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Article

# Solid-State Anaerobic Digestion of Dairy Manure from a Sawdust-Bedded Pack Barn: Moisture Responses

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**Abstract:** Bedded pack manure has long been considered an unsuitable feedstock for conventional anaerobic digestion systems due to its high solids content. However, solid-state anaerobic digestion (SS-AD) provides an opportunity to generate methane from such high-solids feedstocks. This study was conducted to determine the influence of moisture content on the digestion of bedded pack dairy manure using SS-AD. Mixtures of sawdust bedding and dairy manure were prepared with moisture contents (MCs) of 70%, 76% and 83% and digested at 37 °C for 85 days. The performance of digesters containing manure at 83% MC was 1.3 to 1.4-fold higher than that of digesters containing 70% MC manure in terms of volatile solids (VS) reduction and biogas production. VS reduction rates were 55 to 75% and cumulative methane yield ranged from 64 to 90 NmL g VS<sup>-1</sup>. These values are lower than those from SS-AD of fresh manure and this is likely due to the partial decomposition of biodegradable materials during the two to three-month period before the manure was removed from the barn. However, in terms of efficient management of farm odors and providing a renewable energy source for heating, SS-AD of bedded pack manure offers a potential alternative to the conventional composting systems currently in use.

**Keywords:** dairy manure; solid-state anaerobic digestion; moisture content; biogas; methane

## 1. Introduction

Manure production from Korea's 430,000 dairy cows was approximately 6 million wet tons in 2014 and constitutes about 13% of the country's total animal manure production. Bedded pack barns, in which animals are bedded using a 10 cm layer of sawdust that is removed every two to three months, are the most common type of dairy operation in Korea [1]. Although composting is an accepted method for treatment of dairy manure and bedding material mixtures, the majority of Korean dairy farms lack the resources and financial incentives to compost this material. For this reason, most farms simply stack manure after collection and apply this untreated material to fields [2]. This practice causes potential environmental problems (such as soil and groundwater pollution) as well as odor complaints.

Anaerobic digestion (AD) has emerged as an alternative technology to conventional composting processes for treating high-solids wastes. The AD is now considered an attractive and innovative

technology for reduction of odor, stabilization of manure with mass and volume reduction, generation of green electricity, and reduction of emissions of the greenhouse gas methane [3–6].

In 2013, seven full-scale liquid-phase manure digesters were in operation in Korea, mainly for handling high-moisture swine manure. These systems are not suitable for treating manure containing low-moisture wastes such as dairy and beef manure collected from bedded pack barns. Solid-state AD (SS-AD) systems can be used to treat dairy manure with high solid content (>15%) and can treat more biomass than liquid-phase systems in a given digester volume because of the high dry matter content of the feedstock [7]. With respect to bedded pack dairies, SS-AD has the potential to integrate well with typical farm operations since its batch mode operation period matches the period of bedding generation. Studies related to the AD of slurry-type dairy manure collected from free-stall barns have been performed by a number of previous researchers, but despite the advantages of SS-AD, as mentioned above, there have been few cases of an AD of dairy manure collected from bedded pack barns [8–12].

Despite the potential advantages of SS-AD, the process poses a number of practical challenges. Slow startup performance of SS-AD leads to relatively low rates of anaerobic decomposition [13]. In addition, SS-AD requires about threefold longer retention times than liquid systems because the feedstocks of SS-AD typically contain large amounts of recalcitrant materials such as lignin, cellulose, and hemicelluloses [14–16]. In order to overcome slow startup and prolonged digestion times, SS-AD also requires larger amounts of inoculum than do liquid systems [16].

Feedstock moisture content is one of the most important factors affecting the efficiency of SS-AD [17]. The moisture content of bedded pack materials, as collected, typically ranges between 70% and 83% wet basis depending on the frequency with which the bedding is replaced. Although results from previous studies suggest that the efficiency of SS-AD increases as feedstock moisture levels increase [18], there is no information on optimum moisture levels for SS-AD of dairy manure and sawdust bedding mixtures from bedded pack barns. The objective of this experiment was to examine the suitability, in terms of VS reduction and methane production, of bedded pack dairy manure with varying moisture contents as a feedstock for SS-AD. It is very meaningful and important to develop an SS-AD technology for bedded pack dairy manure that can be used as an energy source as well as suggesting an alternative method of composting to that typically used.

