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Ammonia Air Stripping from Different Livestock Effluents Prior to and after Anaerobic Digestion

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Abstract: Livestock digestate provides nutrients and organic matter to the soil while increasing agricultural sustainability. Nevertheless, nitrogen (N) losses due to the nutrient surplus in regions characterized by intensive animal farming activities still represent an unsolved issue. For this purpose, digestate needs proper treatment and management to avoid N losses in the environment. In the livestock farming context, anaerobic digestion (AD) can be accompanied by an ammonia stripping (AS) process for N recovery. This paper aims to investigate the feasibility AS prior to and after AD of the manure, focusing on two different livestock farms, representative of dairy cattle and pig breeding in southern Italy. AS was performed at a lab scale by injecting microbubbles of air, which allowed the pH to increase, and thus the removal of ammonia. The results show that treating a dairy raw slurry with high intermediate alkalinity (IA) (6707 mg $CaCO_3$ L^{-1}) with AS may not be convenient in terms of total ammonia nitrogen (TAN) reduction. As a matter of fact, the loss of buffering capacity during the stripping process resulted in a pH never exceeding the value of 9, which could not promote free ammonia volatilization, whereas integrating AD with AS allowed us to obtain a 34% higher TAN reduction under the same stripping conditions at a temperature (T) of 38 °C and a gas-to-liquid ratio (G/L) of 1:1. Therefore, the AS removal efficiency strongly depends on the characteristics (mainly IA) of the treated matrix. High IA values suggest a possible high concentration of volatile fatty acids, which hinders pH increases and, thus, enables ammonia stripping. Despite the initial matrix origin, a low IA compared to the total alkalinity (TA) (<20% of TA) ensures a greater ammonia removal efficiency, which could be similar between digestate and raw manure in the same operative process conditions. Nonetheless, the amount of ammonia stripped is related to the initial TAN concentration of the specific matrix.

Keywords: ammonia stripping; nitrogen recovery; anaerobic digestion; digestate; manure management

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1. Introduction

The increase in inorganic fertilizer costs, together with environmental safeguard aspects, has pushed towards the necessity of a circular N recovery from agricultural subproducts. A possible strategy is to implement suitable techniques for the treatment of livestock effluents, which also allows for the fulfilment of the other EU concerns about renewable energy production and GHG emission reductions [1]. In this context, a treatment strategy is considered as a process or a set of processes that could modify the amount of manure to be managed and its physical–chemical characteristics, such as N content, in order to enhance manure management [2].

According to Flotats et al. [1], it has been estimated that animal farming produces 1400×10^6 tonnes of livestock effluents every year in Europe. These include animal faeces and urine, together with bedding materials (usually straw if adopted) and water, that can substitute a major part of mineral fertilizers in areas where livestock farms are located if utilized properly [3]. Generally, livestock effluents could be defined as manure, if dry matter (DM) content is higher than 12%, or as slurry, which can be drained or pumped, if it is characterized by high moisture (>88%) [3]. Considerable variations exist among livestock effluents depending on the manure-handling practice [4]. For instance, the adoption of a slatted floor leads to slurry with low DM content due to the water used for washing. Usually, cattle slurry has a higher DM content than pig slurry (i.e., 5–7%), but it is similar to buffalo cows (i.e., 8.1–8.3%), ranging from 7 to 9% [5]. Another manure parameter is the volatile solid (VS) content, which indicates the content of organic material to be converted into methane and carbon dioxide via anaerobic degradation [6]. Generally, it is expected that 80% of DM is volatile in animal manure [3]. This high VS to DM ratio, together with the availability and affordability of animal manure, has widely encouraged its use as a substrate for biogas production via anaerobic digestion (AD). Compared to the initial manure, the digestate is less putrescible, thus being more easily manageable during farm storage and spreading activities due to the decrease in spontaneous organic transformation as well as odour and methane emissions.

Regarding breeding conditions and different animal species, livestock effluents can be characterized by higher or lower N content, which can affect the efficiency of the AD process. The inhibition of methanogens, with the consequent accumulation of volatile fatty acids (VFA), was reported due to the presence of rather high levels of ammonia, which accounts for a range of 1.7–14 g TAN L^{-1} [7]. Specifically, at lower concentrations (1.7–2 g TAN L^{-1}), inhibition occurs for unacclimated bacteria, while 50% inhibition of acclimated microbes has been reported at a concentration of 12–14 g TAN L^{-1}.

