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# Alkaline Wood Ash, Turbulence, and Traps with Excess of Sulfuric Acid Do Not Strip Completely the Ammonia off an Agro-waste Digestate

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## Abstract

The present study combined two nutrient management strategies to improve the marketability of a waste-derived fertilizer: (a) isolation of ammoniacal nitrogen and (b) preparation of a bulk soil amendment. The wood fly ash with low content of pollutants was added to an agrowaste anaerobic digestate as alkaline stabilizer, which promoted the volatilization of ammonia and adsorption processes, and as nutrient supplement. The 39.71 ± 1.44 g blend was incubated for 60 hours at 20°C and 100 rpm in a closed chamber (250-mL Schott Duran® bottle) with a 5.21 ± 0.10 mL sulfuric acid trap of 10 different concentrations (0.11, 0.21, 0.32, 0.43, 0.54, 0.64, 0.75, 0.86, 0.96, and 1.07 mol/L). For analytical purposes, the sulfuric acid, water-soluble, and water-insoluble fractions of the blend were isolated after the incubation. The 1.07 mol/L sulfuric acid solution contained 23.69 ± 5.72 % more of ammonical nitrogen than the 0.11 mol/L solutions. However, in all cases the amount of nitrogen in the $H_2SO_4$ compartment was lower than the one in the water-soluble and water-insoluble fractions. Only the 15.52 ± 2.13 % of the nitrogen accounted after the incubation was found in the $H_2SO_4$ trap. The bottleneck of the $NH_3$ stripping process was the rate of mass transfer at the interface between the blended fertilizer and the headspace of the closed chamber. The organic phosphorus was more susceptible to be adsorbed during the alkaline treatment with non-intrusive acidification than the nitrogen and carbon. Activation of the ash as adsorbent before mixing with the digestate should improve the properties of the blend as slow release fertilizer, since more nutrients would end in the water-insoluble fraction.

**Keywords:** Waste-derived fertilizer, Alkaline stabilization, Ammonia volatilization, Adsorption, Closed chamber

**Abbreviations:** $C_{org}$-Organic Carbon, C/N-Carbon to Nitrogen Ratio, $NH_4^+$-N-Ammoniacal Nitrogen, $N_{org}$-Organic Nitrogen, $NO_3$-N-Nitric nitrogen, $P_{org}$-Organic Phosphorus, $PO_4^{3-}$-P-Phosphorus in the form of Orthophosphate, PVWD-Post-Harvest Vegetable (Agro-Industrial) Waste Digestate, WFA-Wood Biomass Derived Fly Ash, WS-Water-Soluble, WI-Water-Insoluble, $H_2SO_4$-Sulfuric Acid.

## Introduction

The commercialization of waste-derived fertilizers is constrained by the level of contaminants [1]. It might be easier to isolate the plant nutrients contained in the residues and sell them as conventional fertilizers [2]. The addition of ashes to organic manures, such as anaerobic digestates, could be proposed for: (a) improving the properties of these organic amendments as fertilizers; (b) reducing the greenhouse emission and phosphorus leaching associated to the management and use of these materials; (c) manufacturing of a granular fertilizers; and (d) increasing the pH to promote the volatilization of ammonia and subsequent capturing the $NH_3$ in a sulfuric acid trap. The ammonium sulfate could be sold as liquid fertilizer (40–60% $(NH_4)_2SO_4$ solution) or as solid after crystallization. On the other hand, the use of clean materials with low content of pollutants, such as biomass ash and agro-industrial digestate, enables the end-of-waste status and the marketability of the blend as bulk soil amendment [3-9].

Mixing wood ash and agro-waste digestate to get an approximate carbon, nitrogen, phosphorus ratio (C/N/P) of 40/2/1 could enhance the use efficiency of these elements upon spreading the blend in the soil [10,11]. Given the high pH of the blend ash-digestate, this material could be used as liming agent, after the removal of the ammoniacal nitrogen ($NH_4^+$-N) [12]. This nutrient management strategy might offer better results than the preparation of the blend under acid conditions to promote the adsorption and to minimize the loss of $NH_4^+$-N [13]. In fact, Miranda et al. (2021) found that the direct addition of sulfuric acid to a 4.5% (w/v) blend of biochar and cattle slurry mitigated less the $NH_3$ emissions than just applying the 0.3 mL $H_2SO_4$ (98%) to 50 g of cattle slurry to reach a pH 5.5. The stepwise mechanism of acidification, dehydration, and adsorption or flocculation is widely used to improve the management of anaerobic digestates [6,14]. The present study analyzed how the $H_2SO_4$ non-invasive acidification) affects the alkaline stabilization of a blend of Wood Fly Ash (WFA) and Post-Harvest Vegetable Waste Digestate (PVWD) in terms of: (a) $NH_4^+$-N recovery, (b) N, C, and P availability and, (c) overall C/N/P. The severe $H_2SO_4$ non-invasive acidification was meant to decrease the amount of $NH_3$ in the gas phase and create a greater gradient of concentration, which would be enough to overcome the mass transfer resistance at the layer between the blend WFA+PVWD and the headspace. The $H^+$ that remains in the blend WFA+PVWD due to the dissociation of WS $NH_4^+$ and subsequent volatilization of $NH_3$, might promote dehydration and adsorption processes affecting the availability of nitrogen, carbon, and phosphorus.

