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ORIGINAL ARTICLE

Enhancing biogas production from chicken manure through vacuum stripping of digestate

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Abstract
The vacuum stripping's combined ammonia removal and disintegration effect on chicken manure digestate was evaluated for the first time at different pH values (8.5, 9.5, and 10.5) and temperatures (30, 50, and 70 °C). In this way, the potential increase in biogas production by recirculating the vacuum-stripped digestate to the anaerobic digester was determined. Experimental results showed that increasing pH and temperature significantly increase TAN removal, but pH is more effective. A significant portion of the ammonia was removed in the first 30 min. Therefore, a second set of stripping tests was performed for 30 min and at 70 °C and pH 10.5. After 30-min tests, a biomethane potential (BMP) assay was performed using the vacuum-stripped digestate to determine how vacuum stripping affects biomethane production. Despite having the lowest disintegration efficiency, the highest biomethane potential (56.2 ± 29.7 mL CH_4/gVS) was obtained with the digestate, which was subjected to vacuum stripping at 70 ℃ without pH adjustment, and 48.7% more methane was produced than the control set. The lower residual biomethane potential in vacuum-stripped digestate at pH 9.5 and 10.5 was attributed to Na^+ inhibition resulting from high NaOH consumption for pH adjustment.

Keywords Methane · Ammonia · Chicken manure · BMP

1 Introduction

Anaerobic digestion (AD) is a well-known process that converts organic waste into bioenergy. Animal manure, particularly chicken manure (CM), is an excellent energy source for AD and is a valuable source of essential nutrients such as nitrogen, phosphorus, and potassium. These nutrients are crucial for plant growth and are commonly found in chemical fertilizers. [1, 2]. Nevertheless, the extensive use of CM in land applications can result in significant environmental issues, including eutrophication, air pollution, the release of greenhouse gases, and the proliferation of pathogens. In the AD process, energy is generated, and CM is stabilized to avoid possible environmental issues [3] that may arise from the direct application of CM to agricultural lands. After anaerobic digestion, stabilized digestate can be applied to agricultural lands to provide nutrients and irrigation water [4, 5] by taking care of nitrate concentration limits given in the EU Nitrate Directive [6]. However, in the AD of CM, the organic nitrogen present in the form of proteins and urea undergoes conversion into ammonia. This conversion process can result in inhibiting the AD process. Elevated levels of ammonia concentration can impede the efficient production of biogas and trigger the accumulation of volatile fatty acids (VFAs). This accumulation can ultimately lead to the complete failure of the anaerobic digestion system [7–9]. Several physicochemical processes such as ammonia stripping [10], ion exchange [11], zeolite adsorption [12], and struvite precipitation [13] have been applied before or during anaerobic digestion to eliminate ammonia inhibition and use the energy content of organic wastes efficiently [14–16]. Ammonia stripping is one of the most common and efficient physicochemical methods. However, the main drawback of it is the fouling of the stripping tower due to scale formation and solid accumulation on the packing materials [17].

Although vacuum stripping is a promising alternative to physio-chemical processes of ammonia stripping that offers advantages over the conventional stripping process, such as a reduced energy requirement, no need for external stripping

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gas under 65°C, and advanced solid-liquid separation, it has only been addressed in a few studies [18–20]. In vacuum stripping, vapor bubbles form in the liquid when vapor pressure overcomes the surrounding pressure, and boiling starts at low temperatures. Dissolved ammonia is carried to the surface with bubbles and transferred to the gas phase. In this way, boiling and bubbling speed up ammonia stripping. Lowering the total ammonia nitrogen (TAN) content reduces the inhibition risk in AD [21–23] and increases the specific biogas yield and biogas generated per volume of digester since more organic matter can be loaded into the digester. In addition to ammonia removal, it was hypothesized that boiling may help the disintegration of slowly biodegradable organic matter in the digestate [18]. In this study, since the effect of vacuum may vary at different temperatures and pH values, we conducted experiments for pH 8.5, 9.5, and 10.5 and temperature values of 30, 50, and 70 °C. Han et al. [24] also reported that vacuum application itself leads to the disintegration of the organic matter in digestate. In addition, ammonia captured during the vacuum stripping process can be recovered and used as a valuable nutrient source in agriculture or for other industrial purposes, and reducing the ammonia content in the digestate helps mitigate the potential for environmental pollution [14]

