

## Article

# Ammonia Recovery from Livestock Manure Digestate through an Air-Bubble Stripping Reactor: Evaluation of Performance and Energy Balance

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**Abstract:** The recovery of livestock manure, rich in nutrients, as fertilizer in agriculture, could pose the risk of an excessive load of nitrogen on the soil. Ammonia stripping is one of the available technologies for reducing the amount of nitrogen in the digestate obtained by the anaerobic digestion of manure. The study investigated the performance and energy consumption of a full-scale ammonia-stripping plant, equipped with a bubble reactor and working without the use of any alkaline reagent under semi-batch conditions. Stripping tests were conducted on the liquid fraction of the digestate, studying the current and optimized operative conditions of the plant. The main variables influencing the process were pH, temperature, airflow, and feed characteristics. In the experimental tests, the pH spontaneously increased to 10, without dosing basifying agents. Higher temperatures favoured the stripping process, the higher tested value being 68 °C. The airflow was kept equal to 15 Nm<sup>3</sup> h<sup>-1</sup> m<sup>-3</sup> digestate in the pre-stripping and to 60 Nm<sup>3</sup> h<sup>-1</sup> m<sup>-3</sup> digestate in the stripping reactors, during all tests. The energy requirement was completely satisfied by the CHP (combined heat and power) unit fed with the biogas produced by manure digestion. Results showed anaerobic digestion coupled with stripping to be a suitable solution for removing up to 81% of the ammonium with neither external energy input nor reagent dosage.

**Keywords:** stripping; ammonia recovery; anaerobic digestion; digestate; livestock manure



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## 1. Introduction

Livestock manure management is one of the most critical aspects of animal husbandry. In the scientific literature [1], the value of manure, rich in nutrients and usable as fertilizers on the soil, is recognized. Since ancient times, these substrates have been used as resources to maintain the fertility of the soil [2]. However, land spreading of organic fertilizers commonly used in agriculture (including animal manure, compost, septic sludge, sewage sludge, municipal biosolids and food-processing wastes) could represent a source of pollution, due to the high concentration of nitrogen, and due to the presence of trace elements, such as perfluorinated alkylated substances (PFASs), brominated flame retardants, pharmaceuticals and other toxic substances [3]. An excessive load of nutrients on the soil may pose the risk of the eutrophication of superficial waters and nitrate pollution of aquifers [4], as well as emissions of ammonia into the atmosphere due to the incorrect management of livestock manure residues [5].

To comply with the limits imposed by European regulations (especially the Nitrates Directive [6]) it is necessary to use technologies that aim to reduce the amount of nitrogen in manure. Among the different treatments, the adoption of anaerobic digestion (AD) of animal manure (bovine, swine, and poultry), mixed with agricultural waste and food

residues, is becoming more important [7] and it will play a vital role in the agricultural and energy value chain [8]. The integration of AD into the manure treatment chain does not achieve an alteration of nitrogen content or the composition of other nutrients, but concerns a purely energy-related aspect. However, the effluent of AD is more suitable for subsequent treatments, to obtain the recovery or removal of nutrients such as nitrogen [9]. The electricity produced in CHP (combined heat and power) units by the combustion of biogas can be sold or used in a closed cycle to meet the energy demand of the treatments for the separation and removal of nitrogen [10].

The nutrient composition of digestate varies with the substrate treated in the biogas plant and may contain compounds that stimulate or inhibit soil microbial activity [11]. Moreover, the operating parameters of the AD process have a significant influence on the physical and chemical characteristics of the digestate [12].

The implementation of an efficient and economical solution to recover and reuse nitrogen from livestock manure is essential and will reduce the reliance on chemical fertilizers [13]. The identification of proper technologies is considered one of the main challenges within a circular economy context [14]. Increased recycling of nutrients back to agriculture from organic waste streams is necessary for increased rural–urban sustainability [15].

Various technologies can be used to treat the digestate in order to reduce and/or recover ammonia. However, prior to applying these technologies, removing particulate matter from the digestate is generally necessary [16]. The solid fraction has a high content of organic nitrogen; it can be spread on soils directly or after a composting process [17]. On the contrary, the liquid phase has high contents of ammonium, phosphorus, and other macro- and micronutrients [18]; its recovery/disposal can be a critical aspect.

As concerns ammonia recovery from the liquid digestate, several treatments are available, such as struvite precipitation [19–21], ammonia stripping [22–25], evaporation [26,27], ion exchange and absorption [20], membrane separation processes [1,28,29] and an innovative biological treatment with the use of a membrane bioreactor in thermophilic conditions [30,31]. The main advantages and limitations of these ammonia-recovery processes are listed in Table 1.

**Table 1.** Main advantages and limitations of ammonia-recovery methods.

Ammonia Recovery Method	Main Advantages	Main Limitations	Selected References
Ammonia stripping	<ul style="list-style-type: none"> <li>• Easy to operate</li> <li>• Process stable</li> <li>• Remove odorous compounds and dust particles</li> <li>• Small footprint requirements</li> <li>• Can be applied directly to digesters</li> </ul>	<ul style="list-style-type: none"> <li>• Scaling and fouling</li> <li>• High energy and chemical requirements</li> <li>• High costs for energy, alkali dosage, stripping tower</li> </ul>	[32–36]
Membrane Separation	<ul style="list-style-type: none"> <li>• Gas permeable membrane less prone to fouling</li> <li>• Easy to operate</li> <li>• The addition of chemicals not required</li> </ul>	<ul style="list-style-type: none"> <li>• Fouling and clogging</li> <li>• Need for pre-treatment</li> <li>• High investment costs</li> </ul>	[20,33,35,36]
Struvite precipitation	<ul style="list-style-type: none"> <li>• Mature technology</li> <li>• Low energy costs</li> </ul>	<ul style="list-style-type: none"> <li>• High cost for chemicals (salt of magnesium)</li> <li>• Nitrogen recovery percentage lower than other methods</li> </ul>	[33,35,36]
Ion exchange And absorption	<ul style="list-style-type: none"> <li>• Easy to operate</li> <li>• Low cost</li> <li>• Low energy consumption</li> </ul>	<ul style="list-style-type: none"> <li>• Fouling and competition of foreign ions</li> <li>• May be used as an intermediate step in the digestate treatment</li> </ul>	[20,34,35]
Evaporation	<ul style="list-style-type: none"> <li>• Easy to operate</li> </ul>	<ul style="list-style-type: none"> <li>• High thermal energy needs</li> <li>• Low efficiency</li> </ul>	[20,35]

Table 1. Cont.