## Materials and Methods

For the preparation of the blend WFA+PVWD, the 10 mL of ultrapure milli-Q® water was added to each gram of sample before blending (1:10). This improved the fluency of the waste streams and generated the Water-Soluble (WS) fraction of the blend WFA+PVWD. A detailed description of the components of the blend WFA+PVWD is shown in the **Figure 1**:

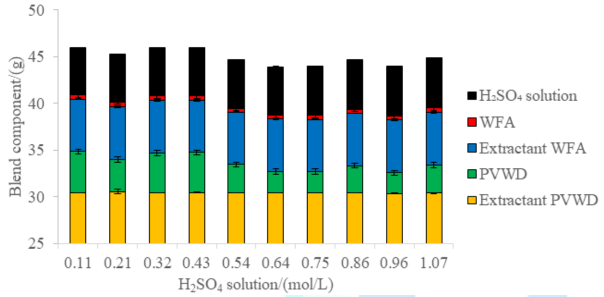

**Figure 1:** Detailed amounts of samples and extractant (ultrapure milli-Q® water) used for the preparation of the blend WFA+PVWD (**Table 1**) incubated in a closed chamber with different concentrations of $H_2SO_4$ in the trap (**Figure 2**).

The 60-hour incubation of the 39.71 ± 1.44 g fluidized blend WFA+PVWD was carried out under 100 rpm continuous shaking at 20 °C in a 250-mL (Schott Duran® bottle) closed chamber with a 5.21 ± 0.10 g $H_2SO_4$ solution to capture the $NH_3$ released, containing an aqueous solution of $H_2SO_4$ (**Figure 2**). To evaluate the effect of the non-intrusive acidification on the alkaline stabilization of the blend WFA+PVWD, the following concentrations of $H_2SO_4$ were tested: 0.11, 0.21, 0.32, 0.43, 0.54, 0.64, 0.75, 0.86, 0.96, and 1.07 mol/L. The setup employed was a modification of the procedure developed by Velthof et al. (2005), who performed 90-day incubation of manures. At every sampling point, they refreshed the $H_2SO_4$ solution and flushed the bottle containing the manure with $N_2$ gas for 10 minutes, to avoid any interference of the previous $NH_3$ release in the next measurement. A similar procedure was followed by Van der Stelt et al. (2007) for a 223-day incubation of dairy farm slurry. Destructive sampling was more convenient for the present work due to the shorter incubation. In this way, 40 experimental units (i.e. 4 repetitions for each of the 10 $H_2SO_4$ non-intrusive acidifications) were prepared. This methodology also offered more realistic results about the potential of the $H_2SO_4$ non-invasive acidification to affect the composition of the blend WFA+PVWD. The way of conducting this experiment was based on a previous study [15] (Unpublished) and aimed that, by the end of the treatment, all the fractions of the blend were in equilibrium, including the $H_2SO_4$ fraction. It is important to mention that the stoichiometric amount required to capture all the nitrogen of the blend WFA+PVWD (**Table 1**) would correspond to a 3.43-mL solution of 0.11 mol/L $H_2SO_4$.