## 2. Materials and Methods

### 2.1. Feedstock

In Korea, pine tree sawdust is the most common bedding material for bedded pack dairy barns. In this study, a mixture of dairy manure and pine tree sawdust bedding material (hereafter termed manure) was collected from Chungnam National University's animal farm and stored at 4 °C prior to use. Manure characteristics are shown in Table 1.

**Table 1.** Characteristics of dairy manure from a sawdust-bedded pack barn. Data represent means  $\pm$  SE ( $n = 4$ ) (<sup>a</sup> w.b.: wet basis; <sup>b</sup> d.b.: dry basis).

| Parameters                             | Bedded Pack Dairy Manure (as Collected) |
|--|---|
| Moisture Content (% w.b.) <sup>a</sup> | 69.4 $\pm$ 0.1                          |
| Volatile solids (% d.b.) <sup>b</sup>  | 84.2 $\pm$ 0.1                          |
| Water holding capacity (% w.b.)        | 83.1 $\pm$ 2.3                          |
| Bulk density (kg/m <sup>3</sup> )      | 648.4 $\pm$ 3.9                         |
| Free air space (%)                     | 43.3 $\pm$ 0.7                          |
| Total Carbon (% d.b.)                  | 39.2 $\pm$ 3.5                          |
| Total Nitrogen (% d.b.)                | 1.5 $\pm$ 0.2                           |
| Cellulose (% d.b.)                     | 24.0 $\pm$ 1.2                          |
| Hemicellulose (% d.b.)                 | 5.6 $\pm$ 2.3                           |
| Lignin (% d.b.)                        | 29.1 $\pm$ 1.8                          |

The moisture content (MC) of the manure was 70% and the water holding capacity (WHC) was 83%. The typical moisture content of bedded pack dairy manure collected on the farm ranges from 70 to 83% (saturated condition) wet basis depending on the frequency with which the bedding is replaced. Prior to the experiment, two aliquots of the collected manure were removed and water was added to the aliquots to achieve MC values of 76% and 83%, respectively. These aliquots of manure, along with an aliquot of non-amended manure, were incubated at 37 °C for 2 days before use in order to acclimate the microbial community to mesophilic conditions.

## 2.2. Experimental Design

Lab-scale anaerobic reactors with a volume of 1.8 L (working volume 1.5 L) were constructed with PP (Polypropylene) and a fine-mesh screen was installed on the reactor bottom. A completely randomized design with three manure feedstocks (327 g at 70% MC, 389 g at 76% MC, and 507 g at 83% MC) were placed in each reactor with three replicates per MC level. Each reactor gas sampling port was connected to a 5 L Tedlar bag. Digesters were placed in a constant-temperature room held at 37 °C. Gas production was measured daily using a graduated syringe.

## 2.3. Analytical Methods

Total solid (TS) and volatile solid (VS) contents were determined using standard methods [19]. Water holding capacity was measured using a modified Hilgard method [20,21]. Bulk density and free air space (FAS) were measured using a method reported by Ahn et al. [22]. Total carbon (TC) and total nitrogen (TN) contents were analyzed using an elemental analyzer (2400 CHNS/O series II system, Perkin Elmer, Waltham, MA, USA). Methane and carbon dioxide contents in the biogas were determined using a gas chromatograph (iGC 7200, DS Science, Gwangju-si, Korea) equipped with a 1.8 m SUS column 80/100 mesh and thermal conductivity detector (TCD). Helium was used as the carrier gas with a flow rate of 25 mL min<sup>-1</sup>. The temperatures of the injection, detector, and column were 50 °C. The GC was calibrated with an external standard containing CH<sub>4</sub>/CO<sub>2</sub> in volume ratios of 25:75, 50:50, and 75:25. Methane volume values are reported at standard temperature and pressure (STP) conditions.

