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5

6 **Phosphorus fertiliser equivalent value of dairy processing sludge-derived**
7 **STRUBIAS products using ryegrass (*Lolium perenne* L.) and spring wheat**
8 **(*Triticum aestivum*)**

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27 **Highlights**

- 28 • STRUBIAS products derived from dairy sludge were tested for agronomic performance.
- 29 • Not all of the products tested showed good agronomic performance.
- 30 • High Fe content limits the fertiliser value of struvite and hydrochars.
- 31 • Generic guidelines based on a particular group of bio-based fertilisers are flawed.
- 32 • Removal of Fe from STRUBIAS products will improve agronomic performance.

33

34 **Abstract**

35 Struvite, biochar and ash products (collectively known as STRUBIAS) derived from different waste
36 streams are used as fertilisers in agriculture. Raw dairy processing sludge (DPS) shows promise as
37 bio-based fertilisers, but their secondary STRUBIAS-derived products need testing as fertilisers. The
38 objective of this ryegrass (*Lolium perenne L.*) and wheat (*Triticum aestivum*) pot trial was to calculate
39 their phosphorus mineral fertiliser equivalency (P-MFE) using the apparent P recovery (APR) method
40 for Fe-DPS and DPS-derived struvites (Struvite 1 – 4), hydrochars (HC1 – 3) and ash. Results showed
41 that the products can be divided into two groups: (1) a range of products that can (i.e., Struvite 1 – 3)
42 and (2) cannot (i.e., Struvite 4, HC1 – 3, ash and Fe-DPS) be considered as fertilisers. In the first group,
43 the P-MFE ranged from 66.8 to 76.7% for ryegrass and from 77.9 to 93.5% for spring wheat grain. In
44 the second group, the P-MFE ranged from 7.8 to 58.3% for ryegrass and from -34.5 to -151.3% for
45 spring wheat grain. The negative agronomic effects of some products for wheat grain (struvite and
46 HC) in this study were mainly caused by high Fe content which could be overcome by improved
47 treatment processes. Future policy and research must be aware that not all STRUBIAS products are
48 suitable as fertilisers and therefore need to be tested individually.

49 **Keywords:** dairy processing; bio-based fertilisers; phosphorus mineral fertiliser equivalence value;
50 STRUBIAS.

51

52 **1. Introduction**

53 In the European Union (EU) the dairy industry is the largest industrial food wastewater
54 contributor (Shi et al., 2021). This waste is phosphorus (P)-rich and leads to large volumes of solid
55 organic waste, referred to as dairy processing sludge (DPS). There are several types of DPS, with
56 altered chemical characteristics based on the chemicals (i.e. salts containing Al, Fe, or Ca, etc.) used
57 to treat waste, all of which have different nutrient and metal concentrations and mineral fertiliser
58 equivalence value (MFE) (Ashekuzzaman et al., 2019; Shi et al., 2022). Similar to other organic
59 fertilisers, land application of DPS occurs only at certain times of the year (Sommer and Knudsen,
60 2021). This results in storage requirements and may result in nitrogen (N) losses through gaseous
61 emissions. Therefore, technologies that process raw DPS on site are cost-efficient ways to recover
62 nutrients from waste. Conversion of DPS (with the addition of other feedstock) into struvite, biochar
63 (char-based materials) or ash (collectively called STRUBIAS) before land application is one such
64 technology (Hu et al., 2022; Shi et al., 2021). STRUBIAS materials are recognised as fertilisers in the
65 EU (EC, 2019; Huygens et al., 2018) and are expected to be certified to trade on the EU fertiliser
66 market by 2030 (Huygens et al., 2018). DPS-derived STRUBIAS products are a new subset of products
67 which, to date, have only been characterised in terms of their nutrient and metal concentrations (Shi et
68 al., 2021), while its agronomic performance has rarely been reported (Shi et al., 2022).