Different manure characteristics are reported in the literature. Moreover, as said above, a critical variation can exist among farms rearing the same animal categories [3], also due to different feeding strategies. Heidarzadeh Vazifehkhoran et al. [8] reported a DM content (g kg^{-1}) of 48.0 ($\pm$8.2) and 42.9 ($\pm$0.2) and total nitrogen (TN) value (g kg^{-1}) of 5.6 ($\pm$0.1) and 2.8 ($\pm$0.1) for a liquid fraction of pig slurry from a fattening pig farm and cattle slurry from a dairy cattle farm, respectively. Similarly, Borowski et al. [9] indicated a DM content (g kg^{-1}) of 123.96 $\pm$ 28.20 and 277.18 $\pm$ 20.24 and a TN value (g kg^{-1}) of 6.40 $\pm$ 2.17 and 13.09 $\pm$ 4.16 for swine manure and poultry manure, respectively. In particular, these values referred to a pig farm using no bedding and a laying hen farm with a cage system. On the other hand, Ahn et al. [10] found a DM content of 45, 30 and 429 (g kg^{-1}) and TN values of 2 $\pm$ 0, 2 $\pm$ 0 and 21 $\pm$ 4 (g kg^{-1}) for dairy manure, swine manure and poultry manure, respectively.

Generally, the organic N entering the digesters is converted into total ammonia nitrogen (TAN = NH_3 + NH_4^+) [11]. Thus, a higher N content in the manure influences the TAN concentration in the digestate, as AD does not reduce the TN content of the digesting mixture but increases the TAN/TN ratio. This higher TAN increases the potential N losses (mainly as NH_3) [12] in the environment during the storage and the land spreading of digestate. For this reason, digestate needs further processing [13] and management efforts to mitigate NH_3 emissions [14] and be potentially considered as an effective fertilizer [15], enabling circular nutrient and organic management, thus increasing agricultural sustainability [16]. For this purpose, the most common treatment following AD is solid–liquid separation, which allows for the partitioning of the digestate into two fractions (liquid and solid) with different characteristics. The solid fraction has a lower quantity of water and a higher concentration of organic carbon that can be used for biochar, bio-oil and ethanol production. Conversely, the liquid fraction is characterized by a high amount of soluble nutrients and thus could be used for microalgae cultivation or the extraction of struvite and ammonium phosphate [17]. Among N recovery treatment technologies that can be applied to the liquid phase of digestate [18], such as gas-permeable membrane, ion exchange and adsorption, struvite precipitation, wet scrubber, reverse osmosis or biological treatment (nitrification/denitrification [19]), ammonia stripping (AS) is a robust technique enabling the separation of free ammonia (NH_3), only requiring pH control and a certain amount of energy for aeration [20]. AS could be an easy operating process for N recovery, as NH_3 can be then concentrated in the form of ammonium sulphate. AS is often coupled with AD, both to mitigate the ammonia inhibition on AD and to comply with the energy demand of the AS process. According to the specific aim, an AS tower could precede the AD reactor or come in succession. Nevertheless, AS could be affected by the formation of fouling during the process and elevated energy and chemical requirements for an increase in pH [21].

Based on the above-reported background, the purpose of this paper is to verify the feasibility of ammonia air stripping before and after the AD treatment. For this purpose, the effluents of two different livestock farms, rearing dairy cows and pigs, were considered. The digestate effluents were characterized and compared with the corresponding manure samples to discuss possible differences in each trial and provide suggestions for improving the AS treatment.

2. Materials and Methods

2.1. Source of Raw Slurry and the Liquid Fraction of the Anaerobic Digestate

Cattle raw slurry and the liquid fraction of the digestate were collected from a dairy farm located in the province of Caserta (Campania region, southern Italy). The cattle digestate came from a mesophilic (38 °C) AD plant with a hydraulic retention time (HRT) of 40 days. This plant is fed principally with cattle slurry (78.2%) and with other farm by-products, namely silage (10%), cattle litter (6.6%) and the solid fraction of the raw manure (5.2%). After the AD process, the digestate is sent to the solid/liquid separation. The obtained solid fraction of the digestate is used as bedding material, whereas the liquid fraction of the digestate is currently spread on fields as organic fertilizer for farm crop production.