## 2.4. Ultimate Biodegradability

Total volatile solids (TVS) consist of biodegradable volatile solids (BVS) and nonbiodegradable solids (NBVS). BVS can easily be converted to biogas while NBVS (usually lignin, hemicelluloses, and cellulose) are converted slowly or not at all. Thus, BVS is a better metric than TVS for evaluating organic matter decomposition rates during the AD. The ultimate biodegradability value (UB, BVS/TVS) was determined using a graphic statistical analysis [23]. This method uses a linear regression of the remaining volatile solids (TVSe) from the initial total volatile solids (TVSo) at time *t*, as the test was continued to infinity. The TVS remaining at infinity represents the refractory fraction of the feedstock (R<sub>O</sub>) and the nonbiodegradable portion of TVSo. The value of the *y*-intercept of TVSe/TVSo versus 1/*t* is defined as the refractory fraction (R<sub>O</sub>). The UB of feedstock is estimated using Equation (1) [24,25]:

$$UB = (1 - R_O) \times 100 \quad (1)$$

where R<sub>O</sub> is the refractory fraction. VS mass removal values were determined from biogas production values. Normalized biogas at the STP was assumed to be an ideal gas which occupies 22.413 L mol<sup>-1</sup> of dry biogas. Removed biomass (BMR) is presented in Equation (2) [24]:

$$BMR = V_O \times \{1.963 - (0.0124 \times CH_4)\} \quad (2)$$

where BMR (g) is removed biomass, V<sub>O</sub> (L) is normalized biogas volume, CH<sub>4</sub> (%) is biogas normalized methane content.

## 2.5. Biogas Production Simulation

Biogas production rates of dairy manure and sawdust mixtures in SS-AD were simulated using the Gaussian and Gompertz equation. The methane production rate was simulated using the modified Gaussian equation. This equation describes the daily methane production in batch reactors assuming that microbial kinetic growth and destruction of methanogens follow a normal distribution over the anaerobic digestion stages [23]. The Gaussian equation is presented in Equation (3):

$$y = a \times \exp \left[ -0.5 \left( \frac{t - t_0}{b} \right)^2 \right] \quad (3)$$

where  $y$  is the methane production rate,  $\text{NmL g VS}^{-1} \text{ day}^{-1}$  at any time  $t$ ;  $t$  is the time over the digestion period, in days;  $a$  ( $\text{NmL g VS}^{-1} \text{ day}^{-1}$ ) and  $b$  (day) are constants; and  $t_0$  is the time where the maximal methane production rates occurred, in days. The parameters  $a$ ,  $b$ , and  $t_0$  were calculated by using the “Solver” in MS Excel.

Assuming that methane production is a result of anaerobic bacterial growth, the Gompertz equation describes cumulative methane production from batch reactors [26]. The modified Gompertz equation is presented in Equation (4):

$$M = P \times \exp \left\{ - \exp \left[ \frac{R_m \times e}{P} (\lambda - t) + 1 \right] \right\} \quad (4)$$

where  $M$  is the cumulative methane production,  $\text{NmL g VS}^{-1}$  at any time  $t$ ;  $P$  is the methane yield potential,  $\text{NmL g VS}^{-1}$ ;  $R_m$  is the maximum methane production rate,  $\text{NmL g VS}^{-1} \text{ day}^{-1}$ ;  $\lambda$  is the duration of the lag phase, in days; and  $t$  is the time at which the cumulative methane production  $M$  is calculated. The parameters  $P$ ,  $\lambda$ , and  $R_m$  were estimated by using the “Solver” feature in MS Excel. The parameter values which minimized the sum of the square of errors between fit and experimental data were determined.

## 2.6. Statistical Analysis

Digestion treatments were conducted in triplicate, and average values are reported. The software SAS 9.1 (SAS 9.1, SAS Institute Inc., Cary, NC, USA) was used for analysis of variance (ANOVA) and  $t$ -testing. Differences were considered statistically significant when  $p \leq 0.05$ .

## 3. Results and Discussion

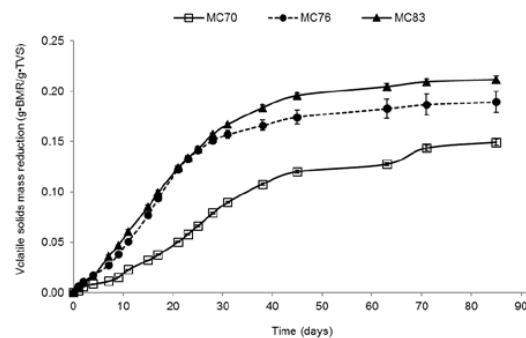
### 3.1. Volatile Solids Removal

Results showed that volatile solids removal values increased approximately 30% as the moisture content increased from 70 to 83% (Figure 1). Volatile solids mass removal values in digesters containing 70, 76, and 83% MC were 0.16, 0.19, and 0.21  $\text{g (g TVS)}^{-1}$ , respectively.