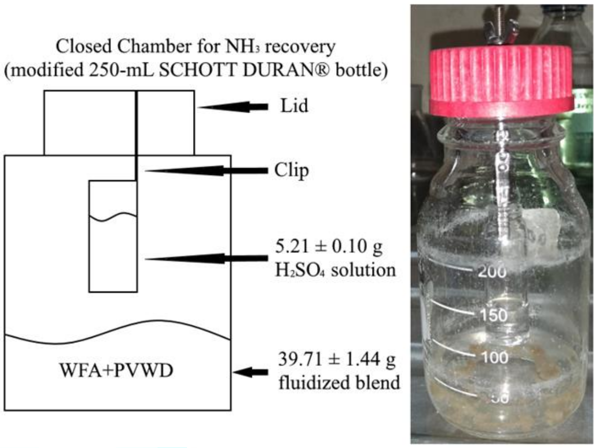

**Figure 2:** Experimental setup: Closed chamber for isolation of ammoniacal nitrogen of the blend WFA+PVWD.

**Table 1** shows the initial composition of the blend WFA+PVWD is expressed in terms of the amount of each element in the WS extract and Water-Insoluble (WI) material. Moreover, Table 1 includes the amount of nitrogen that would end in the $H_2SO_4$ compartment ([$H_2SO_4$] $NH_4^+$-N) at time zero. It is important to mention that there is a share of the WS $NH_4^+$-N, which has been volatilized and was not accounted as [$H_2SO_4$] $NH_4^+$-N due to the mass transfer resistance. The estimations of the organic forms of nitrogen ($N_{org}$), carbon ($C_{org}$), and phosphorus ($P_{org}$) were based on the nature of the samples and the empirical data of the segmented flow analysis (SEAL analytical), TOC-L (Shimadzu), and elemental analysis (Elemental vario EL cube). The $C_{org}$ and $P_{org}$ were relevant species in the blend, in the same way the concentration of WS $N_{org}$ was greater than the WS $NH_4^+$-N and the WS $NO_3^-$-N. The WS Norg was calculated as the difference between the WS N and the sum of WS $NH_4^+$-N and WS $NO_3^-$-N. All concentrations in this manuscript were expressed in fresh basis of the masses of WFA and PVWD used to prepare the blend. The calculation of the concentrations of the chemical species of N, C, and P was done using the amounts of $H_2SO_4$ solution, WS extract, and WI material isolated after the incubation **(Figure 3)**.

A 3-µm filtration was required for the solid-liquid separation of the WS extract and the WI material. The three fractions of the blend were weighed with a precision balance and the calculation of the volumes of $H_2SO_4$ solution and WS extract was done assuming a density of 1 g/mL. The availability of an element was defined as the ratio of the WS form to the WI form. The average recovery effectiveness of an element was calculated as its final amount measured after the incubation divided by its initial amount before starting the incubation. The trap effectiveness of stripping the $NH_3$ off the headspace was calculated as the ratio of the [$H_2SO_4$] $NH_4^+$-N to the nitrogen which could not be found in any of the WS and WI fractions. The One-Way Analysis of Variance (ANOVA) was performed with Microsoft Excel ($p<0.05$) to decide whether the $H_2SO_4$ non-invasive acidification affected significantly the composition of the blend WFA+PVWD.

## Results and Discussion

### Fractionation of the blend after the incubation

The mass of the 5 mL $H_2SO_4$ aqueous solutions in the traps increased in agreement with the content in sulfuric acid (**Figure 3a**). The 5 mL $H_2SO_4$ solutions of 0.64 and 0.96 mol/L had lower density than

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expected. The reason could be that in non-ideal solutions, the volumes are not strictly additive. However, the data reported by Hovey & Hepler [16] did not agree with the excess partial molar volume in that range of concentrations for the mixtures of sulfuric acid and water. On the other hand, it was unlikely that less volume or less concentrated H2SO4 solutions were used instead because these trends have not been seen in any of the other results of this study.

| Parameter | Units | Average | Standard deviation |
|---|---|---|---|
| Dry matter | % | 14.26 | 3.55 |
| [$H_2SO_4$] $NH_4^+$-N | mg/kg | 3.84 | 0.74 |
| WS $NH_4^+$-N | mg/kg | 102.59 | 26.96 |
| WS $NO_3^-$-N | mg/kg | 0.18 | 0.06 |
| WS N | mg/kg | 483.6 | 92.59 |
| WI N | mg/kg | 1,037.01 | 376.97 |
| WS C | mg/kg | 3,763.92 | 778.7 |
| WI C | mg/kg | 22,243.23 | 8,283.15 |
| WS $PO_4^{3-}$-P | mg/kg | 5.42 | 2.3 |
| WI P | mg/kg | 636.01 | 140.53 |

**Table 1:** Initial characterization, expressed in fresh basis, of a blend (n=4) of 0.51 ± 0.07 g WFA and 3.40 ± 0.41 g PVWD prepared in a closed chamber with a 4.39 ± 0.02 g trap of 0.11 mol/L $H_2SO_4$ aqueous solution. The amounts of milli-Q® water added to WFA and the PVWD were 5.58 ± 0.02 g and 30.43 ± 0.05 g, respectively [17].

