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1 **New insights into biochar ammoniacal nitrogen adsorption and its correlation to aerobic**  
2 **degradation ammonia emissions**

3 Matheus Vieira Firmino<sup>a\*</sup>, Anne Trémier<sup>a</sup>, Annabelle Couvert<sup>b</sup>, Anthony Szymczyk<sup>c</sup>

4 <sup>a</sup> INRAE, OPAALE, F-35044, Rennes, France. Email addresses: matheus.vieira-firmino-  
5 silva@inrae.fr and anne.tremier@inrae.fr

6 <sup>b</sup> Univ Rennes, Ecole Nationale Supérieure de Chimie de Rennes, CNRS, ISCR - UMR 6226, F-  
7 35000 Rennes, France. Email address: annabelle.couvert@ensc-rennes.fr

8 <sup>c</sup> Univ Rennes, CNRS, ISCR (Institut des Sciences Chimiques de Rennes) – UMR 6226, Rennes,  
9 France. Email address: anthony.szymczyk@univ-rennes1.fr

10 \*Corresponding author at: INRAE, OPAALE, F-35044, Rennes, France. E-mail address:  
11 matheus.vieira-firmino-silva@inrae.fr

12 **Abstract**

13 One of the technical barriers to the wider use of biochar in the composting practices is the  
14 lack of accurate quantification linking biochar properties to application outcomes. To address  
15 this issue, this paper investigates the use of ammonia nitrogen adsorption capacity by biochar  
16 as a predictor of ammonia emission during composting in the presence of biochar. With this  
17 in mind, this work investigated the use of ammonia nitrogen adsorption capacity of biochar  
18 when mixed with solid digestate, and the reduction in ammonia emissions resulting from the  
19 addition of biochar during aerobic degradation of solid digestate. A biochar synthesized at  
20 900°C, another synthesized at 450°C, and two derivatives of the latter biochar, one chemically  
21 modified with nitric acid and the other with potassium hydroxide, were tested. This study  
22 concluded that the chemical characteristics of the biochar, including pH and oxygen/carbon

23 atomic ratio, had a greater influence on the adsorption of ammonia nitrogen than physical  
24 attributes such as specific surface area. In this regard, nitric acid modification had superior  
25 performance compared to hydroxide potassium modification to increase biochar chemical  
26 attributes and reduce ammonia emissions when applied to aerobic degradation. Finally, a  
27 significant linear correlation ( $p$ -value  $< 0.05$ ,  $r^2 = 0.79$ ) was found between biochar ammonia  
28 nitrogen adsorption capacity and ammonia emissions along composting, showing the  
29 potential of this variable as a predictive parameter. This study provides insights for future  
30 explorations aiming to develop predictive tests for biochar performance.

### 31 **Abbreviations**

32 ANOVA = analysis of variance

33 AW = solid digestat issued from mix of agricultural and agro-industrial wastes

34 BC = biochar

35 BC\_C = biochar distributed by a commercial company

36 BC\_C\_HNO<sub>3</sub> = BC\_C modified with HNO<sub>3</sub>

37 BC450 = biochar produced at 400-450°C

38 BC450\_HNO<sub>3</sub> = BC450 modified with HNO<sub>3</sub>

39 BC450\_KOH = BC450 modified with KOH

40 BC900 = biochar produced at 700-900°C

41 CV = coefficient of variation

42 DM = dry matter

43 FTIR = Fourier transform infrared spectroscopy

- 44 FW = fresh weight
- 45  $\text{HNO}_3$  = nitric acid
- 46 KCl = potassium chloride
- 47 KOH = potassium hydroxide
- 48 n = number of observation
- 49  $\text{N}_2$  = atmospheric nitrogen
- 50  $\text{N}_2\text{O}$  = nitrous oxide
- 51 NA = not available
- 52 ND = not detected
- 53  $\text{NH}_3$  = ammoniac
- 54  $\text{NH}_{3e}$  =  $\text{NH}_3$  emissions
- 55  $\text{NH}_{3er}$  =  $\text{NH}_3$  emissions reduction compared to control
- 56  $\text{NH}_4^+$  = ammonium
- 57  $\text{N-NH}_4^+$  = ammoniacal nitrogen, defined as the sum of nitrogen in the form of ammonia and  
58 ammonium in a sample.
- 59 OFMSW = solid digestat issued from organic fraction of municipal solid waste
- 60 r = Pearson correlation coefficient
- 61 SD = standard deviation
- 62 TKN = total Kjeldahl nitrogen
- 63 VS = volatile solids

## 64 1 Introduction

65 Composting is a biological aerobic process used for treating a large variety of organic wastes,  
66 from animal manures to industrial residues (Gajalakshmi and Abbasi, 2008). This process  
67 changes the organic waste's properties through a reduction in moisture, pathogens  
68 inactivation, stabilization of organic matter and synthesis of humic substances, leading to a  
69 high-value material for soil application (Haug, 1993). Despite these benefits, composting  
70 generates environmental impacts, notably through nitrogen emissions. Indeed, along the  
71 composting treatment the organic nitrogen can be mineralized to its ammonia ( $\text{NH}_3$ ) form  
72 and then stripped out of the composting medium. These  $\text{NH}_3$  emissions are generated by the  
73 displacement of the equilibrium  $\text{NH}_4^+/\text{NH}_3$  to the gaseous form and favoured by the  
74 composting medium aeration supplied either by forced aeration (generally air blowing) or  
75 turning operations (de Guardia et al., 2010). The inherent composting conditions, such as high  
76 temperature ( $>40^\circ\text{C}$ ), and the organic waste's properties, such as high pH ( $>8$ ), contribute to  
77  $\text{NH}_3$  production (Sanchez-Monedero et al., 2018). Thus,  $\text{NH}_3$  emissions are an inevitable  
78 aerobic degradation characteristic. On the other hand, the nitrification/denitrification  
79 microbiological process generates nitrous oxide ( $\text{N}_2\text{O}$ ) and atmospheric nitrogen ( $\text{N}_2$ ). Along  
80 composting, nitrification occurs through the oxidation of ammonium ( $\text{NH}_4^+$ ) by ammonia-  
81 oxidizing bacteria and archaea, which generates hydroxylamine ( $\text{NH}_2\text{OH}$ ), nitrite ( $\text{NO}_2^-$ ) and  
82 nitrate ( $\text{NO}_3^-$ ) anions (Zeng et al., 2013). These oxidized nitrogen forms can be transported to  
83 micro spots of anoxic environment, favouring the development of anaerobic microorganisms  
84 that use oxidized nitrogen as their final electron acceptor and produce  $\text{N}_2\text{O}$  and  $\text{N}_2$  (Cáceres  
85 et al., 2018). These nitrogen losses occur mainly as gaseous emissions in the form of  $\text{NH}_3$ ,  $\text{N}_2\text{O}$   
86 and  $\text{N}_2$ , contributing to environmental problems such as acid rain (Leip et al., 2015), the  
87 formation of fine particles and global warming (Sekar et al., 2021)

88 Different strategies have been investigated to reduce nitrogen emissions, such as finding  
89 optimal operational conditions, such as aeration, or the use of various bulking agents. In  
90 recent years, supplementing the composting mixture with biochar (BC), i.e. chars intended  
91 for environmental applications, has proven to be a promising strategy to capture nitrogen in  
92 composts. The literature reports nitrogen losses and NH<sub>3</sub> emission reductions of 6-86% and  
93 7-98%, respectively (Sanchez-Monedero et al., 2018).

94 Despite these advantages and more than ten years of research into the addition of biochar  
95 during composting, its use in composting practices has not yet been adopted. An important  
96 obstacle to biochar broader utilization lies in the absence of precise quantification linking  
97 biochar properties to application outcomes (Hu et al., 2021). This deficiency undermines  
98 buyers confidence in the investment made through the purchase of biochar, emphasizing the  
99 need for a clearer understanding of its effects in order to build trust in its application. In  
100 addition, the characteristics of biochar vary considerably (Vieira Firmino, M., Trémier, 2023),  
101 because of the heterogeneity of feedstock (Zhao et al., 2013), as well as pyrolysis operational  
102 parameters such as heating temperature and holding time (Shaaban et al., 2014). The  
103 heterogeneity of the final product can be an obstacle for biochar suppliers who wish to  
104 guarantee uniform and consistent results from biochar application. The development of a  
105 straightforward test that empower biochar users to anticipate its effects before incorporating  
106 it into composting processes might ease its adoption.

107 To develop such a test, it is first necessary to identify the pertinent biochar effect to be  
108 predicted. Within the context of biochar application in composting, a primary focus is on its  
109 ability to mitigate nitrogen losses, given its significant environmental and agronomic  
110 implications. Ammonia emissions is the main pathway of nitrogen losses in composting (Pardo

111 et al., 2015). Consequently, it was judge pertinent to selected  $\text{NH}_3$  emissions as the parameter  
112 to be predicted in the development of the test.

113 To identify viable strategies for developing a prediction test for  $\text{NH}_3$  emissions, the current  
114 mechanistic understanding of biochar's impact on the nitrogen cycle in composting was  
115 accessed. Various authors have attributed the reduction in nitrogen losses with biochar  
116 amendment to its adsorption capacities. In this regard, Joseph et al. (2018) hypothesized that  
117 when a biochar is mixed with organic wastes, it adsorbs moisture with dissolved inorganic  
118 nitrogen, especially  $\text{NH}_4^+$ , before its surface is covered with soluble carbon. Subsequently,  
119 microbiological or chemical reactions could oxidize the trapped  $\text{NH}_4^+$ . In this way, biochar  
120 would reduce the amount of free  $\text{NH}_4^+$ , which could otherwise be volatilized as  $\text{NH}_3$ . In  
121 addition, there are indirect benefits associated with the adsorption of  $\text{NH}_4^+$  by biochar.  
122 Indeed, the high concentration of ammonium in some organic wastes such as digestate (Manu  
123 et al., 2021) can be toxic to nitrifying microorganism (Cáceres et al., 2018). Thus, the  
124 adsorption of  $\text{NH}_4^+$  onto biochar would create a better environment for nitrification (Li et al.,  
125 2023), that also leads to a reduction of  $\text{NH}_4^+$  available for volatilization. As so,  $\text{N-NH}_4^+$   
126 adsorption on biochar has often been suggested to be related to  $\text{NH}_3$  emissions reduction in  
127 aerobic degradation (Agyarko-Mintah et al., 2017; Li et al., 2023; Steiner et al., 2010).