Ammonia Recovery Method	Main Advantages	Main Limitations	Selected References
Thermophilic aerobic Membrane reactor	<ul style="list-style-type: none"> <li>The dosage of external alkaline reagents not required</li> <li>Almost complete ammonification of organic nitrogen</li> </ul>	<ul style="list-style-type: none"> <li>High energy requirements for membrane</li> <li>May be used as an intermediate step in the digestate treatment</li> </ul>	[30,31]

Many aspects affect the choice of a suitable technology: (i) the required pre-treatments, which are mainly related to the qualitative characteristics of the digestate, (ii) the robustness of the process and technology, (iii) the need for maintenance and (iv) the investment and operation costs.

Among these abovementioned treatments, ammonia stripping is an easy-to-operate technology, which has already been implemented in full-scale plants and has proven to have low energy costs compared to membrane technologies [20]. In this process, ammonium is removed from the liquid phase with the use of a gas flow. The volatility of ammonium in an aqueous solution can be enhanced by increasing temperature (thus with an energy input) and pH (usually by dosing alkaline chemicals). The recovery of ammonia from flue gas is subsequently obtained in an absorption tower (scrubber) with an acidic solution. Using sulphuric or phosphoric acid, a fertilizer for agricultural use (ammonium sulphate or phosphate, respectively) is obtained.

The efficiency of ammonia stripping is mainly dependent on pH, temperature, air-to-liquid ratio, and feed characteristics [22].

The design of the stripping reactor is another significant aspect that affects the performance of the process: the aim is to maximize the contact between air and digestate (maximum rate of mixing and highest specific surface area) while minimizing energy costs [24]. The two main technological approaches are the packed bed column and the bubble reactor. The first one has been studied and implemented widely; it has been shown that the main issue is related to clogging due to the suspended particles in the treated digestate. Instead, bubble aeration is a simpler technology, easy to implement, despite foaming and the energy balance having been revealed to be the main issues [25]. Hence, only a few applications of air stripping are documented in the scientific literature, although in theory this is a suitable solution. In particular, results of full-scale experiences are lacking. The objective of the research work is to contribute to filling this gap, through the assessment of the process performance and energy balance of a full-scale ammonia-stripping plant in a bubble reactor, working in semi-batch conditions and without the dosage of alkaline reagents. The results of this work, carried out on a full-scale plant that has been running for 4 years and with the use of a real digestate, will be very useful to researchers and farmers in solving the critical issues related to the content of nitrogen in livestock manure.

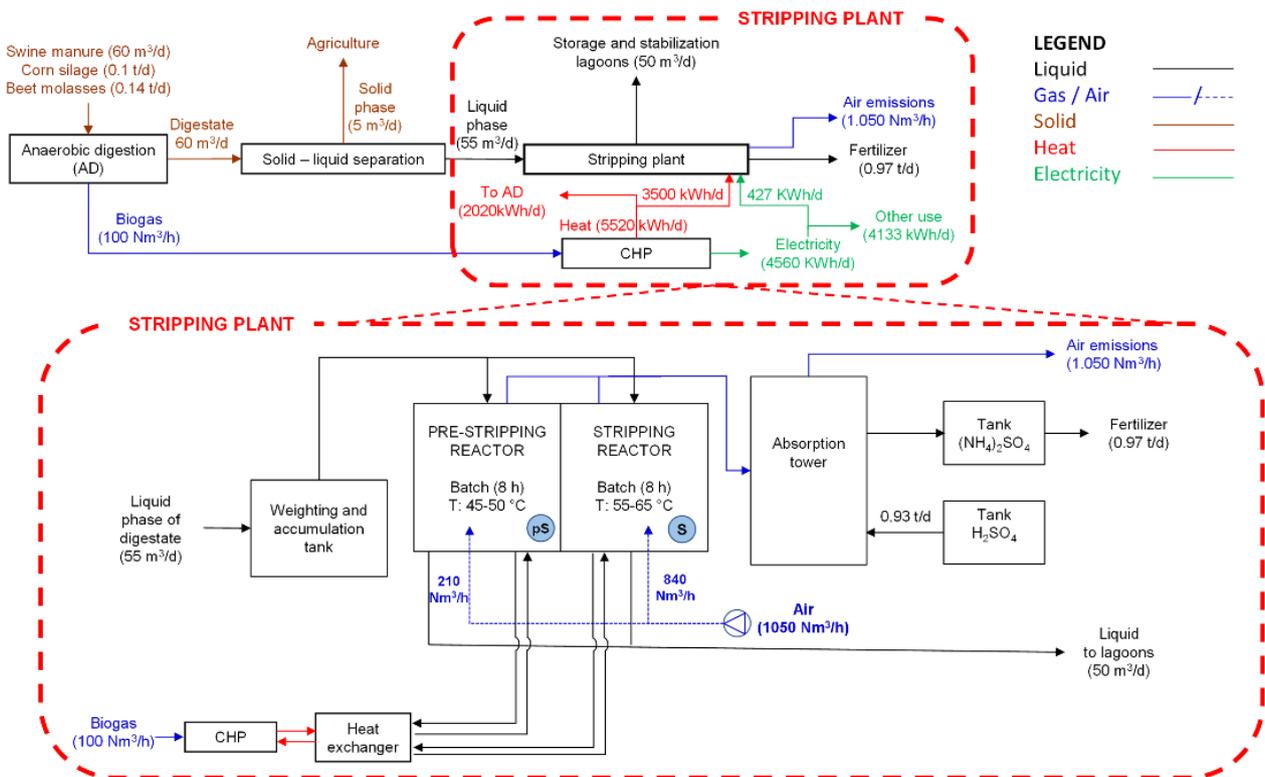
## 2. Materials and Methods

### 2.1. Description of the Study Site

The full-scale ammonia-stripping plant is located at a livestock farm in Lombardy, North Italy. The company's main activities are swine (7400 animals) and bovine (210 animals) breeding, and cultivation (corn, grain, rapeseed and vegetables). The plant was installed to reduce the nitrogen content in the outflow from the anaerobic digestion, by means of the recovery of nitrogen as fertilizer from the liquid digestate; the solid digestate is used for land application. The anaerobic treatment and ammonia-recovery process (Figure 1) includes:

