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Nitrogen Recovery from Different Livestock Slurries with an Innovative Stripping Process

Ali Heidarzadeh Vazifehkhoran, Alberto Finzi *, Francesca Perazzolo, Elisabetta Riva, Omar Ferrari and Giorgio Provolo

Department of Agricultural and Environmental Sciences, University of Milan, Via G. Celoria, 2, 20133 Milano, Italy; ali.heidarzadeh@unimi.it (A.H.V.); fraperazzolo@gmail.com (F.P.); elisabetta.riva@unimi.it (E.R.); omar.ferrari@unimi.it (O.F.); giorgio.provolo@unimi.it (G.P.)
* Correspondence: alberto.finzi@unimi.it

Abstract: Ammonia (NH_3) emissions deriving from the management of livestock manure have a significant environmental impact, and therefore it is important to reduce them. Among the available options, the process of NH_3 stripping is promising to remove NH_3 from manures and digestates recovering it as a mineral fertilizer (e.g., ammonium sulfate) that is more widely adoptable on farms. The traditional stripping process takes place in batches; however, in this study, a continuous process was evaluated using a lab scale plant in which four reactors were used in series with different hydraulic retention times (HRTs) of 12 or 20 days. The NH_3 recovery of each reactor was studied for the liquid fraction of pig slurry, dairy cattle slurry and digestate, applying simple headspace aeration. For 20 days of HRT, totals of 92%, 83% and 67% of NH_3 were stripped from the digestate, pig slurry and dairy cattle slurry, respectively. For 12 days of HRT, total NH_3 recoveries were 83%, 60% and 41% for the digestate, pig slurry and dairy cattle slurry, respectively. The inlet NH_3 concentration and inlet total alkalinity had a positive and negative effect, respectively, on the specific NH_3 removal rate for each reactor. Stripping NH_3 on farm scale can abate NH_3 emissions in response to the environmental concerns of European policies.

Keywords: manure management; nitrogen recovery; aeration; ammonia stripping; alkalinity

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1. Introduction

The proliferation of the human population and food demands has led to a rapid expansion of the livestock industry and consequently massive production of animal manure [1], which has been associated with the pollution of water resources and soil [2,3]. Fresh and digested manure contain recoverable nutrients including nitrogen (N), phosphorous (P) and potassium (K) that are fundamental macronutrients for satisfactory crop productions [4]. Although effluents are already available on livestock farms with no costs, the application of manure and digestate rich in N (mostly in form of NH_3) might lead to environmental problems, particularly in areas that already have excess N [5,6]. Therefore, the adoption of treatments able to reduce the N content in animal manure slurries is necessary to abide by environmental legislation and make the materials safely utilizable on fields [5,6].

The development of various treatment technologies for manure management (e.g., anaerobic digestion and solid–liquid separation) has led to the mitigation of several aspects, including the odor problem, nutrient recovery or removal, energy production, improvement of fertilizer value, and the reduction of the pollution potential and GHG emission [7–9]. In most treatment processes, the removal of Carbon (C), N and P has been the main objective, which can be achieved through the nitrification–denitrification process that releases C as CO_2 and N as N_2 to the air [10,11].

However, in view of sustainable processes to be listed within the circular economy principles, the main goal should be the recovery of nutrients and not only the reduction

of nutrients and of emissions to the atmosphere [10,12]. In this context, N recovery plays a fundamental role because it is beneficial in two ways: first, for the minimization of the negative environmental impacts (acidification and eutrophication) and the consequent social benefit for human health (PM2.5 formation); second for the revenue generation by the transformation of N to nitrogenous fertilizers [13], thus, enhancing the relationship of livestock manures with a circular economy [14].

Various technologies can be used to recover NH_3 from slurries, such as air/steam stripping, chemical precipitation, and membrane separation [15,16]. However, prior to the application of these technologies, the removal of particulate matter from manure and digestate is generally necessary [17]. Regarding air stripping via direct aeration, the use of thermal vacuum stripping and of gas-permeable membrane stripping seem to be the most practical alternatives due to their efficiencies, limited disadvantages, favorable costs and ability to produce ammonium fertilizers [13]. Therefore, these processes are particularly used for the treatment of waste streams that contain high NH_3 concentration, such as pig manure and digestate produced from the anaerobic treatment of animal and other wastes [6,18].

Furthermore, due to its stability and technical simplicity, air stripping technology is suitable and economical for use by both farmers and industry specialists alike and often coupled with acid absorption. Its possible future implementation on farm scale will lead to important effects on the nutrient management of livestock slurries, which is in line with the objectives of the European Commission defined in the Farm to Fork strategy (reduce nutrient losses by at least 50% by 2030) and specifically with the principle of recycling of organic waste into renewable fertilizers.

Moreover, transforming N from livestock origin to a N-form with higher absorption efficiency from the cultivated crops on field represents a relevant improvement for the livestock sector and for the related negative environmental impact attributed to slurry management practices [19,20]. Moreover, using efficiently this substrate already available on farms could help reduce the need to purchase mineral fertilizers from the market, and particularly of urea, which is produced through an energy-intensive process [21].

To properly perform an air stripping process, there are several important factors that need to be evaluated and that impact on NH_3 recovery; these are temperature, pH and air flow rate [22,23] as well as the design and process costs [24]. In the literature, many studies have evaluated the effect of liquid temperature on NH_3 removal by air stripping, generally indicating NH_3 recovery from 60% to 90% already at temperatures around 30–40 °C [6,13,25]. However, it is known that with higher temperatures (70–80 °C), the process is much faster (i.e., a duration of few hours vs. days) [26].

Generally, electricity or surplus heat from cogeneration biogas plants present on livestock farms can be used to address the energy required for heating the materials to be stripped [24]. Similarly to temperature, an increased pH shifts the NH_3 equilibrium reaction towards the production of molecular NH_3, which is favorable for stripping. Many researchers have proven the positive influence of pH increase on NH_3 stripping [23], commonly by using sodium hydroxide, lime, calcium or sodium carbonate [27].

Although increased pH values from 8.5 to 10 improved the removal of NH_3 dramatically, a negligible effect was observed by increasing pH beyond 10 [22]. Provolo et al. [6] observed that adjusting the acidity of digestates to pH 9 increased the NH_3 recovery by 23% and 18% in comparison to the digestates without pH adjustment at 30 and 40 °C, respectively. However, some studies have demonstrated that without adding chemicals, the pH adjustment accomplished by only CO_2 stripping can achieve more than 90% NH_3 removal from digestates [28].

Instead, air flow has no effect on the concentration of free NH_3 in the liquid phase. Rather, high aeration rate reduces the mass transfer resistance by rapidly depleting the boundary layer above the liquid surface, thus, allowing a higher diffusion of gaseous NH_3 due to the increase in the interfacial area [23,29]. A high aeration rate might reduce the liquid temperature and cause foaming and evaporation of the liquid phase [23], thus,

affecting the process performance. The aeration time might also play an important role in the NH_3 stripping process depending on the aeration regime and strategy [28].

Another important aspect to consider is the design of the stripping reactor because it provides the contact between the gas and liquid phases. The two main reactor designs are the packed bed column and the bubble aeration reactor. Packed bed technology has been studied and implemented widely and has been shown to be prone to clogging due to fouling caused by the suspended particles in the treated substrate [13,23]. Instead, bubble aeration is easy to implement and extensively used, although foaming and cooling are the main obstacles when this approach is used to treat digestate. Provolo et al. [6] developed a slow-release stripping process that was simple and easy to implement as they extracted NH_3 in gas phase while it blew over the surface of the liquid being treated.

Most of the previous studies on the stripping process were conducted in the batch and fed-batch mode and conducted at very low HRTs, e.g., 180 min [30] or 24 h [1]. Instead, in the present study, four reactors were positioned in series to achieve NH_3 stripping with a continuous process using headspace aeration without pH adjustment of the liquid phase. With respect to the batch process, the option of conducting NH_3 stripping in a continuous process and, in this study, has yet to be adequately evaluated for stripping animal slurries.