69 The characterisation and agronomic performance of different STRUBIAS products varies
70 considerably. Struvite (magnesium ammonium phosphate hexahydrate ($\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$)) is a
71 mineral of P formed at treatment plants during the anaerobic digestion process whereby the pH and
72 Mg levels are increased (Hertzberger et al., 2020). Struvite is considered to be a good slow-release
73 fertiliser, as it normally has similar fertiliser efficiency to common mineral P fertilisers such as super
74 phosphate (SP) and triple superphosphate (Johnston and Richards, 2003). However, the chemical
75 composition of waste-recovered struvite is not consistent with pure struvite (Hall et al., 2020), leading

76 to a variation in fertiliser performance. In addition, Al, Ca, Fe, and other toxic heavy metals can also
77 precipitate along with struvite and affect the fertiliser efficacy (Li et al., 2019). Biochar is made from
78 the thermochemical conversion of biomass in an oxygen-depleted atmosphere (Atallah et al., 2020)
79 with different thermochemical pre-treatment processes, conditions and feedstocks, resulting in
80 different products (Amoah-Antwi et al., 2020). Hydrothermal carbonisation (HTC) is a wet
81 thermochemical process at the temperature range of 180-260 °C (Kambo and Dutta, 2015) and
82 produces hydrochar. During this process an additional liquor is produced containing small-chain
83 organic acids, ammonium (NH₄) and phosphate (Becker et al., 2019). Ash is produced from the
84 incineration of bio-based materials by oxidation (Huygens et al., 2018) and contains K, P, S, Ca and
85 Mg (Brod et al., 2012; Haraldsen et al., 2011; Knapp and Insam, 2011) and levels of P that are
86 comparable to chemical equivalents (5.98% - 11.2% total P; Xu et al., 2012).

87 The objective of this study is to examine the phosphorus mineral fertiliser equivalent value (P-
88 MFE) of a range of DPS-derived STRUBIAS products, since they are mainly P recovery products. We
89 hypothesised that DPS could be a potential feedstock for STRUBIAS material and will show different
90 agronomic performance depending on their characterisation. Suggestions of processing solutions will
91 be provided when there is a shortfall in agronomic performance of the STRUBIAS materials. The
92 results can give guidance to the fertiliser and agricultural industries with respect to these new emerging
93 bio-based fertilisers and their efficacy.

94 **2. Materials and methods**

95 **2.1 Sample collection, preparation and analysis**

96 In this study four types of struvite (hereafter referred to as Struvite 1, 2, 3, and 4), one type of
97 ash, Fe-DPS, three types of hydrochar (hereafter referred to as HC1, 2, and 3), and one reference
98 mineral P fertiliser (SP) were used. The production process is illustrated in Figure 1. Struvite 1, 2 and
99 3 were precipitated from cheese production wastewater by varying the Ca:P, Mg:P and pH

100 (Numviyimana et al., 2020), and Struvite 4 was precipitated from HTC liquor (Numviyimana et al.,
101 2022). Ash was created using a biochar (Kwapinska et al., 2019) processed in a laboratory furnace at
102 650 °C for 3 hours, cooled to room temperature, and then ground using a pestle and mortar. HC1, 2,
103 and 3 were produced using a HTC process using Fe-DPS with different moisture contents. There was
104 no additional water added in the reactor liner during the HC1 process. One percent H₂SO₄ was added
105 in the reactor vessel with the DPS sample (set at 200 °C) to achieve moisture contents of 85% (HC2)
106 and 90% (HC3), respectively. The liquor from HC2 was the feedstock of Struvite 4. Once the
107 temperature was reached, the stirrer was operated at 25 rpm (HC1) and 36 (HC2 and HC3) rpm for 2
108 hours. Fe-DPS was collected from a dairy processing wastewater treatment plant in Ireland.

109 All DPS-derived STRUBIAS samples (Struvite 1-4, HC1-3 and ash) were characterised to
110 determine their nutrient, metal and carbon (C) contents using the methodology presented in Shi et al.
111 (2022). Briefly, pH was determined using a Jenway 3510 pH meter. Nutrients and metals were
112 examined by an Agilent 5100 synchronous vertical dual view inductively coupled plasma optical
113 emission spectrometer (Agilent 5100 ICP-OES) following the microwave-assisted acid digestion of
114 samples. A high temperature combustion method (LECO TruSpec CN analyser) was used to determine
115 total carbon (TC) and total nitrogen (TN). Mineral N was analysed colorimetrically following with
116 0.1M HCl extraction using an Aquakem 600 Discrete Analyser.