A volume of 17.11 ± 3.45 mL of WS extract was lost during the 60 hours incubation at 100 rpm and 20 °C and subsequent filtration of the blends (**Figure 3b**). According to the ANOVA test ($p<0.05$), there was not significant increase in the amount of WS fraction recovered when using $H_2SO_4$ solutions in the trap with greater concentration than 0.43 M. This effect would be explained by the neutralization of the surface negative charges of the colloids of the PVWD by adding a cationic surfactant or via intrusive acidification, which make feasible their dehydration and flocculation. Similarly, the losses of WI material were 0.13 ± 0.05 g and did not show dependence on the concentration of the $H_2SO_4$ solution in the trap (**Figure 3c**). The explanation for the constant losses could be the procedure followed to achieve the solid-liquid separation. Some of the WI material would have remained stuck to the walls of the closed chamber and any weight gain due to the hydration of the ashes would have been lost during the drying at 105°C, before weighting the mass of the WI fraction recovered [13,14].

## pH of sulfuric acid trap and the blend WFA+PVWD

Since the volume of the traps were 5.21 ± 0.10 mL (Figure 3a), the pH of the $H_2SO_4$ solutions were measured directly (at 23.38 ± 0.36 °C) and also after eleven times dilution, to ensure a better contact with the probe of the Mettler Toledo® Seven CompactTM S220 pH/Ion meter. For the second set of measurements, the pH in the undiluted traps was calculated by increasing an order of magnitude on the concentration of the $H^+$ species determined in the eleven times diluted $H_2SO_4$ solutions. The first thing that needs to be highlighted is that the pH decreased during the incubation. This is opposite to what was expected since the absorption of $NH_3$ should increase the pH of the $H_2SO_4$ solutions. Understanding why the pH of the trap decreased is important to enhance the absorption and recovery of the $NH_3$ in the headspace. It could be possible that the pH of the trap decreased because of the evaporation of the water and subsequent increase in the concentration of $H^+$ ions.

However, the losses of the mass of the traps were negligible. It should be noted that, even when the pH decreased during the incubation, greater values than the theoretical ones were obtained. For example, the 1.07 M $H_2SO_4$ solution should have a pH lower than zero (i.e. -0.03) before the incubation and the value measured was 0.74 ± 0.02. After the incubation, the calculated value of pH from the measurements in the eleven times diluted $H_2SO_4$ solutions (0.03 ± 0.06; **Figure 4a**) was lower than the values measured in the undiluted traps (0.45 ± 0.05; Figure 4a). Thereby, these calculated values of the pH could be considered more accurate than the values of the pH obtained from the direct measurements of the $H_2SO_4$ solutions after the incubation. Therefore, the $H_2SO_4$ solutions needed to be diluted for the measurement of the variations in the concentrations of the $H^+$ species due to the upper detection limit of the pH-meter. The pH of the blend WFA+PVWD was not affected by the level of non-invasive acidification (10.40 ± 0.46; **Figure 4b**).

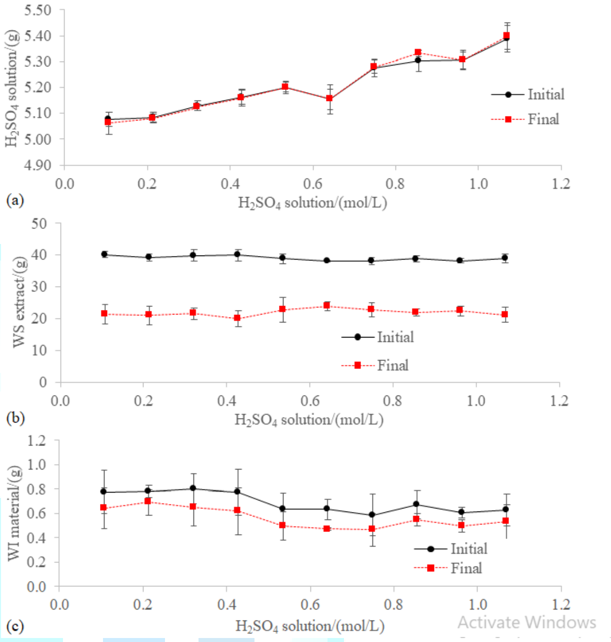

**Figure 3:** Initial (i.e. before the incubation) and final (i.e. isolated after 60 hours of incubation at 100 rpm and 20 °C) masses of the fractions of the blend WFA+PVWD: (a) $H_2SO_4$ solution, (b) WS extract, and (c) WI material. The initial volume of the WS fraction was determined considering the moisture of WFA+PVWD (Table 1) and the milli-Q® water used to prepare the blend (Figure 1). The initial mass of WI material was assumed the dry matter of the blend (Table 1).