The pig raw slurry and the liquid fraction of the digestate were sampled in a fattening pig farm located in the province of Salerno (Campania region, southern Italy). The anaerobic digester of the farm is a mesophilic (42 °C) plant, fed mainly with pig slurry (43.2%) and, when available, cattle/buffalo slurry (26.5%), poultry manure (10.4%), vegetation water (6.2%), wet pomace (12.5%) and tomato husks/seeds (1.2%). The plant has an HRT of 55 days, resulting in a highly stabilized solid fraction of digestate, which is used as a soil conditioner. The liquid fraction of the digestate is used in agriculture.

In both farms, the liquid fraction of the digestate was collected from the storage tank of the farm, while the raw slurry was sampled from the loading tank upstream of the AD plant.

After sampling, all the materials were stored at a temperature of 4 °C, then characterized for the determination of total solid (TS) and VS content; total Kjeldahl nitrogen (TKN); and intermediate (IA), partial (PA) and total alkalinity (TA).

2.2. Experimental Set-Up

This study reports findings from two test sets, focusing on the ammonia air stripping application. The tests differed in the initial materials, coming from two breeding types, i.e., dairy cattle and fattening pig farms, and operating conditions.

The stripping experiments were carried out in duplicate, and each test lasted 24 h. Table 1 summarizes the materials used in the tests.

In the first test, AS was performed on both the liquid fraction of the dairy cattle digestate (DD) and the dairy cattle raw slurry (DRS) collected from the same farm, at a temperature (T) equal to 38 °C and a gas-to-liquid ratio (G/L) of 1:1 to compare the efficiency of the process obtained with the two different materials. The same approach was used for the liquid fraction of pig digestate (PD) and pig slurry (PRS), using a T equal to 45 °C and a G/L of 2:1. In this way, the objective was to evaluate the feasibility and performance of the stripping technique for N removal, performed before or after the AD process in different livestock farms.

Table 1. Sources of raw slurry and anaerobic digestate.

Set		Substrate	Breeding Type	Anaerobic Digestion Condition	Biodigester Feeding
1	DD	Liquid fraction of the dairy cattle digestate	Dairy cattle	Mesophile	Cattle slurry, maize silage, cattle litter and the solid fraction of the raw manure
	DRS	Dairy cattle raw slurry	Dairy cattle	—	—
2	PD	Liquid fraction of the pig digestate	Fattening pig	Mesophile	Pig slurry, cattle/buffalo slurry, poultry manure, wet pomace, olive mill wastewater (vegetation water), husks and seeds
	PRS	Pig raw slurry	Fattening pig	—	—

DD = liquid fraction of the dairy digestate; DRS = dairy raw slurry; PD = liquid fraction of the pig digestate; PRS = pig raw slurry.

2.3. Ammonia Stripping

AS was performed on both raw slurries and digestate samples obtained from the dairy cattle and the fattening pig farms. The process was evaluated in terms of ammonia removal efficiency, also discussing the pH, PA and IA trends. As indicated by Matassa et al. [22], prior to the stripping test, the digestate and raw slurry samples were sieved with a 1 mm filter mesh in order to reduce the presence of coarse parts. According to the procedure described by Scotto di Perta et al. [23], 150 mL of the obtained liquid fraction was placed in 500 mL Drechsel glass bottles, characterized by a pore stone (pore size 40–100 µm), through which air as the stripping agent was insufflated. The mesh and the position (2 cm deep) of the pore stone were chosen to maximize the gas-to-liquid mass transfer by inducing the fractionation of the air stream into microbubbles and promoting ammonia removal [24]. Foam formation was avoided by adding 1 mL of vegetable oil in each reactor before the experiment started. Air was supplied through a peristaltic pump, ensuring an airflow rate of 150 and 300 mL min^{-1} in Tests 1 and 2, respectively. Before the aeration started, liquid materials were preheated for one hour to obtain the desired temperature [24]. In order to maintain the temperature during the stripping process, bottles were placed in a thermostatic bath [23]. The operating temperature (see Section 2.2) was chosen in accordance with the operating conditions of the AD plant in the origin farm.

2.4. Analytical Procedures and Calculation

During the first 6–7 h and at the end of the experiment, pH, IA, PA and TA, and TAN contents were monitored by sampling 2 mL of liquid materials from the glass bottles. Standard methods [25] were considered as a reference for measuring TS, VS, TKN and all alkalinity forms. The TAN concentration was analysed spectrophotometrically through the indophenol blue method [26]. The pH values were measured using an HI-98103 pH meter (Hanna Instruments, Woonsocket, RI, USA).

