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To cite this version:

Eric Dumont, S. Lagadec, N. Guingand, L. Loyon, A. Amrane, et al.. Ammonia removal using biotrickling filters Part A Determination of the ionic nitrogen concentration of water using electrical conductivity measurement. ChemEngineering, 2020, 4 (3), pp.1-9. 10.3390/chemengineering4030049 . hal-02932019

HAL Id: hal-02932019

https://hal.science/hal-02932019v1

Submitted on 29 Sep 2020

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Article

Ammonia Removal Using Biotrickling Filters: Part A: Determination of the Ionic Nitrogen Concentration of Water Using Electrical Conductivity Measurement

Éric Dumont [1,*], Solène Lagadec [2], Nadine Guingand [3], Laurence Loyon [4], Abdeltif Amrane [5], Valérie Couroussé [5] and Annabelle Couvert [5]

1 UMR 6144, Oniris, GEPEA, Université de Nantes, IMT Atlantique, 44307 Nantes, France
2 Chambre d'Agriculture de Bretagne, 35042 Rennes, France; solene.lagadec@bretagne.chambagri.fr
3 IFIP Institut du Porc, 35651 Le Rheu, France; nadine.guingand@ifip.asso.fr
4 INRAE, 35044 Rennes, France; laurence.loyon@irstea.fr
5 ISCR–UMR6226, CNRS, ENSCR, Université de Rennes, 35000 Rennes, France; abdeltif.amrane@univ-rennes1.fr (A.A.); valerie.courousse@ensc-rennes.fr (V.C.); annabelle.couvert@ensc-rennes.fr (A.C.)
* Correspondence: eric.dumont@imt-atlantique.fr; Tel.: +33-025-185-8266

Received: 1 April 2020; Accepted: 7 August 2020; Published: 18 August 2020

Abstract: It is emphasized that a generalized relationship can be used to predict the ionic nitrogen concentration (i.e., sum of ammonium NH_4^+, nitrite NO_2^- and nitrate NO_3^-) of the scrubbing liquid in a biotrickling filter treating ammonia emissions by measuring the electrical conductivity (EC) of the water directly. From measurements carried out on different water samples from six biotrickling filters in operation in pig husbandries, the generalized relationship is: $\Sigma([NH_4^+]+[NO_2^-]+[NO_3^-])_{g\ N/L} = 0.22\ EC_{mS/cm}$. This equation is valid provided the fresh water feeding the biotrickling filter has a low electrical conductivity (<1 mS cm^{-1}). Moreover, since ammonium, nitrite and nitrate ions are the ultra-majority ions in the liquid phase, the balance between NH_4^+ and (NO_2^- + NO_3^-) was confirmed, and consequently the relationship $NH_4^+ = 0.11\ EC_{mS/cm}$ can also be applied to determine the ammonium concentration from the EC. As a result, EC measurement could be applied extensively to monitor operating biotrickling filters worldwide and used to determine ammonia mass transfer in real time, keeping in mind that the accuracy of the generalized relationship is ±20%.

Keywords: ammonia; absorption; mass transfer; air treatment; biofiltration; electrical conductivity

1. Introduction

Air pollution due to ammonia (NH_3) generated by human activities leads to environmental problems that affect the atmosphere (eutrophication, acidification of soils, particle precursors), the neighborhood (odor nuisance) and the health of humans and animals (respiratory diseases) [1–5]. Biotrickling filtration is considered an efficient and economical technique for ammonia removal [6]. In a biotrickling filter, the polluted air flows through an inert packing material sprayed continuously with water, contained in a buffer tank. Once the ammonia has been absorbed into the water, it is first converted into ammonium cations (NH_4^+) which are subsequently oxidized into nitrite ions (NO_2^-), then into nitrate ions (NO_3^-) by the biomass fixed onto the packing material and present in the scrubbing liquid, i.e., ammonia oxidizing bacteria (AOB) and nitrite oxidizing bacteria (NOB) [7–11]. Although ammonia easily dissolves in water, a removal efficiency (RE) of around 70%–80% is expected; however, values of between 10% and 99% are reported in the literature [6]. The large discrepancies between RE values may be due to the operating conditions applied in biotrickling filters, which may vary significantly from one device to another, and to the accumulation of large amounts of nitrogen