## Speciation of nitrogen

The [$H_2SO_4$] $NH_4^+$-N was the only nitrogen species that increased significantly ($p<0.05$) with respect to the initial characterization (Table 1). The concentration of WS $NH_4^+$-N (83.45 ± 56.99 mg/kg; **Figure 5a**), WS $NO_3^-$-N (0.18 ± 0.06 mg/kg; **Figure 5b**), WS N (463.91 ± 87.99 mg/kg; **Figure 6a**), and WI N (1,030.87 ± 185.20 mg/kg; Figure 6a) did not change significantly regarding the initial characterization (102.59 ± 26.96 mg WS $NH_4^+$-N/kg, 0.18 ± 0.06 mg WS $NO_3^-$-N/kg, 483.60 ± 92.59 mg WS/kg, and 1,037.01 ± 376.97 mg WI N/kg; Table 1). High [$H_2SO_4$] $NH_4^+$-N was expected because of the low pH of the $H_2SO_4$ solutions in the trap (0.29 ± 0.24; Figure 4a) were able to absorb the $NH_3$ available in the headspace. The effect of the non-intrusive acidification can be seen in Figure 5a, which shows increase (23.69 ± 5.72 %) in [$H_2SO_4$] $NH_4^+$-N and decrease (41.08 ± 17.46 %) of WS $NH_4^+$-N when the concentration of the $H_2SO_4$ solution in the trap was increased from 0.11 mol/L to 1.07 mol/L. Furthermore, Figure 3b shows the increase in the amount of WS fraction due to the presence of a $H_2SO_4$ trap with a concentration greater than 0.43 M. This significant dehydration of the WI fraction has a p-value of 0.16. In previous work [15] (Unpublished), in which the WFA was acidified with a 1.82 mol/L aqueous solution of hydrochloric acid before mixing it with the PVWD, a level of 1,968.90 ± 588.36 mg WI N/kg blend (HCl-WFA+PVWD) was reached. Since lower concentration of WI N was found in the blend WFA+PVWD of the present study, the $H_2SO_4$ non-

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invasive acidification did not promote the adsorption of WS N as much as the HCl intrusive acidification.

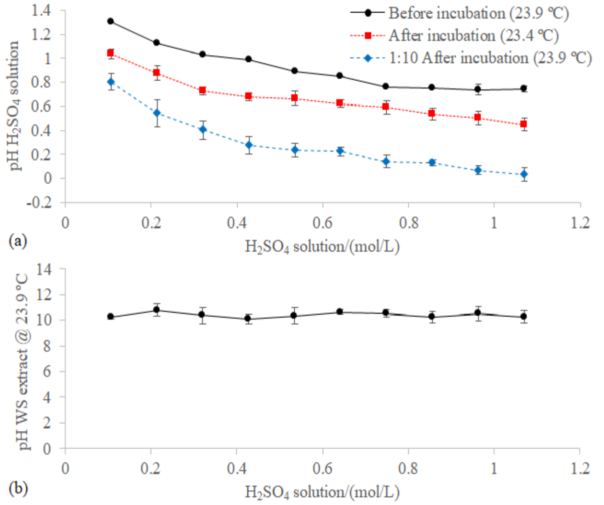

**Figure 4:** (a) pH of the 5.21 ± 0.10 mL $H_2SO_4$ traps (Figure 3a) measured before the incubation at 23.93 ± 0.18 °C, eleven times diluted (1:10) with milli-Q® after the incubation at 23.90 ± 0.16 °C, and without dilution after the incubation at 23.38 ± 0.36 °C. (b) pH of the 21.92 ± 0.15 mL (Figure 3b) extract of the blend WFA+PVWD measured at 23.91 ± 0.15 ºC.

