Volatilization of ammonia in systems of treatment of swine manure with aquatic macrophytes

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Abstract

The usage of aquatic plants represents an alternative in the treatment of residues originating from swine. In these systems, one of the N removal methods is the ammonium (NH_4^+) uptake and volatilization of ammonia (NH_3). In this way, the objective of this work was to evaluate the volatilization rates of NH_3 in waste treatment systems swine fluids (SSF) with aquatic macrophytes, as well as the concentration of NH_4^+ present in the swine fluids. The experiment was carried out at Campus II/UNOESTE. The treatment systems were composed of 16 boxes of PVC and characterized as: T1 = Control sample 50% of SSF/50% of water; T2 = 50% SSF/50% water + *Eichhornia crassipes* (Mart.) Solms; T3 = 50% SSF/50% water + *Pistia stratiotes* L.; T4 = 50% SSF/50% water + *Salvinia auriculata* Aubl. The design was randomized blocks, with 4 treatments and 4 replicates. The hydrogen potential (pH) and the NH_4^+ content of the effluent were analyzed weekly, and the volatilization of NH_3 by means of collectors installed in each treatment unit. The presence of aquatic macrophytes promoted the reduction of NH_4^+ concentration and of the pH values of swine fluids, and this resulted in the reduction of NH_3 volatilization rates to the environment, with emphasis on the system with *Eichhornia crassipes* (Mart.) Solms, which presented the lowest rate of volatilization.

Keywords: greenhouse gases, phytoremediation, wastewater, environmental management.

Volatilização de amônia em sistemas de tratamento de dejetos de suínos com macrófitas aquáticas

Resumo

A utilização de plantas aquáticas representa uma alternativa no tratamento de resíduos oriundos da suinocultura. Nestes sistemas, uma das formas de remoção de nitrogênio (N) é a absorção de amônio (NH_4^+) pelas plantas, entretanto, também ocorre a volatilização de amônia (NH_3). Dessa forma, o objetivo do trabalho foi avaliar as taxas de volatilização de NH_3 em sistemas de tratamentos de dejetos líquidos de suínos (DLS) com macrófitas aquáticas, bem como a concentração de NH_4^+ presente nos dejetos. O experimento foi realizado em área de ambiente aberto no Campus II/UNOESTE. Os sistemas de tratamento foram constituídos de 16 caixas de PVC e caracterizados como: T1 = Testemunha 50% de DLS/50% de água; T2 = 50% de DLS/50% de água + *Eichhornia crassipes* (Mart.) Solms; T3 = 50% de DLS/50% de água + *Pistia stratiotes* L.; T4 = 50% de DLS/50% de água + *Salvinia auriculata* Aubl. O delineamento adotado foi em blocos casualizados, com 4 tratamentos e 4 repetições. Foram analisados o potencial hidrogeniônico (pH) e o teor de NH_4^+ do efluente semanalmente, e a volatilização de NH_3 por meio de coletores instalados em cada unidade de tratamento. A presença das macrófitas aquáticas proporcionou a redução da concentração de NH_4^+ e dos valores de pH dos dejetos líquidos de suínos, e isto resultou na redução das taxas de volatilização de NH_3 ao meio ambiente, com destaque ao sistema com *Eichhornia crassipes* (Mart.) Solms, que apresentou a menor taxa de volatilização.

Palavras-chave: gases do efeito estufa, fitorremediação, efluentes, manejo ambiental.

1. Introduction

Pig farming represents a sector of great economic and social importance for Brazil (Gonzatto et al., 2013), responsible for increasing exports of meat and its industrialized products, and also for the generation of jobs. Brazilian pig farming occupies a prominent position in the world scenario, where Brazil is the fourth largest producer and exporter of pork (ABCS, 2014).

However, pig farming is characterized as an activity with great potential for pollution, due to the effluent generation normally in the liquid form, with high load of organic and

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nutrient matter (mainly nitrogen and phosphorus, and also, potassium, calcium, sodium, magnesium, manganese, iron, zinc and copper) (Steinmetz et al., 2009). The pollutant load of liquid pig slurry may adversely affect environments as the water bodies, promoting the growth of microorganisms and the occurrence of eutrophication (Meade et al., 2011).