The ammonia removal efficiency Ef_t at any given time t (min) was estimated according to Equation (1), as demonstrated by Laureni et al. [27], considering volume reductions due to water evaporation and sampling:

$$Ef_t = \left(1 - \frac{C_t \times V_t}{C_0 \times V_0}\right) \cdot 100 \quad (1)$$

where C_0 $\left(mg_{NH_4}L^{-1}\right)$ and V_0 (L) are, respectively, the initial ammonia concentration and volume of the liquid substrate, and C_t $\left(mg_{NH_4}L^{-1}\right)$ and V_t (L) are the ammonia concentration and volume of the liquid substrate at time t (min).

The free ammonia concentration [FAN] (mol L^{-1}) was estimated by means of Equation (2) [24]:

$$[FAN] = \frac{[TAN]}{1 + [H^+]/K_d} = \frac{[TAN]}{1 + 10^{pKa-pH}} \quad (2)$$

where [TAN] is the total ammonia concentration (mol L^{-1}), $[H^+]$ the hydrogen ion concentration (mol L^{-1}), and K_a is the acid ionization constant of ammonia (mol L^{-1}). pK_a can be valued as a function of temperature T (K) using Equation (3) [24]:

$$pK_a = 0.09018 + \frac{2729.92}{T} \quad (3)$$

According to Scotto di Perta et al. [23], FAN was expressed as a percentage of the TAN in the liquid solution.

3. Results and Discussion

3.1. Substrates Characterization

The characteristics of the substrates used are reported in Table 2. DRS and PRS showed different TS/VS contents and TA and IA. According to Burton and Turner [28], animal feeding, housing, storage and water dilution are the main aspects affecting the final livestock manure characteristics. For instance, PRS resulted in diluted effluent (TS ~1%), probably due to both the pig feeding and the flushing systems used to remove the manure residues under the slatted floor. A certain variability was also observed in the case of digestate samples, i.e., a lower TAN content in DD than in PD. Indeed, pig slurry is generally characterised by a higher nitrogen content compared to cattle slurry and manure [28], and this is generally accompanied by higher alkalinity [8]. As a matter of fact, as shown in Table 2, PD resulted in the highest TAN content (3795 mg N L^{-1}) and TA (22,493 mg $CaCO_3$ L^{-1}). Comparing the raw slurries with the corresponding liquid fraction of digestate, it can be seen that partial alkalinity was higher in the digestate samples, while IA (corresponding to the alkalinity due to VFAs [29]) was lower in DD and PD rather than in DRS and PRS. These values are broadly consistent with the effect of AD, which allows a reduction in VFA concentration. A higher IA was observed in DRS than in PRS. The most likely explanation is the different livestock feeding standards and metabolisms, which differ among farms and animal categories. In fact, in the case of cattle, the main product during ruminal fermentation is a VFA mixture (acetic, propionic and butyric acids), which is the main source of energy for the animal [30]. On the other hand, the PRS dilution, also confirmed by the low TS content, can be responsible for VFA reduction.

Table 2. Characterization of the raw slurries and digestate samples used in this study.

Test	Sample	TS (g kg^{-1})	VS (g kg^{-1})	pH	TA (mg $CaCO_3$ L^{-1})	PA (mg $CaCO_3$ L^{-1})	IA (mg $CaCO_3$ L^{-1})	TAN (mg N L^{-1})	TKN (mg N kg^{-1})
1	DD	26.3 ± 1.3	17.6 ± 0.7	7.1	8379	6973	1406	1008	1563
	DRS	58.3 ± 0.3	42.7 ± 0.4	7.5	14,193	7486	6707	1626	2939
2	PD	51.7 ± 0.9	34.1 ± 0.7	7.8	22,493	20,935	1558	3795	5555
	PRS	11.0 ± 3.2	5.0 ± 2.3	7.6	9451	8263	1188	1710	2342

DD = liquid fraction of the dairy digestate; PD = liquid fraction of the pig digestate; DRS = dairy raw slurry; PRS = pig raw slurry; TAN = total ammoniacal nitrogen; TKN = total Kjeldahl nitrogen; TS = total solids; VS = volatile solids; TA = total alkalinity; PA = partial alkalinity; IA = intermediate alkalinity.

3.2. Air Stripping Efficiency on Raw Cattle Slurry and the Liquid Fraction of the Digestate

Table 3 shows the effect of AS on the final characteristics of DRS and DD after 24 h of Test 1.