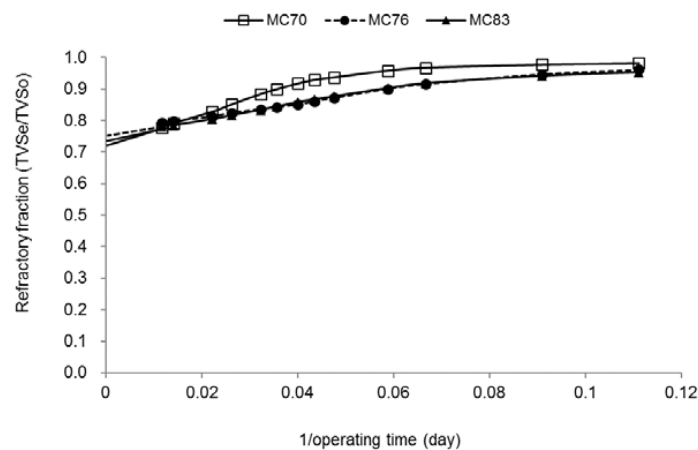
Amon et al. [27] reported a 24–33% TVS reduction of dairy manure (MC 84–87% wet basis (w.b.)) during 6 weeks of anaerobic digestion. One possible explanation for the lower values in the present study is that a portion of the organic material in pack barn manure was degraded during the two- to three-month period before the manure was removed from the barn. In addition, it is likely that the TVS removal rates observed in our study were lower than the values reported in Amon et al. [27] because the dairy manure used in this study contained sawdust bedding which has low biodegradability.

From the graphical statistical analysis, as shown in Figure 2, the refractory fraction of dairy manure and sawdust bedding mixtures ranged between 0.76 and 0.79. These values correspond to ultimate biodegradability (UB) values (the biodegradable portion of VS in the dairy manure/bedding mixture) of 21 to 24% (Table 2). However, Kang et al. [28] reported greater UB values of fresh dairy manure than that of the dairy manure used in this study. Its UB values ranged between 37% and 46%.

As the organic material decomposed during its stay for about two to three months in the barn and the dairy manure mixed with sawdust which has low biodegradability, the dairy manure collected from the sawdust-bedded pack barn showed lower ultimate biodegradability than did fresh dairy manure.



**Figure 1.** Cumulative volatile solids mass reduction (BMR/TVS) during solid-state anaerobic digestion (SS-AD) of dairy manure. Digesters containing non-amended manure containing 70% moisture content (MC) (MC70) and manure amended to 76% and 83% MC (equivalent to the water holding capacity of dairy manure) (MC76 and MC83, respectively) were incubated at 37 °C for 85 days. Values are means ( $\pm$ SE) of three replicates. BMR: removed biomass; TVS: total volatile solids.



**Figure 2.** Ultimate biodegradability values of dairy manure during SS-AD. Digesters containing non-amended manure containing 70% MC (MC70) and manure amended to 76% and 83% MC (equivalent to the water holding capacity of dairy manure) (MC76 and MC83, respectively) were incubated at 37 °C for 85 days. Values are means ( $\pm$ SE) of three replicates. TVSe: remaining volatile solids; TVSo: initial total volatile solids.

**Table 2.** Ultimate biodegradability and volatile solids removal during SS-AD of dairy manure. Data represent means  $\pm$  SE ( $n = 3$ ) (<sup>a</sup> Biodegradable volatile solids removal =  $\frac{\text{Removed biomass (BMR)}}{\text{Initial VS} \times \text{UB}} \times 100$ ).