- The anaerobic digesters fed mainly with swine manure (60 m<sup>3</sup>/d), corn silage (0.1 t/d) and beet molasses (0.14 t/d), with biogas production;
- The solid/liquid separation unit, separating the solid phase (recovered in agriculture) from the liquid phase (undergoing stripping);
- The stripping plant, with the production of ammonium sulphate for agriculture;

- The storage lagoons for the stabilization of the liquid digestate;
- The CHP unit, fed with biogas, for the production of electricity and heat reused in the ammonia-stripping reactor.



**Figure 1.** Scheme of the treatment plant with a focus on the stripping system and sampling points (pS and S). The mass and energy flows are referred to the current operating configuration.

The two anaerobic digesters have a total volume of 1800 m<sup>3</sup>, and work at a temperature of 45 °C. The production of biogas is 100 Nm<sup>3</sup>/h.

## 2.2. Description of the Ammonia-Stripping Plant

The ammonia-stripping plant (Figure 1) is composed of an accumulation and weighting tank receiving the liquid fraction of the digestate (hereafter digestate) from the solid/liquid separator. The digestate is then conveyed to the air-bubble stripping reactor, composed of two reactors that can work in parallel and in batch, functioning as pre-stripping or stripping reactors, alternatively. Under current operating conditions, the pre-stripping reactor works with digestate at 45–50 °C for 8 h, while the stripping reactor operates with digestate at 55–65 °C for 8 h. The digestate inside the stripping reactor is heated by a heat exchanger supplied with hot water from the CHP unit. The rating plate data of CHP, supplied with a maximum of 130 Nm<sup>3</sup> of biogas with 54% methane, are  $P_t = 306 \text{ kW}_t$  and  $P_e = 249 \text{ kW}_e$ . Heated air from the CHP is conveyed in a small shed where the air blower is located. Therefore, the stripping air inlet to the air blower has a temperature from 20 °C to 45 °C depending on external weather conditions and season. The temperature of the air provided to the stripping process increases as the blower speed raises. The heated air is conveyed 80% (840 Nm<sup>3</sup>/h, equal to 60 Nm<sup>3</sup>/(h m<sup>3</sup><sub>digestate</sub>)) to the stripping reactor and 20% (210 Nm<sup>3</sup>/h, equal to 15 Nm<sup>3</sup>/(h m<sup>3</sup><sub>digestate</sub>)) to the pre-stripping reactor, by the use of a valve. After 8 h, the valve reverses the fluxes, and the stripping reactor works as a pre-stripping reactor and vice versa.

The air enriched by the ammonium removed from digestate is sent to an absorption tower with a dosage of sulphuric acid (H<sub>2</sub>SO<sub>4</sub>) 50% v/v (volume/volume), to produce ammonium sulphate ((NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>), utilized as fertilizer. Treated air is released into the

atmosphere. The digestate from the reactors is collected in storage lagoons (with a total volume of 36,000 m<sup>3</sup>) for stabilization.

Each reactor, pre-stripping and stripping, has a volume of 25 m<sup>3</sup>, and walls with 10 cm of reinforced concrete and 10 cm of thermal insulation material.

### 2.3. Operating Conditions and Plant Monitoring

Three campaigns for a total of seven tests were carried out to verify the operation and efficiency of the stripping plant, run at the current operating conditions and at different possible implementable configurations (Table 2). The first campaign included four tests (A, B, C, D) conducted under the current operating conditions: the two reactors were working, one as a stripping and the other as a pre-stripping reactor, at the same time, for 8 h, with each supplied 80% and 20% of air, respectively.

**Table 2.** Summary of campaigns and tests carried out and their operating conditions.

Campaign	Test	Operating Conditions		
		Reactors' Modes of Functioning	Heat Settings	Monitoring Time [h]
First	A, B, C, D	Pre-stripping and stripping reactors working simultaneously	Pre-stripping reactor not heated, stripping reactor heated (starting from the temperature of the pre-stripping one)	8
Second	E	Pre-stripping and stripping reactors working simultaneously	Pre-stripping reactor not heated, stripping reactor heated (setting the initial temperature equal to 68 °C)	8
Third	F, G	One reactor working in pre-stripping and then in stripping	Heating activated at the beginning of the pre-stripping phase	16

The second campaign included one test (E) and was carried out under current operating conditions, but keeping a higher temperature (68 °C) at the beginning of the stripping phase.

The third campaign included two tests (F, G), in which one reactor was used, for pre-stripping for 8 h and then for stripping for an additional 8 h, for a total of 16 h, heating the pre-stripping phase from 45 to 65 °C and keeping constant the temperature of 65 °C in the stripping phase. The difference with respect to the first campaign was that here we had the same digestate along the whole test (total duration 16 h), while in the first and second campaigns, the digestate in the pre-stripping and stripping reactors (monitored simultaneously for 8 h) came from two different loading cycles of the accumulation and weighting tank.

Samples were collected at two sampling points (Figure 1). During the first and second campaigns, the following samples were collected:

- from the pre-stripping reactor (pS): from tap, 5 samples at different time intervals (0 h, 0.5 h, 1 h, 3 h, 8 h);
- from the stripping reactor (S): from tap, 5 samples at different time intervals (0 h, 0.5 h, 1 h, 3 h, 5 h, 8 h).

