Baseline Study of Greenhouse Gas Emission from Stored Digested Slurry after Separation of Biogas Plant for Dairy Slurry in Spring Snowmelt Period

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The purpose of this research was to provide an accurate estimate of greenhouse gas (GHG) emissions during the storage of liquid digested slurry after separation (LDSS) produced by a dairy-manure-fed biogas plant in Hokkaido, Japan, in order to create a reliable gas inventory for life-cycle assessment (LCA) of the system. To the best of our knowledge, there have been no case studies measuring GHG generation by anaerobic digesters in such snowy cold regions. Therefore, a GHG measurement method adapted to snowfall and freeze-thaw conditions was newly developed: we measured the amounts of GHGs produced before and after the melting of frozen LDSS at the surface layer of the storage tank during the snowmelt period. The concentrations of CO2, CH4, N2O, and NH3 in the LDSS storage tank (above ground, open-type, diameter: 44 m, capacity (effective volume): 6,839 m3 (5,471 m3)) were measured with a multi-gas monitor (infrared photoacoustic detector manufactured by INNOVA) using an open-chamber technique. The measurements were conducted at the LDSS storage tank for a biogas plant in Ebetsu City, central Hokkaido, Japan from February 20 to April 3, 2018. This plant processes 29 m3/day, has a fermentation temperature of 40°C, and hydraulic retention time (HRT) of 40 days. The results revealed that almost no GHG emissions were observed from the frozen storage tank. In addition, when the frozen LDSS began to melt, the GHG emission pattern showed diurnal fluctuations that generally moved in tandem with the ambient temperature. The GHG emissions greatly changed during the period immediately after the frozen LDSS began melting until it melted completely. After the frozen LDSS was melted, CH4, CO2, and N2O emissions were 113.0 mg CH4/m2/day, 88.8 mg CO2/m2/day, and 0.021 mg N2O/m2/day.

Key Words
Greenhouse gas, Biogas plant, Dairy slurry

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1. Introduction

Under the Paris Agreement (2015), signed after the release of the Fifth Assessment Report (AR5) of the Intergovernmental Panel on Climate Change (IPCC) in 2013, all signatory countries agreed to work to reduce GHGs with the goal of limiting the average global temperature increase to 2 °C (“pursue effort” target of 1.5 °C). Accordingly, Japan is working actively to reduce GHG emissions, and the Cabinet approved a “Plan for Global Warming Countermeasures” in 2016. Methane (CH₄) from rice cultivation is the greatest GHG emission in Japan. However, the amount of GHGs produced from livestock production is comparable. In livestock agriculture, methane produced by gastrointestinal fermentation in ruminant livestock such as cattle is the largest source of GHGs. This is followed by N₂O and CH₄ from livestock manure (GIO 2017⁵).

The amount of livestock manure produced in Japan is 82.95 million tons per year. In terms of the major types of livestock, dairy cattle produce 23.57 million tons per year, beef cattle produce 24.42 million tons per year, and pigs produce 22.38 million tons per year; these three types of livestock produce about 85% of the total livestock manure. Treatment methods for these livestock manure can be divided into composting treatments and slurry treatments. Composting treatment is used mainly to treat the manure of beef cattle and tie stall-housed dairy cattle. Slurry treatment is used mainly to treat the manure of pigs and free-stall-housed dairy cattle. Free stalls are used by about 20% of all feeding facilities ⁵. Assuming that 20% of the calculated amount of manure in Japan comes from free stall-housed dairy cattle, we can surmise that about 2.26 million tons per year of manure are slurry-treated ⁵.

It is believed that in general, the amount of GHGs emitted from the large volume of stored dairy manure slurry fluctuates greatly depending on the duration of the storage and the weather conditions. Therefore, emission factors from storage tanks should be evaluated in light of each country’s treatment procedures for livestock manure. It is especially critical that each country have appropriate GHG emission measuring techniques. These measurements are indispensable not only for creating inventory data, but also for developing GHG regulations and reduction technologies.