The use of reactors in series has allowed the study of NH_3 removal at a broad range of HRTs, based on the inlet quality of substrates that changed from one reactor to another. Six different slurries (e.g., pig slurry, dairy cattle slurry and digestate) were used as substrate to examine NH_3 recovery in relation to the inlet quality and composition of the substrates at different HRTs of 12 and 20 days. The objective of the study was to evaluate the effect of HRT, type of substrate and substrate physicochemical properties on NH_3 recovery in a continuous process with a series of simple mixed tanks that used headspace aeration as an inexpensive slow-release and capture technology.

2. Materials and Methods

The experiments were conducted using a set of four stripping reactors (named R1, R2, R3 and R4) positioned in series as reported in Figure 1.

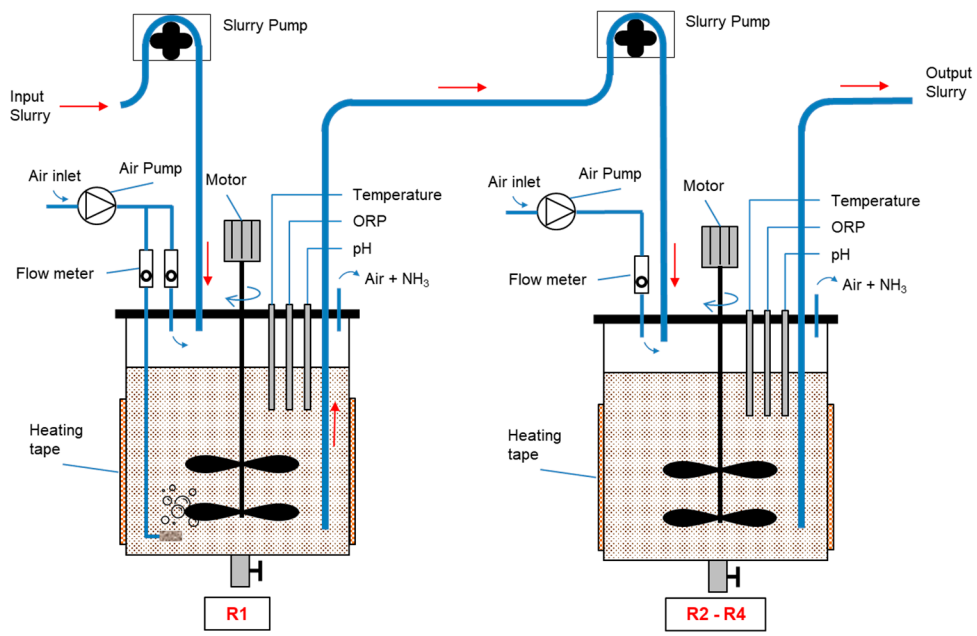

Figure 1. Reactors configuration for continuous ammonia stripping. The slurry or digestate was added to R1 (where the stripping process started) and then was discharged from R4 (where the stripping process ended), passing through R2 and R3. Daily, in each reactor, the same quantity of slurry or digestate was discharged (downstream) and then recharged (upstream).

Each reactor consisted of a cylinder (net volume of 50 L, diameter and height of 40 cm) made from poly-methyl-methacrylate and fitted with a polyvinyl chloride lid. Each reactor and its lid were bolted together and a neoprene gasket between them ensured an airtight seal. All the probes and the inlets and outlets of the air and of the treated substrate were fitted through the lid. The reactors were continuously mixed at 60 rpm using a vertical mixer with two pairs of blades. The reactors were maintained at 40 °C using a heating mat (RS Components, Corby, UK) to promote NH_3 volatilization.

Four peristaltic pumps (M1500, Verderflex, Castleford, United Kingdom) allowed the transfer of digestate in sequence from R1 to R4 through pipes inserted in the reactors. No chemical additives were used to adjust pH. The NH_3 was removed in a flow of air (30 L min^{-1}) through the headspace in reactors R2–R4, while in R1 the airflow was subdivided between the headspace (20 L min^{-1}) and the bottom of reactor (10 L min^{-1}). In R1, the air into the bottom was blown for 8 h per day into the slurry through air stones in order to strip CO_2 and to achieve a natural increase of pH [17].

The air was pumped into the reactors using an air pump (EVO30 series, ELECTRO A.D., Barcelona, Spain) and the air flow was adjusted using manual flow meters (2–30 L min^{-1}, Key Instruments, Trevose, PA, USA). The air flow in the reactors was adequate to inhibit the production of methane and to avoid nitrification. The four-reactor treatment plant was fully operational after a full cycle of HRT (12 or 20 days), after which the experiments began. In the startup period, the process was monitored to confirm stable performance.

Each slurry (i.e., substrate) consisted of the liquid fraction of either pig manure, cattle manure or digestate. Pig slurry (PS) was collected from a fattening pig farm, dairy cattle slurry (DCS) was collected from a dairy farm, and digestate (D) was collected from a biogas plant fed with pig and cattle slurries. Every substrate was tested for 12 and 20 days of HRT, i.e., 3 and 5 days of HRT for each reactor. During an experiment, R1 was loaded with the raw material at four equally spaced times each day. The same quantity was shifted from one reactor to the next until it was ultimately discharged.

The process was monitored using both continuous and periodic measurements. The pH (PHE-1335, Omega Engineering), oxidation/reduction potential (ORP) (ORE-1311, Omega Engineering), and temperature (Pt1000, Italcoppie, Cremona, Italy) of reactors were continuously monitored. These probes were connected to a control unit for each reactor, monitored and managed by specifically developed software using Labview® (National Instruments, Austin, USA). The sampling was conducted daily until the total ammoniacal N (TAN) reached a steady state.

The samples taken on the last three days were used to determine the performance of the process. All samples were analyzed using standard methods [31] for the content of ash (ASH), total solids (TS), volatile solids (VS), total Kjeldahl N (TKN), TAN, alkalinity (ALK), pH and ORP. Organic N was calculated by subtracting TAN from TKN. Treatments designated PS12, DCS12 and D12 were pig slurry, dairy cattle slurry and digestate, respectively, treated with 12 days of HRT. Likewise, pig slurry, dairy cattle slurry and digestate used for experiments with 20 days of HRT were named PS20, DCS20 and D20, respectively. The experimental conditions are summarized in Table 1.

In addition to NH_3 removal of the whole treatment process (which is independent from time and volume of liquid slurry), the specific NH_3 removal rate (SARR) was utilized to indicate the volumetric daily NH_3 removal rate of each reactor in relation to inlet and outlet TAN and HRT. SARR (g kg^{-1} d^{-1}, Equation (1)) was calculated as:

$$\mathrm{SARR} = (\mathrm{TANi} - \mathrm{TANo})/\mathrm{HRT} \quad (1)$$

where TANi and TANo (g kg^{-1}) are the total NH_3 concentration of substrate into and out of each reactor, respectively, and HRT (day) is the individual hydraulic retention time of each reactor (3 or 5 days).

Table 1. Experimental conditions of the study.

Test	Slurry	Reactor	Individual HRT per Reactor (Days)	Overall HRT (Days)	T (°C)	Headspace Airflow Rate (L min^{-1})	Intermittent Injection Airflow Rate (L min^{-1})
1	Pig (PS12)	R1	3	3	40	30/20	0/10 [1]
		R2		6		30	0
		R3		9			
		R4		12			
2	Pig (PS20)	R1	5	5		30/20	0/10 [1]
		R2		10		30	0
		R3		15			
		R4		20			
3	Cattle (DCS12)	R1	3	3		30/20	0/10 [1]
		R2		6		30	0
		R3		9			
		R4		12			
4	Cattle (DCS20)	R1	5	5		30/20	0/10 [1]
		R2		10		30	0
		R3		15			
		R4		20			
5	Digestate (D12)	R1	3	3		30/20	0/10 [1]
		R2		6		30	0
		R3		9			
		R4		12			
6	Digestate (D20)	R1	5	5		30/20	0/10 [1]
		R2		10		30	0
		R3		15			
		R4		20			

[1] In R1 the air was injected for 8 h per day at the bottom of the reactor.