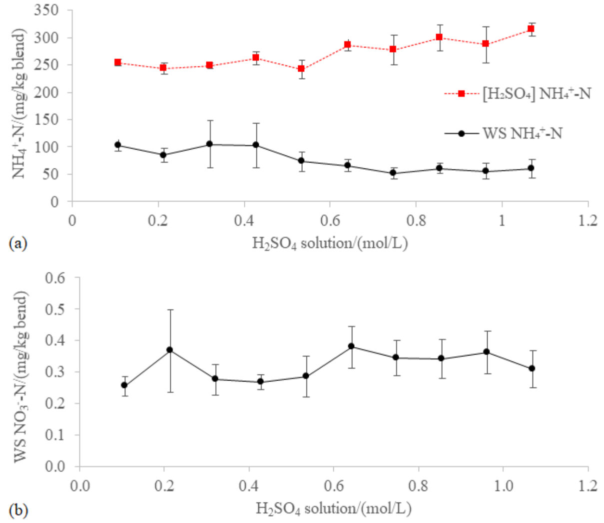

**Figure 5:** (a) $[H_2SO_4]$ $NH_4^+$-N and WS $NH_4^+$-N and (b) WS $NO_3^-$-N in the blend WFA+PVWD after a 60-hour incubation at 100 rpm and 20 °C with different concentrations of $H_2SO_4$ in the trap of the closed chamber for scrubbing the $NH_3$ off the headspace.

A calibration procedure is typically required for measuring the concentration of $NH_3$ in the air using $H_2SO_4$ solutions [18]. Nevertheless, in the present study, the trap effectiveness in all the conditions evaluated was 100% (i.e. all the $NH_3$ released to the headspace was absorbed in the $H_2SO_4$ solutions in the trap) and what limited the depletion of the WS $NH_4^+$-N, was the mass transfer resistance at the film between the fluidized blend and the gaseous phase. This constant value of trap effectiveness was calculated without considering the amount of sulfuric acid used, which ranged from 0.05 ± 0.00 g to 0.54 ± 0.01 g of $H_2SO_4$. Otherwise, there would be a difference of one order of magnitude between the effectiveness of the less concentrated and the most concentrated traps.

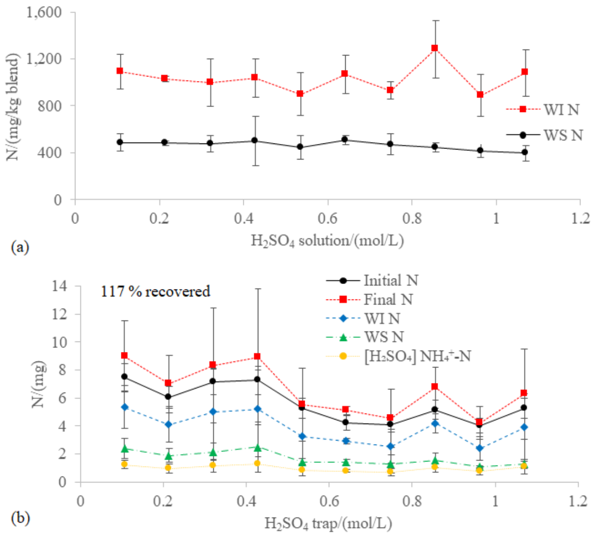

**Figure 6:** (a) WS N and WI N in the blend WFA+PVWD and (b) mass balance of nitrogen after a 60-hour incubation at 100 rpm and 20 °C with different concentrations of $H_2SO_4$ in the trap of the closed chamber for scrubbing the $NH_3$ off the headspace. The overall recovery effectiveness of the nitrogen is shown in the (b) graph.

The concentration of the $H_2SO_4$ trap did not affect the volatilization of $NH_3$ from the blend WFA+PVWD. Similar amounts of $[H_2SO_4]$ $NH_4^+$-N were found in all the conditions evaluated (0.99 ± 0.38 mg; **Figure 6b**). Most of the nitrogen recovered was in the form of WI N and only the 15.52 ± 2.13% of the nitrogen accounted after the incubation was in the form of $[H_2SO_4]$ $NH_4^+$-N. The overall recovery effectiveness of nitrogen was 117% (Figure 6b), as more nitrogen was accounted after the incubation than in the initial characterization. Most of the volatilization of $NH_3$ took place immediately after the blending and the absorption in the $H_2SO_4$ trap continued progressively during the course of the incubation, due to the mass transfer resistance [16]. Thereby, in processes with short contact time between the gas and the $H_2SO_4$ solution, it might be convenient to use bubbling systems to increase surface area between the two phases and thus the rate of transfer of $NH_3$ [19].