The aim of the study was to evaluate the increase in biogas production by recirculating the vacuum-stripped digestate to the anaerobic digester. The combined ammonia removal and disintegration effect of vacuum stripping on chicken manure digestate was evaluated at different pH values (8.5, 9.5, and 10.5) and temperatures (30, 50, and 70 °C). Thus, for the first time, it was investigated how much the biogas potential of nitrogen-rich chicken manure could be increased by vacuum stripping.

2 Material and methods

2.1 Batch vacuum stripping tests

2.1.1 Chicken manure digestate

The digestate used in vacuum stripping tests was collected from a full-scale anaerobic chicken manure digester in Afyonkarahisar, Türkiye. The digestate was characterized as soon as it arrived at our laboratory and stored at 4 °C throughout the study. pH, electrical conductivity (EC), TAN, total Kjeldahl nitrogen (TKN), total solid (TS), volatile solid (VS), acidity, and alkalinity values of the digestate were 8.5, 34.2 mS/cm, 4644±475 mg/L, 6226±476 mg/L, 3.68%,1.99%, 2577 mg$CaCO_3^-$/L, and 28500 mg$CaCO_3^-$/L, respectively.

2.1.2 Vacuum stripping setup and operational conditions

Batch vacuum stripping tests were carried out using a 1-L airtight, vacuum-resisted glass vessel connected to a vacuum pump (Artiko, VP250). The temperature of the vacuum vessel was controlled with a heating jacket connected to a digital temperature controller. The digestate was mixed with a magnetic stirrer, and the vacuum pressure was monitored by means of a manometer during the tests. The vacuum stripping test setup is shown in Fig. 1.

In vacuum experiments, two different retention times (120 and 30 min) were tested in order to evaluate the effects of duration on ammonia removal and disintegration of digestate (increase in soluble COD). In addition, the effects of temperature (35, 50, and 70 °C) and pH (8.5–9.5–10.5) were also assessed in 120-min vacuum experiments. Thirty-minute experiments were only performed at 50 and 70 °C (Table 1). pH of the digestate was adjusted with 32% NaOH solution