ions in the water (mainly NH_4^+ + NO_2^- + NO_3^-), which can lead to a decrease in the ammonia transfer rate. To avoid the accumulation of nitrogen ions in the liquid phase, some of the water must be discharged regularly and replaced with fresh water, which indicates good management of the equipment. The large discrepancies between RE values may also be due to the accuracy of the RE determination itself. One widely-used method of determining NH_3 concentration in air emissions at pig farms is InfraRed PhotoAcoustic Spectroscopy (IR-PAS) [1,12–15]. However, the management of this analytical apparatus is complex and the non-compensated interferences between gases (ammonia NH_3, nitrous oxide N_2O, carbon dioxide CO_2, methane CH_4 and water vapor H_2O) contribute to the uncertainty in emissions measurement (under- and over-estimations [13]). Since RE determination can be unreliable, the real amount of ammonia transferred from gas to water cannot be known with any certainty. Therefore, the objective is to develop a new, simple way of monitoring the amount of nitrogen transferred between phases in biotrickling filters over time. The new method is based on continuous measurement of the electrical conductivity (EC) of the scrubbing liquid. This measurement, which may already be carried out at industrial scale in different countries—mainly Northern Europe—is used to control the water quality [6,15,16]. Consequently, the new method (developed in part B) could allow the nitrogen transferred in the washing water (in g_N day^{-1} or in g_N week^{-1} animal^{-1}) of a large number of in situ biotrickling filters to be quantified cheaply and easily. To demonstrate that this new method can be applied to any industrial biotrickling filter, it was first necessary to prove that a generalized relationship can be used to predict the nitrogen concentration by measuring the electrical conductivity of the scrubbing liquid directly. The objective of part A of this paper is therefore to show that the theoretical relationship EC vs the ionic nitrogen concentration in the liquid phase can be applied to any livestock facility. Water samples from six different industrial biotrickling filters located in Brittany were analyzed for this purpose and compared with the available literature data.

2. Rationale

A few works have reported a relationship between the amount of nitrogen ions in the scrubbing liquid and the electrical conductivity [17–20]. According to [8], half of the ammonia absorbed in the washing liquid is oxidized while the other half remains dissolved as ammonium. Consequently, the combined NO_2^- + NO_3^- molar concentration should correspond closely to the NH_4^+ molar concentration, since ammonium is the only cation available to balance the anions produced, the nitrogen species being the ultra-majority in the water for biotrickling filters used in pig and poultry facilities. From the molar ionic conductivity of NH_4^+, NO_2^- and NO_3^- (73.5, 71.8 and 71.42 S cm^2 mol^{-1}, respectively) [21], Melse et al. [19] showed that the electrical conductivity of these three ions in an ideal solution are similar, i.e., 5.25, 5.13 and 5.10 mS cm^{-1} per g_N dissolved in one liter. Similarly, Ottosen et al. [8] indicated that a 7 mM aqueous solution of NH_4NO_x has a conductivity of 1 mS cm^{-1}. In other words, 5.1 mS cm^{-1} corresponds to 1 g_N L^{-1}, or 0.196 g_N L^{-1} corresponds to 1 mS cm^{-1}. As a result, it can be argued that the nitrogen concentration in water could theoretically be deduced by measuring the electrical conductivity, using the following relationship:

$$\Sigma\,([NH_4^+] + [NO_2^-] + [NO_3^-])_{\,gN/L} = 0.196\ EC_{\,mS/cm} \quad (1)$$

From the measurement of electrical conductivity and the concentrations of ammonium ions, nitrite ions and nitrate ions in the water of a biotrickling filter treating ammonia effluent from a pig farm in the Netherlands, Melse et al. [19] obtained a linear trend between the two parameters ($EC_{mS/cm}$ = 4.6011 $\Sigma\,([NH_4^+] + [NO_2^-] + [NO_3^-])_{gN/L}$ + 1.8345; R^2 = 0.945). This relationship can be rewritten as $\Sigma\,([NH_4^+] + [NO_2^-] + [NO_3^-])_{gN/L} = 0.22EC_{mS/cm} - 0.40$). Compared with Equation (1), the slope is therefore slightly higher than expected (+ 11%). In addition, it can be observed that the intercept determined by the authors (i.e., +1.8345 mS cm^{-1}) is very high, indicating that the ground water used to feed the biotrickling filter was salty. Note that typical values for ground water would be lower than 1 mS cm^{-1}, but higher values are possible [22,23]. Another linear regression was proposed by