As the WFA+PVWD blend had a pH of 10.40 ± 0.46 (Figure 4b), the 90 % of the WS $NH_4^+$-N was in the form of $NH_3$ [20]. The closed chamber continuously shaken at 100 rpm was not enough to enable the equilibrium between the three fractions of the blend WFA+PVWD. This setup was chosen for its low capital and operating cost but the use of an excess of $H_2SO_4$ in the trap could not be justified technically and economically. It might be possible to attain the depletion of the WS $NH_4^+$-N in the blend WFA+PVWD using advanced equipment, which allow to operate at higher temperatures under vacuum conditions (e.g. 65 °C and 25.1 kPa; [7]) or perform hydraulic cavitation [21]. Another processing option would be to reduce the moisture content of the blend, for the production of the granular fertilizer. As the surface area of the dewatered material is greater than the fluidized blend WFA+PVWD, the emissions of ammonia increase [5,22,23].

## Speciation of carbon

Similarly to nitrogen, most of the carbon was in the WI form (**Figure 7a**). Despite the variability of the results of WI C obtained with $H_2SO_4$ traps with concentrations greater than 0.54 M, there was clear difference between the concentration of the WI and the WS species of carbon. The level of WS C (4,022.26 ± 883.39 mg/kg blend; Figure 7a) and WI C (25,350.70 ± 185.20 mg/kg blend; Figure 7a) after the incubation was the same as in the initial characterization (3,763.92 ± 778.70 mg WS C/kg blend and 22,243.23 ± 8,283.15 mg WI C/kg blend; Table 1). Therefore, the $H_2SO_4$ non-intrusive acidification did not promote the adsorption of the WS $C_{org}$ onto the WFA, the release of $CO_2$ or emission of volatile organic molecules. It is important to mention that, Ukwuani & Tao (2016) reported the flux of other compounds different from $NH_3$, such as cyclohexene, towards the $H_2SO_4$ trap. In the present study, the concentration of the $H_2SO_4$ solution in the trap was not responsible of any phenomena which affected the distribution of carbon between the fractions of the blend WFA+PVWD and did not affect the recovery effectiveness of carbon after the incubation. The alkaline pH (Figure 4b) of the blend prevented the losses of carbon and the recovery effectiveness after the incubation was 115% (**Figure 7b**), which could be related to the fact that the blend was a sink of carbon.

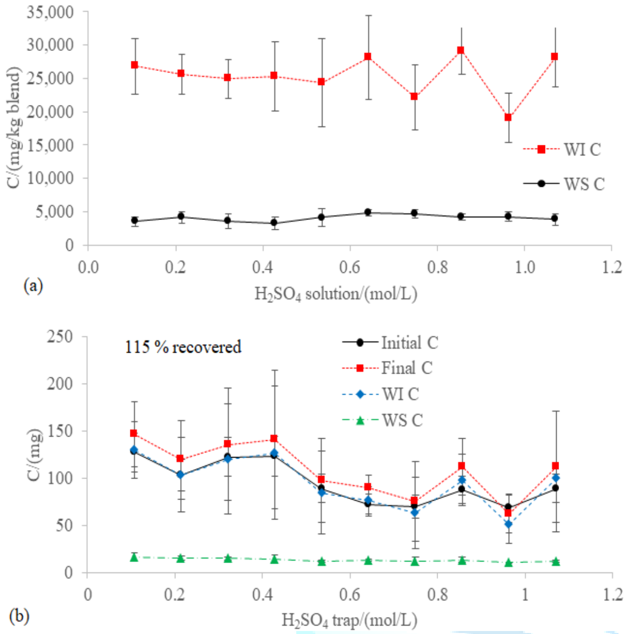

**Figure 7:** (a)WS C and WI C in the blend WFA+PVWD and (b) mass balance of carbon after the 60-hour incubation at 100 rpm and 20 °C with different concentrations of $H_2SO_4$ in the trap of the closed chamber for capturing the $NH_3$ in the headspace. The overall recovery effectiveness of the carbon is shown in the (b) graph.