117

118 **2.2 Pot design for P-MFE of STRUBIAS products**

119 Soil samples were collected at Teagasc, Johnstown Castle Environmental Research Centre (52°
120 17'N, 6° 29'W) in Ireland and physically and chemically characterised for dry bulk density, water
121 holding capacity (WHC), moisture content, soil mineral N, soil pH, organic matter (OM), total
122 concentrations of nutrients and metals, and Morgan's P using the methodology presented in Shi et al.
123 (2022) and the results are shown in Table 1. Briefly, bulk density and WHC were measured using the
124 method of Wilke (2005). The moisture content was determined using BS 1377-1 (BSI, 2016). Soil

125 mineral N was analysed colorimetrically after extraction by 1M KCl. Soil OM was measured by loss
126 on ignition using BS 1377-3 (BSI, 1990). Soil pH, total concentrations of nutrients and metals was
127 measured using the same methodology as for STRUBIAS samples. Plant available P was measured
128 with Morgan's P extracting solution (Morgan, 1941). The result of soil Morgan's P indicated that the
129 soil was P deficient ($< 3.0 \text{ mg L}^{-1}$) (Teagasc, 2020). The soil used in the pot trial was air dried for a
130 week before sieving to $<4 \text{ mm}$. Pot trials, comprising two crops, ryegrass (*Lolium perenne L.*) and
131 wheat (*Triticum aestivum*), were set up to examine the P-MFE following the methodology of Sigurnjak
132 et al. (2017), whereby two litre-capacity pots were filled as follows: a 2 cm-deep layer of gravel was
133 added to the pots followed by 0.5 kg of soil and the remaining soil (1.3 kg) was mixed with the
134 respective DPS-derived STRUBIAS materials and then added. Each layer of soil was compacted using
135 a circular disk to a target dry bulk density of 1.2 g cm^{-3} . Finally, distilled water was added to reach a
136 70% WHC target.

137 The results of a previous study conducted by Shi et al. (2022) indicated that an application rate
138 equivalent to 40 kg P ha^{-1} for ryegrass and 50 kg P ha^{-1} for spring wheat was optimal for plant growth.
139 All the application rates were transformed to the pot experiment depending on taking the surface area
140 of the pot into account. The final P rate was 91 mg P pot^{-1} for ryegrass and $113 \text{ mg P pot}^{-1}$ for spring
141 wheat. Therefore, these rates were used in the current study. STRUBIAS treatments (i.e., Struvite 1 –
142 4, with Struvite 4 only applied on spring wheat due to experimental logistical issues), ash, HC1-3, raw
143 Fe-DPS, and SP were applied at one application rate for each crop. A study control (without P fertiliser)
144 for each crop was also included in the experiments. Mineral fertilisers (i.e., calcium ammonium nitrate
145 (CAN), potassium chloride (MOP) and sulphate of potash (SOP)) were applied to all pots to ensure
146 that P was the only limiting nutrient (Table S1 and S2). Mg fertiliser was not added since extra Mg
147 fertiliser is only advisable if soil Mg is less than about 50 mg/l , while the Morgan's Mg of the soil used
148 was 177 mg/l (Teagasc, 2020). Every treatment had three replications.

149 For ryegrass pots, 0.6 g of seeds (equivalent to 28 g m⁻²) were seeded per pot. For wheat, 10
150 germinated wheat seeds were seeded in each pot (Darch et al., 2019). The pots were placed in a
151 randomised block layout within a controlled growth chamber (Teagasc, Johnstown Castle) and
152 operated under the following conditions: (1) 16-hour light photoperiod (2) daytime temperatures of
153 14 °C and night-time temperatures of 8 °C, with respective relative humidities of 85% and 75%, and
154 (3) photosynthetically active radiation of 450 ± 50 μmol m⁻² s⁻¹. All pots were held between 70 and
155 80% WHC by regularly weighting them. The grass was manually cut 4 cm above soil level after
156 reaching a length of 22 – 26 cm. The grass pot trial lasted 16 weeks and 3 cuts were taken. The wheat
157 plants were grown to maturity (20 weeks) and then separated into grain and chaff + straw after
158 harvesting (Darch et al., 2019).