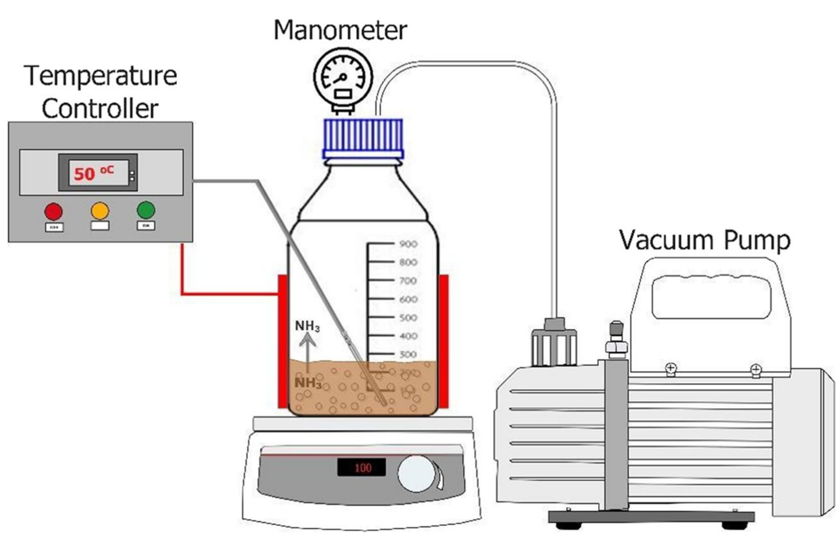

Fig. 1 Batch vacuum stripping test setup

Table 1 Vacuum stripping test conditions

Duration (min)	Temperature (°C)	pH	Influent TAN (mg/L)	Influent FAN (mg/L)	FAN/TAN (%)
120	35	8	4113	416	10.1
		9.5	4422	3451	78.0
		10.5	4596	4470	97.3
	50	8	4567	1026	22.5
		9.5	4391	3959	90.2
		10.5	4565	4516	98.9
	70	8	4509	2136	47.4
		9.5	4936	4769	96.6
		10.5	3555	3543	99.7
30	50	8	4667	1048	22.5
		9.5	5212	4699	90.2
		10.5	4566	4517	98.9
	70	8	4887	2315	47.4
		9.5	4454	4303	96.6
		10.5	5694	5674	99.6

FAN, free ammonia nitrogen

to 9.5 and 10.5. Free ammonia nitrogen concentration was calculated with the formulae given by Hansen et al. [25].

2.2 Biomethane potential (BMP) tests

2.2.1 Inoculum

The inoculum used in BMP tests was collected from a laboratory-scale anaerobic digester in our laboratory fed with CM for more than 200 days. The pH, EC, TAN, TKN, TS, and VS values of inoculum were 7.7, 20.7 mS/cm, 2864±19 mg/L, 2.74%, and 1.63%, respectively.

2.2.2 BMP test setup and conditions

To investigate the effect of vacuum stripping on residual biomethane potential, BMP tests were carried out with 30-min vacuum-stripped and non-stripped digestate. Although 120 min-vacuum stripping was found to be more effective than 30-min stripping in terms of ammonia removal and disintegration of organic matter, 120-min vacuum stripped digestates were not used in BMP tests due to economic concerns.

BMP tests were carried out in 500-ml glass bottles with 400 ml active volume in duplicate under mesophilic conditions (36±1 ℃). Test bottles were continuously stirred on an orbital shaker (PSU-20i Multi-functional Orbital Shaker) placed in an incubator (WTW TS 606-G/2-i). The pH of vacuum-stripped digestates was adjusted with 37% HCl before the BMP test. All BMP bottles were flushed with nitrogen (N_2) gas to provide anaerobic conditions.

2.3 Analytical methods

TS, VS, and TKN analyses were performed according to standard methods [26]. TAN was determined with the nesslerization method using a spectrophotometer (WTW 6100, Germany). pH was measured using a pH meter (WTW 3310, Germany). The biogas formed in the test bottles was collected in the gas balloons and measured periodically by the water displacement method. The biogas composition was determined by a gas chromatograph coupled with a thermal conductivity detector (Shimadzu GC-TCD, Japan) according to method described by Molaey et al. [27].

Water loss due to evaporation (Eq. 1) and the TAN removal performance (Eq. 2) were determined for each experiment. Disintegration efficiency (Eq. 3) was also evaluated by the increase in the concentration of soluble chemical oxygen demand (sCOD) during the vacuum stripping.

$$\text{Evaporation, \%} = \frac{\text{Volume left after stripping, ml}}{\text{Initial volume, ml}} * 100 \tag{1}$$

$$\text{TAN removal Efficiency, \%} = \frac{\text{Initial TAN, mg} - \text{Final TAN, mg}}{\text{Initial TAN, mg}} * 100 \tag{2}$$

$$\text{Disintegration Efficiency, \%} = \frac{\text{Final sCOD, mg} - \text{Initial sCOD, mg}}{\text{Initial sCOD, mg}} * 100 \tag{3}$$

3 Results and discussion

3.1 Batch vacuum stripping tests

Initially, batch vacuum stripping tests were performed at 35, 50, and 70 °C for 120 min, and the effect of pH on disintegration and ammonia (TAN) removal was examined at pH 8.5, 9.5, and 10.5 at each temperature. The TAN removal, the disintegration efficiency, and the amount of water evaporated (evaporation, %) were monitored during the experiments.