## Speciation of phosphorus

The concentration of WS $PO_4^{3-}$-P (3.75 ± 1.45 mg/kg; **Figure 8a**) and WI P (792.88 ± 218.92 mg/kg; Figure 8a) after the incubation was the same as before the incubation (5.42 ± 3.84 mg WS $PO_4^{3-}$-P/kg and 636.01 ± 140.53 mg WI P /kg; Table 1). Thus, most of the phosphorus was in the form of WI P, regardless the concentration of the $H_2SO_4$ solution in the trap. Both the fact that there were no losses of phosphorus via gaseous emissions in the studied conditions and the adsorption of the WS $P_{org}$ could explain the 115 % of average recovery effectiveness (**Figure 8b**). Any change in the amount of adsorbed WS $P_{org}$ would be accounted by the colorimetric analytical method (i.e. molybdenum blue reaction) followed. The reason is that the sulfuric-peroxide digestion of the WI fraction led to the formation of WS $PO_4^{3-}$-P, which was measured with the segmented flow analysis. The availability of phosphorus went from 0.0083 ± 0.0021 mg WS $PO_4^{3-}$-P/mg WI P at the beginning of the incubation to 0.0048 ± 0.0015 mg WS $PO_4^{3-}$-P/mg WI P at the end. This tiny difference in the availability could prevent losses via leaching in an open system, for example, when applying phosphorus to land at a rate of 26 kg/ha [10].

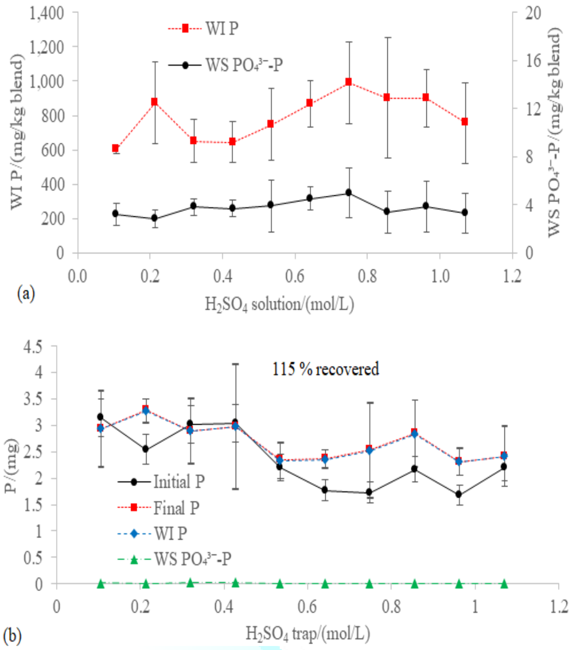

**Figure 8:** (a) WS $PO_4^{3-}$-P and WI P of the blend WFA+PVWD and (b) mass balance of phosphorus after the 60-hour incubation at 100 rpm and 20 °C with different concentrations of $H_2SO_4$ in the trap of the closed chamber for stripping the $NH_3$ in the headspace. The overall recovery effectiveness of the phosphorus is shown in the (b) graph.

The fluctuations of the amount of WI N (Figure 6b), WI C (Figure 7b), and WI P (Figure 8b) were related to the size of the system (Figure 1) and the losses during the incubation and subsequent isolation of this fraction (Figure 3c). The relatively small variations seen in the WS N, $[H_2SO_4]$ $NH_4^+$-N, WS C, and WS $PO_4^{3-}$-P could be explained by the fact that less amount of nitrogen, carbon, and orthophosphate ended up in the WS extract.

## Conclusions

Exceeding the $H_2SO_4$ non-intrusive acidification beyond the stoichiometric limit was not an efficient way of stripping the WS $NH_4^+$-N off the blend WFA+PVWD. The depletion of the $NH_3$ in the headspace of the closed chamber and the turbulence created with the 100 rpm rotary mixing did not sort out the bottleneck of $NH_3$ transfer from the WFA+PVWD blend to the gas phase. Advanced processing conditions (e.g. vacuum thermal stripping and hydraulic cavitation) or dewatering of the blend are required to increase the rate of $NH_3$ volatilization. Although a 10 times increase of the concentration of the solution in the trap resulted in 23.69 ± 5.72 % more $[H_2SO_4]$ $NH_4^+$-N, this only represented an increase of 3.57 ± 2.05 % in the overall fate of nitrogen in this fraction. The C/N/P:38.74 ± 17.56/2.38 ± 0.69/1 found after the incubation was the same as the intended C/N/P:40.55 ± 15.72/2.38 ± 0.80/1 (Table 1), as there was no losses during the treatment. The greatest share of these elements was found in the WI fraction and the WS $P_{org}$ was more susceptible to be adsorbed under the studied conditions than the WS $N_{org}$ and WS $C_{org}$. This controlled-release fertilizer should minimize the greenhouse gas emissions, eutrophication of underground waters upon land application, and pollution swapping. Activating the WFA as adsorbent, for example via calcination at temperatures greater than 500 °C, could improve the valorization of PVWD by enhancing the retention of nutrients in the WI fraction of the blend.

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