 4% T563 (dark blue line).

In digested cow slurry (Figure 8), pH was reduced by about 0.3-0.4 units with addition of the finest marble, and this effect was quite stable during the study. However, the pH decrease by marble could not counteract the general pH increase over time.

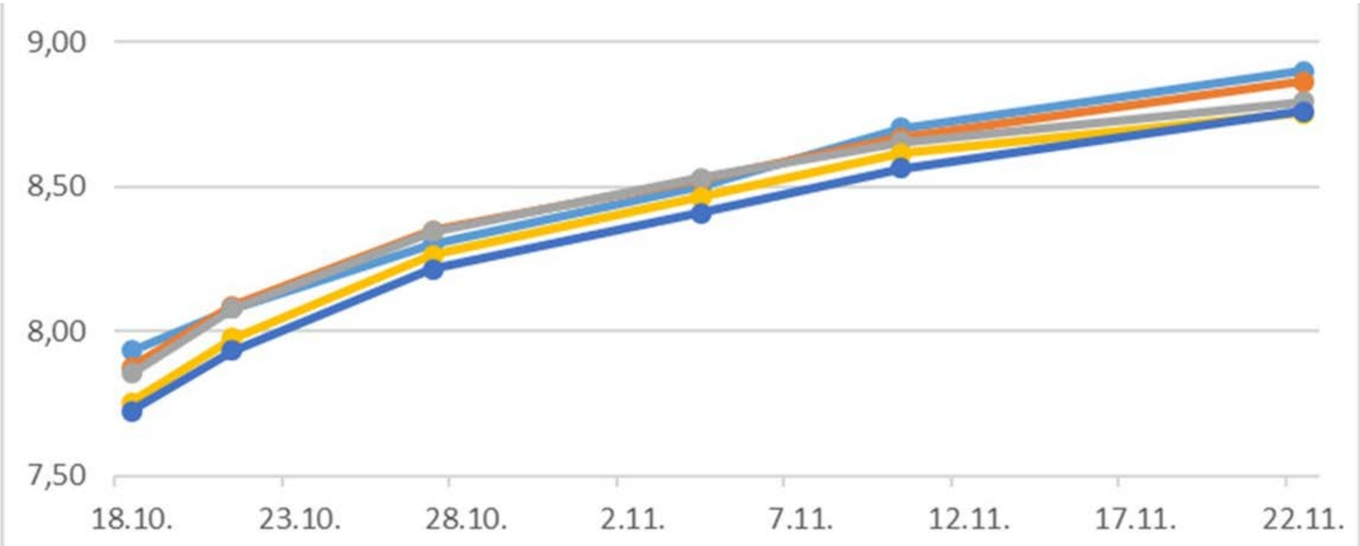


Figure 8. Values of pH during storage of digested cow slurry with no marble addition (light blue line), addition of 2% coarser marble T509 (orange line), 4% T509 (grey line), 2% very fine marble T563 (yellow line) and 4% T563 (dark blue line).

For pig slurry (Figure 9), addition of marble had almost no effect on the pH.

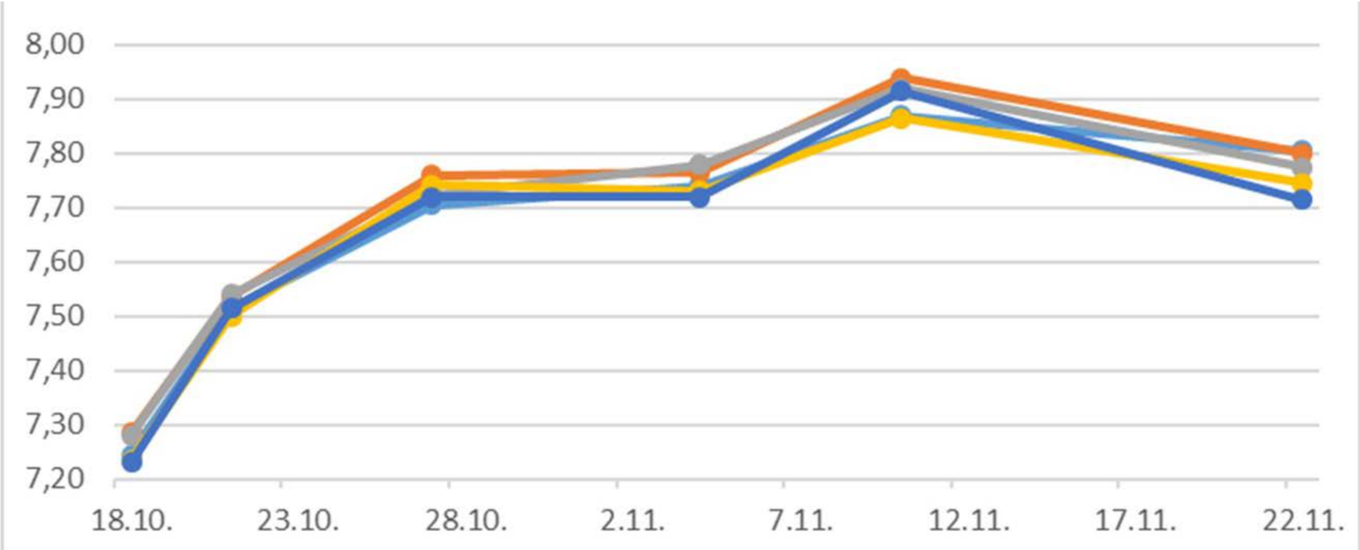


Figure 9. Values of pH during storage of pig slurry with no marble addition (light blue line), addition of 2% coarser marble T509 (orange line), 4% T509 (grey line), 2% very fine marble T563 (yellow line) and 4% T563 (dark blue line).

For undigested cow slurry (Figure 10), marble addition had no clear effect on manure pH.