A stepwise linear regression model was conducted for statistical analysis to evaluate the significance of each independent variable on SARR from all the slurries in each reactor (R1, R2, R3 and R4). Individual HRT, inlet VS, inlet ASH, inlet TAN, inlet organic N, inlet pH and inlet ALK were chosen for further statistical and linear regression analysis as independent variables. They were analyzed using IBM SPSS® 27 software (IBM Corp., Armonk, NY, USA). The independent variables were chosen by stepwise linear regression analysis to avoid collinearity.

3. Results and Discussion

3.1. Physicochemical Characteristics of Input Slurries and Reactor Outputs

The physicochemical characteristics of untreated slurries used as inputs as well as of these substrates inside the reactors at the steady state phase are reported in Table 2. The characteristics of the input slurries and digestate are representative of the livestock context of the study area [32].

Table 2. The characteristics of the separated liquid fractions of pig slurry, dairy cattle slurry and digestate (input and inside the R1, R2, R3 and R4 reactors). The mean values are shown with standard deviation (in brackets).

	Pig Slurry					Dairy Cattle Slurry					Digestate				
	Input PS12	**R1**	**R2**	**R3**	**R4**	**Input DCS12**	**R1**	**R2**	**R3**	**R4**	**Input D12**	**R1**	**R2**	**R3**	**R4**
HRT (Days)	**-**	**3**	**6**	**9**	**12**	**-**	**3**	**6**	**9**	**12**	**-**	**3**	**6**	**9**	**12**
TS ($g\ kg^{-1}$)	48.0 (±8.2)	57.7 (±0.9)	52.7 (±3.7)	49.6 (±4.4)	50.9 (±2.7)	42.9 (±0.2)	36.1 (±0.6)	37.7 (±1.7)	38.7 (±0.8)	35.2 (±0.9)	33.1 (±3.7)	37.7 (±0.8)	40.7 (±1.5)	45.3 (±0.6)	51.5 (±1.3)
VS ($g\ kg^{-1}$)	26.5 (±5.7)	32.6 (±0.7)	29.4 (±3.1)	26.8 (±3.0)	27.1 (±2.9)	29.1 (±0.2)	24.2 (±0.4)	25.1 (±1.3)	25.5 (±0.8)	22.9 (±1.2)	20.9 (±2.0)	23.7 (±0.7)	24.9 (±0.5)	28.0 (±0.7)	31.8 (±0.6)
Ash ($g\ kg^{-1}$)	21.6 (±2.1)	25.1 (±0.5)	23.3 (±1.1)	22.7 (±1.9)	23.7 (±0.7)	13.9 (±0.1)	11.9 (±0.3)	12.6 (±0.4)	13.2 (±0.5)	12.3 (±0.3)	12.2 (±0.9)	14.0 (±0.2)	15.8 (±1.0)	17.4 (±0.2)	19.7 (±0.8)
VS (%TS)	54.8 (±3.1)	56.5 (±0.6)	55.8 (±1.2)	54.1 (±0.6)	53.3 (±2.5)	67.7 (±0.3)	67.0 (±0.2)	66.6 (±0.9)	65.8 (±1.5)	64.9 (±2.2)	63.1 (±0.7)	62.8 (±0.7)	61.2 (±1.3)	61.7 (±1.1)	61.8 (±0.8)
Ash (%TS)	45.2 (±3.1)	43.5 (±0.6)	44.2 (±1.2)	45.9 (±0.6)	46.7 (±2.5)	32.3 (±0.3)	33.0 (±0.2)	33.4 (±0.9)	34.2 (±1.5)	35.1 (±2.2)	36.9 (±0.7)	37.2 (±0.7)	38.8 (±1.3)	38.3 (±1.1)	38.2 (±0.8)
TKN ($g\ kg^{-1}$)	5.6 (±0.1)	3.5 (±0.6)	3.4 (±0.4)	3.2 (±0.5)	2.9 (±0.7)	2.8 (±0.1)	2.6 (±0.1)	2.2 (±0.1)	2.1 (±0.0)	2.0 (±0.0)	3.5 (±0.1)	2.4 (±0.3)	2.0 (±0.2)	1.7 (±0.1)	1.6 (±0.0)
TAN ($g\ kg^{-1}$)	4.0 (±0.1)	2.4 (±0.6)	2.1 (±0.3)	1.9 (±0.2)	1.6 (±0.2)	1.3 (±0.0)	1.2 (±0.0)	1.1 (±0.2)	0.9 (±0.0)	0.8 (±0.1)	2.2 (±0.0)	1.1 (±0.1)	0.7 (±0.1)	0.5 (±0.0)	0.4 (±0.0)
Organic N ($g\ kg^{-1}$)	1.6 (±0.1)	1.1 (±0.1)	1.3 (±0.2)	1.4 (±0.3)	1.3 (±0.4)	1.4 (±0.1)	1.3 (±0.1)	1.1 (±0.1)	1.3 (±0.0)	1.2 (±0.1)	1.3 (±0.1)	1.3 (±0.1)	1.3 (±0.1)	1.2 (±0.1)	1.2 (±0.1)
TAN/TKN (%)	71.6 (±1.5)	69.5 (±5.2)	62.4 (±0.3)	57.7 (±3.3)	55.1 (±4.5)	48.0 (±1.5)	48.3 (±1.5)	49.0 (±6.5)	40.5 (±1.3)	39.6 (±4.3)	61.9 (±1.2)	47.4 (±1.6)	36.1 (±4.0)	29.3 (±2.4)	23.6 (±1.9)
pH	7.9 (±0.1)	8.7 (±0.0)	8.4 (±0.0)	8.3 (±0.1)	8.0 (±0.1)	7.3 (±0.1)	8.4 (±0.1)	8.6 (±0.1)	8.5 (±0.1)	8.5 (±0.1)	8.3 (±0.1)	8.7 (±0.0)	8.5 (±0.1)	8.3 (±0.0)	8.3 (±0.0)