128 Based in these results, it is plausible to hypothesize that a significant statistical correlation  
129 might exist between biochar adsorption of ammoniacal nitrogen in compost and its capacity  
130 to reduce  $\text{NH}_3$  emissions. Consequently, estimating biochar's adsorption of ammoniacal  
131 nitrogen in compost could serve as a reliable predictor of its capability to reduce  $\text{NH}_3$   
132 emissions. However, the capacity of biochar to adsorb ammoniacal nitrogen ( $\text{N-NH}_4^+$ ), i.e. the  
133 sum of nitrogen in the form of ammonia and ammonium in a sample, in a solid environment has

134 rarely been estimated and the statistical correlation between N-NH<sub>4</sub><sup>+</sup> adsorption and the  
135 reduction of ammonia emissions has not been demonstrated. Indeed, the works available in  
136 the literature dealing with the capacity of biochar to adsorb NH<sub>4</sub><sup>+</sup> mainly present adsorption  
137 results in liquid solution using isothermal models (Zhang et al., 2020). To our knowledge, no  
138 work has yet attempted to estimate and optimize the potential of biochar to adsorb N-NH<sub>4</sub><sup>+</sup>  
139 in solid organic waste. Yet, solid organic waste represents a complex environment with  
140 varying moisture content and heterogeneous chemical concentrations, soluble organic  
141 matter and biological activity that could make previous models inaccurate.

142 Moreover, the confirmation of the correlation between N-NH<sub>4</sub><sup>+</sup> adsorption onto biochar and  
143 the reduction of NH<sub>3</sub> emissions during composting with biochar demands testing biochars  
144 with diverse N-NH<sub>4</sub><sup>+</sup> adsorption capacities. In this regard, biochar chemical modifications  
145 could be a helpful tool. Biochar modifications aim to increase properties usually related to  
146 adsorption, such as surface area, ion exchange capacity and surface functional groups.  
147 Literature reviews show that, in liquid solutions, modified biochar can adsorb more N-NH<sub>4</sub><sup>+</sup>  
148 (mean = 22.79 mgN.gBC<sup>-1</sup>, number of observations = 12) than unmodified ones (mean = 11.9  
149 mgN.gBC<sup>-1</sup>, number of observations = 65) (Zhang et al., 2020). It would thus be interesting to  
150 apply this approach to N-NH<sub>4</sub><sup>+</sup> adsorption on biochar in solid organic waste. Closer  
151 investigation of the different modifications available shows that nitric acid (HNO<sub>3</sub>) and  
152 potassium hydroxide (KOH) modifications are interesting alternatives. It was reported that  
153 HNO<sub>3</sub> and KOH modifications increased biochar NH<sub>4</sub><sup>+</sup> adsorption capacity by 36% (Vu et al.,  
154 2017) and 39% (Song et al., 2020), compared to the unmodified biochar, when tested in 40  
155 and 10 mgNH<sub>4</sub><sup>+</sup>.L<sup>-1</sup> solutions, respectively. Vu et al. (2017) suggested that the HNO<sub>3</sub>  
156 modification improves NH<sub>4</sub><sup>+</sup> adsorption by increasing biochar surface chemical groups and  
157 total acidity, while Song et al. (2020) attributed KOH modification efficiency to an increase in



158 biochar surface area and pore structure. These results suggest that physical and chemical  
159 adsorption mechanisms co-exist in biochar, as shown by Song et al. (2020) who studied  
160 biochar  $\text{NH}_4^+$  adsorption in wastewater. Some authors highlighted that chemical  
161 characteristics such as cation exchange capacity and surface acidity (Gai et al., 2014; Yang et  
162 al., 2018) had higher influence over biochar  $\text{NH}_4^+$  adsorption in wastewater than the surface  
163 area (Takaya et al., 2016). Thus, it is essential to understand which biochar's characteristics  
164 are related to its capacity to adsorb  $\text{N-NH}_4^+$  in organic waste in order to identify the main  
165 reactional mechanisms and optimize biochar production for supplementation of organic  
166 waste composting.

167 In this context, the present work had three objectives. Firstly, to investigate whether there is  
168 a statistically significant correlation between the ammoniacal nitrogen of biochar in organic  
169 waste and its ability to reduce  $\text{NH}_3$  emissions. In addition, this work assesses whether the  
170 chemical modifications of biochar KOH and  $\text{HNO}_3$  can improve the potential of biochar to  
171 reduce  $\text{NH}_3$  emissions during aerobic degradation. Finally, this work seeks to identify the main  
172 characteristics of biochar linked to the adsorption of ammoniacal nitrogen in organic waste  
173 and to  $\text{NH}_3$  emissions.

## 174 2 Materials and methods

### 175 2.1 Studied substrates

176 Solid digestates, which are residues of anaerobic digestion, were studied in this work because  
177 one of their characteristics is the high content in  $\text{N-NH}_4^+$ . Two types of digestate were used.  
178 The first one was produced from the anaerobic digestion of a mix of agricultural and agro-  
179 industrial wastes (AW), such as cattle manure (20t) and slurry (20t) and cheese whey (12t).  
180 The complete list of organic wastes used in AW production is available in Supplementary

181 Materials. The second one was collected in an industrial anaerobic digestion plant treating  
182 sorted organic fraction of municipal solid waste (OFMSW). In both cases, the solid digestate  
183 was collected directly after solid/liquid separation by a screw press, and it was stored at -20°C  
184 to preserve its chemical characteristics.

185 Three biochars were considered in this study. Two of these were produced in a rotary kiln 4.2  
186 m long and 0.2 m in diameter using green waste pallets made from beech branches. Pyrolysis  
187 was carried out with a biomass feed rate of 6 kg.h<sup>-1</sup>, a rotation speed of 3 RPM, an inclination  
188 of 3°, an incoming nitrogen flow of 4 NL.min<sup>-1</sup>, and a residence time of 40 minutes. Two  
189 temperatures of pyrolysis were considered: one biochar was produced in a low temperature  
190 range, 400-450°C (BC450), and a second at high temperature range, 700-900°C (BC900). The  
191 kiln was fed with 6kg.h<sup>-1</sup> of biomass and had a residence time of 40 minutes. The third biochar  
192 is a retail biochar (5kg) distributed by a commercial company (BC\_C).

## 193 2.2 Biochar modifications

194 In order to increase the diversity of properties of the studied biochar, the raw biochars  
195 presented above have been subjected to chemical modifications. A preliminary study was  
196 carried out to select the most relevant chemical modifications (data not shown). In this study,  
197 all biochars were subjected to modifications using HNO<sub>3</sub>, KOH, H<sub>2</sub>SO<sub>4</sub> and HCl. The  
198 modifications that induced the most significant changes in the infrared spectra of the biochars  
199 were selected for the present work. In this regard, an acid modification (HNO<sub>3</sub>) was used to  
200 activate BC450 and BC\_C, generating BC450\_HNO<sub>3</sub> and BC\_C\_HNO<sub>3</sub>. Moreover, BC450 was  
201 also activated using a basic attack (KOH), generating a third modified biochar (BC450\_KOH).  
202 These modifications were based on Song et al. (2020) and Shang et al. (2018) works. Previous

203 experiments (data not presented) showed that BC900 chemical and physical properties  
204 cannot be altered by any of the tested modifications.

205 The acid and basic activations consisted in soaking the biochars, at room temperature and  
206 under constant agitation, into a 6 or 3 mol.l<sup>-1</sup> HNO<sub>3</sub> or KOH solution for 16 or 24h at 1:10 or  
207 1:20 ratio (mass of biochar/volume of reactant solution) respectively. Afterward, the soaked  
208 biochar was washed until the washing solution achieve pH close to neutral. Biochars were  
209 then dried at 105°C for 24h and stored in closed opaque recipients and at ambient  
210 temperature.

### 211 2.3 Measurement of biochar capacity to adsorb NH<sub>4</sub><sup>+</sup>

212 Biochar was mixed with AW or OFMSW at 10% biochar to digestate ratio (fresh weight) in a  
213 250ml beaker for 48h at room temperature. The beakers were sealed with Teflon to avoid  
214 NH<sub>3</sub> volatilization and drying. The contact period of 48h was chosen based on the work  
215 reported by Prost et al. (2013). The authors showed that pores of biochar mixed with  
216 farmyard manure clogged with dissolved organic matter after two days, preventing major  
217 adsorption after this date.

218 After 48h, the biochar granules were separated from the digestate using tweezers and the  
219 solid digestate fibers and debris attached to the biochar particles were manually removed.  
220 The adsorbed NH<sub>4</sub><sup>+</sup> was then extracted based on the norm NF ISO 14.256-2. The extractions  
221 consisted in soaking the biochar granules in 1M potassium chloride (KCl) solution for 1h under  
222 constant agitation at ambient temperature. Afterwards, the soaked biochar was separated by  
223 centrifugation at 12000 rpm. The extracted solution was filtered through a 0.45µm  
224 polypropylene filter with glass fiber. The NH<sub>4</sub><sup>+</sup> content of the solution was then measured  
225 by ionic chromatography using Metrohm 850 Professional IC.

226 This procedure was applied to BC450, BC450\_KOH, BC450\_HNO<sub>3</sub> and BC900 in quadruplicate,  
227 but not to BC\_C and BC\_C\_HNO<sub>3</sub>. Indeed, the granulometry (powder) of these latter biochars  
228 prevented their use in this test.

#### 229 2.4 Aerobic degradation test

230 The influence of biochar properties over NH<sub>3</sub> emission reduction during aerobic degradation  
231 was tested using mixtures of digestate AW and biochar with a biochar to digestate ratio of 5%  
232 (wet weight).