Liu et al. [17] in a study dedicated to 13 field-scale bioscrubbers in northern Germany. These authors reported the following relationship: $EC_{mS/cm} = 3.34\Sigma\ ([NH_4^+] + [NO_2^-] + [NO_3^-])_{gN/L} + 5.48$; $R^2 = 0.83$ (corresponding to $\Sigma\ ([NH_4^+] + [NO_2^-] + [NO_3^-])_{gN/L} = 0.30EC_{mS/cm} - 1.64$)). The significant difference between the relationships proposed by Melse et al. (2012) and Liu et al. (2017) may be due to differences in the water they analyzed, the relationship proposed by [19] being obtained for only one pig farm. Nonetheless, the slope obtained by [17] was 53% greater than the theoretical value (on the basis of the slope: i.e., 0.3 vs. 0.196) and the intercept (+5.48 mS cm^{-1}) seems unrealistic. A similar finding was also reported by Van der Heyden et al. [18] for a pig fattening facility located in Belgium: $EC_{mS/cm} = 3.5\ \Sigma\ ([NH_4^+] + [NO_2^-] + [NO_3^-])_{gN/L} + 2.0$; $R^2 = 0.978$ (corresponding to $\Sigma\ ([NH_4^+] + [NO_2^-] + [NO_3^-])_{g\ N/L} = 0.29EC_{mS/cm} - 0.57$). The authors indicated that the large deviation between the theory and the experiment result could be due to non-ideal conditions occurring in water that contains a high concentration of nitrogen species. From a study conducted on 31 field-scale bioscrubbers in operation between 2003 and 2010, Lagadec et al. [20] measured the electrical conductivity and concentration of ammonium ions in the water (nitrite ions and nitrate ions were not considered). A good trend between EC and $[NH_4^+]$ was obtained ($[NH_4^+]_{mg\ N/L} = 123.75EC_{mS/cm} - 432.07$; $R^2 = 0.9806$). Using g_N L^{-1} as the unit for ammonium and considering that ammonium concentration is balanced by nitrite and nitrate concentrations, the slope is therefore $2 \times 123.75/1000 = 0.247$, i.e., 26% higher than the theoretical value.

Taking into account that (i) nitrite and nitrate ions were not considered in the study conducted by Lagadec et al. [20]; (ii) the relationships provided by Liu et al. [17] and Van der Heyden et al. [18] are far from the theory, it therefore appears necessary to take new measurements for different biotrickling filters in order to confirm the relationship obtained by Melse et al. [19].

3. Materials and Methods

3.1. Water Samples

Water samples from 6 different biotrickling filters at pig farms located in Brittany (France) were analyzed in 2 measurement campaigns (spring 2017 for site #1, 2, 3, 4 and autumn 2017 for site #1, 2, 5, 6). For each measurement campaign and each biotrickling filter, two volumes of water were sampled, one from the surface of the tank and one from the bottom (respectively, 'top water' and 'bottom water' in the text). For site #4, the number of samples was doubled. For each water sample, the following parameters were determined: (a) electrical conductivity; (b) ammonium ion concentration; (c) nitrite ion concentration; (d) nitrate ion concentration; (e) total nitrogen concentration.