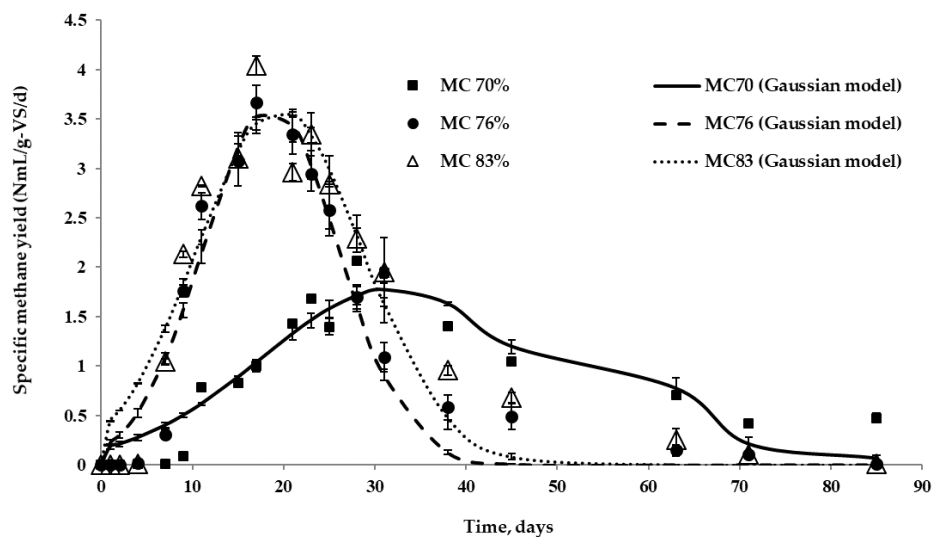
| Parameters   | MC70           | MC76           | MC83           |
|--|----------------|----------------|----------------|
| Ultimate biodegradability (UB) (%)                     | 23 $\pm$ 0.0   | 21 $\pm$ 0.0   | 24 $\pm$ 0.0   |
| Total volatile solids (VS) removal (%)                 | 15.5 $\pm$ 0.3 | 18.9 $\pm$ 1.0 | 21.2 $\pm$ 0.4 |
| Biodegradable volatile solids removal (%) <sup>a</sup> | 54.7 $\pm$ 1.1 | 66.9 $\pm$ 3.7 | 74.6 $\pm$ 1.3 |

After 85 days of digestion, BVS removal values were 55%, 67%, and 75% at 70%, 76%, and 83% MC, respectively (Table 2). Results showed that calculated biodegradable volatile solids (BVS) removal values improved by up to 36% by increasing the MC from 70 to 83%.

### 3.2. Methane Production

#### 3.2.1. Maximum Methane Production Rate

Our results suggest that a feedstock moisture content near saturation (above 90% of WHC) is optimal for decreasing the startup period and improving the methane production rate. Experimental and Gaussian plots of the daily methane production rate under three different moisture conditions are shown in Figure 3.



**Figure 3.** Influence of initial feedstock moisture content on specific methane yields from dairy manure. Digesters containing non-amended manure containing 70% MC (MC70) and manure amended to 76% and 83% MC (equivalent to the water holding capacity of dairy manure) (MC76 and MC83, respectively) were incubated at 37 °C for 85 days. Values are means ( $\pm$  SE) of three replicates. Experimental and model-derived results are shown.

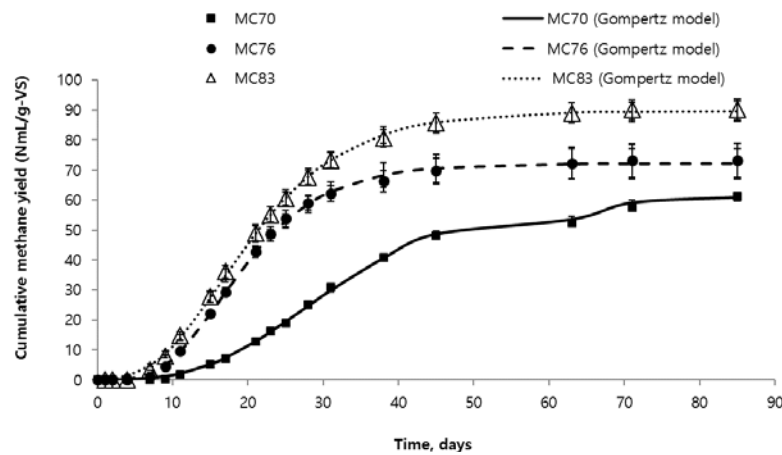
Using the Gaussian kinetic model, the calculated maximum specific methane yield rates were approximately 1.6, 3.7, and 3.6 NmL g VS<sup>-1</sup> day<sup>-1</sup> at 70, 76, and 83% MC, respectively (Table 3). There were no significant differences between 76% and 83% MC test units ( $p > 0.05$ ). However, there was a significant difference between digesters operated at 70% MC and digesters operated at the two higher MCs ( $p < 0.05$ ). The maximum methane yield rates were increased 2.3-fold by increasing the MC from 70 to 83%. These results are in general agreement with those from previous SS-AD studies using other feedstocks. Forster-Carneiro et al. [13] and Fernández et al. [29] showed that biogas production and volatile solids decomposition rates increased as moisture content in anaerobic digesters increased from 70 to 80%.