159 **2.3 Crop and soil sampling and analysis**

160 Fresh harvested crop samples were oven-dried at 70 °C for 72 hours in perforated plastic bags.
161 Once dried, dry weight was recorded for dry matter (DM) analysis and, subsequently, dried samples
162 were grounded and sieved to < 2 mm for nutrient and metal analysis. Soil samples before and after the
163 pot trial were oven-dried at 40 °C for 72 hours and then sieved to <2 mm and analysed for nutrients
164 and metals as for the field soil (Table 1).

165 **2.4 P-MFE and statistical analysis**

166 Shi et al. (2022) examined different methods to determine the agronomic performance of DPS.
167 As a result of that study, the P-MFE (equation 2) calculated from apparent P recovery (APR) (equation
168 1) was deemed most suitable to present agronomic performance of P and is used in the current study.

$$170 \text{ APR}(\%) = \frac{P \text{ uptake}_{\text{Treatment}} - P \text{ uptake}_{\text{Control}}}{\text{Total P applied}_{\text{Treatment}}} \quad (1)$$

171 where APR is the difference in P uptake between treatment (P uptake_{Treatment}) and unfertilised pots (P
172 uptake_{Control}) (Murphy et al., 2013).

173

$$174 \quad \text{P-MFE (\%)} = \frac{APR_{Treatment}}{APR_{Reference}} \times 100 \quad (2)$$

175 where P-MFE is the ratio between the apparent nutrient recovery of organic residues ($APR_{Treatment}$) and
176 the mineral fertiliser applied at the same rate ('reference') (Sigurnjak et al., 2019).

177

178 Statistical analysis was performed using SAS statistical software (SAS, Statistical Analysis System,
179 2013). One-way analysis of variance (ANOVA) was performed for every dataset of crop yield and
180 crop P uptake to determine if differences were seen as a function of treatment. Statistically
181 significant differences were considered at a p-value ≤ 0.05 and where significance was found, a
182 Fisher's Least Significant Difference (LSD) test was used to determine statistical differences in
183 means as a function of treatment for each variable at each harvest.

184

185 **3. Results**

186 **3.1 Characterisation of nutrients and metals**

187 The DPS-derived STRUBIAS products differed in their nutrient and heavy metal contents
188 (Table 2). Comparing with the minimum nutrients requirements in the EU fertiliser regulation i.e. (1%
189 by mass of TN and 1% by mass of P_2O_5 for solid organic fertiliser; 2% by mass of TN and 2% by mass
190 of P_2O_5 for solid organo-mineral fertiliser) (EC, 2019), all products had high P contents and therefore
191 met the minimum P requirement. However, the TC content of struvite 4 and ash was too low and
192 therefore these products cannot be considered as organic fertilisers under the EU fertiliser regulation.
193 The characterisation results suggested that all products had potential as fertilisers from at least a
194 nutritional perspective. The heavy metal content of the ash was much higher than that of the other
195 products. However, all products had heavy metal content below EU regulated limits (Cu, Ni, Pb, Cd,
196 Zn, Hg and As) (EU, 2019).

197

198 **3.2 Crop yield and P uptake**

199 In the ryegrass study, cumulative yields and P uptake of DPS or STRUBIAS treatments were
200 significantly higher than those of the control (no P treatment), except for Struvite 4 and ash (Table 3).
201 The lowest ryegrass yields were measured in these two treatments, while high yields were achieved
202 with Struvite 1 and 3, and HC1 and 3, which also had a similar yield to mineral P fertiliser. For P
203 uptake, only Struvite 1 – 3 and HC1 treatments were significantly higher than the control and for
204 Struvite 2 and 3, P uptake was in the same order of magnitude as for SP. In the spring wheat study,
205 there was no significant difference between chuff + straw and grain yields of the control and all
206 treatments, except ash for chuff (Table 3). This result indicated that the recommended P application
207 rates might still not be enough to increase plant-available P in deficient soils (Croffie et al., 2020).
208 Therefore, higher P concentrations should be applied in future studies to achieve a higher crop yield
209 response and P uptake result. The lowest grain yield was found in the ash treatments, with the highest
210 grain yield achieved with Struvite 1 and 2. All treatments had similar yields to mineral P fertiliser.