233 To perform aerobic degradation, a pilot composed of six 10 L stainless steel reactors was used.  
234 The reactors are operated with controlled temperature (40°C) and aeration (70 L.h<sup>-1</sup>). Part of  
235 the reactor outlet gas recirculates in the system to allow the homogenization of the air within  
236 the reacting cell. The recirculation airflow was set to approximately 5 L.min<sup>-1</sup>. The composition  
237 of the inlet and outlet gas was analysed using an inline analyser ABB EL3020 that measures  
238 the concentration of carbon dioxide (CO<sub>2</sub>), oxygen (O<sub>2</sub>), methane (CH<sub>4</sub>) and nitrous oxide  
239 (N<sub>2</sub>O). In addition, the NH<sub>3</sub> emitted by the cells was quantified in a parallel circuit containing  
240 two ammonia traps with 200 mL of sulphuric acid 1M. The ammonia traps were analysed by  
241 distillation in a Gerhardt vapodest 50. The aerobic degradation device and operational  
242 conditions were similar to those of Berthe et al. (2007) and Zeng et al. (2013). The complete  
243 scheme of the aerobic degradation pilot can be visualized in Supplementary Materials.

244 Each experiments lasted two weeks.

#### 245 2.5 Digestates characterization

246 Analyses of the digestates included the determination of total Kjeldahl nitrogen (TKN), total  
247 N-NH<sub>4</sub><sup>+</sup>, easily extractable N-NH<sub>4</sub><sup>+</sup>, dry matter (DM), volatile solids (VS) and pH. The dry matter  
248 (DM) was obtained by drying the sample at 105°C for 48h, and the volatile solids (VS)

249 concentration by loss-on-ignition at 550°C for 5h. The total Kjeldahl nitrogen (TKN) was  
250 determined by distillation in a Gerhardt Vapodest 50 after sample mineralization with  
251 sulphuric acid 98% (v:v) and copper tablets in a Mineralization Bloc Kjeldatherm KT followed,  
252 according to the norm NF EN 13342.

253 The total ammoniacal nitrogen content was determined in Büchi distillation Unit K-350. To  
254 perform Büchi distillation, samples were basified with magnesium hydroxide right before  
255 being inserted into the device. The distillation was performed with water vapour for  
256 approximately 3 minutes. Easily extractable ammoniacal nitrogen was calculated through 1M  
257 KCl extraction at 1:10 (w:w, wet weight) ratio during 1h. The extracted solution was filtered  
258 through a 0.45 µm polypropylene filter with glass fiber and analysed using Metrohm 850  
259 Professional ionic chromatography. It is expected that the harsher extraction conditions used  
260 in Büchi distillation compared to KCl extractions, might estimate the total N-NH<sub>4</sub><sup>+</sup>. At the same  
261 time, it is expected that KCl extractions would estimate the more labile fraction of total N-  
262 NH<sub>4</sub><sup>+</sup>.

263 The pH was measured by mixing the digestate with deionised water in a ratio of 1:5 (w:w, wet  
264 weight) and stirring for 1 hour, in accordance with standard NF EN 13037. Afterwards, the pH  
265 was measured using Mettler Toledo Five Easy. All previous mentioned analyses were  
266 performed in triplicate (if not attested otherwise), except for ammonia traps which were  
267 analysed in duplicate. The ammonia traps samples were homogeneous, and the individual  
268 analyses differed less than 0.5% from the mean.

## 269 2.6 Biochar characterization

270 Biochars were characterized for TKN, DM, VS and pH using the same methodology applied to  
271 digestates. In addition, biochar's elemental composition (carbon, nitrogen, sulfur and

272 oxygen), surface area, Fourier transform infrared spectroscopy (FTIR) and zeta potential were  
273 analysed. The elemental analysis was performed using an elemental analyser flash 2000.  
274 Additionally, the total oxygen and total carbon results from the elemental analysis were  
275 converted to number of atoms and the atomic ratio TO/TC was calculated. The specific surface  
276 area was determined using a Micromeritics 3Flex analyser with nitrogen as the adsorbent gas  
277 and applying the model Brunauer-Emmett-Teller (BET).. The FTIR analysis was done in the  
278 wavelength's region from 4000 to 400 cm<sup>-1</sup> with a spectral resolution of 4 cm<sup>-1</sup> using a Nicolet  
279 iS5 with ATR iD7 module.

280 Biochar zeta potential was inferred from streaming potential measurement, which is a  
281 versatile technique for characterizing the electrical surface properties of materials of different  
282 geometries such as polymer films (Szymczyk et al., 2007), porous materials (Fievet et al., 2004)  
283 or particles (Szymczyk et al., 2002). Experiments were carried out with a SurPASS  
284 electrokinetic analyser (Anton Paar GmbH). A porous plug was formed with biochar particles  
285 of millimetric dimensions. It was further surrounded by two Ag/AgCl flow electrodes allowing  
286 circulation of the measuring solution (0.001 M KCl) between the particles and measurement  
287 of the streaming potential generated by the application of pressure ramps (0-300 mbar)  
288 through the porous plug. All experiments were performed at T = 20 ± 2 °C and duplicated. The  
289 pH of the measuring solution was adjusted with 0.05 M HCl and KOH solutions. The zeta  
290 potential ( $\zeta$ ) of biochars was determined from experimental streaming potential ( $\Delta V_s$ ) values  
291 from the well-known Helmholtz-Smoluchowski expressed as:

292 
$$\zeta = \frac{\Delta V_s \eta \lambda_0}{\varepsilon_0 \varepsilon_r \Delta P} \quad (1)$$

293 Where  $\eta$  is the viscosity of the measuring solution,  $\lambda_0$  its electric conductivity,  $\varepsilon_r$  its dielectric  
294 constant,  $\varepsilon_0$  is the vacuum dielectric permittivity and  $\Delta P$  is the hydrostatic pressure difference  
295 applied through the porous plug formed by the biochar particles.

296 Some biochars were found to release colloidal particles (generated during the pyrolysis  
297 process or post modifications (Yang et al., 2020) into the measuring solution during streaming  
298 potential experiments. The zeta potential of these particles was determined from their  
299 electrophoretic mobility measured by electrophoretic light scattering (Wallis zeta potential  
300 analyzer, Cordouan Technologies), considering the Smoluchowski equation for  
301 electrophoretic mobility ( $u_E$ ):

$$302 \quad \zeta = \frac{u_E \eta}{\varepsilon_0 \varepsilon_r} \quad (2)$$

303 Where  $\eta$  is the viscosity of the measuring solution,  $\varepsilon_r$  its dielectric constant,  $\varepsilon_0$  is the vacuum  
304 dielectric permittivity.

## 305 2.7 Data analysis

306 At the end of the adsorption tests, the influence of digestate type (AW or OFMSW), biochar  
307 type (BC450, BC450\_HNO<sub>3</sub>, BC450\_KOH, BC900) over biochar N-NH<sub>4</sub><sup>+</sup> adsorption was studied  
308 using one-way and two-way ANOVA. The differences in biochar N-NH<sub>4</sub><sup>+</sup> adsorption capacity  
309 obtained in the adsorption tests and the differences in aerobic degradation NH<sub>3</sub> emissions  
310 were investigated using two tailed welch t-test.

311 To compare nitrogen losses from different reactors, the NH<sub>3</sub> emissions (NH<sub>3e</sub>) and NH<sub>3</sub>  
312 emissions reductions (NH<sub>3er</sub>) were defined according to:

313

314 
$$NH_{3e} = \frac{NH_{3\text{ stripped}} \cdot 100}{TKN_i} \quad (3a)$$

315 
$$NH_{3\text{ er}} = \frac{(NH_{3c} - NH_{3bc}) \cdot 100}{NH_{3c}} \quad (3b)$$

316 Where  $TKN_i$  is the Total TKN initially present in the cell,  $NH_3$  stripped is the  $NH_3$  captured in  
317 acid traps,  $NH_{3c}$  is the  $NH_3$  emissions in control (%),  $NH_{3bc}$  is the  $NH_3$  emissions in biochar  
318 treatment (%),  $NH_{3e}$  is the  $NH_3$  emissions and  $NH_{3\text{er}}$  is the  $NH_3$  emissions reduction.

319 Furthermore, the influence of biochar  $N-NH_4^+$  adsorption (e.g. estimated by the adsorption  
320 tests) over aerobic degradation  $NH_3$  emissions (e.g. estimated through aerobic degradation  
321 tests) was estimated using a Pearson correlation and linear regression. Additionally, the  
322 investigation of biochar characteristics related to biochar  $NH_4^+$  adsorption and aerobic  
323 degradation  $NH_{3\text{er}}$  was done using a Pearson Correlation Matrix.

324 The confidence level of all statistical analysis was defined at 90%. All statistical analyses were  
325 done using the software R Studio version 3.5.1.

## 326 3 Results and Discussion

### 327 3.1 Substrates characteristics

328 The substrate characteristics are presented in Table 1.

329 **Table 1.** Physicochemical characterization of substrates

330

331 Analysis with less than three repetitions had all their values showed in the table.

332 Studied digestates differed in their nitrogen composition. Digestate AW had higher TKN and  
333 total  $N-NH_4^+$  than OFMSW. Results show that AW TKN is composed of a higher proportion of  
334  $N-NH_4^+$  (45.6%) compared to OFMSW (37.5%). In addition, Table 1 shows that the values of



335 total N-NH<sub>4</sub><sup>+</sup> and easily extractable N-NH<sub>4</sub><sup>+</sup>. AW had higher total N-NH<sub>4</sub><sup>+</sup> but lower easily  
336 extractable N-NH<sub>4</sub><sup>+</sup> compared to OFMSW. Thus, it is likely that AW N-NH<sub>4</sub><sup>+</sup> is more linked to  
337 the organic matter structure than OFMSW N-NH<sub>4</sub><sup>+</sup>.