In Japan, the manure of dairy cattle and pigs is mainly subjected to slurry treatment. For dairy cattle, it is applied in natural downflow-type feeding facilities and free-stall feeding facilities. For pigs, it is applied to excrement flowing from slatted floor-type pig barns. According to the GIO 2017⁵, in the mixed urine/feces treatment category, slurry treatment of dairy manure (15.4%) was the third most frequent treatment method after piling (50.9%) and composting (22.9%). Slurry treatment as the main livestock manure treatment method assumes mixed urine/feces treatment and consists of slurry tank storage and methane fermentation in biogas plants. GHGs emitted from the storage tanks of livestock manure, as exemplified by slurry tanks, make up the bulk of GHGs emitted from these two treatment methods. In Japan, large-scale free-stall dairy farms such as those that comprise much of Hokkaido’s agriculture, must store slurry for at least six months total, in tanks or pretreatment facilities ⁶. Also, as open-type storage tanks are directly affected by rainfall, they are constructed with designs that anticipate this effect. It is said that in general, the amount of GHGs produced from the storage of manure slurry produced in large amounts by dairy cattle varies greatly depending on the amount of slurry stored over the long term and weather conditions.

In the case of Japan, regulation of methane (CH₄) and nitrous oxide (N₂O) emissions is almost entirely based on actual measurements from field surveys and lab experiments, and levels are established for each livestock manure treatment method according to the type of livestock. However, with regard to slurry storage in Japan, IPCC’s 1990 default values are still used for the amounts of GHGs produced during storage of digested slurry. Refinement based on actual measurements is thus necessary. Various methods to measure GHGs from manure slurry in storage tanks have been developed. These methods include the open chamber method (Peu et al., 1999⁷) and the closed chamber method (Sommer et al., 2000⁸). However, to increase the number of measurements, a method that is easy to establish on the grounds of different farms and which takes into account prevention of communicable diseases is necessary. Digested slurry produced from biogas plants has a higher temperature during production time than general dairy manure slurry. The surface layer of the digested slurry storage tank freezes at the height of winter. As a result, the material of the chamber used year-round to measure GHGs can be damaged through contact with blocks of frozen digested slurry and contraction of the chamber body. There is therefore a need for strengthening the chamber and improving its resistance to cold. In addition, the temperature of digested slurry discharged from biogas plants as the time of discharge is higher than that of ordinary dairy manure slurry, which results in the generation of much steam during storage. Thus, in the height of winter, there are times when the year-round GHG measurement system becomes inoperable due to clogging of the GHG sampling line by frozen steam adhering to the inner side of the line near the exhaust ventilation blower. These countermeasures
are therefore essential for a GHG measurement system used in severely cold regions.

In Chapter 5.3B “AGRICULTURE: Manure Management” of GIO 2017, there is no information given about emission factors of GHGs emitted during livestock manure treatment by biogas plants. We therefore referred to Chapter 7.3.2 “WASTE: Anaerobic Digestion at Biogas Facilities (5.B.2)” to consider emission factors of GHGs from agricultural biogas plants. Here, methane fermentation facilities for anaerobic digestion treatment are reported to the GIO 2017 as airtight structures that do not emit gases from applicable sources. From this statement, it can be said that there are no GHG emissions from the fermentation facilities of agricultural biogas plant, mainly anaerobic digestion tanks. However, the digested slurry produced from agricultural biogas plants is returned to the environment, and storage facilities for digested slurry produced by biogas plants before it is returned to the environment are open-type. Thus, when considering an agricultural biogas plant as a livestock manure treatment system, GHGs emitted from digested slurry storage tanks cannot be ignored.

As with slurry, the relationship between the amounts of GHGs emitted from digested slurry and the temperature of the digested slurry is a key feature to analyze. The biggest difference between slurry and digested slurry is that digested slurry produced after methane fermentation in a biogas plant contains huge populations of methanogens, or anaerobic microorganisms. Also, while the amounts of formic acid and acetic acid in livestock manure, which serve as the substrate for methanogens, decrease in the methane fermentation tank, the bacteria are both discharged from the methane fermentation tank in a state of vigorous methanogenic activity and retained in the storage tank.