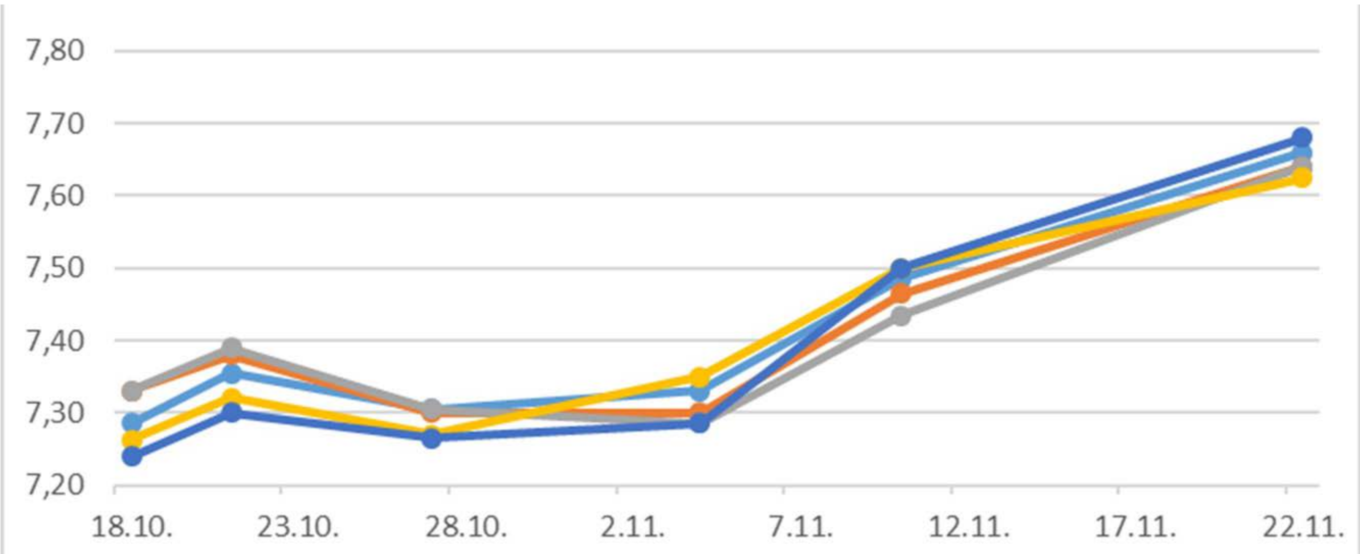


Figure 10. Values of pH during storage of cow slurry with no marble addition (light blue line), addition of 2% coarser marble T509 (orange line), 4% T509 (grey line), 2% very fine marble T563 (yellow line) and 4% T563 (dark blue line).

For de-ionized water mixed with finely ground calcium carbonate in small batches on November 1 and kept open in the same room as the manure buckets, pH was about 10 immediately after addition of the marble. The samples were stirred well before pH measurement on totally 8 dates. After 3 days, pH was 9.25 with marble and 9.20 in the control (water only). On November 22, pH was 8.6-7.7 in all treatments. This shows that the marble does not affect the pH of de-ionized water significantly, over periods longer than a week.

Red-ox potential

The redox potential (pE) is commonly studied in soil, or composts, to inform about the aeration characteristics. It may be measured very easily in liquids by a hand-held electrode. The number -100 mV is used as a limit between anaerobic and aerobic conditions. Degradation of organic matter is a form of oxidation, and makes the red-ox potential less negative. In a Canadian study of bacteria in pig slurry (Leung & Topp 2001), the red-ox potential was -190 mV in unaerated slurry with pH 8.0 and DM 0.6%. After 49 days of sealed storage or continuous aeration, the red-ox potential was -180 mV with anaerobic conditions (pH 8.1), and +280 mV with aerobic conditions (pH 9.5). Positive red-ox values were found already after 2 days (+170 mV, pH 9.3). Three other studies are referred in Weinfurtner (2011), and show that pig slurry commonly has a red-ox potential of about -350 mV, with some seasonal variation. Hjorth et al. (2012) found the same value for cow slurry, and about -400 mV for pig slurry. These values are slightly lower than the levels found here, which were a bit lower in cow slurry than the other substrates (Figure 11). The differences between treatments with and without marble were small, and inconsistent. The general trend was decreased (more negative) red-ox potentials over time. However, our buckets were not continuously stirred. The red-ox values were recorded immediately after thorough stirring, and this has likely affected the values. Even if this effect should be similar for all treatments for each substrate, the values should be interpreted with care.