Excess nutrients, applied to the soil at rates higher than those for capture, may get in surface and groundwater due to the flow and leaching (Stone et al., 1998). To reduce the nutrient load into the environment, alternative or additional ways of treating wastewater should be implemented. An option for the additional treating is phytoremediation, that is, the use of plants and associated microorganisms as an instrument for containment, isolation, removal or reduction of contaminant concentrations in solid, liquid or gaseous media (EPA, 2000), at safe levels compatible with the protection of human health, as well as preventing the spread of harmful substances to the environment (Andrade et al., 2007).

The systems made with aquatic macrophytes are used to reduce the nutrient concentration of pig slurry, minimize the impacts on aquatic ecosystems (Poach et al., 2003), and provide an operationally passive form of wastewater treatment (Hunt and Poach, 2001; Kadlec and Knight, 1996). Furthermore, they can effectively treat great amounts of animal excrements (Knight et al., 2000), especially in the nitrogen (N) removal (Poach et al., 2003).

According to Vesilind and Morgan (2011), the N is an important element in biological reactions, and may be connected to components that produce a lot of energy, such as amino acids and amines, also known as organic N. An intermediate element formed during the biological metabolism é the ammoniacal N. In accordance to Peng et al. (2005), the organic and ammoniacal N are the main forms presented in wastewater, and are considered indicators of recent pollution. The organic N is converted to ammoniacal under anaerobic and aerobic conditions, with the reduction of ammoniacal nitrogen concentrations contributing to the reduction of total nitrogen, and the temperature and pH have an impact on the bioactivity and volatilization processes.

The wetlands, systems artificially designed for utilizing aquatic macrophytes, remove N through sedimentation, absorption, organic matter accumulation, microbial assimilation, nitrification/denitrification and volatilization of ammonia (Brix, 1993; Johnston, 1991; Poach et al., 2003). Sooknah and Wilkie (2004) verify the reduction of N because of the direct absorption by the aquatic macrophytes to the nitrification carried out by the nitrifying bacteria and the volatilization of non-ionized ammonia (NH_3) which occurs predominantly in high pH environments. Ammonia emissions characterize a major threat to the environment, due to their implications, such as changes in the rainfall pH, contributions to the greenhouse effect, as well as effects on human and farmed animal health (Felix and Cardoso, 2004). Thus, monitoring these emissions is something fundamentally important for the control of atmospheric pollution.

Based on the previously said, this work aims at evaluating the losses of N to the atmosphere by volatilization of ammonia (NH_3) in pig slurry treatment systems, with and without the presence of aquatic plants, as well as the concentration of ammonium (NH_4^+) present in pig slurry, with the hypothesis that aquatic plants are able to reduce the concentration of NH_4^+ present in swine manures, as well as to reduce losses of N by NH_3 volatilization.

2. Material and Methods

The experiment, carried out in May and June of 2016, was conducted in an open environment at Campus II/UNOESTE, whose geographic coordinates are: Latitude 22° 07' S e Longitude 51° 09' W, Presidente Prudente, São Paulo, Brazil. Located in a defined climate region, according to the climatic classification of Köppen, as Aw – tropical wet with dry winter, and average annual temperature of 29.2 °C. The average annual rainfall is 1254.9 mm (CEPAGRI, 2017).

The treatment systems were composed of 16 polyvinyl chloride (PVC) containers with dimensions of 76.5 cm in height, 101.5 cm in superior diameter and 73.0 cm in inferior diameter, making a volume of 0.32 m³, in which only 0.25 m³ are used per container, margin adopted to avoid overflow.

Pig slurry was used, coming from the zootechnical center of Campus II/UNOESTE, for the composition of the treatments, which are: T1 = Control sample – 50% pig slurry and 50% water; T2 = 50% pig slurry and 50% water + *Eichhornia crassipes* (Mart.) Solms (Figure 1); T3 = 50%

Figure 1. *Eichhornia crassipes* (Mart.) Solms (Lutzenberger, 1985).

pig slurry and 50% water + *Pistia stratiotes* L. (Figure 2); T4 = 50% pig slurry and 50% water + *Salvinia auriculata* Aubl. (Figure 3), distributed in a randomized block design (RBD), in split plots, with four repetitions. The plots are represented by the treatments (with and without the presence of aquatic macrophytes) and the split plots, by the collection periods.