211

212 **3.3 P-MFE for ryegrass and spring wheat**

213 The APR and the corresponding P-MFE results of the ryegrass and spring wheat studies are
214 presented in Table 4. The highest APR was observed for SP treatment in both ryegrass and spring
215 wheat pot trial, which demonstrated that mineral P fertiliser is more readily available for plant uptake.
216 The P-MFE of the DPS-derived STRUBIAS materials ranged from 7.8 to 76.7% for ryegrass and from
217 -151.3 to 93.5% for spring wheat grain. Struvite 1-3 treatments had the highest P-MFE (66.8-76.7%
218 for ryegrass and 77.9-93.5% for spring wheat grain), while ash had the lowest among all types of
219 STRUBIAS materials examined in this study. Negative P-MFE results were found in ash, HC and Fe-
220 DPS treatments in the spring wheat grain trial.

221

222 **4. Discussion**

223 **4.1 Variation in chemical characteristics**

224 The chemical characteristics of all STRUBIAS products are different and are mainly caused
225 by the feedstock and treatment process, so generic fertiliser guidelines, based solely on the type of end
226 products, are flawed. Struvite products had high concentrations of P and Mg, with metal concentrations
227 lower than legal limits (EU, 2019). Struvites 1 - 3 were generated from cheese production wastewater
228 (whey) with different pH and salt dosages, resulting in different nutrient concentrations (Numviyimana
229 et al., 2020). Struvite 1 was produced under optimal conditions (highest struvite content) and contained
230 the highest amounts of nutrients, while Struvite 3, produced with a high dose of calcium salts, had low
231 nutrient but a high Ca content. The P recovery of Struvite 3 was improved by chemical precipitation
232 with Ca. However, this resulted in lower fertiliser quality as the Ca addition caused the loss of
233 ammonium and P availability. Struvite 4 was precipitated from the HC2 liquor and contained high
234 amounts of Fe due to the feedstock used. Both Ca and Fe are known to negatively affect the availability
235 of P in soil (Ashekuzzaman et al., 2021). All ash and HC samples contained a significant amount of
236 nutrients and metals, except NH₄-H, because P and metals are most likely to remain and concentrate
237 in solid residues during thermo-chemical process (Shackley et al., 2010). Three HCs in this study were
238 produced from a Fe-DPS and different initial acidity was used, which can affect HC yield (Khalaf et
239 al., 2022) but did not affect the HC characteristics.

240

241 **4.2 Problems and solutions for the tested STRUBIAS products**

242 The results of this study suggest that not all STRUBIAS products of dairy waste are suitable
243 as fertilisers. For example, struvite is normally considered to be an excellent fertiliser, because it has
244 similar fertiliser efficiency to common mineral P fertilisers (Johnston and Richards, 2003). However,
245 in this study, only three of the four struvites tested showed good potential as fertilisers. Struvite 4,

246 precipitated from HC2 liquor, produced a low ryegrass yield and consequently had a low P-MFE.
247 Numviyimana et al. (2020) conducted a citric acid nutrient release assay on Struvite 3 (the same
248 product as used in the current study) and their results showed lower nutrient availability (P, Mg, NH_4^+)
249 in that product, which was also observed in the current study. Furthermore, Numviyimana et al. (2020)
250 also found that Struvite 1 had slow P release properties, which may explain the higher grass yields and
251 P uptake in the last ryegrass harvest in the current study (Table 3). The results of the literature show
252 that struvite derived from different feedstocks exhibits a range of agronomic performance (Table S3).
253 Szymańska et al. (2020) conducted a long-term pot experiment with struvite derived from cattle slurry.
254 Higher P-MFE (~150% in silty loamy soils and ~140% in loamy sandy soils) was obtained in the
255 second year of the experiment, with overall results outperforming commercial ammonium phosphate.
256 The results indicated that struvite was an excellent slow-release P fertiliser and might have better
257 agronomic performance than mineral P fertilisers in the long term. González-Ponce et al. (2021)
258 conducted a 90-day pot experiment with struvite derived from anaerobically digested sewage sludge
259 on grass. Increased APRs (~10%) were obtained from these samples and the highest APRs (11.5 ± 3.8
260 and 15.7 ± 5.5) were obtained from treatments with struvite of a larger particle size. All these results
261 suggested that the plants efficiently used the P contained in the struvite.