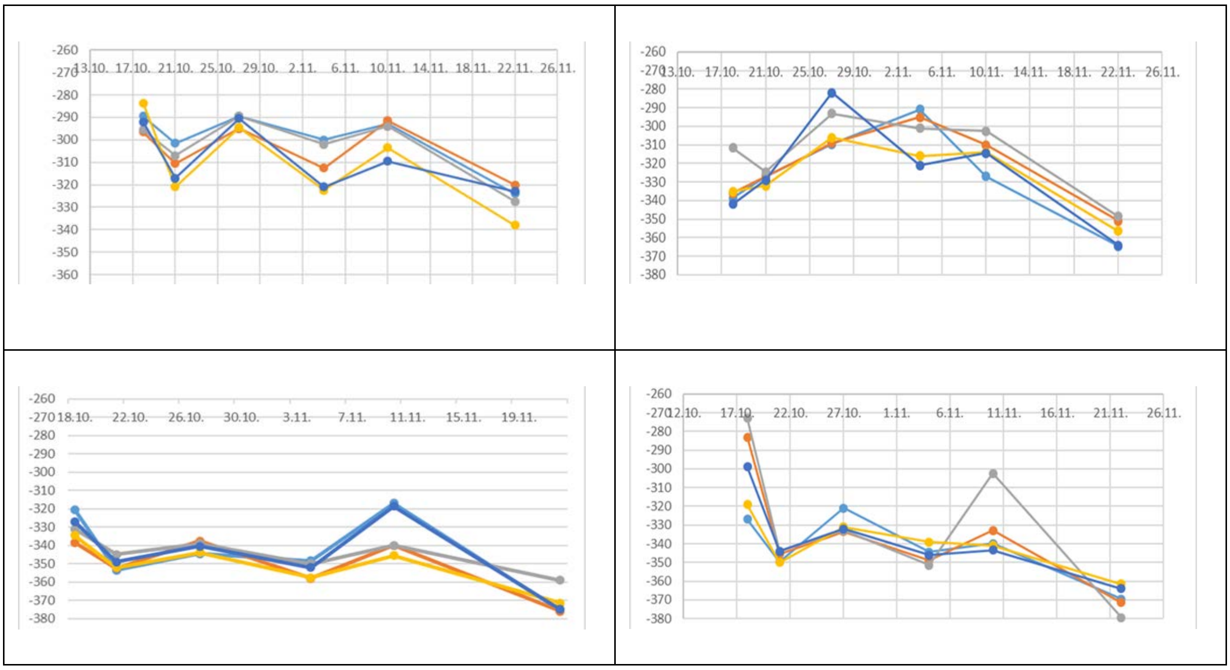
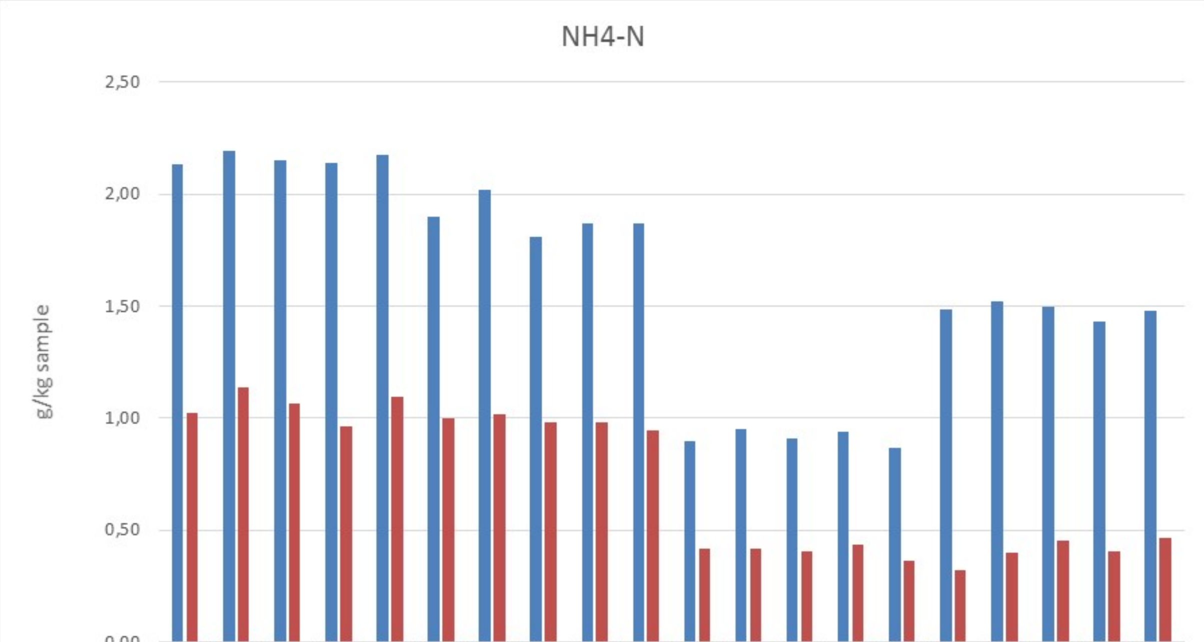


Figure 11. Red-ox potentials in Ecopro digestate (top left), pig slurry (top right), digested cow slurry (bottom left) and undigested cow slurry (bottom right). For all diagrams, control = light blue, 2% coarser marble T509 = orange, 4% T509 = grey, 2% very fine marble T563 = yellow, and 4% T563 = dark blue line.

N emissions

Emissions of N were not measured directly in this study. However, conditions supported N emissions, with quite high temperatures and repeated stirring. The difference in ammonium contents, concentrations or amounts per container in/of the substrates from the beginning to the end of the experiment is a good indicator of the actual N emissions. As shown in Figure 12 for ammonium contents (g per kg substrate), emissions were significant with the conditions provided. About 50% of the ammonium was lost from Ecopro digestate, pig manure and cow slurry digestate, whereas for undigested cow slurry, about 70% of the initial ammonium was emitted. Since the dry matter (DM) content of the substrates may change during a study of organic matter, we have also shown these data as concentrations (Figure 13). Since addition of marble increased the DM content significantly, the DM value of control treatments were used to calculate the proportions of ammonia in the treatments with marble applied. For the ammonia concentration (% of substrate DM), again no effect was found that added marble reduced N emissions.

The third possibility of calculation was the N balance from start to end. Since there was some variation in evaporation between the containers, this might have affected the N loss. This balance is shown for total N in Figure 14. The losses of total N were smaller than found for ammonium. On average for all treatments, losses of total N per container were 18% from digested cow slurry, 28 % from pig slurry, 36% from undigested cow slurry and 42% from Ecopro digestate. For digested cow slurry, the N loss was slightly lower with addition of marble (Figure 12), and pig slurry showed some of the same tendency. A variance analysis was conducted separately for the CD samples, which was the substrate showing the most consistent trend. The effect of marble addition (control, c or f) was not statistically significant for total N ($p= 0.19$) or ammonium ($p= 0.21$). Interval plots are shown as Figure 15. When the vertical lines overlap (in the vertical direction), the differences between treatments are not significantly valid. Hence, irrespective of assessment method, the effect of addition of marble was insignificant. Still, it is interesting that digested cow slurry showed a consistent pH decrease as well as a trend towards decreased N emissions with added marble.



	D0	Dc2	Dc4	Df2	Df4	P0	Pc2	Pc4	Pf2	Pf4	C0	Cc2	Cc4	Cf2	Cf4	CD0	CDc2	CDc4	CDf2	CDf4
NH4-Nstart	2,14	2,19	2,15	2,14	2,17	1,90	2,02	1,81	1,87	1,87	0,90	0,95	0,91	0,94	0,87	1,49	1,52	1,50	1,43	1,48
NH4-Nend	1,02	1,14	1,07	0,96	1,10	1,00	1,02	0,98	0,98	0,95	0,42	0,42	0,40	0,44	0,36	0,32	0,40	0,46	0,41	0,47

Figure 12. Ammonium contents (g per kg substrate) in substrates before and after six weeks of storage at about 12 °C with repeated stirring. Substrates are indicated by large letters to the left of the treatment abbreviation. D indicates Digestate from Ecopro, P indicates Pig manure, C indicates undigested Cow slurry, CD indicates Cow slurry which is Digested. Additions of calcium carbonate are indicated by small letters in the middle of the abbreviations, where c indicates somewhat more coarse material (T 509), and f indicates finer material (T563). Amounts of additions are indicated as numbers, where 0 = no addition (control), 2 = 2% and 4 = 4% by weight.