Studies on GHGs emitted from stored digested slurry include Wang et al. (2014), Thomas et al. (2011), Umetsu et al. (2005), and Sommer et al. (2000). However, as in the case of slurry there have been few reports on actual-scale emissions in humid regions like Japan. Also, because digested slurry at the surface layer of a storage tank in a snowy cold region like Hokkaido is frozen, when the frozen digested slurry block at the surface layer melts during the snowmelt period, there is concern that GHGs, chiefly methane, are immediately released. It is therefore necessary to carry out actual measurements.

The purpose of this research is to create a gas inventory or an LCA assessment of the anaerobic digestion systems of biogas plants. Toward this end, we determined the amounts of GHGs produced during the storage of liquid digested slurry after separation (LDSS) produced by a biogas plant that uses dairy manure as the raw material. For GHG measurements, we used a dynamic floating chamber measurement system, which is suited for snowy cold regions. In this paper, we report on the amounts of GHGs produced before and after the melting of frozen LDSS at the surface layer of the storage tank during the snowmelt period. There have been very few cases of such measurements despite their importance to accurate GHG emission estimates.

2. Experimental

2.1 Location of measurement survey and storage conditions of digested slurry

To develop and popularize renewable energy, Japan passed into law a feed-in tariff (FIT) incentive scheme, which began in July 2012. This scheme obligates electric utilities to purchase electricity produced by a renewable energy producer at a fixed price over a long-term period (maximum 20 years). Among other sources of electricity eligible for the FIT requirement is that generated by biogas as the fuel. Biogas is produced by biogas plants using livestock manure, a biomass, as the raw material. Thus, livestock farmers can use the manure effluent to earn income from the sale of electricity as well as recycle the nutrients as fertilizer. Due to this incentive, the number of biogas plants has been on the rise since 2012. In Hokkaido, where more farms raise cattle in free stalls compared with other prefectures, there are presently about 80 biogas plants in operation.

We measured the amounts of GHGs emitted from the storage tank (diameter, 44 m; depth, 4.5 m; capacity (effective volume), 6,839 m$^3$ (5,471 m$^3$)) of LDSS (liquid digested slurry after separation) produced by a biogas plant (hydraulic retention time: 40 days, fermentation temperature: about 40°C). The plant is located in Ebetsu city, central Hokkaido, Japan. Measurements of the surface area of the frozen LDSS at surface layer thickness were conducted from February 20 to April 3, 2018. The floating chamber to the frozen LDSS block was set up on March 14, 2018. The thickness of the frozen LDSS block was measured by drilling ice cubes when setting up the floating chamber. GHG’s measurements were conducted from March 14 to April 3, 2018.

After being discharged from the biogas plant’s anaerobic digestion tank, LDSS is separated into solids and liquid by solid-liquid separation. Only the liquid portion is sent to the LDSS storage tank once a day at a fixed time by a transfer pump. Table 1 shows the composition of the raw materials, digested slurry and digested slurry after separation into liquid (LDSS) and solid. All sample analyses were performed in duplicate and the values averaged...
2.2 Overview of measurement system and gas sampling method

Fig. 1 shows the system used for measuring GHGs emitted from the LDSS storage tank. The floating chamber, the key to this measurement system, was created using a PVC float (diameter: 2.15 m, height: 0.6 m, volume: 530 L), rigid stainless pipes, and a stainless steel tank.

The GHG measurement system consists of the aforementioned floating chamber, a multipoint sampler (Model 1309; Innova), a photoacoustic multi-gas monitor (Model 1312; Innova), an exhaust ventilation pressure gauge, and a differential pressure gauge. The effective LDSS surface area at the floating chamber was 1.17 m².

Fresh air (background; taken at a position 4 m above ground at a location about 10 m from the LDSS storage tank) is drawn from outside of the storage tank into the tank at the upper portion of the chamber through a duct hose (φ50). It is supplied to the LDSS surface layer through six stainless steel pipes. GHGs emitted from the effective surface in the floating chamber are drawn from the central portion of the floating chamber by the exhaust blower and automatically sampled by the multipoint sampler. The methane (CH₄), carbon dioxide (CO₂), nitrous oxide (N₂O), and ammonia (NH₃) concentrations in the gases are then sequentially measured by the photoacoustic multi-gas monitor. Also, a pH meter, Eh (reduction potential) meter and thermometer are placed at a depth of 30 cm in the stored LDSS. These values are measured every hour.