338 The analysis of biochar characteristics, presented in table 1, shows that the higher  
339 temperature used for the production of the BC900 contributes to degrade more OM during  
340 pyrolysis, consequently creating a larger amount of ashes, a lower percentage of oxygen, a  
341 higher percentage of carbon and a higher specific surface area compared to BC450. Similar  
342 results were reported in the literature (Shaaban et al., 2014; Tag et al., 2016). BC\_C had an  
343 elemental composition (TC, TH, TN and TO) and pH similar to BC900 (Table 1), suggesting that  
344 similar pyrolysis conditions might have been employed in its production.

345 The chemical reactions applied to biochars effectively changed BC450 and BC\_C chemical  
346 characteristics. Chemical modifications (HNO<sub>3</sub> and KOH) reduced the ash contents compared  
347 to the original biochars, generating an increase in the proportion of VM (Table 1). The  
348 reduction in ash content might have contributed to decrease biochar pH. Moreover, Table 1  
349 shows that KOH modification had small impact over BC450 elemental analysis and surface  
350 area. The KOH modification applied in this study differed from the one presented in the  
351 literature by avoiding catalysis step usually performed at high temperature (~500°C) and in  
352 an oxygen-limited environment (Ma et al., 2022; Song et al., 2020). The lack of a catalysis step  
353 in this study might have limited the effect of KOH modification.

354 The pH analysis of biochars (Table 1) shows that BC900 and BC\_C had the highest pH with  
355 values close to 9. The lack of acid groups and the ashes content might have contributed to  
356 this result. Additionally, BC450\_KOH reduced BC450 pH from 6.7 to 5.5. This result could be  
357 due to a reduction in ash content or functionalization of biochar surface. In addition, the HNO<sub>3</sub>

358 modifications reduced the pH of the biochar to 2.5 and 3.0 for BC450\_HNO<sub>3</sub> and BC\_C\_HNO<sub>3</sub>  
359 respectively. Yakout (2015) reported similar results concerning HNO<sub>3</sub> modifications. The  
360 authors modified rice straw with 65% nitric acid at 60°C. The modification reduced biochar  
361 pH to values ranging from 3.4 to 3.2, while the non-modified biochar had pH close to 9. The  
362 pH is affected by the deprotonation of acid and basic groups on the biochar surface, in liquid.  
363 Thus, this analysis suggests that studied biochars differed by their surface functions.

364 The elemental analysis supports this idea. Table 1 shows that HNO<sub>3</sub> modification increased  
365 TO and TN and reduced TC and TH percentages when applied to BC450 or BC\_C. These  
366 changes increased the atomic ratio TO/TC by 41% and 111% for BC450 and BC\_C, respectively,  
367 suggesting that HNO<sub>3</sub> modification increased biochar oxidation (Wang et al., 2019). Similar  
368 results were reported by Shang et al. (2018). The authors modified spruce sawdust biochar  
369 produced at 400°C using 6M HNO<sub>3</sub> chemical modification. Their elemental analysis showed  
370 that HNO<sub>3</sub> modified biochar increased TO and TN in 7.4 and 3.3% compared to the original  
371 biochar. In addition, it has been shown that HNO<sub>3</sub> can functionalize the biochar surface  
372 (Mehmood et al., 2021; Shang et al., 2018), resulting in increased oxygen and nitrogen  
373 contents.

374 A more detailed analysis of biochar functionalization can be achieved through FTIR (Fig. 1).

375

376 **Fig. 1.** Fourier transform infrared spectra from (A) BC\_C, BC\_C\_HNO<sub>3</sub> and BC900. (B) BC450, BC450\_HNO<sub>3</sub> and BC450\_KOH.

377 Fig. 1A firstly shows that BC900 and BC\_C had similar FTIR spectra marked by the lack of  
378 detectable peaks. The similarity between both spectra reinforces the idea that these biochars  
379 had similar production conditions. Moreover, it can be assumed that BC900 high pyrolysis

380 temperature degraded the majority of surface chemical functions. This hypothesis could be  
381 verified by ATG-DSC analysis in future studies.

382 Contrastingly, FTIR spectrum obtained with BC450 (Fig. 1B) showed three identifiable peaks  
383 in the functional group region. The first peak at 3000-2800  $\text{cm}^{-1}$  is attributed to the  
384 asymmetric and symmetric C-H stretching vibrations of the methyl ( $-\text{CH}_3-$ ) and methylene  
385 ( $-\text{CH}_2-$ ) (Vu et al., 2018). The second peak close to 1700  $\text{cm}^{-1}$  is attributed to carboxylic and  
386 lactonic groups ( $\text{C}=\text{O}$ ) (Tran et al., 2018; Vu et al., 2018). Lastly, the peak close to 1650  $\text{cm}^{-1}$   
387 is attributed to ( $\text{C}=\text{C}$ ) double bonds in aromatic rings (Tran et al., 2018; Vu et al., 2018).

388 Additionally, BC450\_KOH FTIR spectrum was not notably different from the spectrum  
389 obtained with BC450 concerning the previous described peaks. On the other hand,  
390 BC450\_HNO<sub>3</sub> reduced the methyl ( $-\text{CH}_3-$ ) and methylene ( $-\text{CH}_2-$ ) peak and increased  
391 carboxylic and lactonic peak, compared to BC450. Furthermore, the HNO<sub>3</sub> modification  
392 applied to BC\_C generated a detectable but minor change on the carboxylic and lactonic peak.  
393 Vu et al. (2018) reported similar results. The authors modified corncob biochar with 4M HNO<sub>3</sub>  
394 and observed the appearance of a FTIR peak at 1705  $\text{cm}^{-1}$ , which was attributed to carboxylic  
395 acid. The FTIR reinforces the conclusion of the elemental and pH analyses, suggesting that  
396 modification with HNO<sub>3</sub> increases the acid groups on the biochar surface.

397 Since these acid groups can deprotonate, their presence can also be revealed by determining  
398 the zeta potential of biochars. Fig. 2 indeed shows that biochars are negatively charged over  
399 the whole pH range considered in the streaming potential experiments.

400

401 **Fig. 2.** Zeta potential of BC450, BC450\_KOH, BC450\_HNO<sub>3</sub> and BC900 inferred from streaming potential measurements vs.  
402 pH.

403 By definition, the isoelectric point is the pH at which a particle carries no net electrical charge,  
404 and consequently, its zeta potential is zero (Sujith et al., 2022). Moreover, a decrease in pH  
405 favours the protonation of acid groups, reducing the particle's electronegativity and  
406 increasing the zeta potential. Considering that no biochar has reached its isoelectric point at  
407 pH 3, it can be concluded that a lower pH would be required to achieve this. This result is in  
408 good agreement with data reported in the recent review article published by Yang et al.  
409 (2020).

410 According to Fig. 2, the BC900 was less charged than BC450 and BC450\_KOH, whatever the  
411 pH of the measuring solution. This result is due to the higher pyrolysis temperature used to  
412 produce BC900, which reduced the density of surface acid groups (Shaaban et al., 2014; Yang  
413 et al., 2020). Thus, zeta potential values corroborate FTIR results. It can be noted, however,  
414 that zeta potential analysis indicates that the surface of BC450\_KOH was more negatively  
415 charged than BC450 while FTIR did not show major differences between these two biochars.

416 Surprisingly, it was found that the zeta potential of BC450\_HNO<sub>3</sub> decreased (in absolute value)  
417 with increasing pH (except for pH below 3.2). Although this behaviour can be explained in  
418 some cases by an increase in the electrical conductivity of the solution, this is only expected  
419 at very high pH values (Mouhoumed et al., 2014), which was not the case in the present work.  
420 This behaviour has already been reported in the literature and attributed to swelling effects  
421 with pH (Luxbacher and GmbH, 2014), which conceptually would be equivalent to a shift of  
422 the hydrodynamic slip plane with pH (Uetani and Yano, 2012). In this case, the concept of zeta  
423 potential loses its meaning. Indeed, during the electrokinetic titrations BC450\_HNO<sub>3</sub> particles  
424 tended to partially disaggregate and colloidal particles were released into the measuring  
425 solution during streaming potential measurements. The zeta potential of a sample of the

426 solution containing colloidal particles from BC450\_HNO<sub>3</sub> was determined by electrophoretic  
427 light scattering. A value of  $-35.4 \pm 1.6$  mV was obtained at pH 3.5, which was higher (in  
428 absolute value) than the one measured on all biochars. Colloidal biochar particles are  
429 expected to bear the same chemical groups as bulk biochar (Yang et al., 2020) as confirmed  
430 by measurements made on colloidal particles released by BC450. Indeed, electrophoretic light  
431 scattering measurements led to a zeta potential value of  $-19.3 \pm 3.1$  mV at pH 3.5, which is in  
432 good agreement with the streaming potential results reported in Fig. 2 (it is worth mentioning  
433 that no colloidal particles were detected in the solutions used to characterize BC450\_KOH  
434 and BC900). Considering the colloidal biochar zeta potential as an estimation of the bulk  
435 biochar zeta potential, it is possible to affirm that these additional electrophoretic light  
436 scattering results suggest that BC450\_HNO<sub>3</sub> was more (negatively) charged than the other  
437 three biochars and that HNO<sub>3</sub> modification increased the amount of acid groups in BC450.  
438 Similar results were reported by (Wang et al., 2019). The authors modified pinewood biochar  
439 produced at 300 and 600°C with HNO<sub>3</sub> and pulverized the chars to study its colloidal  
440 properties. It was reported that HNO<sub>3</sub> reduced zeta potential in approximately 6 mV. This  
441 result was attributed to an increase in negatively charged oxygen-containing functional  
442 groups.

443 Based on the previous analysis, it is possible to affirm that the initial biochars chosen for the  
444 present study and the ones obtained after chemical modifications have high variability in their  
445 chemical characteristics. They are thus expected to generate different N-NH<sub>4</sub><sup>+</sup> adsorption and  
446 NH<sub>3</sub> loss quantities during their utilization with solid digestate.

### 447 3.2 Ammoniacal nitrogen adsorption capacity of biochar in a solid digestate environment

448 The results from biochar adsorption capacity when mixed to solid digestate are presented on  
449 Fig. 3.

450

451 **Fig. 3.** Ammoniacal nitrogen adsorbed in different biochars when applied to digestate made from agricultural wastes (AW)  
452 or digestate made from organic fraction of municipal solid waste (OFMSW).