The *Eichhornia crassipes* (Mart.) Solms is characterized by being a free floating aquatic plant, native of South America, belonging to the monocotyledonous class, Pontederidaceae family, Pontederiales order (Esteves, 1998), and by being an angiosperm with perennial life cycle (Bortolotto and

Figure 2. *Pistia stratiotes* L. (Pott and Pott, 2000).

Figure 3. *Salvinia auriculata* Aubl. (Gomes, 2011).

Guarim Neto, 2005). Exotic in origin, it is distributed in all continents, in tropical semitropical latitudes and in some temperate countries (Metcalf and Tchobanoglous, 1991) and, in the absence of nutritional limitations, it rapidly develops in hot climate regions (Andrade et al., 2007).
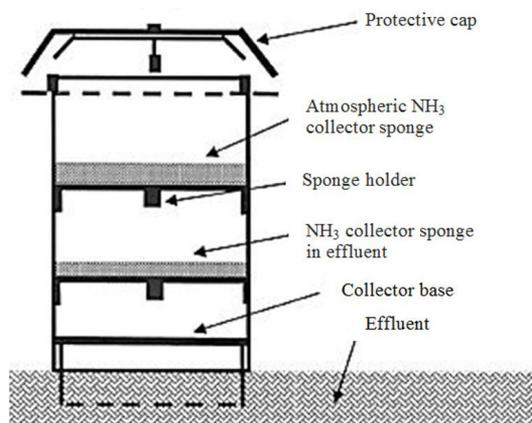
The *Pistia stratiotes* L. is commonly known as water lettuce, belonging to the Araceae family (Kissmann and Groth, 1997), extensively distributed throughout the world. Its origin, which is attributed to Africa or South America (Lorenzi, 1982; Cardoso, et al., 2005), has not yet been fully defined. According to Pott and Pott (2000), *P. stratiotes* is considered a cosmopolitan tropical and subtropical species, being widely distributed throughout Brazil, occurring both in natural ecosystems and in aquatic environments impacted by anthropic activities (Henry-Silva and Camargo, 2000a).

Belonging to the family Salviniaceae, the *Salvinia auriculata* Aubl. is a free floating aquatic plant, of annual or perennial occurrence (Oliveira, 1981). It presents a wide native distribution in the neotropics, extending from Mexico and the Galapagos Islands through Central America and the Antilles and most of South America to the south of Brazil (Sculthorpe, 1967).

The aquatic macrophytes used were collected in lentic lakes from lands located in the West Paulista region, São Paulo, selected as young plants, with an established root system and aerial part of similar appearance. They were initially submitted to the environmental adaptation in boxes containing only water, at the experiment site, for a week. After this period, the treatment was initialized, and the amount of plants inserted in each experimental unit was determined to maintain an occupation of approximately 80% of the experimental units, as described by Henry-Silva and Camargo (2008).

During the experimental period, 16 samples of the effluent to analyse the pH and ammoniacal nitrogen in the ionized form (NH_4^+) were weekly collected, at the periods of 0 (day in which the experiment was implanted), 7, 14, 21 and 30 days. The pH measurements were obtained using the Micronal bench digital pH meter. The determination of the NH_4^+ content was obtained by the Kjeldahl method, consisted of three stages: sample digestion, distillation with the Kjeldahl nitrogen distiller and titration with sulfuric acid, through the method presented by Malavolta et al. (1997). The method is based on the decomposition of organic matter by digestion of the sample with concentrated sulfuric acid at 350 °C, in the presence of catalyst salts that accelerate the oxidation of organic matter. The digestion will be terminated after obtaining a colorless or slightly greenish liquid. The nitrogen present in the resulting acid solution is determined by steam distillation, collected by the boric acid 2% (m/v) solution and indicators, followed by titration with sulfuric acid (0.02 N).

For the quantification of NH_3 volatilization a PVC collector base (Figure 4), with 15.0 cm in diameter and 14.0 cm in height, was placed in each treatment box, fixed to a 4.0 cm thick Styrofoam board, with a center opening beneath the surface of the effluent. Each base was protected

Figure 4. NH_3 collector scheme (adapted from Ros et al., 2005).

Table 1. Values of F for pH, NH_4^+ and the volatilization of NH_3 calculated based on the Tukey Test, at the level of 5% probability, in the treatments with aquatic species, in collection periods.