3.2. Analytical

Electrical measurement was carried out using an EC meter (WTW Cond 340i, Weilheim, Germany) with temperature correction (measurements were normalized at 25 °C). Nitrite and nitrate ions were analyzed using a DIONEX DX120 ion chromatograph (ThermoFischer Scientific, USA) equipped with a conductivity detector, using an anion exchange column AS19 (4 × 250 mm) as the stationary phase, water as the mobile phase and potassium hydroxide KOH as eluent (flow rate 1 mL min^{-1}; elution gradient: (i) 10 mM from 0 to 10 min, (ii) from 10 to 25 min, the concentration of KOH increases from 10 mM to 45 mM; (iii) 45 mM from 25 to 35 min). Ammonium measurement was carried out using the spectrophotometric Nessler method at 420 nm. Total Nitrogen (TN) was measured using a Total Organic Analyzer Shimadzu TOC-$V_{CPH/CPG}$ (Shimadzu Company, Marne-la-Vallée, France). Dissolved nitrogen compounds were burnt and converted to nitrogen oxide (NO) then to nitrogen dioxide (NO_2). The NO_2 was subsequently analyzed by chemiluminescence.

4. Results

4.1. Balance between [NH_4^+] and Σ ([NO_2^-] + [NO_3^-])

According to [8] the ammonium ion being the only cation sufficiently available to counterweight the anions produced, the concentrations of NH_4^+ and (NO_2^- + NO_3^-) in water should be balanced. As observed in Figure 1, equilibrium between the cation and the two anions is usually verified in spite of some possible discrepancies. Similar discrepancies are also reported in the literature [17]. For instance, the concentration of ammonium ions can be twice the concentration of anions (as for site #4), but the reverse was also observed (as for site #1 in autumn). It can also be observed that the concentration of nitrate ions was always significantly lower than that of nitrite ions for all the waters analyzed, which is typical in biotrickling filters treating ammonia emissions [8]. Biological mechanisms controlling the distribution of nitrite and nitrate ions in biotrickling filters must always be clarified, but it seems that incomplete denitrification leading to high nitrite ion accumulation in the water is due to either the absence or inhibition of nitrite oxidizing bacteria. Recent findings have highlighted that NOB was not detected in the case of non-inoculated water [24], which is the case with all the water samples studied here. Nonetheless, even in the presence of NOB, inhibition can occur in water with high nitrite concentrations in relation to the presence of free nitrous acid (HNO_2; [7]). In addition, Figure 1 shows no difference between 'top water' and 'bottom water' indicating that water in the tanks can be considered well-mixed.