Table 2. *Cont.*

	Pig Slurry					Dairy Cattle Slurry					Digestate				
	Input PS12	**R1**	**R2**	**R3**	**R4**	**Input DCS12**	**R1**	**R2**	**R3**	**R4**	**Input D12**	**R1**	**R2**	**R3**	**R4**
HRT (Days)	**-**	**3**	**6**	**9**	**12**	**-**	**3**	**6**	**9**	**12**	**-**	**3**	**6**	**9**	**12**
ALK (mg L^{-1} $CaCO_3$)	22,403 (±474)	16,887 (±1994)	16,042 (±182)	16,131 (±1032)	14,464 (±1069)	11,330 (±524)	10,468 (±196)	9353 (±459)	9064 (±688)	8769 (±587)	13,118 (±55)	8800 (±929)	7859 (±257)	6688 (±301)	6646 (±237)
ORP	−416 (±12)	−376 (±39)	−570 (±25)	−559 (±30)	−543 (±26)	−417 (±16)	−421 (±9)	−397 (±13)	−369 (±16)	−356 (±14)	−390 (±64)	−147 (±35)	−466 (±13)	−462 (±10)	−448 (±17)
SARR (g kg^{-1} d^{-1})	–	0.51 (±0.16)	0.10 (±0.11)	0.09 (±0.03)	0.09 (±0.01)	–	0.03 (±0.01)	0.04 (±0.06)	0.08 (±0.06)	0.03 (±0.02)	–	0.35 (±0.04)	0.14 (±0.02)	0.07 (±0.02)	0.04 (±0.01)
	Input PS20	**R1**	**R2**	**R3**	**R4**	**Input DCS20**	**R1**	**R2**	**R3**	**R4**	**Input D20**	**R1**	**R2**	**R3**	**R4**
HRT (days)	**-**	**5**	**10**	**15**	**20**	**-**	**5**	**10**	**15**	**20**	**-**	**5**	**10**	**15**	**20**
TS (g kg^{-1})	26.4 (±0.2)	43.8 (±1.2)	42.2 (±2.8)	34.8 (±1.2)	31.2 (±5.8)	48.5 (±0.0)	46.1 (±0.9)	47.4 (±0.8)	46.3 (±0.6)	47.7 (±0.4)	36.1 (±0.6)	40.4 (±0.4)	45.6 (±1.2)	52.9 (±0.2)	66.4 (±0.7)
VS (g kg^{-1})	13.7 (±0. 3)	20.5 (±1.6)	21.0 (±0.9)	16.3 (±1.1)	17.3 (±0.1)	32.8 (±0.4)	30.8 (±1.1)	31.6 (±0.6)	30.3 (±0.4)	31.4 (±1.1)	22.9 (±0.4)	25.2 (±0.5)	28.5 (±0.7)	32.8 (±0.2)	41.5 (±0.4)
Ash (g kg^{-1})	12.8 (±0. 2)	23.2 (±2.5)	21.3 (±3.7)	18.6 (±2.2)	14.0 (±5.8)	15.7 (±0.5)	15.3 (±0.4)	15.8 (±0.3)	16.0 (±0.3)	16.2 (±0.6)	13.2 (±0.2)	15.2 (±0.2)	17.1 (±0.5)	20.1 (±0.4)	24.9 (±0.3)
VS (%TS)	51.6 (±1.3)	47.0 (±5.5)	50.0 (±6.9)	46.9 (±5.6)	57.0 (±11.5)	67.6 (±1.2)	66.8 (±1.6)	66.6 (±0.4)	65.5 (±0.6)	65.9 (±2.0)	63.4 (±0.1)	62.4 (±0.7)	62.5 (±0.2)	62.0 (±0.7)	62.5 (±0.2)
Ash (%TS)	48.4 (±1.3)	53.0 (±5.5)	50.0 (±6.9)	53.1 (±5.6)	43.0 (±11.5)	32.4 (±1.2)	33.3 (±1.6)	33.4 (±0.4)	34.5 (±0.6)	34.1 (±2.0)	36.6 (±0.1)	37.6 (±0.7)	37.5 (±0.2)	38.0 (±0.7)	37.5 (±0.2)
TKN (g kg^{-1})	3.3 (±0.2)	1.3 (±0.2)	1.0 (±0.3)	1.1 (±0.2)	0.9 (±0.2)	3.2 (±0.0)	2.9 (±0.0)	2.5 (±0.0)	2.2 (±0.0)	1.9 (±0.0)	3.5 (±0.1)	2.0 (±0.0)	1.6 (±0.1)	1.4 (±0.0)	1.3 (±0.1)
TAN (g kg^{-1})	2.6 (±0.1)	0.8 (±0.2)	0.6 (±0.2)	0.5 (±0.2)	0.4 (±0.1)	1.6 (±0.0)	1.5 (±0.0)	1.1 (±0.0)	0.8 (±0.0)	0.5 (±0.0)	2.2 (±0.0)	0.8 (±0.1)	0.4 (±0.0)	0.3 (±0.0)	0.2 (±0.0)

Table 2. *Cont.*

	Pig Slurry					Dairy Cattle Slurry					Digestate				
	Input PS12	**R1**	**R2**	**R3**	**R4**	**Input DCS12**	**R1**	**R2**	**R3**	**R4**	**Input D12**	**R1**	**R2**	**R3**	**R4**
HRT (Days)	**-**	**3**	**6**	**9**	**12**	**-**	**3**	**6**	**9**	**12**	**-**	**3**	**6**	**9**	**12**
Organic N ($g\ kg^{-1}$)	0.7 (±0.1)	0.5 (±0.1)	0.5 (±0.1)	0.5 (±0.1)	0.5 (±0.1)	1.6 (±0.0)	1.5 (±0.0)	1.4 (±0.0)	1.4 (±0.0)	1.4 (±0.0)	1.3 (±0.0)	1.2 (±0.0)	1.1 (±0.0)	1.2 (±0.0)	1.1 (±0.1)
TAN/TKN (%)	79.0 (±2.7)	60.6 (±2.6)	54.9 (±3.6)	51.5 (±6.4)	46.9 (±2.8)	50.9 (±0.1)	49.6 (±1.2)	45.1 (±1.0)	37.1 (±0.9)	28.5 (±1.1)	63.2 (±1.1)	39.7 (±1.9)	28.4 (±1.2)	18.3 (±0.6)	14.5 (±1.6)
pH	8.4 (±0.1)	9.0 (±0.1)	8.9 (±0.1)	8.8 (±0.1)	8.8 (±0.0)	7.4 (±0.1)	8.2 (±0.1)	8.3 (±0.1)	8.4 (±0.1)	8.5 (±0.1)	8.3 (±0.1)	8.8 (±0.0)	8.6 (±0.1)	8.4 (±0.1)	8.4 (±0.0)
ALK ($mg\ L^{-1}$ $CaCO_3$)	14,750 (±1426)	6561 (±692)	6194 (±272)	6970 (±692)	5404 (±1000)	12,347 (±312)	11,422 (±166)	10,833 (±595)	10,521 (±139)	10,440 (±524)	12,562 (±201)	7615 (±50)	6682 (±188)	5963 (±25)	5774 (±46)
ORP	−384 (±65)	−293 (±63)	−537 (±20)	−532 (±14)	−453 (±66)	−403 (±26)	−401 (±16)	−383 (±12)	−380 (±8)	−384 (±8)	−390 (±64)	−216 (±54)	−485 (±4)	−470 (±1)	−435 (±33)
SARR ($g\ kg^{-1}\ d^{-1}$)	–	0.36 (±0.01)	0.05 (±0.01)	0.01 (±0.00)	0.02 (±0.02)	–	0.04 (±0.01)	0.07 (±0.00)	0.06 (±0.00)	0.05 (±0.00)	–	0.28 (±0.01)	0.07 (±0.01)	0.04 (±0.01)	0.01 (±0.00)

TS concentrations of inputs for PS12, DCS12 and DCS20 were quite similar (mean 46.5 g kg^{-1}). The TS concentrations of inputs for D12 and D20 were 33.0 and 36.1 g kg^{-1}, respectively, whereas the TS concentration of input for PS20 was the lowest of all inputs (26.4 g kg^{-1}). In descending order (from 32.8 to 20.9 g kg^{-1}), the VS concentration of inputs was DCS20, DCS12, PS12, D20, D12 and PS20 (which was 13.7 g kg^{-1}). The ASH concentration of input for PS12 was the highest (21.6 g kg^{-1}), while ASH concentrations ranged from 12.2 to 15.7 g kg^{-1} for the remaining input slurries and showed small variation.

VS and ASH (as a proportion of TS) contributed almost equally in both pig slurries (PS12 and PS20) despite the large differences in TS, VS and ASH concentration. The content of VS as a proportion of TS (66%) was double that of ASH (33%) for DCS12, DCS20, D12 and D20. Generally, the TS, VS and ASH concentrations gradually increased from input to R4 in all experiments, which may have resulted from the evaporation of water. However, as a proportion of TS, the VS (%TS) and ash (%TS) remained almost the same as their input values at each reactor throughout the process for all slurries.