262 The high Fe content of Struvite 4 resulted in its poor agronomic performance. Iron exhibits a
263 high precipitation potential for struvite but limited fertiliser quality of struvite (Numviyimana et al.,
264 2022). This is due to the lower water solubility (K_{sp}) of iron salts such as vivianite ($\text{Fe}_3(\text{PO}_4)_2 \cdot 8\text{H}_2\text{O}$,
265 $K_{sp} = 10^{-35.8}$) than struvite ($K_{sp} = 10^{-13.17}$) (Hanhoun et al., 2011; Priambodo et al., 2017).
266 Numviyimana et al. (2022) conducted cucumber growth experiments using Struvite 4 and observed
267 very low germination rates (32%), which was attributed to phytotoxicity issues associated with metals.
268 However, the fertiliser quality of Struvite 4 could be improved if Fe was removed during the processing
269 chain: Numviyimana et al. (2022) used oxalic acid for better struvite precipitation, which removed Fe
270 from the process chain, resulting in much higher cucumber germination rates (88%). Therefore,

271 struvite from waste streams should not be assumed to be a good fertiliser without testing, and, where
272 needed, processing modifications can be implemented to overcome shortfalls in its agronomic
273 performance.

274 Although the ash had a high P content, it produced the lowest crop yield (and therefore P-MFE)
275 in both the ryegrass and spring wheat trials. Compared to the study control, ash inhibited the growth
276 of spring wheat. The negative P-MFE in the spring wheat trial also implied a slow P release and a low
277 P uptake. This was because P in ash normally occurs as Fe, K, and Ca phosphate (Tan and Lagerkvist,
278 2011), and therefore the solubility of P is likely to be low. In some cases, ash has been reported to
279 increase the yield or P-MFE of agricultural crops (Battisti et al., 2022; Kuligowski et al., 2010), while
280 other studies reported that ash did not significantly affect or even inhibited, plant growth (Kominko et
281 al., 2019; Ochecova et al., 2014) (Table S3). These varying results may be attributed to the difference
282 in the type of feedstock or the post-treatment process, which affects the solubility of P (Møller et al.,
283 2007; Rubæk et al., 2006). For example, acidification can transform P in ash into a more soluble form.
284 Kuligowski et al. (2010) found that using sulfuric acid as an extractant and potassium hydroxide as a
285 neutraliser is capable of making ash P highly available. Buneviciene et al. (2020) found that granulated
286 biofuel ash significantly increased spring barley grain and straw yields compared to ash powder.

287 Positive and negative agronomical effects were observed for HC treatments, with HC 1 – 3
288 performing significantly better in ryegrass when compared to the spring wheat study. The HTC process
289 improved the agronomic value (yield and P-MFE) compared to its feedstock (Fe-DPS), and the
290 different initial acidities did not affect its agronomical performance. The experiments indicated that
291 HC can be (depending on its individual properties) a good fertiliser for ryegrass, but the negative P-
292 MFE for spring wheat implied a slow release of P and low crop P uptake compared to the control of
293 the study. The fertiliser potential of HC is very complex and depends on many variables, such as the
294 type of soil, type of crop, application rates, HTC process conditions, feedstock, time in the soil, and
295 experimental conditions (field/pot) (Melo et al., 2018). Many studies have observed different

296 agronomic performances of HC (Table S3). For example, Melo et al. (2018) reported a positive
297 Phaseolus bean yield response after application of sewage sludge derived HC with soil fertility and
298 soil quality benefits. Furthermore, a longer residence time of HC in the soil enabled better nutrient
299 uptake by the crop due to the slow release of nutrients. Gajić and Koch. (2012) applied HC derived
300 from sugar beet pulp and beer draff in the field with different mineral N fertiliser treatments and found
301 that HC, especially with its high C/N ratio, inhibited sugar beet growth due to its high N immobilizing
302 potential. Xia et al. (2020) found that HC derived from pinewood sawdust inhibited the growth of
303 paddy rice in both root and stem. On the contrary, Xia et al. (2020) observed a significant positive
304 effect on rice treated with aminofunctionalised hydrochar (by polyethylenimine grafting) and this HC
305 product effectively reduced heavy metal uptake by the plant. Therefore, although HC derived from
306 DPS has potential as a fertiliser, more research is still needed to identify suitable feedstocks, possible
307 risks, inhibiting mechanisms and substances, and technologies to reduce risks or improve nutrient
308 availability.