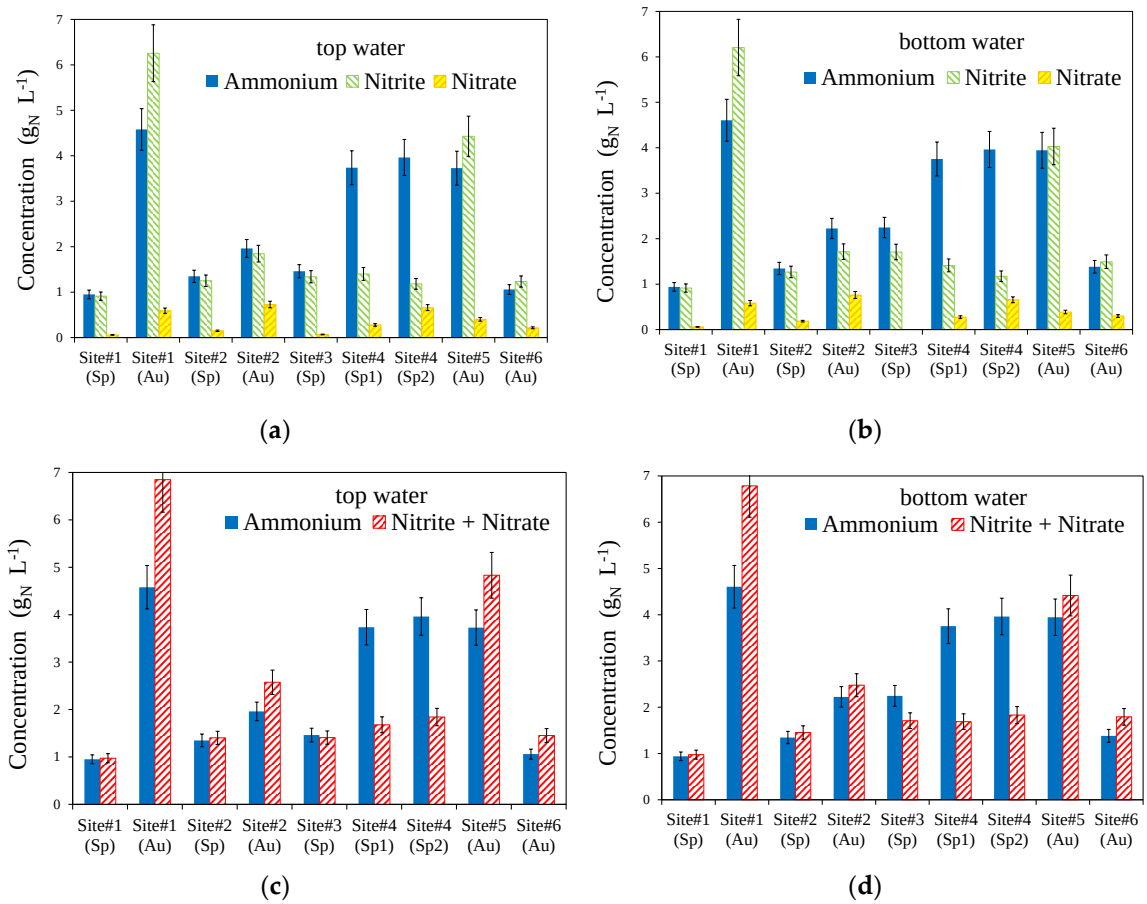

Figure 1. Concentration of nitrogen ions measured in water: (**a**) NH_4^+, NO_2^- and NO_3^- in 'top water'; (**b**) NH_4^+, NO_2^- and NO_3^- in 'bottom water'; (**c**) NH_4^+ and Σ (NO_2^- + NO_3^-) in 'top water'; (**d**) NH_4^+ and Σ (NO_2^- + NO_3^-) in 'bottom water' (Sp = spring; Au = autumn).

The comparison between Total Nitrogen and the sum of ammonium, nitrite and nitrate ions in water is shown in Figure 2. It appears that high concentrations of nitrogen salts in water samples

can be encountered. Due to the possible presence in water of nitrogen species other than ammonium, nitrite and nitrate ions (free ammonia, organic compounds, hydroxylamine, etc.), the Total Nitrogen should be higher than (or at least equal to) the sum of the ammonium, nitrite and nitrate ions. As observed in this figure, the TN measurement can be sometimes lower than Σ ([NH_4^+] + [NO_2^-] + [NO_3^-]), which provides information on the accuracy of the measurements. The difference between the two parameters measured can be estimated at ±15%.