Variation factors	pH	NH_4^+	NH_3
		(mg L^{-1})	
Treatment (a)	27.58*	12.69*	15.05*
Collection periods (b)	239.36*	299.37*	28.67*
Interaction (a) × (b)	3.62*	0.90ns	4.00*
CV (a) (%)	1.49	10.47	31.25
DMS (a)	0.22	0.96	91.04
CV (b) (%)	1.47	20.81	30.33
DMS (b)	0.21	0.94	81.62

*significant at the level of 1% probability (p<0.01); ns = not significant.

at the top with a polyethylene plate with 20.0 m in diameter, and a 2.0 cm opening between the plate and the collector base was kept, for establishing a steam pressure deficit and, thus, the NH_3 volatilization could happen. Inside the PVC base a screen with an effluent height of 4.0 cm was place, fixed to a glass petri dish which housed in its interior a sponge with 2.0 cm thick and 7.0 cm width.

The sponges, moistened with 30.0 mL of phosphoric acid (0.167 mol L^{-1}) to capture the volatilized NH_3 of the effluent, were replaced at the periods of 1, 4, 8, 12, 19 and 27 days after placing the collectors. In the collections, the sponges were gathered, stored in a plastic box with a lid and immediately taken to the laboratory of clinical analysis of vegetal tissues/UNOESTE to extract the ammonium phosphate solution (($NH_4)_3PO_4$) formed from the chemical reaction between NH_3 and phosphoric acid (H_3PO_4). Their washings were performed with 500.0 mL deionized water, in five continuous washings of 100.0 mL each. Finally, an aliquot of 20.0 mL of the solution was submitted to distillation with the Kjheldad distiller by the method described by Cantarella and Trivelin (2001).

The efficiencies of the treatments in the reduction of the content of NH_4^+ were calculated according to Equation 1:

$$E(\%) = [(Ci - Cf) / Ci] \times 100 \qquad (1)$$

In which: E = Removal efficiency (%); Ci = Initial concentration (mg L^{-1}); Cf = Final concentration (mg L^{-1}).

The results were put under analysis of variance by the Test F and the comparison of the means by the Tukey Test, at the level of 5% probability, with software Assistat 7.7.

3. Results and Discussion

The values of Test F, obtained by the contrast of means between the treatments (Table 1), show that there was a significant difference between the plant species used and the collection periods for the following variables: pH ammonium (NH_4^+) and ammonia (NH_3).

The pH values (Figure 5) decreased, throughout the collection periods, in all treatments. It was possible to notice that in the period of 7 days, the *Eichhornia crassipes* (Mart.) Solms provided a significant reduction in the pH compared to the others, and according to Granato (1995), *Eichhornia crassipes* (Mart.) Solms, in contact with alkaline solutions, has the ability of decreasing the pH of these solutions, due to the absorption of the potassium, calcium and magnesium nutrients, respectively, which are all present in the medium. According to Esteves (1998), the organic matter decomposition process reduces the pH, since there is an increase in the carbon dioxide (CO_2) concentration in the medium and, at the same time, the consumption of dissolved oxygen. Lin et al. (2005) also observed a reduction in the pH of the effluent from de *Litopenaeus vannamei* nurseries treated with constructed wetland. According to Shah et al. (2015), a pH of 6.0-9.0 is the most adequate for the aquatic macrophytes performance and, in accordance with CONAMA (2011) Resolution no. 430/2011, the pH values of the effluent from any polluting source, to be directly released in the receiving body, must be between 5.0 and 9.0. Thus, the results found in the treatments with plant species are in agreement with the ones required by the Brazilian legislation.

For Henry-Silva and Camargo (2000b), the highest pH found only in the effluent is probably related to the photosynthesis of the phytoplankton present in the site, which, by assimilating the CO_2 available in the water, increases the pH values of the medium.