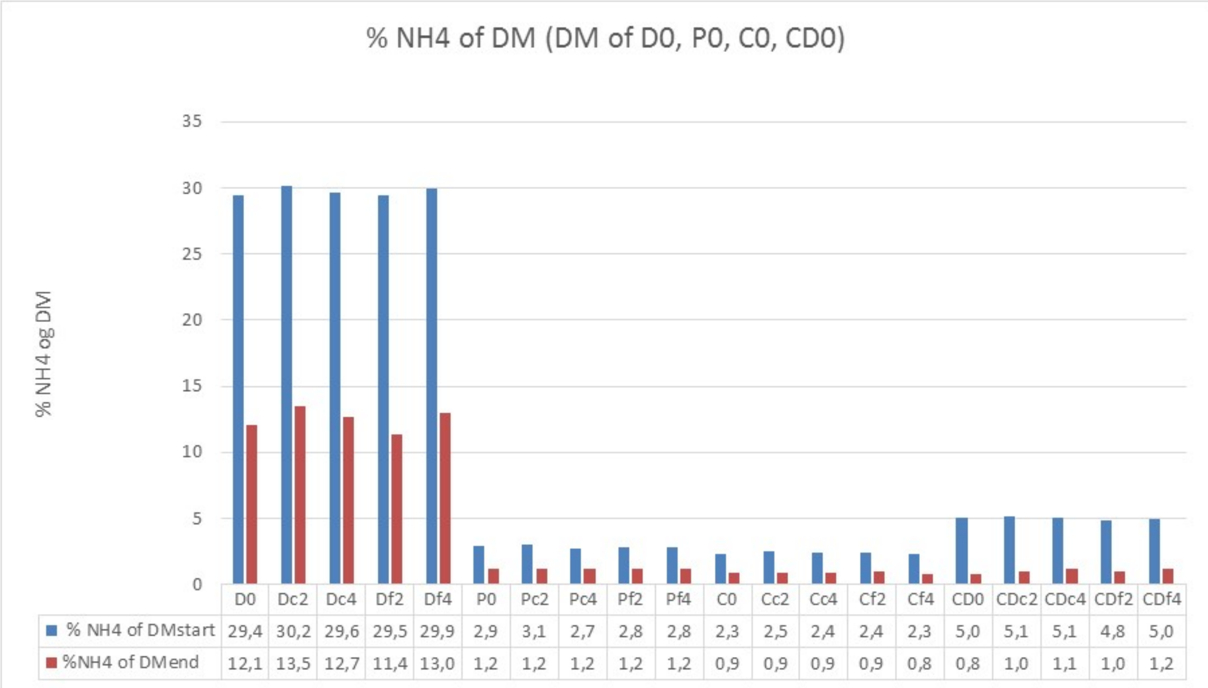


Figure 13. Ammonium concentrations (% of dry matter) in substrates before and after six weeks of storage at about 12 °C with repeated stirring. Treatment abbreviations are explained in Figure 12.

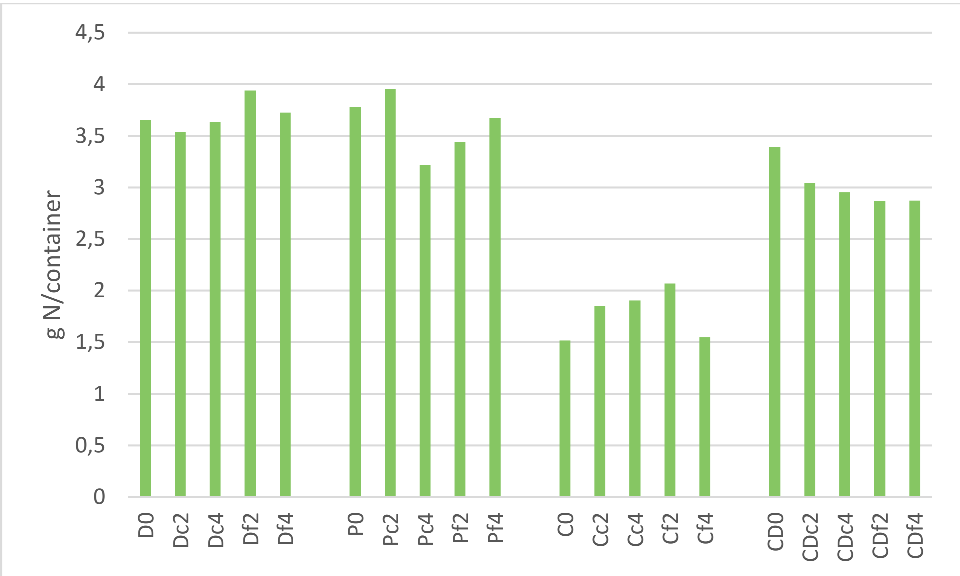


Figure 14. Losses of total N (g per container) from start to end of the experiment. Treatment abbreviations are explained in Figure 12.

The conditions in this study were set to make sure we achieved detectable differences in ammonium concentrations. However, no farmer would stir the manure storage repeatedly during summer, for good reasons, as demonstrated here. Hence, the conditions are not much representative for a real farm situation. It would have been interesting to expand this study by a short-time study of N emissions that were more comparable to manure application in field, e.g. by measuring manure ammonium concentrations before and after storing the manure for 1-2 days in a thin layer. Possibly, such a design could have been more useful to reveal a possible effect of marble on N emissions.