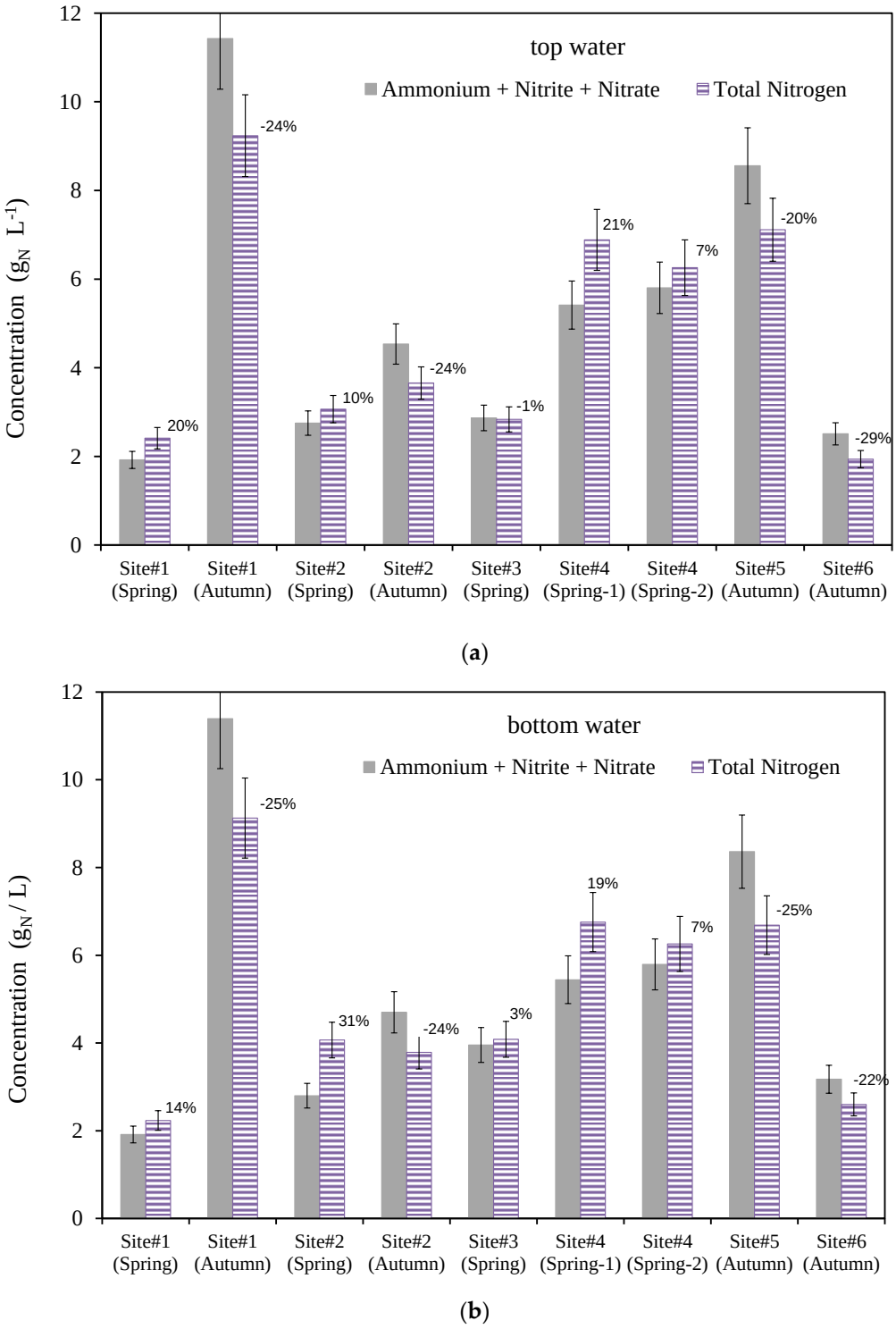

Figure 2. Total Nitrogen (TN) and Σ (NH_4^+ + NO_2^- + NO_3^-). The percentages indicated correspond to the relative difference between TN and Σ (NH_4^+ + NO_2^- + NO_3^-): (**a**) 'top water'; (**b**) 'bottom water'.

4.2. Electrical Conductivity vs. Ion Concentration

The results shown in Figure 3 confirm the linear trend which exists between the electrical conductivity and the concentration of nitrogen ions in the scrubbing liquid (sum of the ammonium, nitrite and nitrate ions). The linear regression calculated from points of the experiment (Σ ([NH_4^+] + [NO_2^-] + [NO_3^-])$_{gN/L}$ = 0.23EC$_{mS/cm}$ − 0.06) is close to the relationship obtained by [19]. Nevertheless, experimental values are usually higher than the expected values calculated from Equation (1), i.e., +15%. The linear trend between EC and the ammonium concentration in water is also confirmed (Figure 4). Additionally, the ratio between the slopes of the linear regressions calculated from Figures 3 and 4 is around two, indicating that the balance between NH_4^+ and (NO_2^- + NO_3^-) is confirmed (the discrepancies previously mentioned balancing each other out). As a result, a generalized relationship could be used to determine the concentration of nitrogen ions from direct EC measurement of any water sample, provided the fresh water feeding the biotrickling filter has a low electrical conductivity. In this case, ammonium, nitrite and nitrate ions are the ultra-majority ions in water for ammonia treatment, and consequently the generalized relationship would be suitable. Basically, if no ions are present in the water then the EC value is zero and the intercept value of the generalized relationship must be equal to zero. In addition, the results shown in Figures 3 and 4 suggest that the following relationship could be used:

$$\Sigma\ ([NH_4^+] + [NO_2^-] + [NO_3^-])_{gN/L} = 0.22\ EC_{mS/cm}\ (\text{accuracy} \pm 20\%) \quad (2)$$