The average VS (%TS) values in the four reactors for PS12, PS20, DCS12, DCS20, D12 and D20 were 55% (±1), 50% (±4), 66% (±1), 66% (±0), 62% (±1) and 62% (±0), respectively. By comparison, the VS (%TS) values were 55% (±3), 51% (±1), 68% (±0), 68% (±1), 63% (±1) and 63% (±0) in input slurries for PS12, PS20, DCS12, DCS20, D12 and D20, respectively. This comparison shows that the VS (%TS) of each input slurry was very close to the average VS (%TS) in the contents of the four reactors for the same slurry. The standard deviations of the average VS (%TS) of the four reactors for each slurry were very low.

The organic N content in most input slurries was in the range of 1.3–1.6 g kg^{-1}; the content for PS20 was much lower (0.7 g kg^{-1}). The organic N did not change drastically during any experiment, decreasing only 0.1–0.3 g kg^{-1} from input of R1 to output of R4. These results indicated that the organic N degradation at 40 °C was negligible in the three types of slurries, corroborating the evidence that this process occurs at temperatures exceeding 40 °C [6]. The TAN concentration ranged from 1.3 to 4.0 g kg^{-1} in the input's slurries.

Input pig slurries had the highest TAN concentration followed by that of digestate and cattle slurry. TKN was the highest for PS12 (5.6 g kg^{-1}) and the lowest for DCS12 (2.8 g kg^{-1}), however, was similar in the inputs for the other treatments (3.2–3.5 g kg^{-1}). ALK of input pig slurries varied significantly as a function of the TAN concentration, as found also in Wang et al. [33]. The higher the TAN, the higher the ALK ($p < 0.05$, data are not shown). TAN concentration and alkalinities are plotted in Figure 2 and show very good agreement with each other ($R^2 = 0.89$). None of the ORPs during the experiments were in the range for nitrification and denitrification; therefore, NH_3 was not removed through these processes.

3.2. TAN Removal

In Figure 3, TAN and TKN removals for dairy cattle slurry are depicted along with the corresponding pH and ALK changes. As expected, the NH_3 removal at HRT of 20 days was higher than at HRT of 12 days in all reactors. The TAN removal after 3 and 5 days of HRTs was not very high; however, it increased to 41% and 67% after HRTs of 12 and 20 days, respectively. Cattle slurry is frequently treated with the stripping process after being digested anaerobically because the TAN content increases. In fact, in the cattle undigested slurry the TAN content is lower than in other manure, such as swine. However, Tao et al. [34] treated the filtered cattle slurry in a stripping process but at high temperatures (>76 °C) and high pH (>9), obtaining 50% of ammonia stripped in 5 h. Clearly, the process conducted under these conditions is much more expensive and less easy to manage although it achieves high performance in a short time.

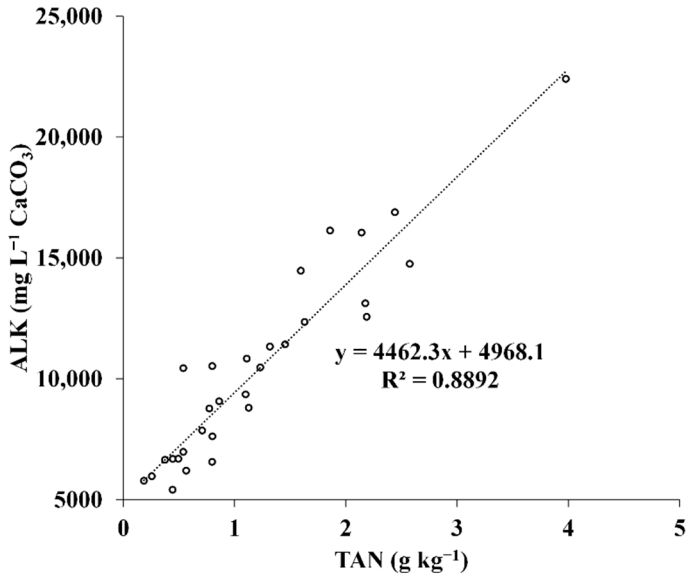

Figure 2. TAN versus ALK of inputs and output of all reactors for all substrates.

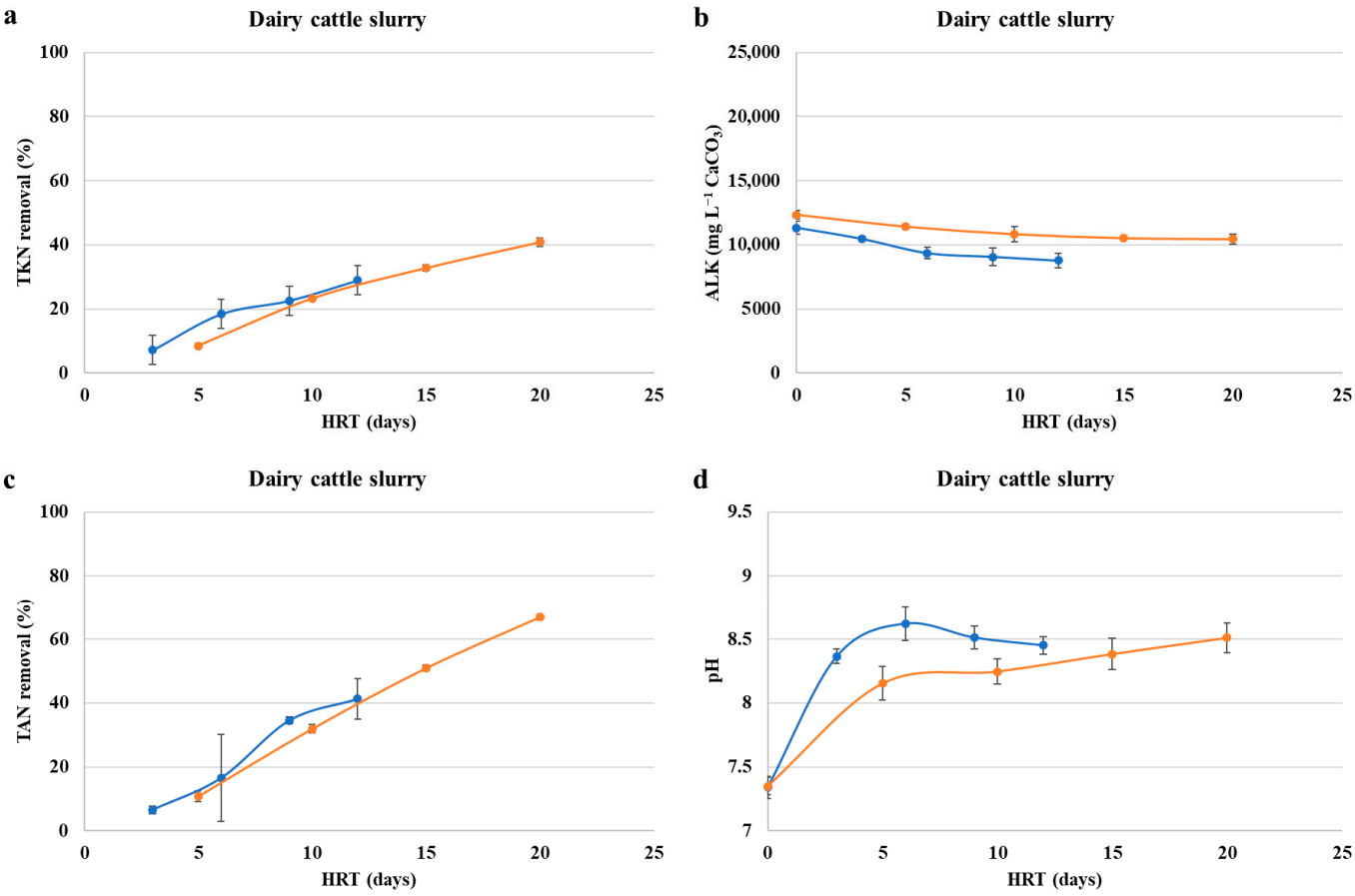

Figure 3. Removal of TKN (**a**) and TAN (**c**) and changes in ALK (**b**) and pH (**d**) as a function of HRT during stripping process of dairy cattle slurry. (Blue line = DCS12 treatment; and orange line = DCS20 treatment).