For the third campaign only, in order to get more data, we increased the number of samples:

- from the pre-stripping reactor (pS): from tap, 7 samples at different time intervals (0 h, 0.5 h, 1 h, 2 h, 4 h, 6 h, 8 h);
- from the stripping reactor (S): from tap, 8 samples at different time intervals (0 h, 0.5 h, 1 h, 2 h, 4 h, 6 h, 7 h, 8 h).

Temperature and pH were measured in the field (by means of a WTW SenTix<sup>®</sup> 940 probe) for each sample. Total nitrogen (N<sub>tot</sub>) and N-NH<sub>4</sub><sup>+</sup> were measured in each sample by an external laboratory, according to the DIVAPRA et al. [37] protocols. Additional parameters were analysed only in the first sample of each test, in order to determine

the characteristics of the inlet, and were: residue at 105 °C and residue at 600 °C. These parameters were measured following the IRSA CNR standards [38].

During the campaigns, the following parameters were monitored: electric consumption ( $A_e$ ) of the system (a current clamp meter was used), airflow and electric power of the air blower, thermal power produced by the CHP and biogas feed.

Results were presented by campaign; for the first and third campaigns, the results of single tests were averaged, and the variation was calculated.

The results of the campaigns were tabulated and graphically elaborated using Microsoft Excel (2018). Results were analysed in order to identify the main variables for the stripping process.

The efficiency of ammonium as N ( $\eta_1$ ) and total nitrogen ( $\eta_2$ ) removal were calculated as, respectively:

$$\eta_1 = \frac{(N - NH_4^+_{out} * V_{out}) - (N - NH_4^+_{in} * V_{in})}{(N - NH_4^+_{in} * V_{in})} \quad (1)$$

$$\eta_2 = \frac{(N_{totout} * V_{out}) - (N_{totin} * V_{in})}{(N_{totin} * V_{in})} \quad (2)$$

where:

- $V_{in}$  is the volume of the manure fed to the reactors, equal to 14 m<sup>3</sup>;
- $V_{out}$  is the volume of manure in the reactors at the end of the process, considering the evaporation. The details for the estimation of the evaporated volume are reported in the Supplementary Material.

Based on the results obtained from the three campaigns, a mathematical model was developed to describe the decrease in ammoniacal nitrogen over time. To eliminate the variability over time of the main process parameters, digestate temperature and pH, for each test, both in the pre-stripping phase and in the stripping phase, time intervals were identified in which both the pH and the temperature could be considered constant. For each identified interval, we calculated a time-weighted average of pH and digestate temperature. In each identified interval, the following mathematical model was applied, where  $N - NH_4^+(t)$  and  $(N - NH_4^+)_0$  are the ammoniacal nitrogen concentrations at time  $t$  and time zero, respectively,  $t$  being time and  $k$  the reaction rate constant. A first order reaction has been assumed:

$$N - NH_4^+(t) = (N - NH_4^+)_0 \cdot e^{-k \cdot t} \quad (3)$$

The  $k$  values were calculated using the Excel solver tool, by identifying the best fit curve to experimental data.

#### 2.4. Energy Balance

The power consumption ( $P$ ) was calculated as:

$$P = \sqrt{3} * A_e * 380 * \cos\varphi \quad (4)$$

where  $A_e$  [A] is the electric absorption, 380 V is the 3-phase voltage, and the value of  $\cos\varphi$  (power factor) adopted was equal to 0.9.

The electric energy consumed was calculated as:

$$E_{e,consumed} = P \cdot t \quad (5)$$

where  $t$  is time and corresponds to the duration of a test.

The thermal energy used to raise the temperature of the digestate in the stripping and/or pre-stripping reactor was assessed considering:

- The heating of the digestate mass in the stripping and/or pre-stripping reactor and, at a minimum, in the weighting tanks ( $H_m$ )—assuming that the specific heat of the digestate is equal to that of water;

- The compensation of heat dissipation from stripping and pre-stripping reactors to the external environment ( $H_d$ );
- The compensation of the latent heat of phase transition of the evaporating water ( $H_l$ );
- The heating of the air entering the blower ( $H_a$ ); this was included in favour of safety, as the air is heated with the heat dissipated by the engine, which would not be transferred to the water recovery circuit.

The consumption of thermal energy was calculated as the sum of the above-listed contributions. The details of the calculations are reported in the Supplementary Material.

The amount of electric energy produced by the CHP unit was read on the display of the system, as well as the biogas flow rate. The thermal energy produced ( $E_{t, \text{produced}}$ ) was calculated from the rating plate data of CHP unit.

### 3. Results

In this Section the results of the experimental tests are reported (Section 3.1). In Sections 3.2 and 3.3 the model for residual fraction ammonium decrease and the energy balance are reported, respectively.

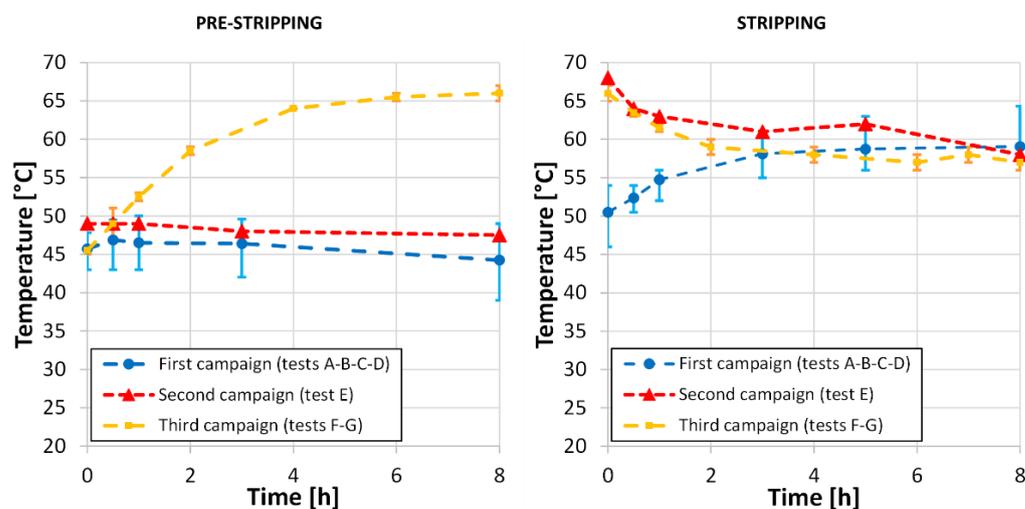
#### 3.1. Nitrogen-Removal Efficiency

The main characteristics of the digestate used in the three campaigns are reported in Table 3.