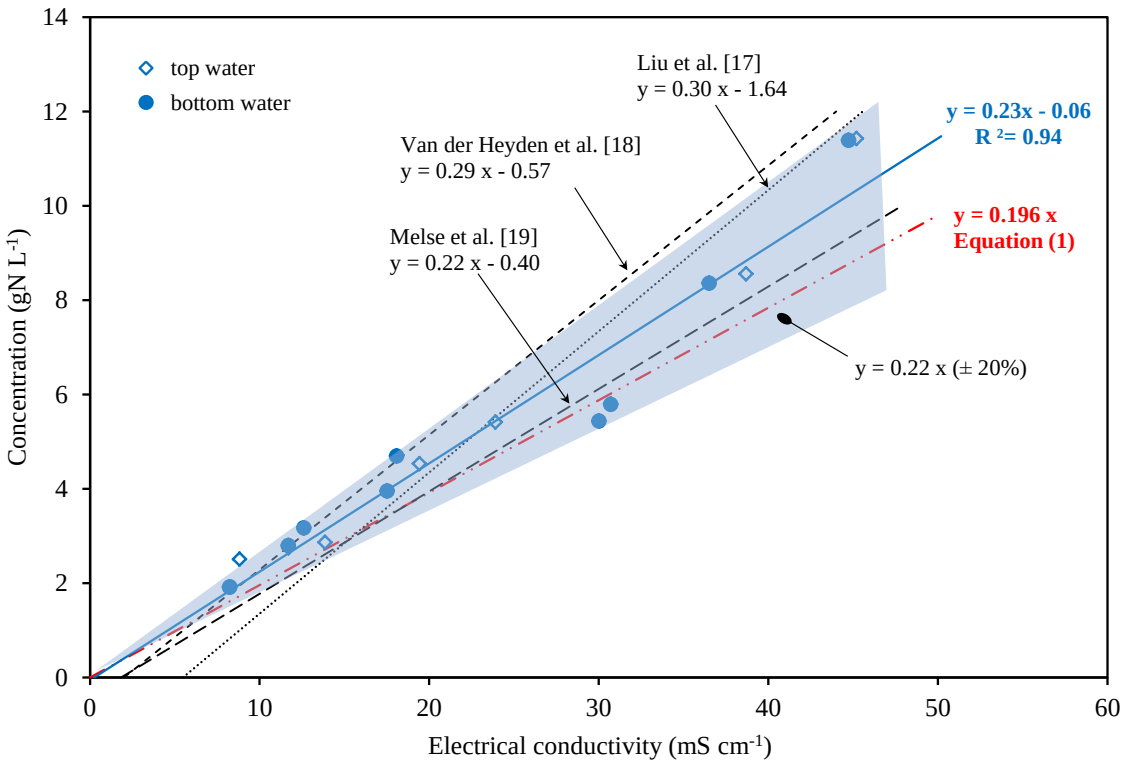

Figure 3. Electrical conductivity vs concentration in nitrogen ions ([NH_4^+] + [NO_2^-] + [NO_3^-]) in water samples.