To calculate emission factors, we determine the emission rates of CH₄, CO₂, and N₂O from the ventilation rate and the difference in concentrations between air samples at the exhaust and inlet air (that is, the background) of the measuring system. NH₃, It is not a GHG, ammonia that cause acid rain, an important substance that affects the environment, is also measured using the same method to determine its emission rate.

For analysis of the composition of the LDSS in storage, slurry at the depth of 10 cm from the surface during experimental period.

### Table 1 Composition of raw materials (Biogas plant), digested slurry (Effluent slurry), digested slurry after separation (liquid; LDSS & solid) during experimental period

|                  | raw materials | digested slurry | digested slurry after separation |
|------------------|---------------|----------------|----------------------------------|
|                  |               |                | Liquid (LDSS)                    | solid |
| pH               | 7.4 ~ 7.6     | 7.8 ~ 8.0      | 7.9 ~ 8.6                       | 7.9 ~ 8.9 |
| TS (%)           | 8.1 ~ 8.8     | 6.1 ~ 6.4      | 21 ~ 3.2                        | 20 ~ 21.2 |
| VS (%)           | 6.4 ~ 7.1     | 4.3 ~ 4.8      | 16 ~ 2.6                        | 17 ~ 20.2 |
| T-N (mg/L)       | 3,800 ~ 4,000 | 3,700 ~ 3,800  | 3,100 ~ 3,600                   | 5,800 ~ 6,200 |
| P (mg/L)         | 590 ~ 650     | 510 ~ 550      | 410 ~ 440                       | 1,200 ~ 1,500 |
| K (mg/L)         | 3,800 ~ 4,800 | 3,800 ~ 4,100  | 1,500 ~ 1,800                   | 4,700 ~ 4,700 |

TS: total solids, VS: volatile solids, TN: total nitrogen, P: Phosphoric, K: Potassium, LDSS: liquid digested slurry after separation.
was sampled to measure the LDSS's pH, total solids (TS), volatile solids (VS), total nitrogen (TN), phosphorus (P) and potassium (K). TS content was determined after samples had been dried for more than 24 h at 105 °C; VS content was measured after samples had been ashed for 4 h at 550 °C. TN, P and K were measured by JIS K0102 (JIS: Japanese Industrial Standard).

3. Results and discussion

3.1 Physicochemical composition of LDSS during the measurement period

Table 2 shows the chemical composition of the LDSS (liquid digested slurry after separation) in storage during the measurement period. For the sampling of the frozen portion of LDSS at depth 0 cm to 20 cm, unfrozen portions were created by crushing frozen LDSS as needed and sampled. Using sensors, the pH and reduction potential (Eh) of the LDSS in storage were continually measured at one-hour intervals. Samples for chemical composition analysis were mixed well and sent to a laboratory for analysis. The TS had a dry matter content between 1.4 and 1.9%.

The pH and Eh of slurry stored at the depth of 1 cm during the measurement period remained stable at 77 ~ 84 and -420 ~ -350 mV, respectively. The physicochemical composition also remained stable. Measured redox potentials were typical of environments in which methanogenesis and sulfur reduction occur (> -100 mV) (Tate, 1995) 10).

3.2 Temperature of LDSS in storage and condition of melting of LDSS at frozen surface layer

3.2.1 Temperature of LDSS in storage and ambient temperature

Fig. 2 shows the ambient temperature during the measurement period and the temperature at each depth of the LDSS in storage. The average ambient temperature during the measurement period was -2.6 °C with 13.3 °C as the highest temperature and -10.7 °C as the lowest temperature. The temperatures of unfrozen LDSS in the storage tank were stable with an average of 7.5 °C at the depth of 30 cm and 23.2 °C at the depth of 400 cm.