**Table 3.** Characteristics of the digestate entering the reactor for each campaign (sample time 0). n.a.: not available.  $N_{\text{tot}}$  = total nitrogen.

Campaign	pH	Residue at 105 °C [% as is]	Residue at 600 °C [% as is]	$N_{\text{tot}}$ [mg/L]	$N\text{-NH}_4^+$ [mg/L]
First	$8.5 \pm 0.1$	$2.0 \pm 0.2$	$0.9 \pm 0.2$	$3569.0 \pm 422.3$	$2890.0 \pm 376.9$
Second	8.4	1.4	0.8	2786.0	2234.0
Third	$8.5 \pm 0.1$	n.a.	n.a.	$3300.0 \pm 141.4$	$2900.0 \pm 141.4$

Figure 2 reports the trend of temperature of the digestate in the three campaigns for the pre-stripping and stripping phases. For the first campaign (tests A, B, C, D) in the pre-stripping reactor, the temperature trend is stable, around 45 °C, due to the supply of heated air that compensates for the heat loss. In the stripping reactor, the temperature rose to an average value of 59.1 °C.

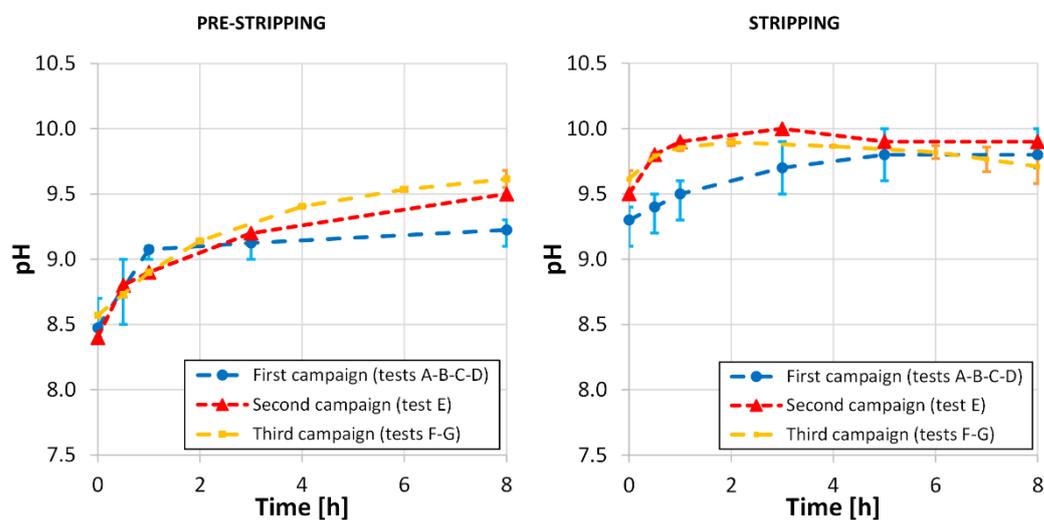


**Figure 2.** Trend of temperature during the pre-stripping and stripping phases for the three campaigns. Baffles show the variation among tests of the same campaign.

In the second campaign (test E) in the pre-stripping reactor the temperature trend was again stable, around 48 °C. In the stripping reactor, starting at a higher temperature, we observed a decrease from an average value of 68 °C ( $t = 0$ ) to 58 °C ( $t = 8$  h). This test, under different operating conditions, was conducted in order to assess possible improvements in the performance of the system, as a consequence of a modification in the heating system.

In the third campaign (tests F and G), the temperature rose from 45.5 °C to 66 °C in the pre-stripping phase and decreased progressively during the stripping phase, reaching 57 °C after 8 h. The pre-stripping temperature registered in the third campaign was very different from the others: the average temperature (weighted upon time) during the third campaign was 61 °C, while it was 46 °C in the first and second campaigns. In the stripping phase, after 8 h the temperature approached to 58 °C for all campaigns.

In Figure 3, the pH trends are shown. In the pre-stripping reactor, the average pH for the first campaign rose from 8.5 to 9.2, and from 9.3 to 9.8 in the stripping reactor. Moreover, we can observe that in the first hour of the pre-stripping, the pH increased by about 50%. In the second campaign, the pH rose from 8.4 to 9.5 in the pre-stripping reactor, and from 9.5 to 9.9 in the stripping reactor. In the third campaign, the pH rose from 8.6 to 9.6 in the pre-stripping reactor, and in the stripping reactor rose to 9.9 to decrease to 9.7 after 8 h.