Surprisingly, TAN removal increased quite linearly over time and did not reach a plateau at 20 days of HRT, indicating that more days of retention are needed to strip away greater quantities of NH_3, using the type of stripping system employed in this study. The TAN removal progress for both dairy cattle slurry treatments (DCS12 and DCS20)

corresponded closely, which might be due to the almost identical characteristics of the input slurries. At 3 and 5 days of HRT in R1, there was a slight decrease in ALK and a noticeable rise (with the average of 0.9 unit) in pH compared to the measurements in input slurries due to the CO_2 stripping by intermittent air injection, which can induce NH_3 removal from the slurry.

However, the decrease in ALK for R1 mostly came from CO_2 stripping because the NH_3 removal was not high, and the organic N concentration did not change, indicating that negligible mineralization occurred. pH fluctuation was not noticeable throughout the other reactors (R2–R4), and ALK decreased slowly and steadily. The ALK reductions at HRT exceeding 6 days might have been due to higher NH_3 removal. The fact that pH did not decrease as NH_3 removal increased in reactors R2–R4 could have been due to the relatively high ALK of slurries throughout the reactors even after NH_3 was released from the liquid phase. The ALK in the input slurries for treatments DCS12 and DCS20 was 11,330 and 12,347 mg L^{-1} $CaCO_3$, respectively, and decreased to 8769 and 10,440 mg L^{-1} $CaCO_3$ after 12 and 20 days, respectively.

For cattle slurry, the NH_3 removal percentages after 12 and 20 days were almost six-times higher than those at 3 and 5 days of HRTs, demonstrating that the reactors in series (which led to higher retention time) had a huge positive impact on NH_3 removal. As the characteristics of input slurries in treatments DCS12 and DCS20 were quite similar, NH_3 removals were comparable according to HRTs. NH_3 removal after 10 days (DCS 20–R2) was 32%, which was very close to the removal of 35% for 9 days (DCS 12–R3). The same pattern can be noticed for NH_3 removal after 5 (DCS 20–R1) and 6 days (DCS 12–R2). The results show that TAN removal from dairy cattle slurry was less than 50% at HRT less than 15 days.

In the case of digestates (Figure 4), almost 50% and 66% of NH_3 was removed at 3 days and 5 days of HRT, respectively. The highest NH_3 removal of 91.51% was achieved after 20 days of HRT compared to 83% after 12 days. The physicochemical properties of both digestates (D12 and D20) used in this study were quite similar, therefore NH_3 removal could be reliably compared based on their HRTs. The NH_3 removal after 5 days (63%) was very close to the removal at HRT of 6 days (67%). The same removal pattern occurred at HRT of 10 and 9 days with NH_3 removals of 79.67% and 77.23%, respectively.

These results were comparable with those of Provolo et al. [6] who obtained 69% NH_3 removal in 10 days by treating digestate with the same slow-release ammonia system used in this study but in batch conditions and an air flow of 10 l min^{-1}. In Lei et al. [28], NH_3 removal from digestate was conducted at low temperatures (15 °C) and pH adjustment until 12; the achieved efficiencies varied between 25.1% and 95.3%, with no aeration for 24 h and aeration of 10 L min^{-1} for 12 h, respectively. These results are comparable with the findings of other studies on digestate treated with the stripping process, in which NH3 removal efficiencies above 90% were achieved in few hours, with pH adjustment or higher temperatures [22,26,35].

The NH_3 removal did not increase considerably after 12 days because, by then, the NH_3 concentration had decreased substantially. Compared to the values in input digestate, the ALK in R1 at HRTs of 3 and 5 days decreased substantially (by 33% and 40%, respectively). Thereafter, the reduction in ALK was modest and in R4 reached approximately 50% of input ALK (at 12 and 20 days of HRT). Due to the moderately high TAN reduction of digestates in R1, where the HRTs were 3 and 5 days, NH_3 removal in R1 played a key role in ALK reduction as did CO_2 stripping by air injection. However, in the subsequent reactors (R2–R4), NH_3 stripping by headspace aeration appeared to be the main cause for the gradual ALK reduction.

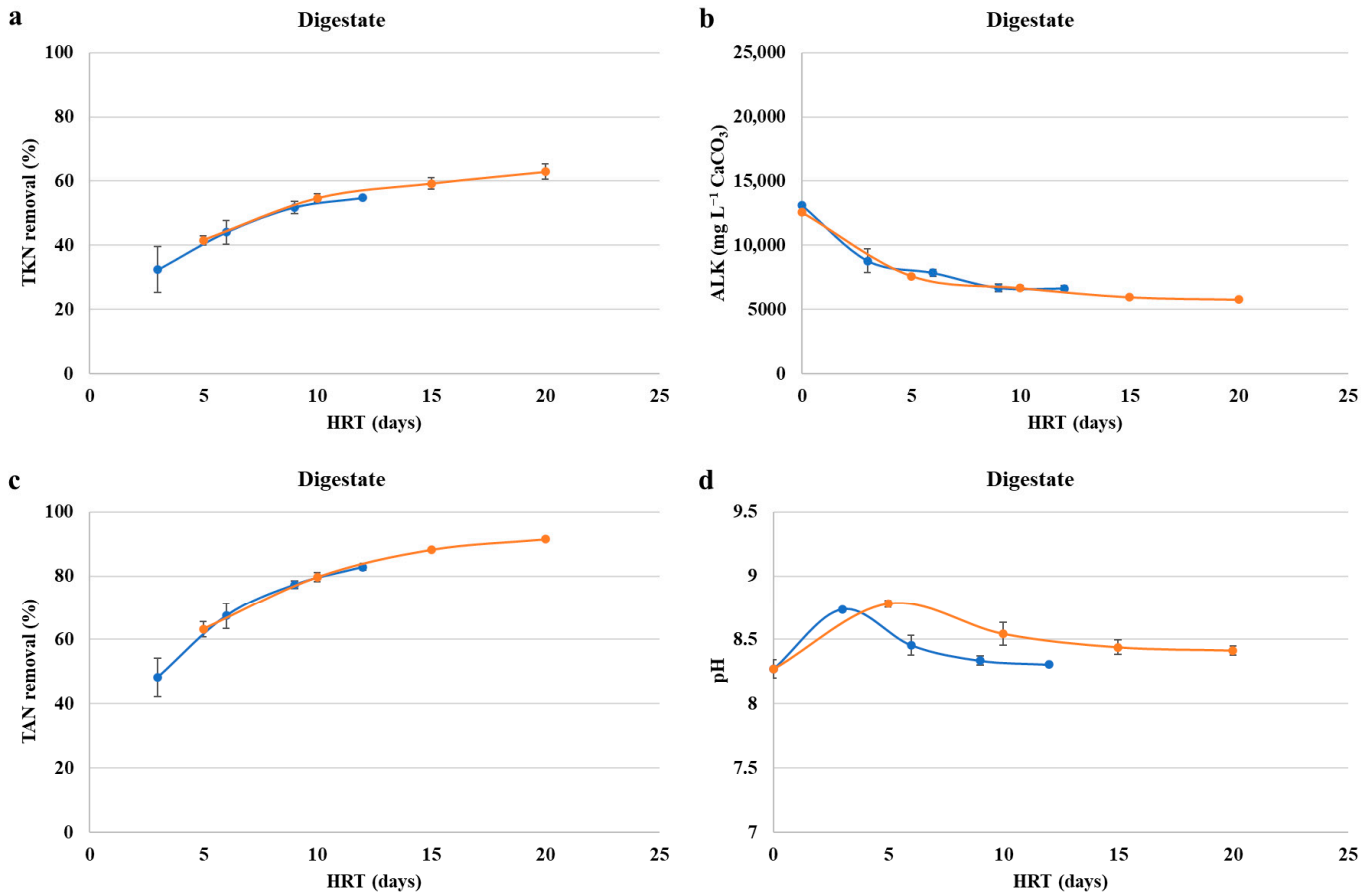

Figure 4. Removal of TKN (**a**) and TAN (**c**) and changes in ALK (**b**) and pH (**d**) as a function of the HRT during the stripping process of the digestate slurry. (Blue line = D12 treatment; and orange line = D20 treatment).