The liquid temperature at the bottom of the storage tank is higher than the ordinary non digestive slurry tank (Minato et al., 2013; Park et al., 2006; Sommer et al., 1993) 11~13. This is because the warm digestive juice discharged from the biogas plant every day was transferred to the bottom of the reservoir every day.

3.2.2 Condition of melting of LDSS at the frozen surface layer

Fig. 3 shows the change in the surface area of the frozen LDSS at surface layer of the storage tank and accumulated day length during measurement periods. The frozen portion of the LDSS's surface layer began to decrease in size beginning February 20, 2018. When setting up the floating chamber, the thickness of the frozen LDSS block was 18.5 cm. It disappeared entirely on April 2, 2018. The daily rate of surface area reduction of the frozen LDSS was approximately 35 m² per day (5.8% per day). The frozen LDSS block thickness was measured by drilling ice cubes when setting up the floating chamber.

3.3 Changes in GHG emissions during the melting period

3.3.1 Condition of GHG measuring system

Typical methods for measuring GHGs emitted from storage tanks include the closed chamber method and the tracer method, in addition to the open chamber method. In this report, we measured GHG and NH₃ emissions using the open chamber method (Minato et al., 2013) 9). We used the same method to measure GHG emissions from compost and dairy manure slurry. Furthermore, the equipment we developed was a measurement system used in cold areas to

| Table 2 The chemical composition of the LDSS in storage during the measurement period | Digested slurry after separation (LDSS) |
|---------------------------------|---------------------------------------|
| pH 77 ~ 84                      |                                       |
| Eh -421 ~ -359                  |                                       |
| TS 1.4 ~ 1.9                    |                                       |
| VS 1.2 ~ 1.6                    |                                       |
| TN 3,100 ~ 3,300                |                                       |
| P 380 ~ 400                     |                                       |
| K 1,400 ~ 1,600                 |                                       |

TS: total solids, VS: volatile solids, TN: total nitrogen, P: Phosphorus, K: Potassium, LDSS: liquid digested slurry after separation
deal with freezing temperatures during the winter period. It was easy to install in the farm slurry storage facility. We implemented measures for minimizing the effects of steam-to-frost by strengthening the chamber material and installing a drain after the blower to reduce the frost adhering to the inner side of the sampling line. During the measurement period, there was no time when the year-round GHG measurement system became inoperable due to clogging of the GHG sampling line by frozen steam adhering to the inner side of the line near the exhaust ventilation blower.

3.3.2 Hourly and daily gas emission rates

1) Hourly gas emission

Fig. 4 shows changes in CH₄, CO₂, NH₃ and N₂O of hourly gas emissions measured during slurry storage.

In the LDSS’s frozen state, CH₄, CO₂, N₂O and NH₃ emissions could not be observed.

With regard to hourly gas fluctuations, the emissions of the four gases being evaluated all had higher concentrations during daytime than during nighttime. The phenomenon indicates that as the temperature of the LDSS at a depth of 30 cm rose, CH₄ emission also increased. In general, gas solubility decreases as the temperature of a liquid rises. The release of CH₄ is thus quickly affected by changes in slurry surface temperature. It has been reported that when measuring gases in dairy manure slurry, CH₄ is released when its concentration in slurry exceeds its solubility (Sneath et al., 2006) ¹². In addition to the effects of daytime, we noted that weather conditions affected gas emissions from stored LDSS. In addition to effects due to weather conditions, management activities related to LDSS storage (e.g. agitation, addition of fresh LDSS) can affect the production of gases (Park et al., 2006; Sommer et al., 1993; Husted, 1993) ¹² ¹³ ¹⁵. At the dairy farm where we conducted the measurements, LDSS was supplied daily from the biogas plant to the bottom of the LDSS storage tank at 10:00. This time overlaps with the time period in which the surrounding temperature rises, so whether the addition of fresh LDSS affects GHG production is unclear.

2) Daily gas emission

Changes in CH₄, CO₂, N₂O and NH₃ emissions on a hourly and daily basis from the storage tank during the measurement period are shown in Fig. 5.