453 A two-way ANOVA was conducted to examine the influence of biochar and digestate type on  
454 N-NH<sub>4</sub><sup>+</sup> adsorption. A statistically significant interaction was found between biochar type and  
455 digestate ( $F(3,24) = 5.46$ ,  $p\text{-value} = 0.005$ ,  $\eta^2_g = 0.41$ ), indicating that the outcome of biochar  
456 N-NH<sub>4</sub><sup>+</sup> adsorption capacity depends not only on biochar properties, but also on digestate  
457 characteristics. Therefore, the analysis of the simple main effects (biochar and digestate type)  
458 was studied individually.

459 A one-way ANOVA was conducted to study how digestate type influenced each biochar  
460 adsorption capacity individually. This analysis shows that there is a significant difference ( $p$ -  
461 value < 0.1) between the amounts of N-NH<sub>4</sub><sup>+</sup> adsorbed by the biochars depending on whether  
462 they are applied to AW or OFMSW. A pairwise t-test analysis shows that all biochars had  
463 significant difference ( $p$ -value < 0.1) in their mean N-NH<sub>4</sub><sup>+</sup> adsorption whether applied to AW  
464 or OFMSW. On this subject, BC450\_KOH, BC450\_HNO<sub>3</sub>, BC450 and BC900 adsorbed 41, 34  
465 and 21 more N-NH<sub>4</sub><sup>+</sup> when applied to OFMSW compared to AW, respectively.

466 The higher biochar N-NH<sub>4</sub><sup>+</sup> adsorption when applied to OFMSW might be explained by the  
467 digestates chemical characteristics. Table 1 shows that OFMSW had higher easily extractable  
468 N-NH<sub>4</sub><sup>+</sup> (mean = 6.02 gN-NH<sub>4</sub><sup>+</sup>.kgDM<sup>-1</sup>) compared to AW (mean = 4.62 gN-NH<sub>4</sub><sup>+</sup>.kgDM<sup>-1</sup>)  
469 whereas an opposite tendency is observed for the total N-NH<sub>4</sub><sup>+</sup> (e.g. 9.16 gN-NH<sub>4</sub><sup>+</sup>.kgDM<sup>-1</sup> and

470 11.45 gN-NH<sub>4</sub><sup>+</sup>.kgDM<sup>-1</sup>, respectively). These results show that the easily extractable N-NH<sub>4</sub><sup>+</sup> is  
471 a good estimator of the organic waste N-NH<sub>4</sub><sup>+</sup> available to be adsorbed by biochar. Thus, the  
472 differences in N-NH<sub>4</sub><sup>+</sup> mobility in the organic waste structure may play an important role in  
473 biochar adsorption performance. It is likely that water mobility analysis with a method such  
474 as fast field cycling nuclear magnetic resonance relaxometry, might help to better understand  
475 the factors affecting biochar N-NH<sub>4</sub><sup>+</sup> adsorption.

476 The differences between biochars capacities to adsorb NH<sub>4</sub><sup>+</sup> were studied for each digestate  
477 individually (AW or OFMSW) using a one-way ANOVA. The results show that biochar type had  
478 a significant effect over N-NH<sub>4</sub><sup>+</sup> adsorption whether applied to AW (F(3, 12) = 313, p-value =  
479 1.17e<sup>-11</sup>, η<sup>2</sup><sub>g</sub> = 0.99) or OFMSW (F(3, 12) = 96, p-value = 1.15e<sup>-8</sup>, η<sup>2</sup><sub>g</sub> = 0.96). A more in-depth  
480 analysis was done by calculating all pairwise comparisons between the different biochars  
481 (BC450, BC450\_HNO<sub>3</sub>, BC450\_KOH and BC900) using a hypothesis test (T-Test). The results  
482 showed that the N-NH<sub>4</sub><sup>+</sup> adsorption was significantly different (p-value adjusted < 0.1) for all  
483 biochars (Fig. 3).

484 Biochar BC900 adsorbed the lowest amount of N-NH<sub>4</sub><sup>+</sup>, with mean adsorption of 0.32 and 0.41  
485 mgN-NH<sub>4</sub><sup>+</sup>.gBC<sup>-1</sup> when applied to AW and OFMSW, respectively. The higher BC900 surface  
486 area compared to other biochars suggested a better BC900 performance than the one  
487 observed. Surface area is considered as a critical parameter for adsorption because it relates  
488 to the density of the adsorption site per unity of mass (Zhang et al., 2020). In spite of its higher  
489 surface area, BC900 was not characterized by a higher density of adsorption sites, compared  
490 to other biochars, due to its considerably lower surface acid functions. This fact might justify  
491 its lower N-NH<sub>4</sub><sup>+</sup> performance.

492 Moreover, the hypothesis test results show that chemical activations applied to BC450  
493 significantly increased the quantity of adsorbed N-NH<sub>4</sub><sup>+</sup>. In this regard, BC450\_HNO<sub>3</sub> and  
494 BC450\_KOH respectively increased NH<sub>4</sub><sup>+</sup> adsorption by 514 and 577%, and 99 and 137%  
495 compared to BC450, applied to AW or OFMSW. Thus, it shows that the acid modification had  
496 superior performances to increase biochar N-NH<sub>4</sub><sup>+</sup> adsorption. These results evidence the  
497 importance of biochar chemical characteristics over N-NH<sub>4</sub><sup>+</sup> adsorption.

498 Furthermore, to optimize biochar N-NH<sub>4</sub><sup>+</sup> adsorption capacity, it is first necessary to identify  
499 the physicochemical properties related to this phenomenon. To answer this question, a  
500 Pearson correlation matrix was generated between biochar N-NH<sub>4</sub><sup>+</sup> adsorption capacity and  
501 biochar characteristics (Table 2).

502 **Table 2.** Pearson correlation results between NH<sub>3</sub> emissions, biochar N-NH<sub>4</sub><sup>+</sup> adsorption and biochar characteristics

503

504 BC sorp. = biochar N-NH<sub>4</sub><sup>+</sup> adsorption, n = number of observations, BC = biochar, TC = total carbon, TH = total hydrogen, TN  
505 = total nitrogen, TO = total oxygen, elemental analysis expressed as mass percentage in dry weight, SA = surface area, DM =  
506 dry matter, FM = fresh matter. ‘\*’ represent significant correlations (p-value < 0.1).

507 Table 2 shows significant correlations between biochar N-NH<sub>4</sub><sup>+</sup> adsorption and biochar pH, TN  
508 and the Atomic ratio TO/TC. These correlations suggest that biochar chemical characteristics  
509 plays an important role in N-NH<sub>4</sub><sup>+</sup> adsorption during solid digestate aerobic degradation. A  
510 more in-depth analysis, shows that lower pH biochars tend to have higher N-NH<sub>4</sub><sup>+</sup> adsorption  
511 capacity, probably thanks to its higher amount of negative surface charges generated by  
512 higher proportion of acid surface functions (as discussed in sections 3.1). This result is  
513 supported by the TO/TC atomic ratio, which is consider as an indicator of the density of acid  
514 functions in biochar. Thus, biochars with higher density of oxygen are more likely to adsorb  
515 more N-NH<sub>4</sub><sup>+</sup> during solid digestate aerobic degradation.



516 Interestingly, no significant correlation between biochar surface area and biochar N-NH<sub>4</sub><sup>+</sup>  
517 adsorption was found. This correlation was expected because higher surface area usually  
518 increases materials' adsorption capacity by increasing their density of adsorption sites per  
519 unit of mass (Zhang et al., 2020), which should reduce NH<sub>3</sub> emissions according to TO/TC  
520 correlation results. However, Table 1 shows that the biochar presenting higher surface area  
521 has the lower TO/TC atomic ratio. Previous discussion identified the increase in pyrolysis  
522 temperature as the cause of these changes (section 3.1). Thus, it is suggested that biochar  
523 surface area, and likely other physical characteristics, can have a role in NH<sub>3</sub> loss reduction,  
524 but they are not the primary determinant parameter.

525 Similar results have been reported in studies investigating biochar N-NH<sub>4</sub><sup>+</sup> adsorption in liquid  
526 solutions. Yang et al. (2018) and Takaya et al. (2016) found that biochars with higher surface  
527 areas (280-180 m<sup>2</sup>.g<sup>-1</sup>) did not had better capacities to adsorb N-NH<sub>4</sub><sup>+</sup> in liquid solution than  
528 lower surface biochars (<10m<sup>2</sup>.g<sup>-1</sup>). Furthermore, Yang et al., (2018) results shows that  
529 biochars with higher O/C ratio (i.e. 0.32) had higher NH<sub>4</sub><sup>+</sup> adsorption capacity (i.e. 5.38 mg.g<sup>-1</sup>)  
530 compared with biochars with lower O/C ratios (i.e. 0.08). The authors highlighted the  
531 importance of electrostatic interactions (Yang et al., 2018) and chemical reaction with oxygen  
532 containing functional groups (Takaya et al., 2016).

533 Based on these results, it is possible to hypothesize that N-NH<sub>4</sub><sup>+</sup> adsorption by biochar in solid  
534 organic waste follows similar mechanisms to those observed in liquid solutions. However,  
535 further research is imperative to discern whether other specific mechanisms govern the N-  
536 NH<sub>4</sub><sup>+</sup> adsorption properties of biochar in solid organic waste.

537 3.3 Performance of ammoniacal nitrogen trapping by biochars in an aerobic degradation test

538 Aerobic degradation experiments were done using digestate AW and biochars BC450,  
539 BC450\_KOH, BC450\_HNO<sub>3</sub>, and BC900 which adsorption capacity was previously estimated.  
540 In addition, BC\_C and BC\_C\_HNO<sub>3</sub> were also tested, aiming to confirm the influence of HNO<sub>3</sub>  
541 modification over NH<sub>3</sub> emissions reduction during aerobic degradation.