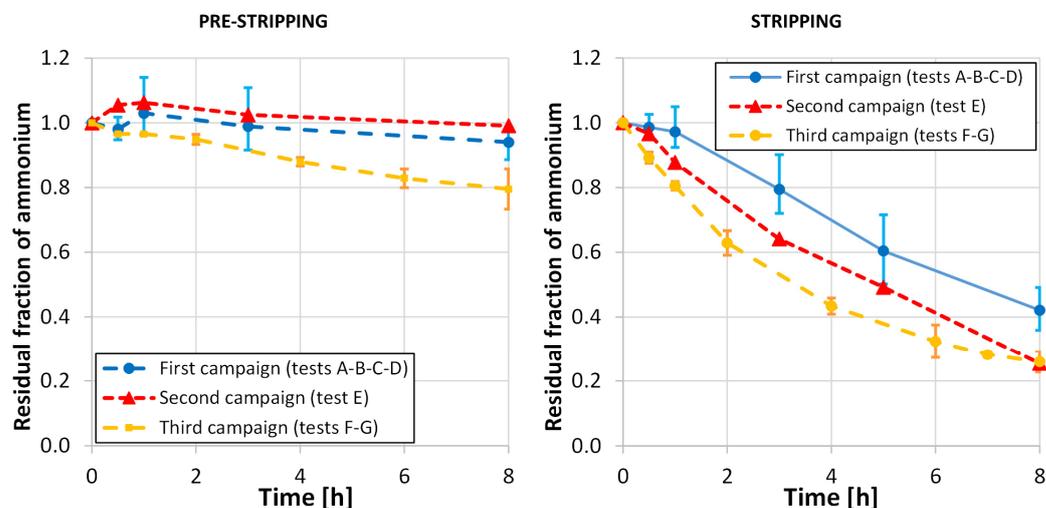


**Figure 3.** Trend of pH during the pre-stripping and stripping phases for the three campaigns. Baffles show the variation among tests of the same campaign.

It is possible to observe that the pH showed an increasing trend in all campaigns along the stripping process. The third campaign shows a higher increase in pH in the pre-stripping phase compared to the others. The second and third campaigns showed higher values of pH than the first campaign in the first part of the stripping, according to the temperature trend (see Figure 2—stripping). Overall, the pH reached similar values among different tests and campaigns, independently from the operating conditions of the plant, at the end of the stripping phase.

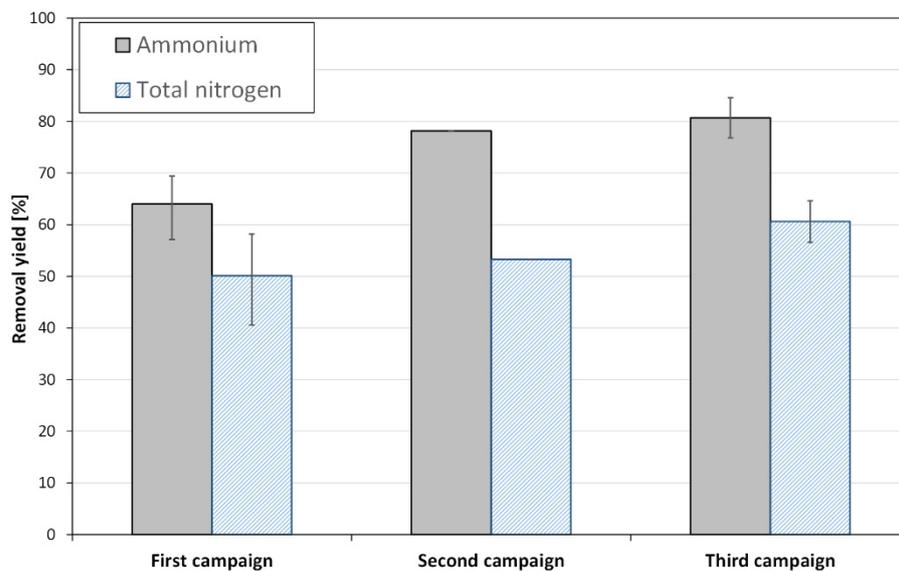
Figure 4 shows the trend of  $\text{N-NH}_4^+$  concentrations normalized with respect to the initial value in the pre-stripping and stripping phases, respectively, for the three campaigns. In the first campaign, values decrease from 1 to 0.94 in the pre-stripping reactor and from 1 to 0.42 in the stripping reactor. In the second campaign, the residual fraction of ammonium was equal to 0.99 after 8 h of pre-stripping, and equal to 0.25 after the following 8 h in the stripping reactor. In the third campaign, the residual fraction of ammonium reached 0.8 after 8 h of pre-stripping, and 0.26 after 8 h of stripping. Overall, the reduction during the pre-stripping phase was lower than during the stripping phase for all campaigns: the reduction was minimal for the first (6%) and second (1%), and it was higher for the third campaign (20%). Ammonium removal was remarkable in the stripping phase (58% in the first campaign, 75% in the second and 74% in the third). In particular, in the second and third campaigns (where the digestate was at a higher temperature at the beginning

of the stripping phase) the overall reduction in ammonium was higher than in the first (corresponding to the current operating conditions) and also faster (an enhanced reduction occurred in the first hours).



**Figure 4.** Trend of the residual fraction of ammonium during the stripping process. Baffles show the variation among tests of the same campaign.

Figure 5 shows the results of the removal efficiency of  $N_{\text{tot}}$  and  $N\text{-NH}_4^+$ , considering the evaporation, for all the campaigns.



**Figure 5.** Removal yield for  $N_{\text{tot}}$  e  $N\text{-NH}_4^+$ , considering evaporation. Baffles show the variation among tests of the same campaign.

For  $N_{\text{tot}}$ , in the first campaign an efficiency of  $50.1 \pm 7.3\%$ , was measured, while it was  $53.3\%$  and  $60.6 \pm 5.7\%$  in the second and third campaigns, respectively.

For  $N\text{-NH}_4^+$ , measured removal efficiencies were  $64 \pm 5.6\%$ ,  $78.1\%$  and  $80.7 \pm 5.5\%$ , during the first, second and third campaigns, respectively.

### 3.2. Mathematical Model of Ammonium Removal

Calculated rate constants of the model reported in Equation (3), for different operating conditions, are summarized in Table 4. Graphical elaborations of the results for the identification of time intervals corresponding to the different operating conditions are presented in Supplementary Material.

**Table 4.** Results of the mathematical model: kinetic constant and initial concentration of ammoniacal nitrogen for each identified time interval in the tests.