**Table 3.** Influence of initial feedstock moisture content on Gaussian parameters for specific methane yield from dairy manure. (<sup>a,b,c</sup> Values are means  $\pm$  SE,  $n = 3$ . Means with different superscripts are significantly different ( $p < 0.05$ )).

| Parameters   | MC70                        | MC76                        | MC83                        |
|--|-----------------------------|-----------------------------|-----------------------------|
| Maximum specific methane yield (NmL g VS <sup>-1</sup> day <sup>-1</sup> ) | 1.6 $\pm$ 0.2 <sup>a</sup>  | 3.7 $\pm$ 0.3 <sup>b</sup>  | 3.6 $\pm$ 0.0 <sup>b</sup>  |
| Kinetic parameters   | -                           | -                           | -                           |
| $t_0$ (days)   | 32.7 $\pm$ 0.6 <sup>a</sup> | 18.6 $\pm$ 0.0 <sup>b</sup> | 19.6 $\pm$ 0.5 <sup>b</sup> |
| $a$ (NmL g VS <sup>-1</sup> day <sup>-1</sup> )                            | 1.6 $\pm$ 0.2 <sup>a</sup>  | 3.7 $\pm$ 0.3 <sup>b</sup>  | 3.7 $\pm$ 0.0 <sup>b</sup>  |
| $b$ (days)   | 15.2 $\pm$ 0.8 <sup>a</sup> | 7.2 $\pm$ 0.0 <sup>b</sup>  | 9.1 $\pm$ 0.3 <sup>c</sup>  |

### 3.2.2. Cumulative Methane Production

The profile of experimental and simulated cumulative methane yield is shown in Figure 4 and coefficients of determination ( $R^2$ ) were 0.99 for all conditions, showing a good simulation. Results showed that the methane yield increased with increasing feedstock moisture content.



**Figure 4.** Influence of initial feedstock moisture content on cumulative methane yields from dairy manure. Digesters containing non-amended manure containing 70% MC (MC70) and manure amended to 76 and 83% MC (equivalent to the water holding capacity of dairy manure) (MC76 and MC83, respectively) were incubated at 37 °C for 85 days. Values are means ( $\pm$  SE) of three replicates. Experimental and model-derived results are shown.

Methane yield potential  $P$ , maximum methane production rate  $R_m$ , and lag phase  $\lambda$  are listed in Table 4. Methane yield potentials ( $P$ ) at 70, 76, and 83% MC were 63, 72, and 90 NmL g VS<sup>-1</sup>, respectively. Although methane yield potential values ( $P$ ) from 70% and 76% MC test units were not significantly different, values from 70% and 83% MC test units were significantly different ( $p < 0.05$ ). The methane yield potential at 83% MC was 40% higher than that at 70% MC.

**Table 4.** Influence of initial feedstock moisture content on Gompertz and experimental parameters of cumulative methane yield from dairy manure. (<sup>a,b,c</sup> Values are means  $\pm$  SE,  $n = 3$ . Means with different superscripts are significantly different ( $p < 0.05$ )).