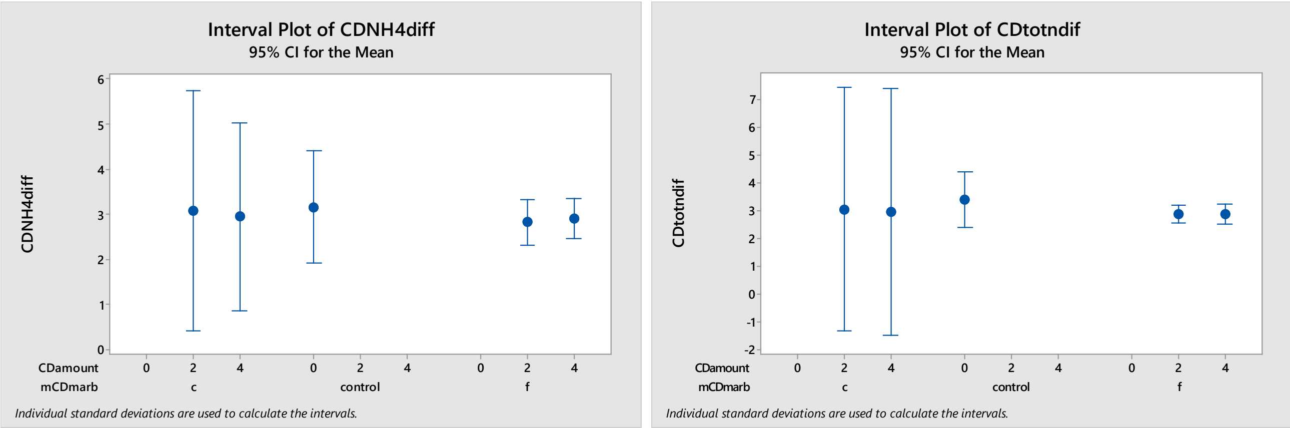
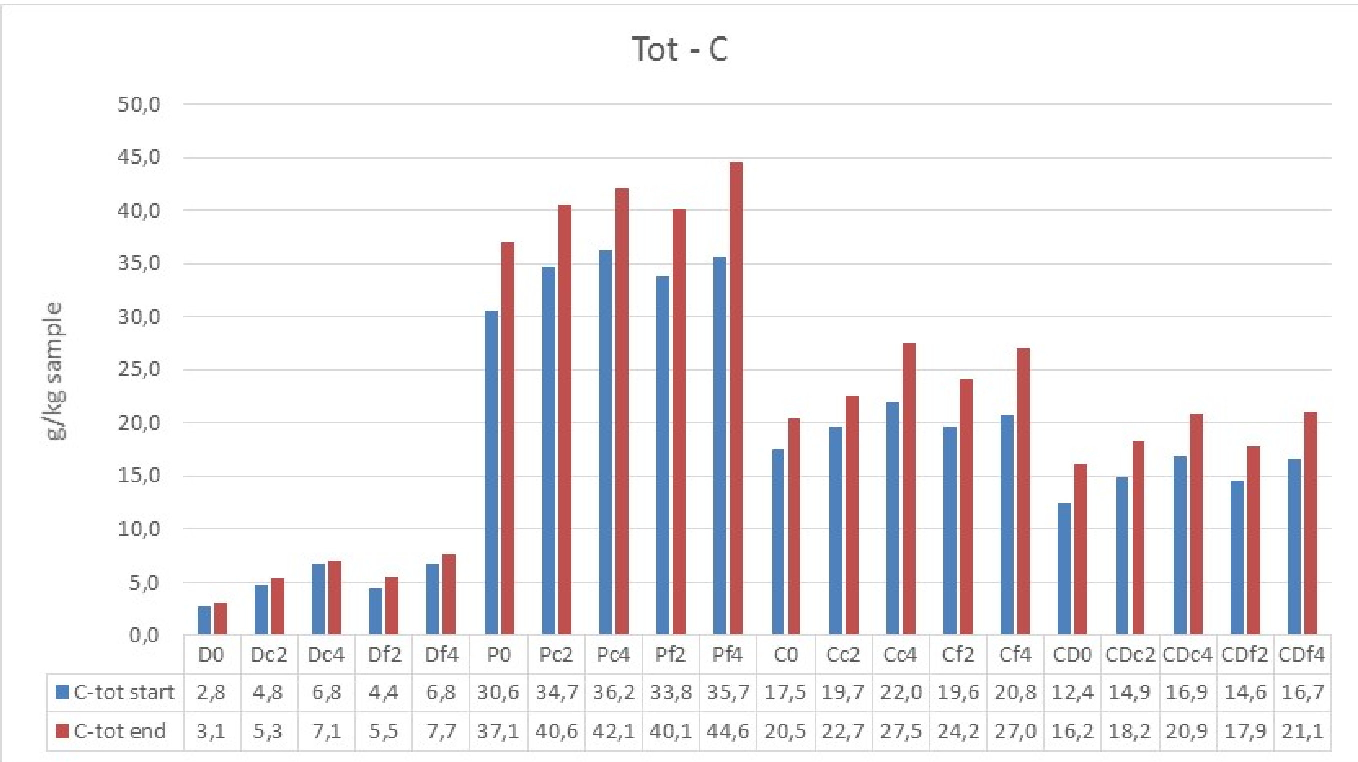


Figure 15. Interval plots with 95% confidence intervals for two replicate samples per treatment of digested cow slurry (CD) without (control) or with addition of coarse or fine ground calcium carbonate (c, f) as 2 or 4 % (by weight) of the substrate. Left side: Ammonium loss, right side: total N loss (start minus end values).

Carbon loss

During storage and stirring of manure, gas is emitted not only as ammonia, but also as various carbon-containing gases such as methane and carbon dioxide. In this study, the observed weight reduction of 20-30% seemed to be mostly due to water evaporation, since the concentration of carbon slightly increased during the study (Figure 16). Some carbon was added to the substrates with the marble, which explains higher bars for these treatments. No losses of carbon indicate that biological decomposition was not pronounced with the conditions provided here.



	D0	Dc2	Dc4	Df2	Df4	P0	Pc2	Pc4	Pf2	Pf4	C0	Cc2	Cc4	Cf2	Cf4	CD0	CDc2	CDc4	CDf2	CDf4
C-tot start	2,8	4,8	6,8	4,4	6,8	30,6	34,7	36,2	33,8	35,7	17,5	19,7	22,0	19,6	20,8	12,4	14,9	16,9	14,6	16,7
C-tot end	3,1	5,3	7,1	5,5	7,7	37,1	40,6	42,1	40,1	44,6	20,5	22,7	27,5	24,2	27,0	16,2	18,2	20,9	17,9	21,1

Figure 16. Total carbon concentrations (g per kg substrate) in substrates before and after six weeks of storage at about 12 °C with repeated stirring. Treatment abbreviations are explained in Figure 12.

Similar to N, sulphur may be lost by emissions of gases such as dihydrogen sulphide, H_2S. Some H_2S was lost from digested cow manure (Figure 17), but not from the other substrates. The applied marble contains about 150 mg S per kg DM, which is reflected in increased S concentrations in the marble treatments.