One hundred twenty-minute-test results are given in Table 2. Results show that the increase in sCOD, which is an indication of disintegration, is very low at temperatures lower than 70 °C [28]. In addition, we determined that pH was as effective as temperature on disintegration in vacuum experiments and sCOD increased considerably at higher temperature and pH values. However, in the tested ranges, high temperature and pH values individually are not very effective. For instance, at 70 °C, the increase in sCOD concentration is negligible at pH 8.4. Literature studies reported that the optimum temperature for disintegration is in the range of 160–180 °C [29]. The disintegration of organic matter at mild temperatures (e.g., 70 °C) was reported to be successful under only alkaline pH conditions [30]. In

Table 2 Results of batch vacuum tests (120 min)

Temperature (℃)	pH	Evaporation (%)	Disintegration efficiency (%)	TAN removal (%)
35	8.3 ± 0.1	11.3 ± 3.6	7.3 ± 1.7	18.5 ± 7.9
	9.5 ± 0.1	11.6 ± 1.9	< 5	32.7 ± 0.3
	10.5 ± 0.1	12.5 ± 4.1	10.6 ± 6.6	67.4 ± 0.2
50	8.5 ± 0.1	19.5 ± 7.7	< 5	47.8 ± 7.2
	9.5 ± 0.1	36.2 ± 1.4	< 5	87.8 ± 3.2
	10.5 ± 0.1	29.7 ± 10.8	8.9 ± 2.0	90.9 ± 6.4
70	8.4 ± 0.1	69.6 ± 1.9	< 5	93.3 ± 3.3
	9.5 ± 0.1	67.3 ± 10.7	54.2 ± 5.9	97.5 ± 0.2
	10.5 ± 0.1	69.1 ± 9.3	85.5 ± 0.2	94.3 ± 0.8

our study, the disintegration efficiency increased only at 70 ℃ and pH above 9.5 (Table 2). The disintegration efficiency and TAN removal were 85.5%±0.2 and 94.3±0.8%, respectively, at 70℃ and pH=10.5 at 120-min stripping tests. Experimental results showed that increasing pH and temperature significantly increase TAN removal, but pH is more effective. The same result was found in different studies applying a thermochemical pretreatment [1] and air stripping [31] to poultry manure to remove TAN. In another study, Han et al. [24] reported similar TAN removal efficiencies of 97–99% at 65 °C and pH= 9.6. In addition, they also reported that vacuum stripping at 65 °C increases solid solubilization compared to thermal stripping at neutral pH. On the other hand, evaporation results show that 120-min vacuum stripping makes the process cost-inefficient due to excessive evaporation regarding high energy loss, even if TAN stripping increases at higher temperatures (Table 2). Anwar et al. [32] used a reflux unit to densify the evaporated water, but it only decreases the water loss, not energy.

The fact that the water loss by evaporation and thus the energy consumption is very high in the 120-min vacuum experiments showed that the vacuum stripping process should be carried out in a shorter time. Figure 2 shows that a significant portion of the ammonia was removed in the first 30 min of the 120-min vacuum experiments. At 70 °C and pH 10.5, 71.1% TAN removal and 45.9% disintegration efficiency was achieved in 30 min.

Due to the high loss of water and energy by evaporation in the 120-min vacuum experiments, new 30-min vacuum stripping experiments were carried out to obtain the digestate to be used in the BMP test. Since 35 °C is ineffective in both disintegration and TAN removal, the 30-min vacuum experiments were carried out at 50 and 70 °C and at pH 8, 9.5, and 10.5. As seen in Table 3, disintegration efficiency and TAN removal were 39.2±2.6% and 67.0±1.3%, respectively, at 70 °C and pH 10.5. Although results were relatively lower than 120-min experiments, the increase in sCOD was about 40% at 70 °C and pH 10.5. In addition, the fact that the water loss due to evaporation and therefore the energy consumption is lower in the 30-min experiments makes the process more applicable in full-scale operations.