Table 3. Final characterization in terms of partial and intermediate alkalinity and soluble ammonia nitrogen of dairy cattle raw slurry and digestate after 24 h of air stripping at T = 38 °C and a gas-to-liquid (G/L) ratio = 1.

Test	Sample	PA (mg $CaCO_3$ L^{-1})	IA (mg $CaCO_3$ L^{-1})	N–NH_4^+ (mg N L^{-1})
1	DD	4341.5	617.5	396.4
	DRS	2935.5	6384	1277.9

DD = liquid fraction of the dairy digestate; DRS = dairy raw slurry.

As it is possible to observe from Figure 1, PA gradually decreased along the treatment for both DRS and DD, while the IA of DRS remained higher than 6000 mg $CaCO_3$ L^{-1} after 24 h. The PA decrease allowed for CO_2 stripping, thus reducing the bicarbonate and carbonate ion content and the buffer capacity of the matrix [13,21]. CO_2 volatilization led to an increase in pH [11] and to a shift in the ammonia dissociation equilibrium ($NH_4^+ \leftrightarrow NH_3 + H^+$), which depends on pH and temperature, towards the free ammonia nitrogen (FAN) production [31]. FAN is the fraction of the N that can be potentially removed via volatilization.

Figure 1. TAN, FAN, IA, PA and pH trend for (**a**) dairy cattle digestate and (**b**) dairy raw slurry during air stripping. (DD = liquid fraction of the dairy digestate; DRS = dairy raw slurry; TAN = total ammonia nitrogen; FAN = free ammonia nitrogen; PA = partial alkalinity; IA = intermediate alkalinity).

A considerable decrease of 66% for DD and 32% for DRS of ammonia nitrogen was achieved after 24 h. In the case of DD (Figure 1a), the decrease in TAN concentration only started 5 h after the beginning of AS, in correspondence with a FAN higher than 60%. On the other hand, FAN never exceeded 56% in DRS, which may have justified the lower TAN reduction. The most likely explanation for the negative result is the high IA, which suggests a high concentration of VFAs in DRS. Indeed, despite the increase, the pH never exceeded the value of 9, reducing NH_3 volatilization [32] and allowing a residual TAN concentration of approximately 1.3 g N L^{-1} at the end of the test of DRS (Figure 1b).

The same results were found by Walker et al. [32], who reported less efficient ammonia removal via stripping from a digestate characterized by high VFA concentrations.

Generally, it has been found that a pH above 9 guarantees a better stripping efficiency [33].

Other authors [11,27] suggested that ammonia removal efficiency can also depend on the solid content. Laureni et al. [27] indicated that a higher N removal rate was obtained with lower organic matter contents, regardless of the slurry's origin or previous treatment. Palakodeti et al. [11] reported that lower TS content avoids the FAN's adsorption onto suspended solids. Thus, the higher TS content of DRS (58.3 g kg^{-1}) compared to DD (26.3 g kg^{-1}) could also contribute to the low N removal efficiency.

3.3. Air Stripping Efficiency on Raw Pig Slurry and the Liquid Fraction of the Digestate

Table 4 summarizes the results related to the characterization of PD and PRS at the end of the trial.

Table 4. Final characterization in terms of partial and intermediate alkalinity and soluble ammonia nitrogen of dairy cattle raw slurry and digestate after 24 h of air stripping at T = 45 °C and a gas-to-liquid (G/L) ratio = 2.

Test	Sample	PA (mg $CaCO_3$ L^{-1})	IA (mg $CaCO_3$ L^{-1})	TAN (mg N L^{-1})
2	PD	5834.4	1438.8	223.0
	PRS	2349.6	488.4	112.1

PD = liquid fraction of the pig digestate; PRS = pig raw slurry.

Despite the big difference in terms of TAN between PD (3795 mg N L^{-1}) and PRS (1710 mg N L^{-1}) at the beginning of the experiment (Table 2), the initial TAN did not affect the N removal efficiency, which accounted for 94% and 95% of PRS and PD, respectively. This result is broadly consistent with Quan et al. [34], who found a TAN removal efficiency of 97% with an initial TAN in the range of 1200–5459 mg N L^{-1}, demonstrating that the feasibility of AS is independent of TAN content, since it does not affect the mass transfer. Not even the different initial TS content seemed to affect N removal.