Test	Operating Conditions	Time Interval [h]	Time-Weighted Average T [°C]	Time-Weighted Average pH	Q <sub>air</sub> [Nm <sup>3</sup> /h]	(N-NH <sub>4</sub> <sup>+</sup> ) <sub>0</sub> [mg/L]	k [h <sup>-1</sup> ]
D	Pre-stripping	1–8	41.07	9.17	210	3367.5	0.006
C	Pre-stripping	1–8	44.30	9.15	210	3052.8	0.021
B	Pre-stripping	0.5–8	47.83	9.21	210	2418.6	0.012
B	Pre-stripping	0.5–3	48.50	9.13	210	2441.5	0.023
E	Pre-stripping	0.5–3	48.60	9.01	210	2371.7	0.014
A	Pre-stripping	1–8	49.44	9.07	210	3001.4	0.011
F	Pre-stripping	4–8	64.75	9.50	210	2603.0	0.042
D	Stripping	3–8	56.10	9.98	840	1940.3	0.075
G	Stripping	6–8	56.50	9.67	840	863.1	0.126
C	Stripping	1–8	56.57	9.72	840	1957.9	0.091
F	Stripping	2–7	58.90	9.87	840	1267.4	0.165
B	Stripping	3–8	59.90	9.78	840	1768.4	0.130
E	Stripping	0.5–5	61.94	9.94	840	1930.0	0.150

The time-weighted average temperature in selected intervals ranged between 41.07 °C and 64.75 °C in the pre-stripping phase and between 56.10 °C and 61.94 °C in the stripping process. The pH ranged between 9.01 and 9.50 in pre-stripping and 9.67 and 9.98 in stripping. The blower was operated at the maximum speed rate in all the campaigns, and, consequently, the airflow was constant and equal to 210 Nm<sup>3</sup>/h in the pre-stripping phase and 840 Nm<sup>3</sup>/h in the stripping phase. The initial concentration of ammoniacal nitrogen ranged from 3367.5 mg/L to 2371.7 mg/L in pre-stripping and 863.1 mg/L to 1957.9 mg/L in stripping.

The calculated rate constant, being affected by operating conditions, assumed values ranging between 0.006 h<sup>-1</sup> (test D) to 0.042 h<sup>-1</sup> (test F) in the reactor operating in the pre-stripping phase, and ranging between 0.075 h<sup>-1</sup> (test D) and 0.165 h<sup>-1</sup> (test F) with the reactor operating in the stripping phase.

In Test F we can observe the highest values of temperature and pH in the pre-stripping phase (64.75 °C and 9.50, respectively); nevertheless, the value of *k* (0.042 h<sup>-1</sup>) was noticeably lower (from about two to four times) than the values of *k* obtained in all tests in the stripping phase. In the stripping phase, the airflow was four times higher than in the pre-stripping phase, and pH values were higher too, for all tests. We can observe that the parameters that controlled *k* were the airflow and the pH. Additionally, higher values of *k* corresponded to higher temperatures in the pre-stripping phase, and also to higher values of pH, due to the fact that temperature and pH influence one another.

Considering the *k* values in the stripping phase for restricted pH ranges and similar initial ammonium concentrations, it is possible to appreciate the influence of temperature. As an example, considering tests D–C and tests B–E, showing a pH difference of 0.26 and 0.16, respectively, and initial ammoniacal nitrogen concentrations between about 1768 and 1957 mg/L, we observe that tests D–C (with a temperature of about 56 °C), showed a lower *k* than tests B–E (having a temperature of about 60 °C). It can be argued, based on collected data, that temperature is a relevant parameter affecting the removal rate (thus *k*), when the pH is higher than 9.7. These first results need to be further investigated and set the basis for further research.

### 3.3. Energy Balance

The electric absorbance measured for all utilities serving the stripping plant (except the solid/liquid separator) was equal to 30 A, corresponding to an electric power of 17.8 kW<sub>el</sub>. The electric absorbance of the blower ranged between 26 to 28 A, representing the highest contribution to the electric consumption compared to other utilities (pumping and dosing sections). The specific consumption was about 10 kWh/m<sup>3</sup> of digestate treated.

The electric and heating power required by the process for each test compared with the amounts made available by the CHP unit are summarized in Table 5. As can be seen, the electric and heating power supplied by the CHP were sufficient to cover the request for all performed tests. The surplus of electric energy is transferred to the national grid, except for the amount used for the biogas plant. The surplus of thermal energy, when available, is used internally for the digesters, for heating the digestate in the stripping plant, and for other utilities.

**Table 5.** Maximum heating requested for the process for each test compared with the heating power produced by the CHP unit (n.a.: not available).

Campaigns	Test	Electric Power Required by the Process [ $\text{kW}_{\text{el}}$ ]	Electric Power Produced by the CHP Unit [ $\text{kW}_{\text{el}}$ ]	Maximum Heating Power Required by the Process [ $\text{kW}_{\text{th}}$ ]	Heating Power Produced by the CHP Unit [ $\text{kW}_{\text{th}}$ ]
First	A	17.8	n.a.	143	n.a.
	B	17.8	221	166	272
	C	17.8	154	127	189
	D	17.8	190	138	233
Second	E	17.8	n.a.	n.a.	n.a.
Third	F	17.8	217	222	267
	G	17.8	191	214	235

#### 4. Discussion

We conducted three experimental campaigns to study the current and optimized operating conditions for an air bubble ammonia-stripping plant. The first and second campaigns were operated under current conditions, with the second having the digestate heated at the beginning of the stripping phase; in the third campaign, the system was operated as a unique reactor.

Overall, during the first campaign (tests A, B, C, D), we observed that:

- The temperature, during the pre-stripping phase, was kept around 40–45 °C, rising to about 60–65 °C in the subsequent stripping phase;
- Although no basifying reagent was dosed, the pH underwent an increase in the order of 1.1–1.3 points, to reach final values in the range 9.7–10.0;
- The ammonium was, overall, removed during the stripping process, with an average efficiency of 64%;
- The total nitrogen was, overall, removed with an average efficiency of 50%.