542 3.3.1 Oxygen consumption and aeration

543 During aerobic degradation, experimental conditions such as aeration can greatly affect  
544 ammonia emissions (de Guardia et al., 2008). High aeration rates can lead to an increase in  
545 NH<sub>3</sub> emissions through stripping. On the other hand, low aeration rates can hinder oxygen  
546 availability in the reactors and slow down microbial metabolism, which can affect in return N-  
547 immobilization and ammonification. To ensure that the differences observed on NH<sub>3</sub>  
548 emissions during the experiments were only related to biochar adsorption, it was firstly  
549 necessary to investigate the impact of the aeration variations (measured through gas  
550 counters) on oxygen consumption, which is closely related to microbial metabolism during  
551 aerobic degradation. The mean aeration and total oxygen consumption are shown in Table 3.

552 **Table 3.** Aeration rate and oxygen consumption

553

554 VM<sub>i</sub> = initial digestate volatile mater.

555 The results presented in Table 3 show that the aeration rate variability within the treatments  
556 was lower than 10.0%. Moreover, the whole treatment dataset presented a mean aeration of  
557 69.2 L.h<sup>-1</sup> with a coefficient of variation of 6.2%. This low variation suggests that this factor  
558 did not had major influence over the results. An in-depth analysis of the results shows that  
559 one repetition from BC450\_HNO<sub>3</sub> presented aeration rate of 58.98 L.h<sup>-1</sup>, which was lower

560 than the dataset mean ( $69.2 \text{ L.h}^{-1}$ ) and lower than the other three repetitions values (e.g.  
561  $72.4$ ,  $73.4$  and  $72.7 \text{ L.h}^{-1}$ ). The anomalous aeration treatment here cited was taken into  
562 account in the following  $\text{NH}_3$  emissions analysis.

563 Furthermore, the total oxygen consumption was analysed. A first observation shows that  
564 variability of results for each treatment condition was smaller than 5%, attesting the good  
565 repeatability of the experiment. In addition, the biochar effect over total oxygen consumption  
566 was investigated using pairwise t-test. No significant difference ( $p\text{-value} > 0.1$ ) was found  
567 between control and biochar treatments. These results show that biochar supplementation  
568 did not influenced the aerobic biodegradability or microbial aerobic activity.

569 The previous analysis shows that operational parameters and composting microbial activity  
570 had small variability between the experiments, which means that differences in  $\text{NH}_{3e}$ ,  
571 discussed in the next session, are likely related to biochar action.

### 572 3.3.2 Impact of biochar adsorption capacities over ammonia emissions during aerobic 573 degradation

574 Fig. 4 presents the results of ammonia emissions depending on the type of added biochar.

575

576 **Fig. 4.** Ammonia emissions expressed as percentage of Total Kjeldahl Nitrogen. Ctrl = control treatment, without biochar (n  
577 = 3), BC450 (n = 3), BC450\_KOH (n = 3), BC450\_HNO<sub>3</sub> (n = 4), BC\_C\_HNO<sub>3</sub> (n = 2), BC900 (n = 2), BC\_C (n = 3), n = number of  
578 observations. Red triangles indicate the treatments means.

579 A pairwise Welch t-test was conducted to study the differences between the experiments.

580 The emissions of  $\text{NH}_3$  measured in experiments with mixtures containing biochars BC450,  
581 BC450\_HNO<sub>3</sub>, BC\_C\_HNO<sub>3</sub> and BC900 were significantly different ( $p\text{-value} < 0.1$ ) from those  
582 measured in the control experiment. The control emitted 21.0% (SD = 1.3) of its initial TKN as

583 NH<sub>3</sub>, while the treatments BC450, BC450\_HNO<sub>3</sub> and BC\_C\_HNO<sub>3</sub> reduced this value from  
584 13.2%, 83.0% and 37.8%, respectively. On the other hand, BC900 increased NH<sub>3</sub> emissions  
585 from 30.2% (SD = 1.9). Additionally, BC450\_HNO<sub>3</sub> experiment with low aeration (previous  
586 identified) did not affect the identification of the previous mentioned significant effect.  
587 Finally, BC450\_KOH and BC\_C experiments did not significantly differ from the control.  
588 Nevertheless Fig. 4. shows that one experiment of the BC450\_KOH triplicate led to extremely  
589 higher NH<sub>3</sub> emissions than the two others. This extreme point was the highest NH<sub>3</sub> emissions  
590 observed during the experiments, achieving 36.7%. For comparison, the other two repetitions  
591 had mean NH<sub>3</sub> emissions of 14.6%, and the mean of the whole experimental dataset was  
592 18.1%. No apparent differences appeared in the experimental conditions between  
593 BC450\_KOH triplicates. However, digestate is a heterogeneous substrate, and the small  
594 reactional volume (10 L) used increases the risks of having the results impacted by this  
595 inherent digestate characteristic. The comparison between control NH<sub>3</sub> emissions and  
596 BC450\_KOH without this outlier point shows a significant difference (p-value = 0.049).

597 Moreover, the comparison between BC450 and its modifications shows that HNO<sub>3</sub> chemical  
598 activation significantly reduced the NH<sub>3</sub> emissions by 80.4%. On the other hand, no significant  
599 difference was found between BC450 and KOH modification, when considering the outlier  
600 point. The efficiency of HNO<sub>3</sub> activation was confirmed by experiments with BC\_C and  
601 BC\_C\_HNO<sub>3</sub>, where the modified biochar reduced NH<sub>3</sub> losses by 92% compared to BC\_C.

602 The utilization of HNO<sub>3</sub> to augment biochar adsorption capacity of N-NH<sub>4</sub><sup>+</sup> has been  
603 technically demonstrated in this study. However, it is important to highlight that a financial  
604 study taking into account cost of production, agronomic benefits and composting market is  
605 necessary before its industrial development can be considered. The analysis of biochar

606 properties revealed significant Pearson correlations ( $p$ -value  $< 0.05$ ) between ammonia  
607 emissions and pH ( $r = -0.91$ ) as well as the atomic ratio O/C ( $r = 0.81$ ). Notably, no significant  
608 correlation was observed between ammonia emissions and biochar-specific surface area.  
609 Interestingly, similar correlations were identified between biochar properties and its N-NH<sub>4</sub><sup>+</sup>  
610 adsorption capacity, supporting the hypothesis that ammoniacal nitrogen adsorption is the  
611 primary factor influencing biochar's ability to mitigate NH<sub>3</sub> emissions. Furthermore, it is  
612 plausible that N-NH<sub>4</sub><sup>+</sup> adsorption by biochar particles contributes to the reduction in the size  
613 of the free ammoniacal nitrogen pool during the composting process. This reduction in NH<sub>4</sub><sup>+</sup>  
614 availability may subsequently limit the potential for NH<sub>4</sub><sup>+</sup> to be volatilized as NH<sub>3</sub>, thereby  
615 contributing to the observed decrease in ammonia emissions. These findings underscore the  
616 intricate relationship between biochar properties, ammoniacal nitrogen adsorption, and their  
617 collective impact on reducing NH<sub>3</sub> emissions during composting.

618 Based on these results, it clearly appears that differences in biochar adsorption capacity led  
619 to a variation in biochar efficiency to reduce NH<sub>3</sub> emissions along aerobic degradation. To  
620 quantify the correlation between biochar adsorption potential and NH<sub>3</sub> emissions, a linear  
621 regression was thus calculated between NH<sub>3</sub> emissions (%TKNi) and biochar N-NH<sub>4</sub><sup>+</sup>  
622 adsorption potential (gN-NH<sub>4</sub><sup>+</sup>.kgBC<sup>-1</sup>). The results are presented in Fig 5.

623

624 **Fig. 5.** Linear regression between Biochar ammoniacal nitrogen adsorption and NH<sub>3</sub> emissions during aerobic biodegradation  
625 of AW. Vertical and horizontal bars represent the standard errors from NH<sub>3</sub> emissions and BC N-NH<sub>4</sub><sup>+</sup> adsorption,  
626 respectively.  $\text{NH}_{3\text{emissions}} = -2.76 \cdot \text{BC}_{\text{NNH}_4\text{adsorption}} + 25.47$ ,  $R^2 = 0.79$ ,  $p$ -value = 0.043. The complete linear regression table can be  
627 visualized in the Supplementary Materials.

628 The Fig. 5 shows a significant ( $p$ -value = 0.043) strong linear correlation ( $R^2 = 0.79$ ) between  
629 both variables. These findings, in conjunction with theoretical considerations, suggest that

630 the biochar  $\text{NH}_4^+$  adsorption capacity serves as good indicator of the biochar's potential to  
631 mitigate  $\text{NH}_3$  emissions in aerobic degradation processes. Regarding the linear regression  
632 model equation, it appears that increasing the biochar  $\text{NH}_4^+$  adsorption capacity by 1  
633  $\text{gNNH}_4^+.\text{kg}^{-1}$  tends to reduce the  $\text{NH}_3$  emissions by 2.76% ( $\text{gN-NH}_3.\text{gTKNi}^{-1}$ ). Thus, optimizing  
634 biochar  $\text{NH}_4^+$  adsorption capacity is a promising strategy to increase its capacity to reduce  $\text{NH}_3$   
635 emissions during aerobic degradation like composting.

636 It is important to note that the linear model presented in Fig. 5 is based on a limited set of  
637 controlled experimental conditions and cannot yet be used as a universal predictive tool.  
638 Despite this limitation, the results show the potential of the methodology presented in this  
639 work. Future researchers interested in further exploring the creation of a predictive test for  
640  $\text{NH}_3$  emissions could enhance the dataset presented in this paper. To increase the robustness  
641 of this correlation as a predictive tool, it would be advisable to incorporate different organic  
642 wastes and biochars made from various substrates, such as straw or bamboo, in future  
643 studies.