| Parameters  | MC70                        | MC76                        | MC83                        |
|---|-----------------------------|-----------------------------|-----------------------------|
| Cumulative methane production (NmL g VS <sup>-1</sup> ) | 64 $\pm$ 1 <sup>a</sup>     | 73 $\pm$ 6 <sup>a,b</sup>   | 90 $\pm$ 3 <sup>b</sup>     |
| $P$ (NmL g VS <sup>-1</sup> )                           | 63 $\pm$ 1 <sup>a</sup>     | 72 $\pm$ 5 <sup>a,b</sup>   | 90 $\pm$ 3 <sup>b</sup>     |
| $R_m$ (NmL g VS <sup>-1</sup> day <sup>-1</sup> )       | 2 $\pm$ 0.0 <sup>a</sup>    | 4 $\pm$ 0.0 <sup>b</sup>    | 4 $\pm$ 0.0 <sup>b</sup>    |
| $\lambda$ (days)  | 13.9 $\pm$ 0.1 <sup>a</sup> | 8.9 $\pm$ 0.1 <sup>b</sup>  | 7.6 $\pm$ 0.2 <sup>c</sup>  |
| T95 (days)  | 66.7 $\pm$ 0.4 <sup>a</sup> | 38.2 $\pm$ 1.6 <sup>b</sup> | 43.2 $\pm$ 1.5 <sup>b</sup> |
| Effective period to produce methane (days)              | 52.8 $\pm$ 0.5 <sup>a</sup> | 29.3 $\pm$ 1.7 <sup>b</sup> | 35.6 $\pm$ 1.4 <sup>b</sup> |

The methane yield ranged from 64 to 90 NmL g VS<sup>-1</sup> at the three different moisture levels. The levels of methane yield are lower than methane yields of general dairy manure reported by other researchers. Amon et al. [27] reported yields of 126 to 159 NmL g VS<sup>-1</sup> and El-Mashad and Zhang [30] observed yields of 200 to 302 NmL g VS<sup>-1</sup>. They used fresh dairy manure that was not mixed with other bedding materials, whereas this study used a dairy manure that was mixed with a large fraction of pine sawdust bedding and which stayed in the barn for a period of two to three months. Readily biodegradable organic material in the excreted dairy manure was likely degraded and the portion of recalcitrant organic matter (sawdust) increased to some extent during the two to three months.



Due to the two elements described above, dairy manure and sawdust bedding mixtures showed a high refractory fraction (ranging from 0.76 to 0.79) and lower methane yield performance than general dairy manure.

### 3.2.3. Effective Period to Produce Methane

Another important AD performance indicator is the time period required to complete digestion. Our results showed that the technical digestion time (T<sub>95</sub>, the time needed to produce 95% of the methane potential) was significantly longer using feedstock at the lowest moisture content (Table 3). Although the 95% value was arbitrarily chosen as the technical digestion time by researchers [23,26,31,32], it is used as a guideline in the design of the hydraulic retention time (HRT) for anaerobic digesters. According to the modified Gompertz equation, simulated T<sub>95</sub> values were 67, 38, and 43 days at 70%, 76%, and 83% MC, respectively. The lowest moisture content condition (70% MC) took 1.6- to 1.8-fold longer for digestion compared with the higher moisture level test units (76% and 83% MC). The lag time at 70% MC was also 1.6- to 1.8-fold longer than the lag times in digesters using 76% and 83% MC conditions. The effective periods for methane production (calculated by subtracting the lag phase from the T<sub>95</sub> value) were 53, 29, and 36 days at 70, 76, and 83% MC, respectively. Although there was no statistically significant difference between the 76% and 83% MC test units with respect to their T<sub>95</sub> values, there was a significant difference between the T<sub>95</sub> values of the units operated at 70% MC and the values from the units operated at 76% and 83% MC ( $p < 0.05$ ).

## 4. Conclusions

This study evaluated the suitability of bedded pack dairy manure as a feedstock for SS-AD at three different moisture levels normally found on farms. After 85 days of digestion, digesters containing dairy manure at 83% MC outperformed digesters containing dairy manure with 70% and 76% MCs in terms of VS reduction and biogas production. VS reduction and methane yield values were lower than those reported for digestion of fresh manure. However, in terms of efficient management of farm odors and providing a renewable energy source for heating, SS-AD of bedded pack manure offers a potential alternative to the conventional composting systems currently in use. Co-digestion of bedded pack manure with highly biodegradable materials available on the farm is considered as an alternative method to improve bioenergy production efficiency. This will need to be further studied in the future.

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**Author Contributions:** Heekwon Ahn designed the study. Eunjong Kim, Seunghun Lee, and Hyeonsoo Jo set up the experimental devices and analyzed samples. Eunjong Kim, Seunghun Lee, and Heekwon Ahn wrote the paper. Jihyeon Jeong, Walter Mulbry, and Shafiqur Rahman reviewed and revised the manuscript. All authors read and confirmed the final manuscript.

**Conflicts of Interest:** The authors declare no conflict of interest.

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