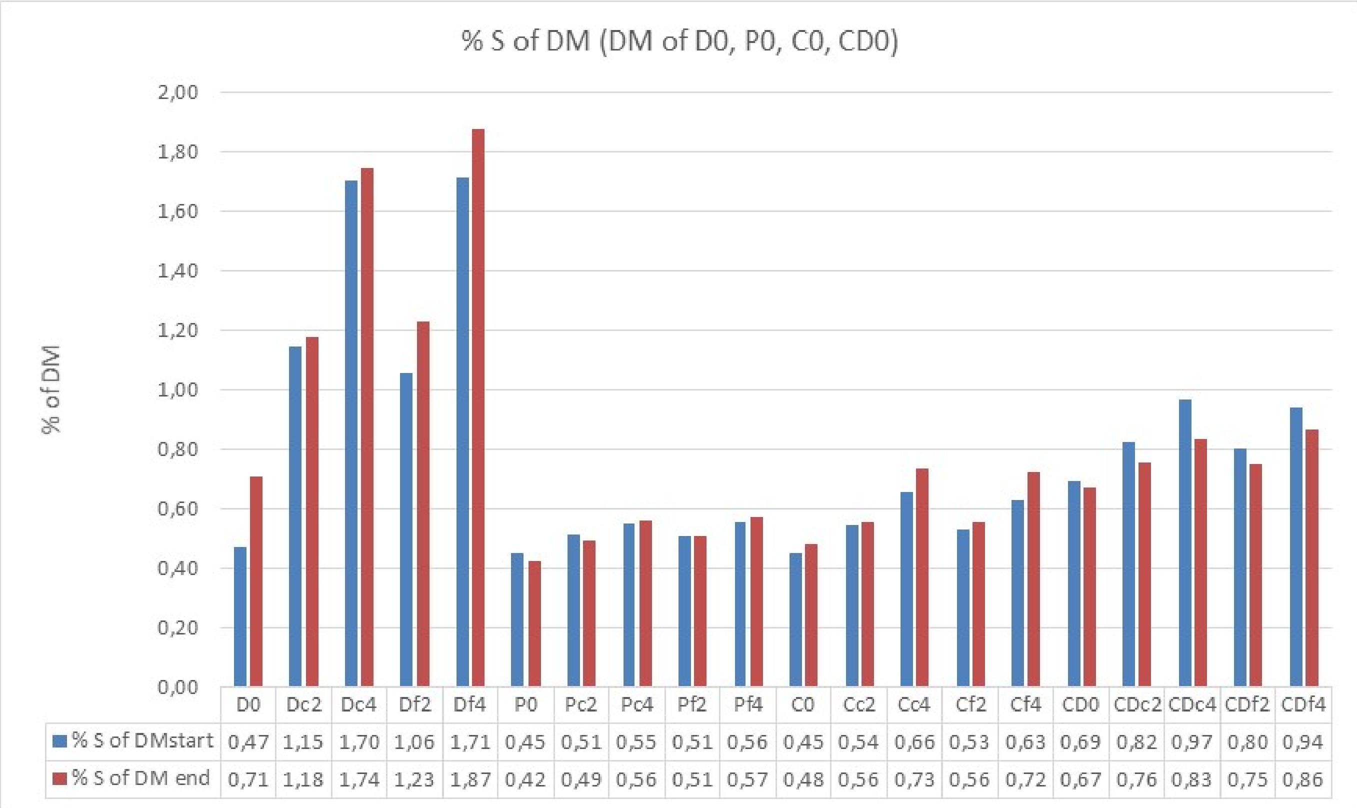


Figure 17. Total sulphur concentrations (% of substrate DM) in substrates before and after six weeks of storage at about 12 °C with repeated stirring. Treatment abbreviations are explained in Figure 12.

Closed container experiment

The barrel T4 with 6% marble T563 was the first to produce gas in the collection bags. This occurred already one day after the barrels were received on January 11. In total 11 gas bags were generated from T4, which corresponds to a total gas amount of 110 litres. Only one more barrel, T2 with 2% of T509, produced gas in the collection bags during the test period. This occurred on January 22, and totally 3 bags where produced. Due to leakage during the quantification of the third bag, only two gas measurements were performed from T2. The leakage was detected due to the abnormally high nitrogen concentration compared with the two other measurements. No gas was generated from T1 (control, no marble) or T3 (2% of T563).

The concentrations of H_2O, CO_2 and NH_3 (a), CH_4 and NO (b) are shown for T4 in Figure 18, and for T2 in Figure 19. For T4, the CO_2 concentration was initially about 10%, rapidly increasing to 25 % for about a week, before it dropped again. Ammonia was stable at 10 ppm for about 10 days, then rapidly increased to approximately 33 ppm before it dropped again. Methane comprised the largest gas volume, starting at 52% and increasing gradually to 82% (Figure 15b). The concentration with NO also increased steadily, from 24 to about 130 ppm at the end of the study. For T2, less trends can be described due to few measurements. The concentrations that were measured are comparable to those from T4.