Once the melting of the frozen LDSS directly under the chamber began, CH₄, CO₂, and NH₃ emissions increased rapidly. Emissions of N₂O, however, were small at this time. Once LDSS began to melt, CH₄ and CO₂ emissions
showed a tendency to increase following an increase in NH$_3$ emission. The generation of N$_2$O, however, was exceedingly small compared with the other GHGs. The CH$_4$, CO$_2$, and N$_2$O emissions after the frozen surface of the LDSS in the chamber completely disappeared were 113.0 mg CH$_4$/m$^2$/day, 88.8 mg CO$_2$/m$^2$/day, and 0.021 mg N$_2$O/m$^2$/day, respectively.

In the generation of GHGs, it is commonly known that the generation of nitrous oxide (N$_2$O) occurs in the process of converting nitrous acid (NO$_2$) to nitric acid (NO$_3$) by nitrifying bacteria activated by ammonia nitrogen (NH$_4$-N) in organic matter. We consider that the reason for the small amount of N$_2$O generated in this test was the low activation of nitrifying bacteria given the low temperature of LDSS and that the digested slurry became increasingly inorganic in the anaerobic fermentation process of the biogas plant.

As for methane (CH$_4$), no significant GHG generation was observed since the temperature in the 30 cm portion of the digested slurry from the surface layer of the storage tank was approximately 10°C or lower, this despite the temperature at the bottom of the storage tank being approximately 20°C or higher. Zeikus et al. (1976) reported that activation of methane bacteria at temperatures below 5°C is remarkably low with no generation of methane. In addition, Umetsu et al. (2005) reported that practically no methane is generated at 5°C under absolute anaerobic conditions. Similarly, Park et al. reported that GHG generation from swine excreta slurry in a state of thin-ice formation (outside air temperature from 0°C to -5°C) is extremely small. Based on these reports, we consider that the generation of methane was small in this test as well.

The relationship between ammonia (NH$_3$) emissions and outdoor temperature, storage-tank surface layer temperature (-30 cm), and day length is shown in Fig. 6.

When LDSS began to melt, the discharge of ammonia, a substance with considerable environmental impact, preceded that of other GHGs. These results show that NH$_3$ emissions from frozen LDSS increased as outside temperature rose and as slurry temperature at 30 cm from the storage-tank surface layer rose due to the effects of sunlight. It can therefore be inferred that ammonia generated from frozen LDSS is influenced by outside temperature and sunlight. In this experiment, however, the temperature at the bottom of the storage tank was approximately 20°C, so the effect of digested-slurry temperature inside the storage tank must be added...
as another factor in the melting of frozen LDSS in addition to outside temperature and day length. There is therefore a need for more data collection and testing. We surmised that the discharge of ammonia preceded that of other GHGs because soluble ammonia contained in the frozen LDSS came to be emitted first as the outside temperature and temperature of surface-layer slurry in the storage tank rose.

Based on the above results, it was found that temperature at the bottom of the storage tank was approximately 20 °C, which generated concern that GHGs dominated by a high concentration of methane would be generated from the lower section of the storage tank. However, experimental results revealed that the amounts of GHGs generated were small.

5. Conclusion

We used a GHG measurement system and the open chamber method to measure storage tank GHG emissions before and after the springtime melting of liquid digested slurry after separation (LDSS) that had been frozen at the surface layer during the winter. The LDSS storage unit was attached to a biogas plant from open-stall dairy, an increasingly popular setup in the Hokkaido region of Japan. The chemical composition of the LDSS in storage during the measurement period did not change greatly. Emission of GHGs from the frozen storage tank was hardly observed. When the melting of frozen LDSS began, the GHG emission pattern showed diurnal fluctuations that generally moved in tandem with the ambient temperature. GHG emissions changed greatly during the period immediately after the melting of the frozen LDSS block began until it melted completely. After the frozen LDSS melted, CH4, CO2, and N2O emissions were 113.0 mg CH4/m2/day, 88.8 mg CO2/m2/day, and 0.021 mg N2O/m2/day. NH3 emission was 0.042 mg NH3/m2/day.

Based on our results we plan to continue measuring GHG emissions from stored LDSS throughout the year and present emission factors from LDSS storage.

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