The pH increased by 0.5 at R1 compared to its value in the input digestates, even though ALK declined substantially. After R1, pH was stable throughout the continuous operation with only a small decrease due to NH_3 removal. In the continuous process of NH_3 removal from digestate, the maximum organic N reductions were 0.1 and 0.2 g kg^{-1} at HRTs of 12 and 20 days, respectively. These removals equated to 10% and 13%, respectively, of the amount in input digestates, and indicated that insignificant mineralization of organic N compounds occurred in the stripping system.

In Figure 5, NH_3 and TKN removals from pig manure are depicted along with pH and ALK changes throughout the continuous stripping process. The physicochemical characteristics of the two pig slurries (PS12 and PS20) were very different at HRTs of 3 and 5 days, reflecting the fact that the PS20 input was more dilute than that for PS12. Although the TAN concentration of the PS20 input (2.6 g kg^{-1}) was lower than that of the PS12 input (4.0 g kg^{-1}), the NH_3 removal was much higher from the former. In fact, approximately 60% of the NH_3 in input slurry for PS12 was removed after 12 days of HRT; however, 83% of NH_3 from the more dilute PS20 was removed after 20 days of HRT.

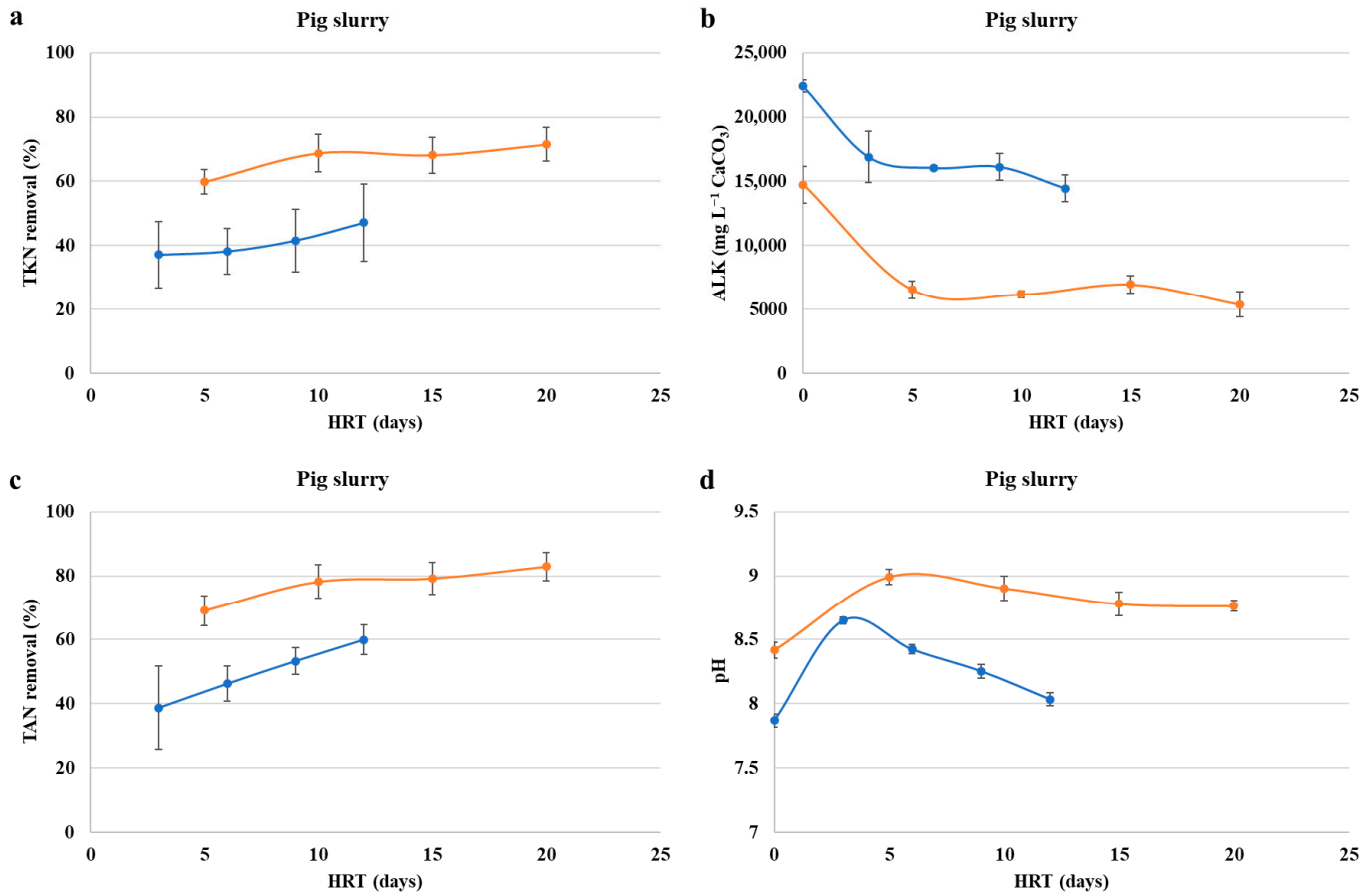

Figure 5. Removal of TKN (**a**) and TAN (**c**) and changes in ALK (**b**) and pH (**d**) as a function of HRT during stripping process of pig slurry. (Blue line = PS12 treatment; and orange line = PS20 treatment.).

These results fall within the range of those of Pandey and Chen [13] and Zarebska et al. [17] reviews, which reported NH_3 removals between 69% and 98.8% for pig slurry treated in a batch process. However, it must be considered that these performances were obtained in few hours due to high temperatures, pH adjustments and high air/slurry ratios, which, in our study, were replaced by a longer treatment time. However, the NH_3 removal from PS20 after 10 days did not increase to a large extent, while in comparison, the removal increased linearly with increasing HRT for PS12.

This indicates that higher HRT can counterbalance the effect of lower TAN concentration. The pH of input pig slurry for PS12 and PS20 was 7.9 and 8.4, respectively, which increased to 8.7 and 9.0, respectively, in R1 after 3 and 5 days of HRT due to CO_2 stripping by the intermittent air injection into this reactor. After, there was a reduction in the pH of PS12 from 8.7 on day 3 to 8.0 at the end of the process on day 12, at which time the NH_3 concentrations were 2.44 g kg^{-1} (day 3) and 1.6 g kg^{-1} (day 12).

On the other hand, the pH of PS20 remained quite constant throughout the other reactors, changing only from 9.0 to 8.8 at the end of the process on day 20, with the corresponding NH_3 concentrations changing from 0.8 g kg^{-1} to 0.4 g kg^{-1}, respectively. In PS20 there was a greater removal of NH_3, particularly at the beginning of the process (R1-R2) which could be due to a higher pH compared to PS12. The initial ALK of PS12 (22,430 mg L^{-1} $CaCO_3$) was much higher than that of PS20 (14,750 mg L^{-1} $CaCO_3$) and decreased to 16,887 mg L^{-1} $CaCO_3$ at HRT of 3 days, whereas the ALK of PS20 decreased to 6561 mg L^{-1} $CaCO_3$ at HRT of 5 days.