309 Currently and more increasingly into the future, farmers and growers will be encouraged to use
310 less mineral fertiliser and to choose bio-based alternatives. As bio-based fertilisers are heterogeneous
311 in nature (differential origin and processing lead to heterogeneous characteristics), a standardised
312 procedure to examine the agronomic performance of each bio-based fertiliser alternative must be
313 applied. As each new bio-based product emerges, the following chain is suggested: (1) documentation
314 of how the product was processed, (2) total and available nutrient and metal concentration must be
315 conducted using standard methods, and (3) elucidation of its N and P-MFE stating in detail the
316 methodology and calculation methods used. Step 3 must be transparent and well documented, as N
317 and P-MFE values differ depending on the methods used, and (4) this process needs to be repeated for
318 each type of bio-based fertiliser and crop combination. Without this thorough chain of investigation in
319 place, assumptions regarding a particular group of bio-based fertilisers may be too generalised. For

320 example, in the current study, not all products defined as struvite were considered potential
321 fertilisers.

322

323 **5. Conclusions**

324 In this study, the agronomic performance of different DPS-derived STRUBIAS materials was
325 determined, but not all the materials tested were deemed suitable as fertilisers to be used in agriculture.
326 Only three of the four struvites tested showed good agronomic performance. The fertiliser value of the
327 fourth struvite and the hydrochars was limited by their high Fe content, which could be overcome by
328 exclusion of the use of iron salts in the removal of P to comply with discharge licence requirements in
329 processing plants. Ash treatments exhibited very low or even negative P-MFEs. These results indicate
330 the importance of testing every bio-based fertiliser alternative to determine their agronomic
331 performance, before making a decision regarding their suitability as fertilisers to be used in agricultural
332 crops. In addition, such testing can guide the processing of STRUBIAS products where low or even
333 negative P-MFEs are determined. Future policy and research must be aware that not all STRUBIAS
334 products will be suitable as fertilisers. Therefore, STRUBIAS products derived from different wastes
335 will continuously need to be evaluated to examine their nutrient and metal concentrations, along with
336 their agronomic performance as fertilisers.

337

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341

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Table 1. Characteristics of the soil used in the pot trial.

Clay	Silt	Fine Sand	Coarse Sand	Organic Matter	Total N	Total P	Total K	Total Al	Total Ca	Total Fe	Morgan's P	pH
%	%	%	%	%	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/l	
15.0	30.1	34.6	20.3	6.5	2700	582	2640	14191	1367	13143	1.9	5.8

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Table 2. Characterisation of dairy processing sludge derived STRUBIAS products