Table 3 Results of batch vacuum tests (30 min)

Temperature (℃)	pH	Evaporation (%)	Disintegration efficiency (%)	TAN removal (%)
50	7.9 ± 0.1	6.8 ± 0.9	7.5 ± 2.9	20.5 ± 0.9
	9.5 ± 0.1	7.8 ± 1.3	15.5 ± 1.1	58.9 ± 5.2
	10.5 ± 0.1	5.4 ± 1.1	16.2 ± 2.2	56.3 ± 11.7
70	8.0 ± 0.1	6.4 ± 2.2	5.8 ± 2.4	38.8 ± 3.7
	9.5 ± 0.1	10.6 ± 0.8	14.4 ± 4.3	64.8 ± 5.6
	10.5 ± 0.1	6.6 ± 0.9	39.2 ± 2.6	67.0 ± 1.3

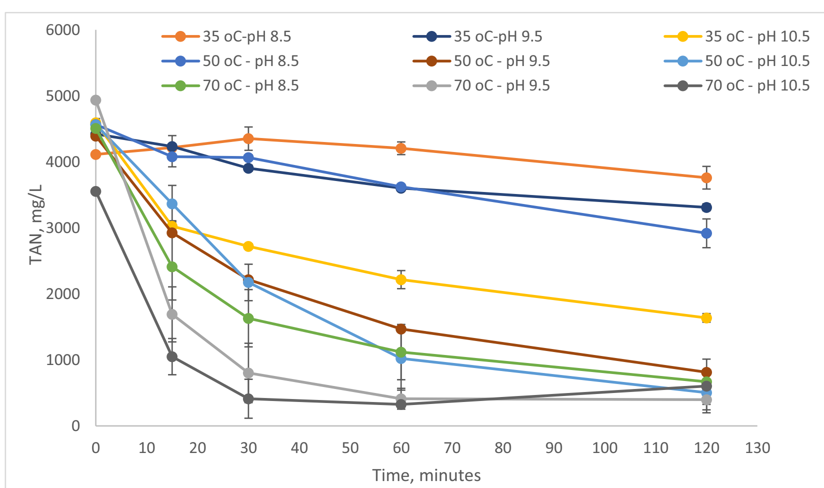

Fig. 2 Ammonia removal in each batch vacuum test

3.2 BMP tests

BMP experiments were performed with the digestate subjected to 30-min vacuum stripping to determine the effect of vacuum stripping on the residual biomethane potential of CM digestate. The BMP test lasted for 43 days, and the conditions of the BMP test and results are presented in Table 4. In the literature, there are studies showing that the treatment processes carried out at mild temperatures and alkaline pH values cause the organic matter to disintegrate slightly and thus increase the biomethane potential [33, 34]. However, our results showed that the increase in sCOD especially at 70 °C and pH 9.5 and 10.5 did not clearly reflect in BMP test results. Although the 39.2±2.6% increase in sCOD of the vacuum stripped digestate at pH 10.5 was significantly higher than that of the stripped digestate at pH 8 and 9.5, the biomethane potential was surprisingly detected below that of other digestates, even the control set. This result is attributed to the inhibition of methane production by sodium, which was added as NaOH to adjust the pH to 9.5 and 10.5 in vacuum stripping experiments. It was determined that the Na^+ concentration increased up to almost 9 g/l, which causes potent inhibition for methanogens, in the sets where the pH was adjusted to 9.5 and 10.5 depending on the amount of NaOH consumed. Methanogens experience moderate inhibition when the concentration of Na^+ ranges from 3.5 to 5.5 g/L. However, a concentration of 8.0 g/L results in a strong inhibition of their activity [32, 35, 36]. On the other hand, some studies also reported that inhibitory compounds, which affect biogas production adversely, might have formed in post-treatment [37, 38]. However, in this study, we did not observe clear evidence of inhibition related to the formation of inhibitory compounds due to alkaline treatment.