During the second campaign (test E), in which current operating conditions were tested under more favourable conditions in terms of temperature (the stripping phase was carried out in full at around 60–65 °C), the ammonium-removal efficiency increased up to 78%. The total nitrogen-removal efficiency increased to 53%. This confirmed that it was possible to improve the process by modifying the operating conditions.

Concerning the third campaign (tests F and G), run under modified operating conditions with the activation of the heating phase already in the pre-stripping phase, the results of the tests (each lasting 16 h) led to the following considerations:

- The temperature of the digestate gradually rose to about 65 °C during the pre-stripping phase, and then lowered during the stripping phase, due to an insufficiency of the current heat-exchange system (although the heating power produced by the CHP could be sufficient);
- Although no basifying reagent was dosed, the pH underwent a significant increase (of the order of 1.2–1.3 points), to reach final values in the range 9.6–9.8;
- The average efficiency removal of ammonium during the stripping process was, overall, about 81%;
- The average efficiency removal for the total nitrogen was about 61%.

In the third campaign, from the first hours in the stripping phase, where the temperature was higher with respect to the current operating conditions (first campaign), the reduction in the ammonium residual fraction was higher. The benefit obtained in the stripping phase due to the increase in temperature, soon after the first moments of treatment, is clearly evident. The concentration of ammonium decreased, in fact, immediately, allowing us to achieve the same efficiency in a shorter time, or to achieve a higher efficiency at the same time. Moreover, during the final hours, the removal trend was slower and tended to stabilize, indicating better exploitation of the process.

In the third campaign, the heating was activated from the pre-stripping phase. This configuration implies that, to operate at a real scale, two heat exchangers are needed, for the pre-stripping and the stripping reactors, respectively, working in parallel. On the contrary, for the first and second campaigns, to operate at a real scale, a single heat exchanger is needed, as in the current configuration, since only one reactor needs to be heated. The configuration used for the second campaign, although appearing favourable, implies heating the stripping reactor to a set temperature, without air, to avoid starting the stripping process. This procedure could require about two hours, which are not counted within the 8 h of operation. This procedure makes the operating conditions used for the second campaign not applicable at full scale. While the operating conditions of the third campaign appear better than the current ones, the only way of implementing them without changes in the plant would be to operate a unique reactor for 16 h; on the other hand, this solution involves an under-exploitation of the potentiality of the plant because a lower flow would be treated, compared to the use of the two reactors in parallel.

The main variables influencing the process were identified as the pH, the temperature of the digestate, and the airflow. In Baldi et al. [24], the temperature was recognized as the main parameter affecting the ammonia-stripping performance, when no alkaline agent is added. The temperature increase in the digestate was demonstrated to successfully increase ammonia recovery in multiple studies [32,39,40]. Moreover, among other variables influencing the process, i.e., the pH and airflow, the latter is the controllable one. These results would set the basis for further experiments aimed at testing the process efficiency under different operating conditions with experimental investigations.

From the results obtained through the kinetic study, we observed that in the pre-stripping phase, the ammonium-removal rate constant took on a value an order of magnitude lower than in the stripping phase. For example, in test B it passed from a value of  $0.012 \text{ h}^{-1}$  in the pre-stripping phase to a value of  $0.13 \text{ h}^{-1}$  in the stripping phase. The latter provides a flow rate of air four times greater than that supplied in the pre-stripping phase, working at higher temperatures and pH, and consequently ammonium decreased faster. It should be noted that other parameters can influence the process, such as the stripping air temperature and the initial concentration of  $\text{NH}_4^+$ , which are not easily controllable in a full-scale plant. Contrarily, the airflow is the controllable parameter among those listed, as it is possible to set the blower speed in the control panel. As mentioned above, the airflow variation can be a parameter to investigate in future experiments.

Overall, across all the campaigns, we observed that the CHP was able to fully satisfy the thermal and electric needs of the plant, with the blower showing the highest electric consumption. It has to be noted that, although there was thermal power available, with the heat exchanger present in the plant, it had not been possible to maintain the temperature constant above  $60\text{--}65 \text{ }^\circ\text{C}$  during the stripping phase. These considerations are valid for the present configuration, and they could not be true anymore if, e.g., a second heat exchanger would be installed, as under hypothetical operating conditions as for the third campaign.

Finally, the main advantage highlighted by the study was that the plant reached good results in terms of ammonium removal without the addition of a basifying agent (i.e., soda). Optimized operative conditions could be foreseen for the use of the plant as a unique reactor (treating less digestate) in batch, or to modify the plant by adding a heat exchanger or a bigger reactor (double volume). In this last case, in addition to the expenses

to modify the plant, the rheology of the fluid should be investigated because of the risk of the formation of foams.

## 5. Conclusions

Under current operating conditions, with the temperature around 45 °C during the pre-stripping phase, and rising to about 59 °C in the subsequent stripping phase, the studied plant showed an ammoniacal nitrogen-removal efficiency of 64%. During the test, which analysed operating conditions more favourable to the stripping process in terms of temperature (the whole stripping phase was conducted around 60–65 °C), the removal efficiency of ammonium rose to 78%. As regards the modified operating conditions with the activation of the heating phase already in the pre-stripping phase (with the temperature rising from 45.5 °C to 66 °C), it was possible to obtain an ammoniacal nitrogen-removal efficiency of 81%. The pH, in all tests, increased without the addition of alkaline reagents. The electricity consumption for the operation of the plant was about 10 kWh/m<sup>3</sup> of treated digestate, with the blower contributing for about 90% of the overall consumption. The CHP unit was able to meet the thermal requirements of the plant in its present configuration. Our results suggested that, simply by modifying the operating conditions, there is room for improving the ammonium-removal rate further.

**Supplementary Materials:** The following supporting information can be downloaded at: <https://www.mdpi.com/article/10.3390/en16041643/s1>, Estimation of the volume of manure evaporated; Consumption of thermal energy; Graphical elaborations of the results for the identification of time intervals for the model for ammonium residual fraction decrease. Reference [41] is cited in the supplementary materials.

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