Figure 2 shows the main parameters monitored during Test 2. As it is possible to observe, the PA and IA trends followed a similar pattern for both PRS and PD. The total alkalinity and TAN concentration decreased proportionally [35]. Overall, TA diminished in both materials, mainly due to the formation and precipitation of salts and/or CO_2 stripping [36]. The TAN concentration started to decrease after 2 and 3 h for PD and PRS, respectively, in correspondence with a FAN close to or higher than 60%. After the first 7 h of AS, TAN decreased from 3795 to 2002 mg L^{-1} and from 1710 to 947 mg L^{-1} for PD and PRS, respectively. Although the N removal efficiency was similar, a higher TAN removal rate was obtained for PD, as it is possible to notice from the slope of the orange curve in Figure 2. Under the same operating conditions, FAN reached 90% of TAN during the stripping of PD. This fact was due to the increase in the pH to 9.7 in PD, whereas the pH was close to 9.0 in PRS.

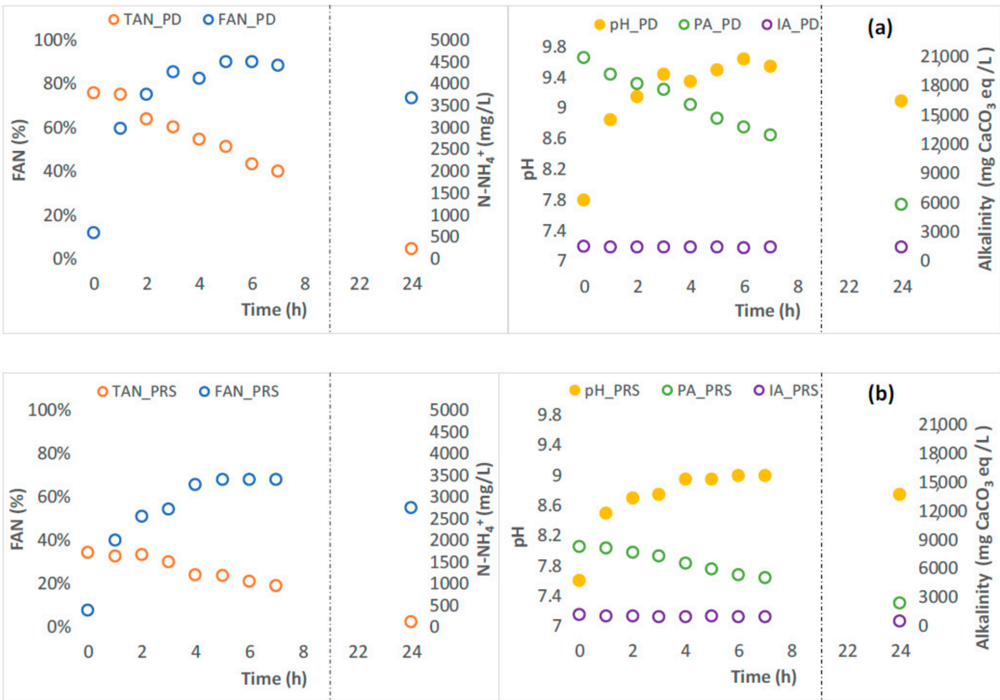

Figure 2. TAN, FAN, IA, PA and pH trend for (**a**) pig digestate and (**b**) pig raw slurry. (PD = liquid fraction of the pig digestate; PRS = pig raw slurry; TAN = total ammonia nitrogen; FAN = free ammonia nitrogen; PA = partial alkalinity; IA = intermediate alkalinity.).

4. Conclusions

The findings of this study are of practical relevance as they provide interesting insights into the effective treatment/recovery of excessive N loads in areas characterized by intensive breeding systems. Furthermore, this study aimed to provide companies with suggestions on what N-rich material to be used for AS in order to obtain a higher N volatilization. Indeed, treating an effluent with high intermediate alkalinity may not be convenient in terms of TAN reduction. Therefore, integrating AD with AS would decrease intermediate alkalinity and enhance the N removal efficiency by up to 34%. Moreover, thanks to the conversion of the produced biogas into electric power, the implementation of AD would cover the operational costs associated with the aeration and thermal power requirement during stripping. Finally, the use of digestate with a higher TAN content resulted in a higher TAN reduction rate in 24 h, implying the possibility of recovering more nitrogen. Further studies focusing on AS application in treating digestate originating from different breeding types would be of interest, as well as a techno-economic analysis of the AS application on a real scale.

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