The lowest disintegration efficiency and the highest biomethane potential (56.2 ± 29.7 mL CH_4/gVS) were obtained for the digestate subjected to vacuum stripping at 70 ℃ without pH adjustment, and 48.7% more methane was produced compared to the control set. Hence, the highest methane yield is thought to be caused by the lowest influent ammonia concentration among the tests without pH adjustment. It is well known that ammonia is an inhibitory compound that reduces biogas production efficiency. Increased biogas production is a natural consequence of the elimination of the ammonia stress factor. On the other hand, sCOD, which increases with the disintegration effect, can also increase the amount of biogas produced. The increase in biogas production may be due to TAN removal or disintegration or the combined effect of both at the same time. It has not been possible to clearly determine which mechanism is effective with BMP tests alone (Table 4). Therefore, we decided to conduct a long-run continuous experiment by applying vacuum stripping without pH adjustment in the internal recirculation line of a daily-fed laboratory scale anaerobic digester.

4 Conclusions

In this study, we investigated laboratory-scale vacuum stripping of chicken manure digestate under controlled conditions, involving moderate temperatures and alkaline pH levels. We hypothesize that the digestate subjected to vacuum stripping could increase the biogas production when circulated back to chicken manure mono-digester. Considering our experimental findings, we conclude that the increase in biogas production after vacuum stripping occurred through the removal of inhibitory ammonia nitrogen and to a lesser extent the disintegration of residual organic matter in the digestate. The following specific conclusions were also made:

- The highest disintegration efficiency and TAN removal were achieved at 70 ℃ and pH 10.5 after 120 min of vacuum stripping
- The disintegration efficiency and TAN removal were 85.5% ± 0.2 and 94.3 ± 0.8%, respectively, at 70 ℃ and pH = 10.5 at 120-min stripping tests.
- Excessive evaporation causing high energy loss made 120-min vacuum stripping cost inefficient above 55 °C.

Table 4 BMP test and methane production yields

Temperature (℃)	Vacuum Stripping pH	BMP test pH	Disintegration efficiency (%)	NH_4-N (mg/l)	CH_4 yield (mL CH_4/gVS)	Increase in CH_4 yield (%)	NaOH (g/L)
Control	-	8.00		3578	37.8 ± 2.0	-	
50	7.9 ± 0.1	8.07	7.5 ± 2.9	3366	47.7 ± 4.0	26.2	
	9.5 ± 0.1	7.99	15.5 ± 1.1	2580	52.3 ± 11.8	38.4	5.4
	10.5 ± 0.1	7.99	16.2 ± 2.2	2508	36.9 ± 3.0	-	8.7
70	8.0 ± 0.1	8.05	5.8 ± 2.4	3006	56.2 ± 29.7	48.7	
	9.5 ± 0.1	8.00	14.4 ± 4.3	2294	30.0 ± 0.6	-	6.5
	10.5 ± 0.1	8.00	39.2 ± 2.6	2450	42.5 ± 3.4	12.4	8.9

- If vacuum stripping lasted for 30 min, water lost with evaporation decreased significantly, and acceptable TAN removal (71.1%) and disintegration efficiency (45.9%) were achieved.
- The highest residual biomethane potential, which was 48.7% more than that of the control set, was obtained with 30-min vacuum stripping test at 70 ℃ without pH adjustment.
- Lower residual biomethane potentials were obtained by vacuum stripping at pH 9.5 and 10.5 as NaOH used to adjust pH caused Na^+ inhibition.
- The optimum conditions obtained through batch experiments will be validated in a vacuum stripper operated on the internal recirculation line of a daily-fed laboratory scale anaerobic digester.

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Author contribution O.S.: writing, original draft preparation; methodology; investigation; and formal analysis. D.A.: writing, review and editing; supervision; and data curation. A.B.: writing, review and editing; supervision; and data curation. B.C.: resources; writing, reviewing and editing; and supervision.

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Data availability All data is contained within the manuscript.

Declarations

Ethical approval This is not applicable.

Competing interests The authors declare no competing interests.

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