#### 644 4 Conclusion

645 This study confirmed the existence of a significant and strong correlation between biochar N-  
646  $\text{NH}_4^+$  adsorption in a solid organic waste environment and its efficacy in mitigating  $\text{NH}_3$   
647 emissions during organic waste aerobic degradation processes, such as composting. This  
648 correlation shows that biochar N- $\text{NH}_4^+$  adsorption is a good predictor of biochar  $\text{NH}_3$   
649 emissions reduction during aerobic degradation. This result offers an insight for future studies  
650 aiming to develop predictive tools assessing the impact of biochar on the nitrogen cycle in  
651 composting. Such tools could assist biochar producers in estimating the potential effects of  
652 their products through small-scale, easy-to-execute tests before commercialization, thereby

653 enhancing the marketability of their products and the trustworthiness of biochar buyers  
654 regarding its effects. Furthermore, this research demonstrate that biochar HNO<sub>3</sub> chemical  
655 modifications improves biochar capacity to reduce NH<sub>3</sub> emissions during aerobic degradation.  
656 Finally, the study demonstrated that certain biochar chemical characteristics, including pH  
657 and TO/TC atomic ratio, exhibit strong and significant correlations with biochar N-NH<sub>4</sub><sup>+</sup>  
658 adsorption. These attributes were identified as influential factors affecting biochar surface  
659 acid groups and electrical properties. The authors recommend that future research seeking  
660 to enhance biochar's capacity to reduce NH<sub>3</sub> emissions during composting should prioritize  
661 the optimization of its chemical characteristics rather than its physical attributes.  
662 Furthermore, to consider an industrial application of these results, a serious economic  
663 analysis, including a sustainable price for biochar as highlighted by Marousek et al. (2017),  
664 will have to be carried out.

665

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808

809 **Table 1.** Physicochemical characterization of substrates

	<b>AW</b>	<b>OFMSW</b>	<b>BC450</b>	<b>BC450_HNO3</b>	<b>BC450_KOH</b>	<b>BC900</b>	<b>BC_C</b>	<b>BC_C_HNO<sub>3</sub></b>
	71.3	62.4	5.0	5.4	3.1	5.2		2.3
<b>Moisture (%)</b>	(SD = 0.3)	(SD = 0.6)	(SD = 0.1)	(SD = 0.4)	(SD = 0.2)	(SD = 0.1)	6.4- 5.9	(SD = 0.5)
	825.4	867.5	899.7	971.1	939.5	710.0	918.9	983.5
<b>VM (g.kgDM<sup>-1</sup>)</b>	(SD =3.9)	(SD = 6.3)	(SD = 2.2)	(SD = 1.7)	(SD = 2.3)	(SD = 35.9)	(SD = 18.2)	(SD = 4.3)
	171.4	126.3	100.2	28.8	60.5	290.0	81.1	16.8
<b>Ashes (g.kgDM<sup>-1</sup>)</b>	(SD = 1.9)	(SD = 3.3)	(SD = 2.2)	(SD = 1.7)	(SD = 2.3)	(SD = 35.9)	(SD = 18.2)	(SD = 4.5)
<b>pH (1:5 water to solid ratio w:w)</b>	9.3	8.7	6.7	2.5	5.5	9.9	9.1	3.0
<b>TC (%)</b>	NA	NA	71.74	62.98	72.72	84.08	85.87	76.45

<b>TH (%)</b>	NA	NA	3.65	2.70	3.47	1.14	1.96	1.69
<b>TN (%)</b>	NA	NA	0.65	3.92	0.52	0.26	0.39	1.94
<b>TO (%)</b>	NA	NA	23.96	30.16	20.22	14.56	8.63	16.52
<b>Atomic TO/TC</b>	NA	NA	0.29	0.41	0.24	0.15	0.09	0.19
<b>Specific surface area</b>								
<b>(m<sup>2</sup>.g<sup>-1</sup>)</b>	NA	NA	1.1	1.5	1.26	157.8	74.9	16.9
<b>Easily extractable N-</b>								
<b>NH<sub>4</sub><sup>+</sup></b>				0.3				
<b>(gN-NH<sub>4</sub>.kgDM<sup>-1</sup>)</b>	4.1-5.1	5.1-6.9	ND	(SD < 0.01)	ND	ND	NA	NA
<b>Total N-NH<sub>4</sub><sup>+</sup></b>	11.45	9.16						
<b>(gN-NH<sub>4</sub>.kgDM<sup>-1</sup>)</b>	(SD = 0.30)	(SD = 0.35)	NA	NA	NA	NA	NA	NA
<b>TKN</b>	25.1	24.4	4.4	32.6	5.1	2.4	2.4	12.7
<b>(gN.kgDM<sup>-1</sup>)</b>	(SD = 1.0)	(SD = 0.4)	(SD = 0.3)	(SD = 0.4)	(SD = 0.1)	(SD = 0.1)	(SD = 0.1)	(SD = 0.2)

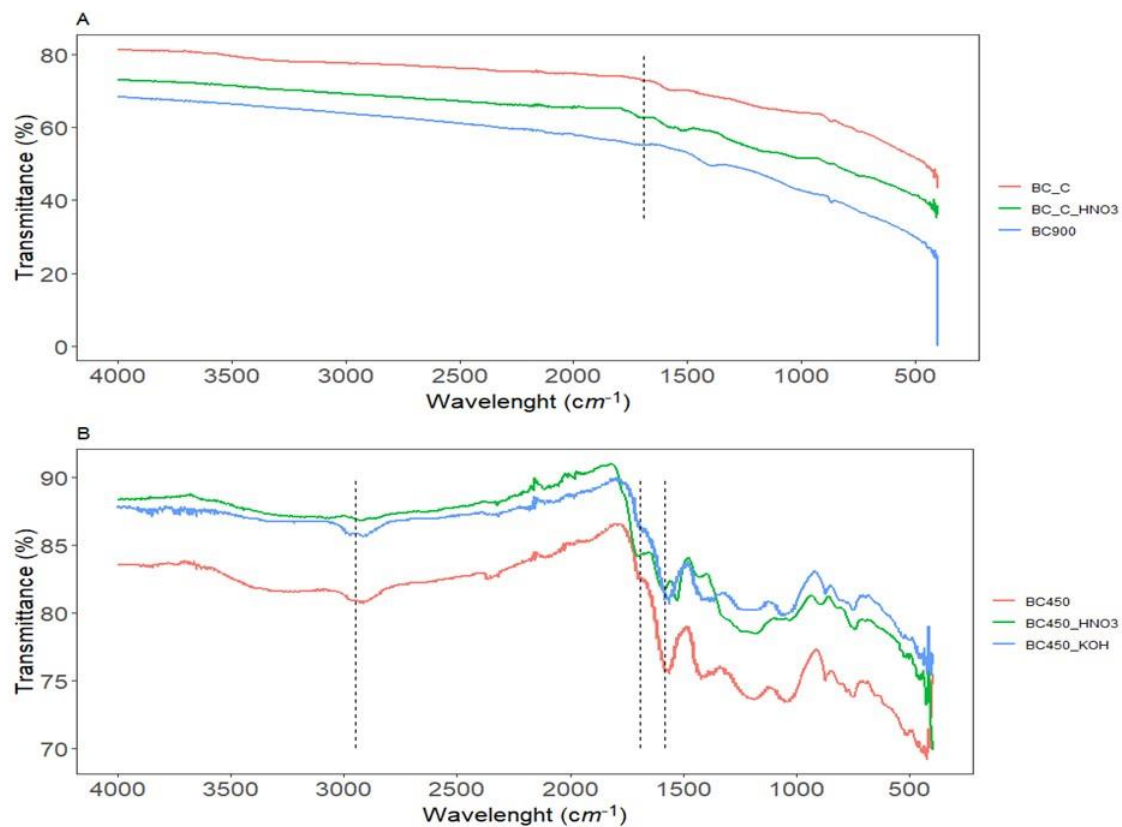
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810 Analysis with less than three repetitions had all their values showed in the table.

811

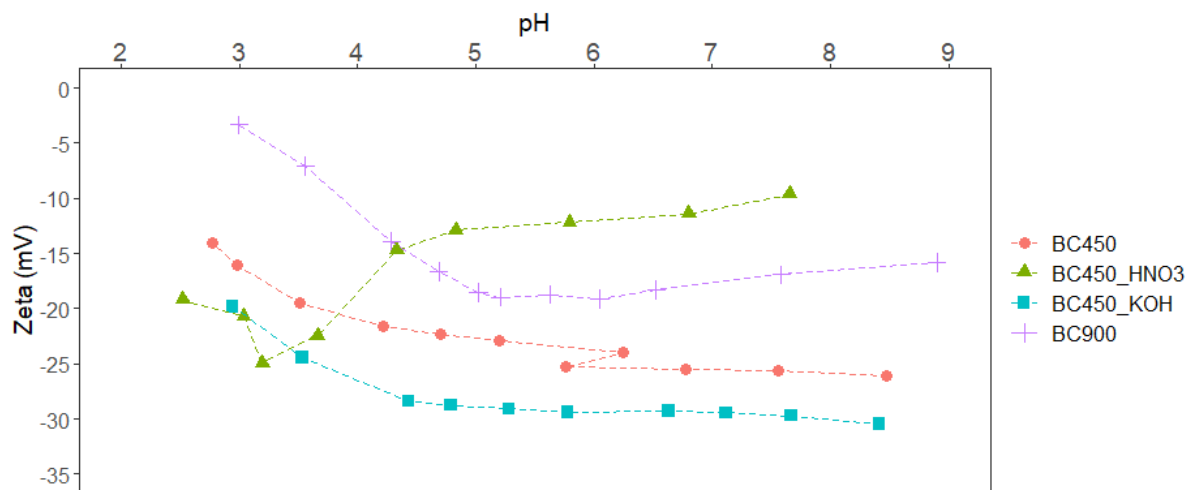
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815 **Fig. 1.** Fourier transform infrared spectra from (A) BC\_C, BC\_C\_HNO<sub>3</sub> and BC900. (B) BC450, BC450\_HNO<sub>3</sub> and BC450\_KOH.