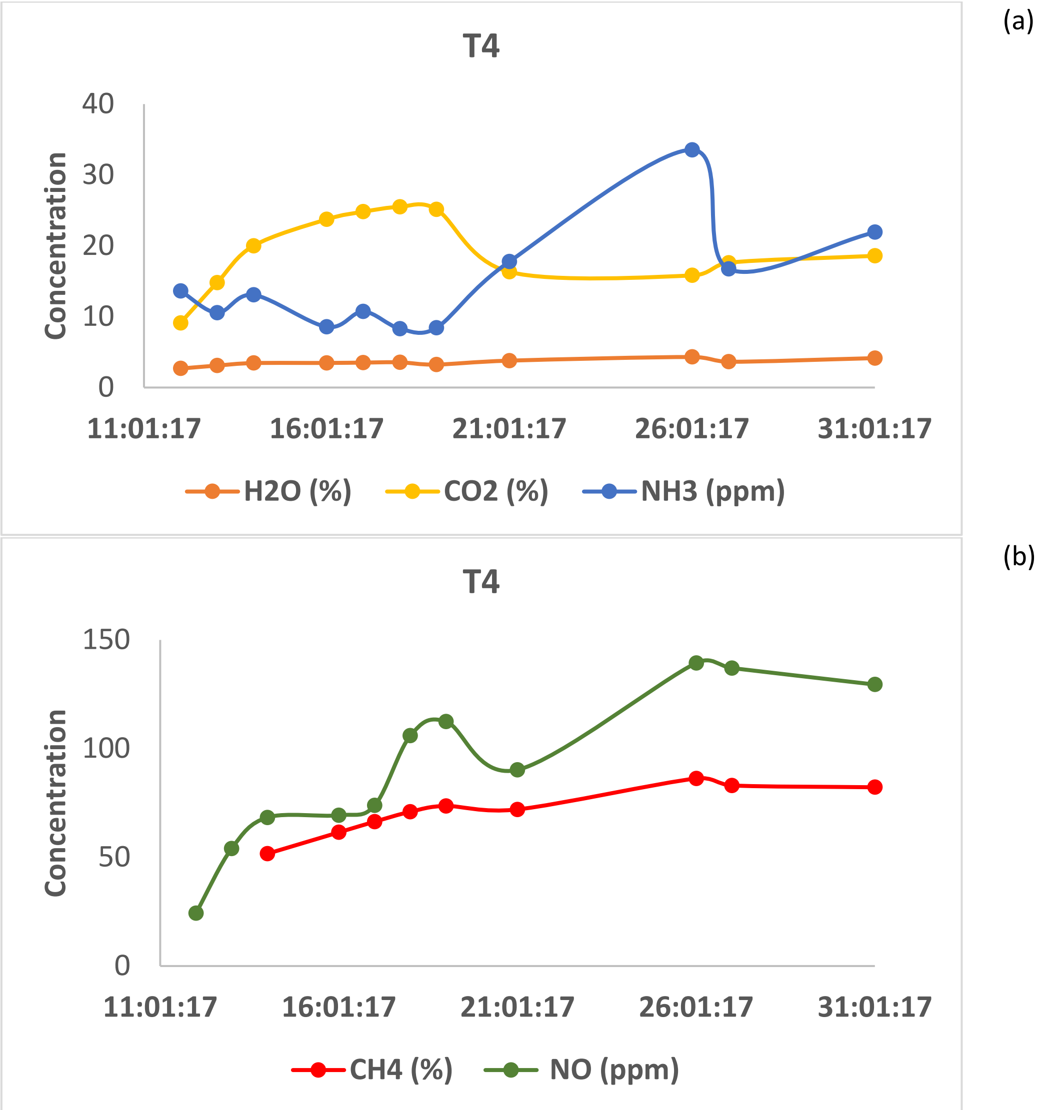
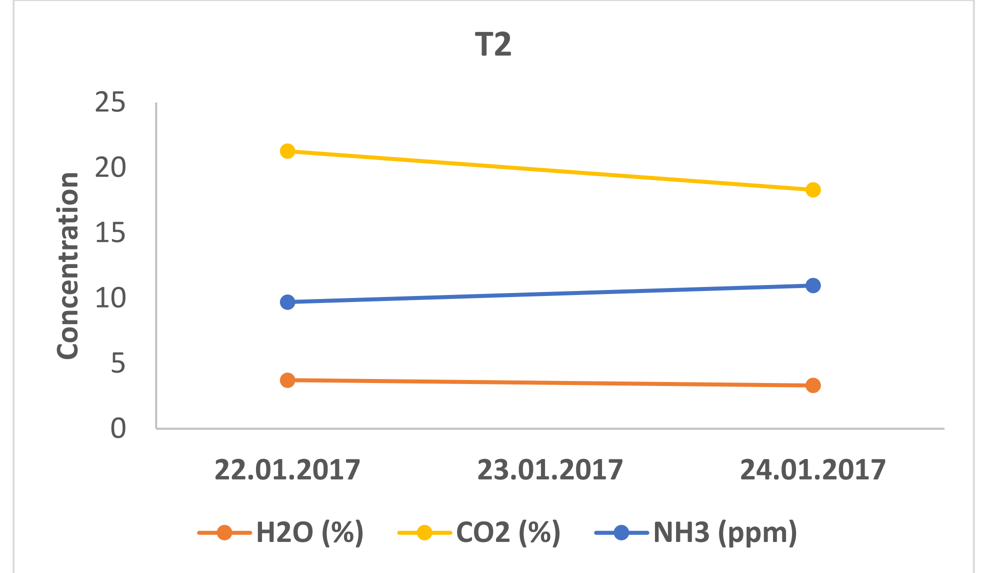
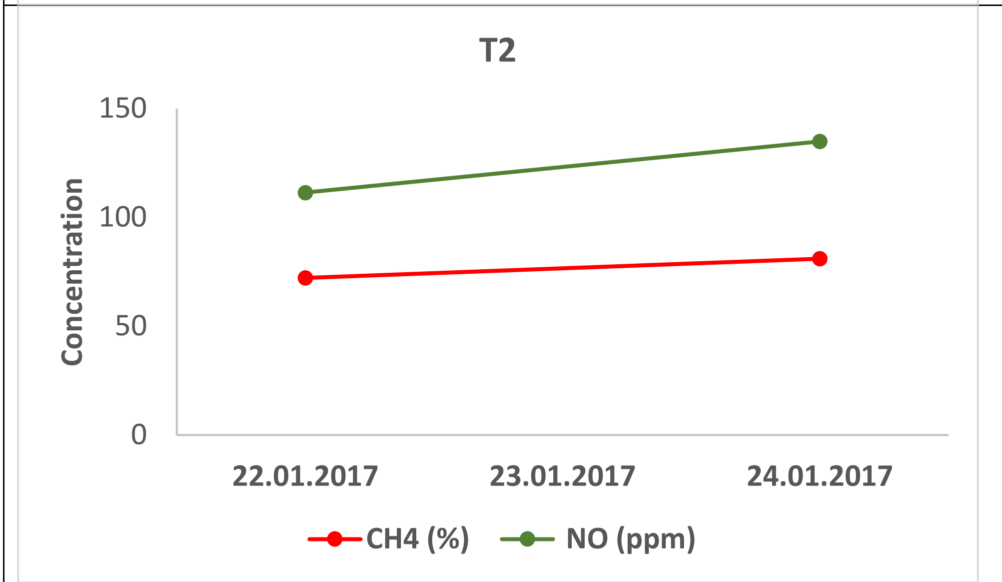


Figure 18. Gas concentrations (% or ppm as indicated for each gas in the legend) in collection bags attached to barrel T4, with 6% calcium carbonate T563.