Silva et al. (2014) found out that the effluent from water supply channels with tambaqui farming treated with *Eichhornia crassipes* (Mart.) Solms showed lower pH values when compared to the treatments without plants. According to the authors, it happens because of the removal of bases that enable the growth of *Eichhornia crassipes* (Mart.) Solms. Gentelini et al. (2008) observed that the organic pisciculture effluent, before passing through the system with aquatic macrophytes, was slightly alkaline, with a pH of 7.03, becoming somewhat

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acid after crossing the system. This same pattern of pH reduction, after passing through the treatment system, was observed by Henry-Silva and Camargo (2006) with three floating macrophytes, *Eichhornia crassipes* (Mart.) Solms, *Pistia stratiotes* L. and *Salvinia molesta* D. S. Mitch., in the treatment of pisciculture effluent.

In relation to the NH_4^+ concentration in the effluent, it should be noted that there was no significant interaction between the treatments (without and with the plants) and between the collection periods. However, after evaluating the means obtained in each treatment (Figure 6), it was possible to notice that the *Eichhornia crassipes* (Mart.) Solms species statically differed from the others by presenting a lower NH_4^+ content in its effluent and, therefore, higher efficiency in the removal of this element (93.4%, according Table 2) in a 30 day detention time. Reidel et al. (2005),

when analysing the refrigerated effluent treatment system with *Eichhornia crassipes* (Mart.) Solms, found that the industrial effluent presented average concentration of 79.85 mg L^{-1} of NH_4^+, and the reductions of this element

**Table 2.** Initial and final values of the content of NH_4^+ in the treatments with aquatic species.

Treatments	NH_4^+ (mg L^{-1})		
	Initial	Final	E (%)
Control sample	9.87	2.38	75.9
Eichhornia crassipes (Mart.) Solms	8.89	0.58	93.4
Pistia stratiotes L.	9.52	3.15	66.9
Salvinia auriculata Aubl.	10.29	2.45	76.2

E(%): Removal efficiency.
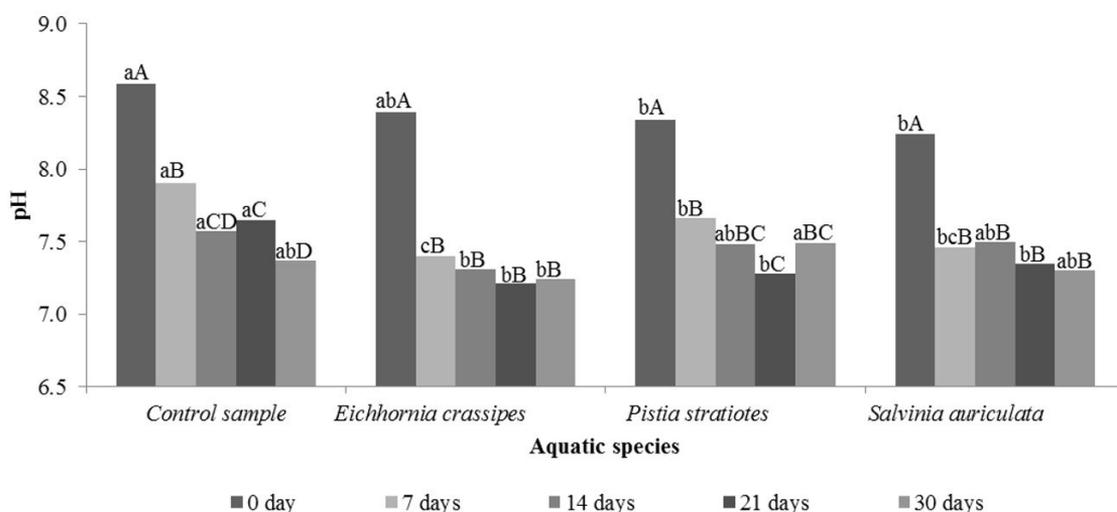
Figure 5. pH values obtained in the treatments with aquatic species, in five collection periods. Reference value according to CONAMA Resolution nº 430/2011 = pH: 5-9. DMS for collection periods = 0.21; CV (%) = 1.47 classification on lowercase letters. DMS for aquatic species = 0.22; CV (%) = 1.49 classification on capital letters. Tukey Test was applied at the level of 5% probability.
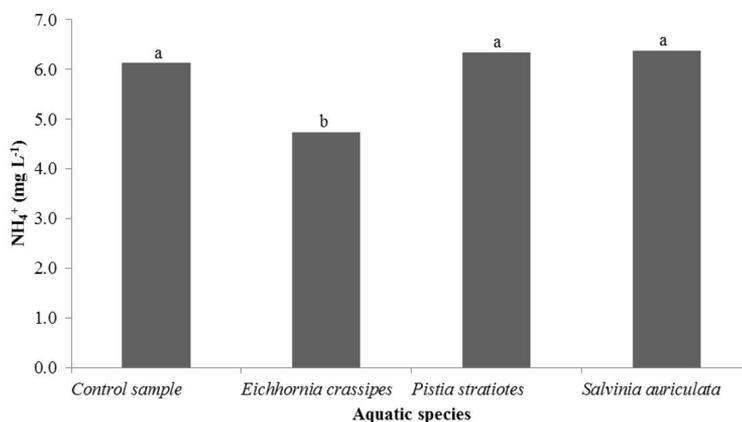
Figure 6. Available NH_4^+ concentration, in mg L^{-1}, in the treatments with aquatic species. Reference value according to CONAMA Resolution nº 430/2011 = NH_4^+: 20.0 mg L^{-1}. DMS = 0.96; CV (%) = 10.47. The means followed by the same letter are not statistically different among themselves. Tukey Test was applied at the level of 5% probability.