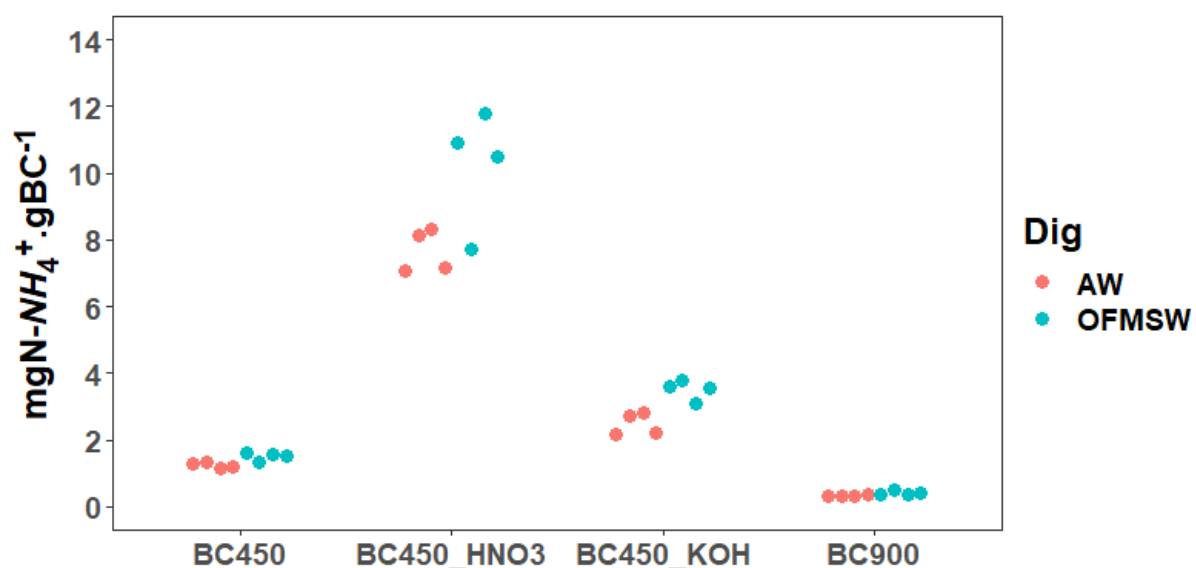


816

817 **Fig. 2.** Zeta potential of BC450, BC450\_KOH, BC450\_HNO<sub>3</sub> and BC900 inferred from streaming potential measurements vs.

818 pH.

819



820

821 **Fig. 3.** Ammoniacal nitrogen adsorbed in different biochars when applied to digestate made from agricultural wastes (AW)  
 822 or digestate made from organic fraction of municipal solid waste (OFMSW).

823

824

825 **Table 2.** Pearson correlation results between  $\text{NH}_3$  emissions, biochar  $\text{N-NH}_4^+$  adsorption and biochar characteristics

							SA		VM	Ashes	
	n	BC pH	TC (%)	TH (%)	TN (%)	TO (%)	Atomic ratio TO/TC	( $\text{m}^2 \cdot \text{g}^{-1}$ )	DM ( $\text{g} \cdot \text{kgFM}^{-1}$ )	( $\text{g} \cdot \text{kgDM}^{-1}$ )	( $\text{g} \cdot \text{kgDM}^{-1}$ )
BC sorp.	4	-0.92*	-0.87	0.19	0.97*	0.86	0.90*	-0.52	0.21	0.71	-0.71

826 BC sorp. = biochar  $\text{N-NH}_4^+$  adsorption, n = number of observations, BC = biochar, TC = total carbon, TH = total hydrogen, TN  
 827 = total nitrogen, TO = total oxygen, elemental analysis expressed as mass percentage in dry weight, SA = surface area, DM =  
 828 dry matter, FM = fresh matter. '\*' represent significant correlations (p-value < 0.1).

829

830 **Table 3.** Aeration rate and oxygen consumption

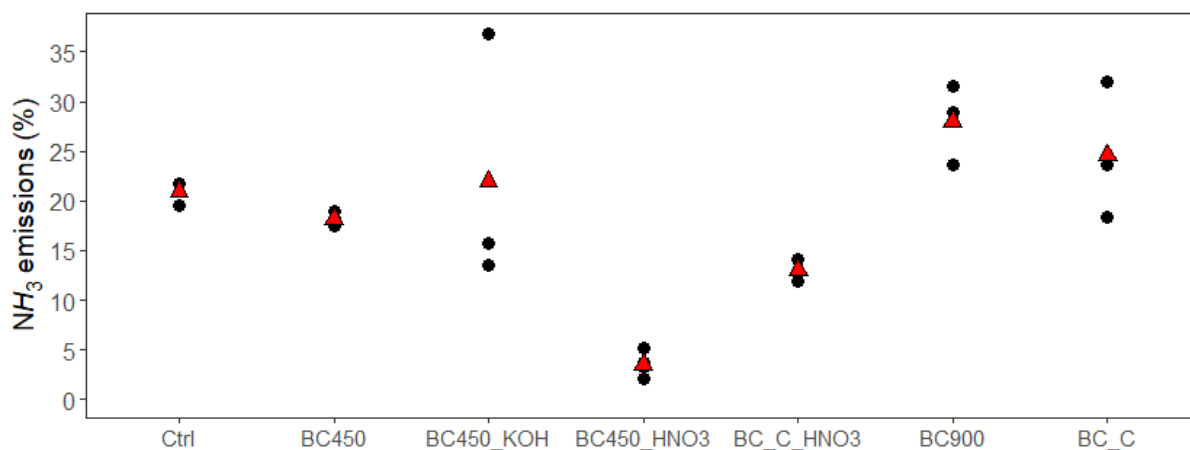


Experiment	n	Aeration rate			Total O <sub>2</sub>	Total O <sub>2</sub>	Total O <sub>2</sub>
		mean (l.h <sup>-1</sup> )	rate SD	rate CV (%)	consumption (mmolO <sub>2</sub> .kgVMi <sup>-1</sup> )	consumption SD	consumption CV (%)
Control	3	69.3	2.9	4.2	5.49	0.06	1.1
BC450	3	68.3	4.1	6.0	5.28	0.2	3.78
BC450_KOH	3	68.3	3.4	4.9	5.44	0.07	1.32
BC450_HNO <sub>3</sub>	4	69.4	7.0	10.0	5.56	0.05	0.83
BC900	3	71.8	4.4	6.1	5.51	0.23	4.23
BC_C	3	66.3	5.0	7.6	5.42	0.11	1.97
BC_C_HNO <sub>3</sub>	2	73.5-72.0	NA	NA	5.42-5.58	NA	NA

831 VM<sub>i</sub> = initial digestate volatile mater.

832

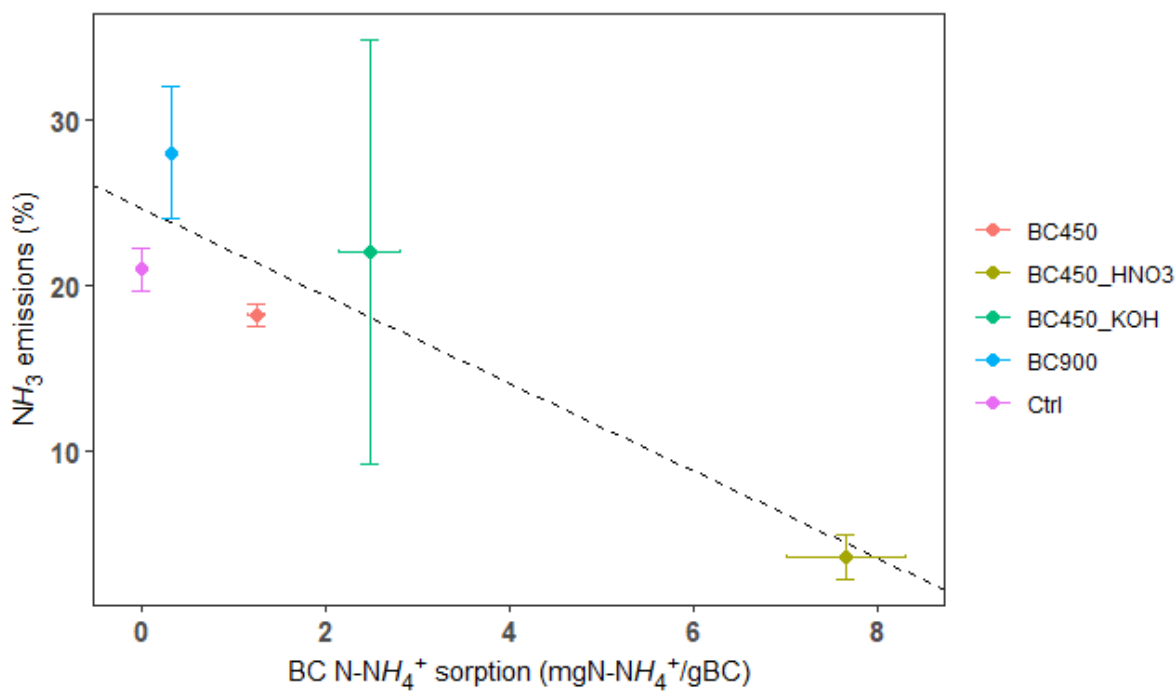
833 .



834

835 **Fig. 4.** Ammonia emissions expressed as percentage of Total Kjeldahl Nitrogen. Ctrl = control treatment, without biochar (n  
836 = 3), BC450 (n = 3), BC450\_KOH (n = 3), BC450\_HNO<sub>3</sub> (n = 4), BC\_C\_HNO<sub>3</sub> (n = 2), BC900 (n = 2), BC\_C (n = 3), n = number of  
837 observations. Red triangles indicate the treatments means.

838



839

840 **Fig. 5.** Linear regression between Biochar ammoniacal nitrogen adsorption and NH<sub>3</sub> emissions during aerobic biodegradation  
 841 of AW. Vertical and horizontal bars represent the standard errors from NH<sub>3</sub> emissions and BC N-NH<sub>4</sub><sup>+</sup> adsorption,  
 842 respectively.  $NH_{3emissions} = -2.76 \cdot BC_{NNH_4sorption} + 25.47$ ,  $R^2 = 0.79$ ,  $p\text{-value} = 0.043$ . The complete linear regression table can be  
 843 visualized in the Supplementary Materials.

844