(a)



(b)

Figure 19. Gas concentrations (% or ppm as indicated for each gas in the legend) in collection bags attached to barrel T2, with 2% ground calcium carbonate T509.

Conclusions and recommendations

Addition of 2% finely ground calcium carbonate from marble to digested and undigested dairy cow slurry increased yields of perennial ley by about 6% in one harvest in 2016. The pH level of animal manures was neglectably affected by this addition. Hence, pH decrease cannot be the cause of the yield increase. Further studies are required to reveal the explanation for higher yields in this case, but as far as we are informed, this is the first time that a statistically higher yield with addition of finely ground calcium carbonate has been found in a Norwegian study. Even so, the former insignificant effects on ley yields in former pot and field studies referred in this report call for carefulness in the interpretation of the yield effect found here, and we recommend that further studies are conducted to clarify whether farmers may expect increased yields with addition of finely ground calcium carbonate.

With addition of marble, pH decreased 0.2-0.3 units in digestate from Ecopro. This digestate has small amounts of organic matter, but high concentrations of ammonium. It could be interesting to study in more detail how addition of finely ground calcium carbonate, with and without dispersion agent, affects the pH of this substrate. If a significant pH decrease can be achieved, this may be an easy way to reduce N emissions from this nutrient rich substrate, and thereby increase its economic value.

Addition of finely ground calcium carbonate did not affect N emissions during storage at about 12 °C with repeated stirring. This is valid for results assessed as ammonium or total-N before and after storage, both as concentrations (g per kg of substrate DM), contents (g per kg of substrate) and amounts per container (g N per bucket). The study conditions may have been biased towards maximizing N losses, possibly hiding an effect of reduced N emissions if the study period had been shorter and more comparable to a real-life situation. However, former studies of N emissions by various methodologies have only found reduced N emission by application of finely ground calcium carbonate in a few cases, and results were inconsistent.

The studies in this project were conducted without dispersion agent, partly because the resources did not allow for a testing of dispersion agent alone, and partly because the dispersion agent is not allowed for use in organic farming and hence could not be tested in the SoilEffects field experiment. It adds to the difficulties of drawing clear conclusions from former studies of marble products from Omya Hustadmarmor AS, that some studies were conducted with dry ground calcium carbonate and other studies with slurry including dispersion agent. Studies to distinguish the effects of the dispersion agent, and of the ground calcium carbonate as such, should be conducted. Such studies were not revealed in scientific literature.

Mixing of finely ground marble into animal manure makes no harm, and may be an easy and agronomically sound way of maintaining or increasing soil pH and to increase the amount of plant nutrients in soil. The content of sulphur and micronutrients (iron and likely others) in addition to calcium which is a plant macro nutrient, adds to the quality of this soil amendment. Especially for organic farmers, who often export plant nutrients in farm products without replacing them by purchased fertilizers, ground calcium carbonate, here called marble, is a valuable and natural resource that should be further utilised.

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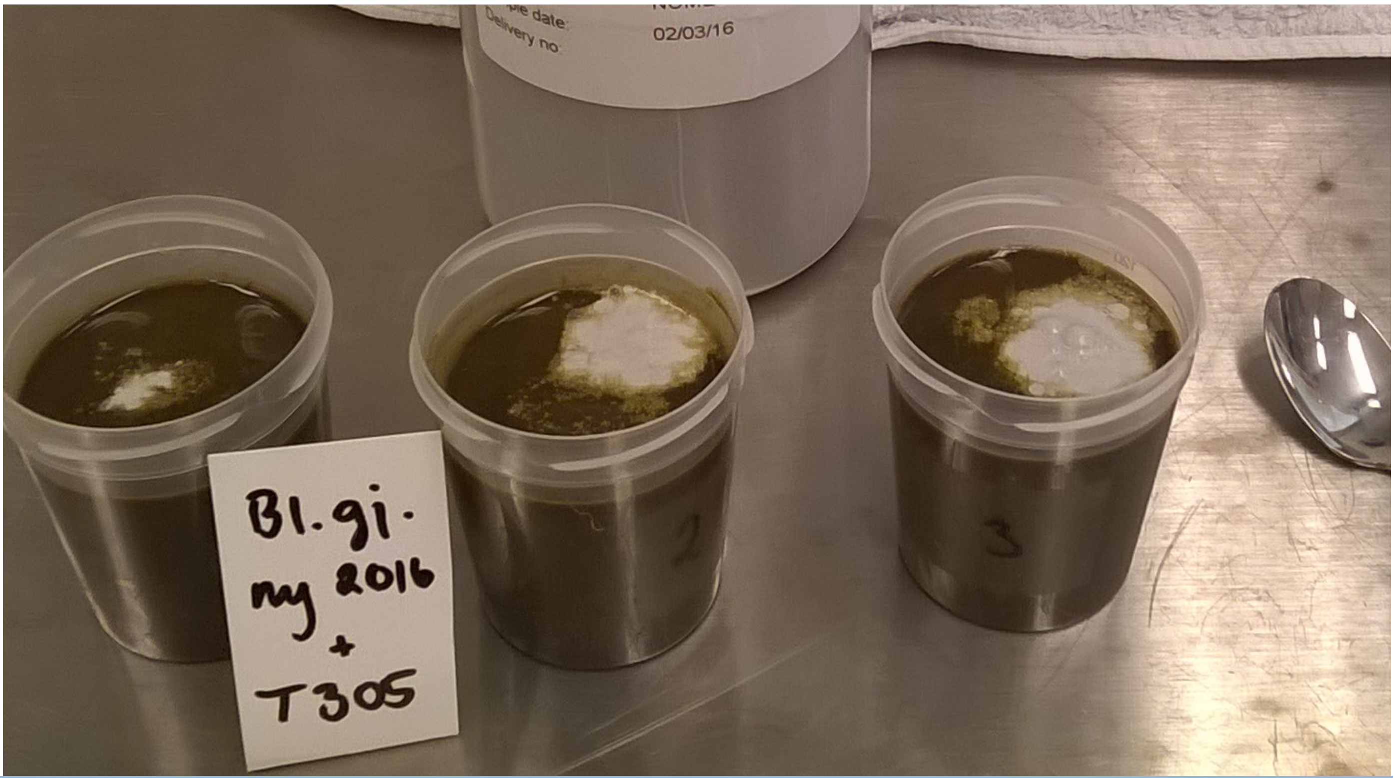

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