were 58.9% for a five day detention time, 86.1% for seven days and 97.7% for ten days, thus increasing the removal efficiency with an increased hydraulic detention time.

Sezerino and Philippi (2000) highlight that, in such treatment systems with plants, approximately 74.0% from the NH_4^+ removal may be associated with the plant uptake. CONAMA Resolution no. 430/2011 puts a limit in the concentration of the NH_4^+ ion to values of 20 mg L^{-1} in effluents from any polluting source. Therefore, the values obtained in the treatments submitted to the aquatic plants (Figure 6) are in accordance with the Brazilian legislation.

The NH_3 volatilization (desorption to the atmosphere) is a physical process of disengaging this gas from the dissociation of the NH_4^+ ion in aquatic environments with high pH values (Assunção, 2009). The NH_3 volatilization was observed in the interaction between treatments with aquatic macrophytes and the evaluation periods (Figure 7).

According to Figure 7, this volatilization was higher in the control sample treatment, stressing that the differentiation occurred, mainly, after the period of 12 days of collection. It is also stated that the systems containing aquatic species provided lower losses of NH_3, with a noticeable decrease during the evaluation period. According to Körner and Vermaat (1998) and Sooknah and Wilkie (2004) in treatment systems with aquatic macrophytes, the removal of N happens by the direct absorption of the plant, by the action of microorganisms fixed in the roots and by the NH_3 volatilization itself.

It is noteworthy that at the initial collection period (1 to 4 days), the NH_3 concentration was higher in the four treatments proposed. According to Assunção (2009), when the pH is high, he balance between the free NH_3 and NH_4^+ ion tends to shift toward the NH_3 formation. For values above 9.26 there's a predominance of NH_3 (practically 100% at pH near 11.0) and for values close to neutrality (between 6.0 and 7.0), practically all NH_3 is in the ionized form.

For Kiehl (1985), the NH_3 losses increase when the compound reaction is in the alkalinity zone and when the pH reaches values above 8.0 and 9.0, a large part of N turned into NH_3, and may be lost in the atmosphere. The balance represented by the reaction $NH_4^+ \leftrightarrow NH_3 + H^+$, at pH values around neutrality, is shifted to the left, and there is a predominance of NH_3 only to pH values above 8.5 (SCHMIDELL et al., 2007). Probably, due to the fact that the pH of the effluent was higher (above 8.0) in the first days (0 to 7 days) (Figure 5), the release of NH_3 was higher. Mkhabela et al. (2009) stated that the initial high pH of the slurry is a factor that contributes to such emissions, and Meade et al. (2011) observed that 95% of the NH_3 emissions happened in the first 24 hours after applying the slurry.

Gonzatto et al. (2013), when evaluating, under field conditions, the NH_3 volatilization and N_2O emission after applying the pig slurry in maize, observed that approximately 80.0% of the N losses by volatilization happened in the first 22 hours after applying the slurry, quickly reducing with time. According to the authors, this kinetic observed in the NH_3 emissions may be attributed to the high content of NH_4^+ of the pig slurry, which is one of the factors that influence the NH_3 volatilization (Sommer and Hutchings, 2001).