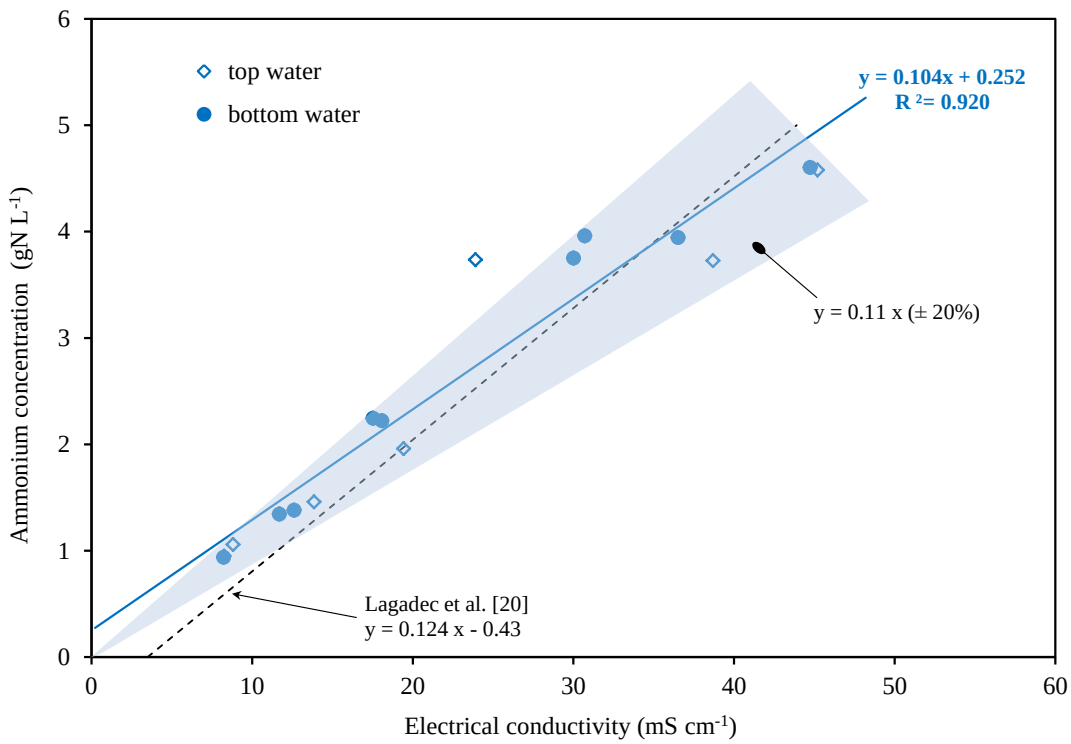

Figure 4. Electrical conductivity vs NH_4^+ concentration in water samples.

As observed in Figure 3, this relationship describes the experiment data satisfactorily over a wide range of nitrogen concentrations (up to 12 g_N L^{-1}), as well as a large part of the literature data (blue shaded zone) [17–19]. For EC values lower than 10 mS cm^{-1}, it can be highlighted that the relationships reported by these authors are less appropriate than Equation (2) for determining the concentration of nitrogen ions, mainly due to the values of the y-intercept. Additionally, the balance between the cation NH_4^+ and the anions (NO_2^- + NO_3^-) implies that Equation (3) deduced from Equation (2) should also validate the experimental data. This validation can be observed in Figure 4. Consequently, it can be concluded that Equation (3) could be used in practice as a generalized relationship applied to any biotrickling filter, keeping in mind that the accuracy is ±20%.

$$[NH_4^+]_{gN/L} = 0.11\ EC_{mS/cm}\ (\text{accuracy} \pm 20\%) \quad (3)$$

5. Conclusions

The concentration of nitrogen ions in the water tanks of biotrickling filters could be deduced by measuring the electrical conductivity of the water directly. A generalized relationship was proposed: $\Sigma([NH_4^+] + [NO_2^-] + [NO_3^-])_{gN/L} = 0.22\ EC_{mS/cm}$. This equation is valid provided the fresh water feeding the biotrickling filter has a low electrical conductivity (<1 mS cm^{-1}). Given that EC measurement is a cheap and easy technique (compared to measuring nitrogen ion concentrations), it could be extensively applied for monitoring biotrickling filters in operation worldwide. In addition, the use of this relationship coupled with EC measurement could advantageously replace the measurement of ammonia concentration in air to determine the ammonia mass transfer in industrial scale biotrickling filters, as demonstrated in Part B.

Author Contributions: All authors conceived and designed the experiments; S.L., N.G., A.C., L.L., A.A. and V.C. performed the experiments; A.C., A.A., É.D. and V.C. analyzed the data and results; É.D. wrote the paper; S.L., N.G., A.C., A.A. and É.D. reviewed the manuscript. All authors have read and agreed to the published version of the manuscript.

Funding: This research was funded by the French Environment and Energy Management Agency (ADEME) (project PRIMEQUAL No. 1660C0014).

Conflicts of Interest: The authors declare no conflict of interest.

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