Thus, the reduction of ALK in PS20 at HRT of 5 days was much sharper than that for PS12 at HRT of 3 days, even though the amounts of NH_3 removed in R1 were very similar for both slurries (1.8 and 1.5 g kg^{-1} from PS20 and PS12, respectively). Throughout the

continuous process after R1 (HRTs of 3 and 5 days), the ALK reduction in PS12 and PS20 was smooth, averaging approximately 16,000 and 6000 for PS12 and PS20, respectively. The decrease in ALK of PS12 at HRT of 3 days (from 22,430 to 16,887 mg L^{-1} $CaCO_3$) was considerable; however, the final ALK of 16,887 mg L^{-1} $CaCO_3$ was still high enough to inhibit NH_3 removal afterward even though TAN concentration of PS12 was quite high.

The organic N of PS12 decreased from 1.6 g kg^{-1} in the input slurry to 1.1 g kg^{-1} after 3 days; however, it increased to 1.3 g kg^{-1} at the end of 12 days (this surprising result might be an experimental error). These results show that organic N did not change noticeably during the process in the PS12 treatment. Likewise, the concentrations of organic N in PS20 were between 0.5 g kg^{-1} (initially) and 0.5 g kg^{-1} (at the end of the whole process), showing almost no mineralization of organic N.

Among the six slurries examined, DCS12, DCS20 and PS12 exhibited NH_3 removal trends that differed from those of the other three slurries throughout the continuous process. The NH_3 removals versus HRT for DCS12, DCS20 and PS12 were quite linear and uniform. In contrast, NH_3 removal tended to level off after a few days of HRT for D12, D20 and PS20. Furthermore, most (more than 75%) of the NH_3 was removed at HRT of 10 days from PS20 and D20 and at HRT of 9 days from D12. Despite the slurry for PS12 having the initial highest TAN concentration, the TAN removal after 3 days was approximately 40%, and it reached only 60% at the end of process after 12 days.

This level of NH_3 removal is fairly low and could be due to a very high ALK in the PS12 slurry (1.5–2-times more than other slurries). In addition to carbonate, bicarbonate and hydroxide ions, alkalinity might come from different ions, such as phosphates, silicates and borates. The results for PS12 suggest that, for slurries with high ALK, it might be better to apply longer HRT if pH adjustment is not utilized, in order to attain higher NH_3 removal. However, such a strategy also reduces SARR. Notably, NH_3 removal could be also inhibited by the slurry matrix with a high content of organic compounds as might be the case in cattle slurries in comparison to digestates and pig slurries.

3.3. Reactor Performance and Statistical Analysis

The performance of individual reactors and their SARR (g kg^{-1} day^{-1}) were investigated independently in relation to the characteristics of the slurries introduced into the reactors. The highest SAAR (Table 2) was achieved in R1 for PS12 and PS20 with values of 0.51 and 0.36 g kg^{-1} day^{-1}, respectively. There was a sharp decrease in SARR after R1. Afterwards, the SARR for PS12 decreased gradually from 0.1 g kg^{-1} day^{-1} (R2) to 0.088 g kg^{-1} day^{-1} (R4). For PS20, the SARR at R3 and R4 was negligible. The SARR of DCS12 and DCS20 exhibited different trends throughout the reactors.

For DCS12, SARR reached its highest value in R3 (0.08 g kg^{-1} day^{-1}) and declined to 0.03 g kg^{-1} day^{-1} in R4. For DCS20, the highest SARR occurred in R2 and R3 with the values of 0.07 and 0.06 g kg^{-1} day^{-1}, respectively. The SARR for D12 and D20 were the highest at R1, equal to 0.35 and 0.28 g kg^{-1} day^{-1}, respectively. There was a noticeable decline in SARR for D12 and D20 from R1 to R2; however, afterwards, it decreased slowly and gradually to reach 0.04 and 0.01 g kg^{-1} day^{-1}, respectively, in R4. The results of stepwise linear regression to evaluate the most effective factors influencing SAAR are shown in Table 3.

Table 3. Statistical analysis and stepwise linear regression for the effect of the independent variables listed in Table 2 on SARR.

ANOVA					
Model	**Sum of Squares**	**Degree of Freedom**	**Mean Square**	**F-value**	***p*-Value**
Regression	0.321	2	0.160	49.722	0.000
Residual	0.068	21	0.003		
Total	0.389	23			
Regression coefficients					
R = 0.909 R^2 = 0.826 Adjusted R^2 = 0.809 Standard error of the estimate = 0.05681	Model	coefficient	Standard error	*t*-value	*p*-value
	Constant	0.120	0.051	2.374	0.027
	Inlet TAN ($g\ kg^{-1}$)	0.294	0.045	6.475	0.000
	Inlet ALK ($mg\ L^{-1}\ CaCO_3$)	-3.790×10^{-5}	0.000	−3.391	0.001

The linear regression model suggested by statistical analysis, where the R2 and adjusted R2 of the model were 0.83 and 0.81, respectively, is given in Equation (2):

$$\text{SAAR} = 0.120 + 0.294 \times \text{Inlet TAN } 3.790 \times 10^{-5} \times \text{Inlet ALK} \quad (2)$$

where the units of Inlet TAN and Inlet ALK are $g\ kg^{-1}$ and $mg\ L^{-1}\ CaCO_3$, respectively.

This model had a high R2 with good predictability. The individual HRT, inlet VS, inlet Ash, inlet organic N and inlet pH were excluded from the model as having insignificant effects on SAAR. That inlet pH was not a statistically important factor was surprising since the inlet pH could be the function of inlet TAN and inlet ALK and was outweighed by these two factors. However, previous studies showed that the pH of feed substrate into stripping reactors is one of the most effective factors influencing NH_3 removal [17,22,23].

To our knowledge, previous studies generally focused on the NH_3 removal percentage and removal rate without considering the volume of liquid slurry treated. SARR is a comprehensive term that can be utilized when scaling up the whole process from laboratory scale to pilot scale. High SARR indicates that a large amount of NH_3 is removed in a short time. Increasing HRT or the stripping time generally results in a higher NH_3 removal percentage but also increases the required working volume and lowers the SARR, resulting in higher capital and maintenance costs.

4. Conclusions

The tested treatments showed different NH_3 recovery behaviors throughout the studied continuous stripping process with four reactors in series. For digestate and pig slurry, about 75% of NH_3 recovery was achieved already after 10 days of HRT, while the additional contribution to NH_3 recovery by a longer HRT was below 12%. The NH_3 removal in the dairy cattle slurries had a different behavior, and an HRT longer than 20 days is required for acceptable TAN removal.

The study results suggest that, for slurries with very high ALK, it might be advantageous to increase HRT in favor of NH_3 recovery percentage, even though this will lower the SARR. The continuous stripping process could be integrated as a post treatment technology into a biogas plant where the digestate is available continuously as well as in a livestock farm with a daily manure removal from the barns. The main limit is related to the difficulties in the management of such a treatment process. In fact, although the achievable performances of recovering NH_3 from batch and continuous processes are comparable, the batch process is easier to manage even if it requires an intermediate storage.

The advantages of the process conducted with a continuous approach are multiple and could make intensive livestock areas more efficient from the economic and environmental point of view and fully comply with the recent EU principles of the farm to fork strategy. In more detail, through the recovery of NH_3 from slurries and digestates, a mineral N fertilizer becomes available for farmers, and its management practices become easier on the field, in terms of distribution technique, efficiency and timely application.

Furthermore, the use of such a fertilizer would allow the use of matrices that are already available locally, thus, reducing the dependence from the market for mineral fertilizers. Reducing NH_3 emissions will further reduce the impact on acidification, eutrophication and particulate matter formation, thus, also benefitting human health. Finally, specific policies should be introduced to incentivize farmers towards the direction of efficient nutrient use and recovery, such as with the air stripping process, as this technique brings advantages not only to farmers and farming practices but also to the environment and society.

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Data Availability Statement: The data presented in this study are available on request from the corresponding author. The data are not publicly available because the experimental activities of the Conserva project are still in progress.

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