Harper et al. (2004) state that the main factors that can be correlated to the NH_3 volatilization in pig ponds are the wind speed, temperature, NH_4^+ concentration and the effluent pH. Thus, it is observed that the NH_4^+ concentrations found in the pig slurry (Figure 6) were higher in the beginning of the experiment; besides, in the aquatic macrophytes treatment, the NH_3 volatilization may have been reduced from the 12 days of collection, compared to the control sample treatment (Figure 7), due to the high absorption of NH_4^+ by the plants, as can be observed in Figure 6. In the treatment with *Eichhornia crassipes* (Mart.) Solms,
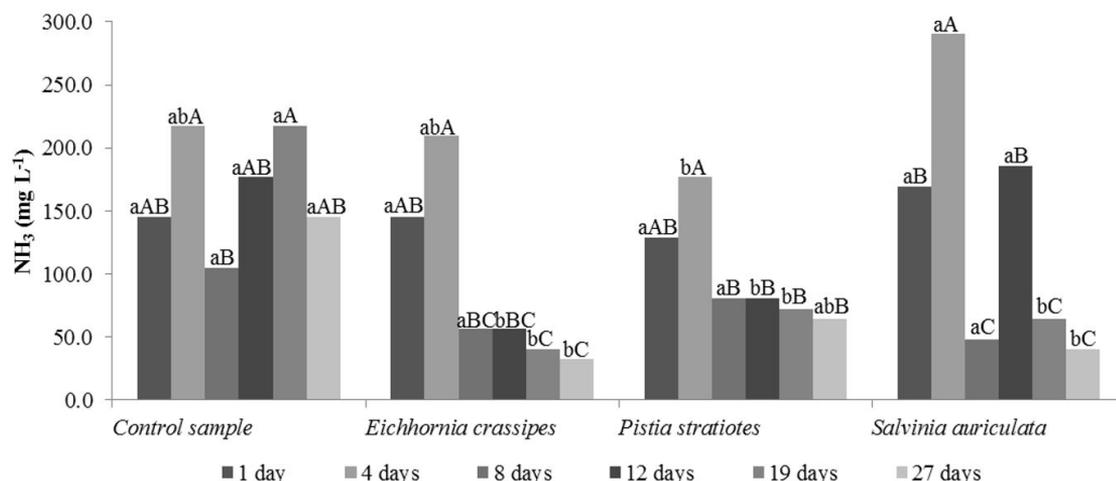
Figure 7. Available NH_3 concentration, in mg L^{-1}, in the treatments with aquatic species, in six collection periods. DMS for collection periods = 81.62; CV (%) = 30.33 classification on lowercase letters. DMS for aquatic species = 91.04; CV(%) = 31.25 classification on capital letters. Tukey Test was applied at the level of 5% probability.

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the NH_4^+ concentrations in the effluent were lower and statistically differ from the others.

Zimmo et al. (2003) verified that the NH_3 volatilization rates in ponds with algae were higher than in ponds with aquatic plants (*Lemna gibba*). According to the authors, it can be explained by the lower NH_3 values in ponds with plants due to the shadowing and to the lower pH values. In this case, the volatilization rate correlated to the free NH_3 concentration in the water of the pond.

When considering the percentages of reduction of the NH_3 volatilization rates, it is possible to notice that the treatment systems with the species *Eichhornia crassipes* (Mart.) Solms and *Salvinia auriculata* Aubl. were more expressive, 77.8% and 76.2% in a 27 day retention time. This reduction in the NH_3 volatilization shows the importance of the presence of aquatic plants in these treatment systems. Oron et al. (1988), when evaluating the treatment of domestic sewage under cultivation of floating aquatic plants of the family Lemnaceae, obtained NH_3 removal efficiency of 90.0%, with a 10 day retention time and initial concentration of 520 mg L^{-1} of COD. Researches carried out in wetlands that treated pig slurry waters mention that the NH_3 volatilization represented less than 20.0% of the N removed by the wetlands (Poach et al., 2002).

4. Conclusions

The presence of aquatic macrophytes promoted the reduction of NH_4^+ concentration and of the pH values of swine fluids, and this resulted in the reduction of NH_3 volatilization rates to the environment, with emphasis on the system with *Eichhornia crassipes* (Mart.) Solms, which presented the lowest rate of volatilization.

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