Parameters	Struvite1	Struvite2	Struvite3	Struvite4	Ash	HC1	HC2	HC3	Fe-DPS	Min.	Max.	Mean \pm SD
pH	7.9	8.3	8.8	9.0	9.3	6.9	7.9	7.7	7.6	6.9	9.3	8.2 \pm 0.72
TN (g kg ⁻¹)	43.7	29.4	11.2	4.99	1.1	37.5	29.4	36.5	68.3	1.1	68.3	29.1 \pm 19.9
NH ₄ -H (g kg ⁻¹)	40.4	15.4	0.33	1.1	0.092	0.0026	0.0031	0.0025	0.35	0.0025	40.4	6.4 \pm 12.9
TP (g kg ⁻¹)	104.2	80.2	47.0	59.0	99.3	78.9	85.4	79.9	57.2	47.0	104.2	76.8 \pm 18.1
TC (%)	10.7	25.9	38.8	0.20	0.90	22.6	18.4	21.2	32.7	0.20	38.8	21.4 \pm 12.6
K (g kg ⁻¹)	7.1	7.5	6.5	7.0	26.7	13.5	8.5	12.6	15.3	6.5	26.7	11.6 \pm 6.1
Mg (g kg ⁻¹)	101.3	62.2	18.8	72.8	17.0	3.7	3.7	3.5	2.9	2.9	101.3	31.8 \pm 35.0
S (g kg ⁻¹)	0.16	0.46	0.62	0.07	11.9	3.2	12.8	8.2	4.3	0.07	12.8	4.6 \pm 4.8
Na (g kg ⁻¹)	2.6	8.8	31.7	65.2	20.5	2.8	1.8	2.6	3.0	1.8	65.2	15.4 \pm 20.1
Ca (g kg ⁻¹)	14.7	34.5	66.9	21.2	227.5	68.0	72.0	65.7	49.2	14.7	227.5	68.9 \pm 59.6
Cr (mg kg ⁻¹)	2.2	2.8	3.3	2.6	41.2	6.5	6.8	6.8	5.3	2.2	41.2	8.6 \pm 11.7
Cu (mg kg ⁻¹)	1.8	0.21	0.38	0.82	92.7	47.8	6.1	5.4	4.2	0.21	92.7	17.7 \pm 30.1
Ni (mg kg ⁻¹)	<0.6	<0.6	<0.6	<0.6	27.4	7.6	9.4	9.1	7.0	<0.6	27.4	6.8 \pm 8.3
Pb (mg kg ⁻¹)	<2	<2	<2	<2	32.6	5.9	5.9	5.3	4.3	<2	32.6	6.1 \pm 9.7
Zn (mg kg ⁻¹)	30.1	34.4	36.2	6.9	482.4	186.1	185.9	171.7	136.0	6.9	482.4	141.1 \pm 139.1
Al (g kg ⁻¹)	0.02	0	0	0.05	82.1	8.0	8.5	7.8	6.1	0	82.1	12.5 \pm 24.9
Fe (g kg ⁻¹)	0.07	0.17	0.39	31.4	7.5	177.3	199.7	183.4	128.7	0.07	199.7	80.9 \pm 84.0

Co (mg kg ⁻¹)	<0.3	<0.3	<0.3	<0.3	4.9	11.0	11.3	11.0	9.6	<0.3	11.3	5.4 ± 5.0
Mo (mg kg ⁻¹)	<0.5	<0.5	<0.5	<0.5	11.1	<0.5	<0.5	<0.5	<0.5	<0.5	11.1	1.2 ± 3.5
Mn (mg kg ⁻¹)	0.53	0.57	2.24	10.2	609.6	234.7	247.9	230.3	181.7	0.53	609.6	168.6 ± 188.1
Cd (mg kg ⁻¹)	<0.15	<0.15	<0.15	<0.15	0.68	<0.15	0.25	<0.15	<0.15	<0.15	0.68	<0.15
As (mg kg ⁻¹)	<1.5	<1.5	<1.5	<1.5	4.1	<1.5	<1.5	<1.5	<1.5	<1.5	4.1	<1.5
B (mg l)	2.0	2.7	3.0	7.4	74.0	3.1	2.0	2.4	1.7	1.7	74.0	10.9 ± 22.4
Se (mg kg ⁻¹)	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1

Abbreviations used in table: HC=hydrochar; DPS=dairy processing sludge; SD=Standard deviation; TN=total nitrogen; TP=total phosphorus; TC=total carbon.

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Table 3. Effect of treatment and P rate on the grass and spring wheat dry matter yield and P uptake over the course of the P-MFE experiment.

Treatment*	P rate kg ha ⁻¹	Yield (g)				P uptake (mg pot ⁻¹)			
		1*	2*	3*	cumulative	1*	2*	3*	cumulative
<i>Ryegrass</i>									
Control	0	2.6 ^d (0.8)	2.0 ^f (0.5)	1.9 ^f (0.3)	6.5^f (1.0)	728 ^d (250)	605 ^c (146)	390 ^f (43)	1723^d (190)
SP	40	5.1 ^{bc} (1.8)	5.3 ^a (1.5)	8.8 ^a (1.5)	19.2^a (2.9)	1982 ^a (685)	1331 ^a (400)	1168 ^a (156)	4482^a (857)
Struvite1	40	5.7 ^{bc} (0.8)	4.4 ^{abc} (1.1)	7.6 ^{bcd} (0.1)	17.7^{ab} (2.2)	1282 ^{bc} (292)	1230 ^{ab} (430)	1054 ^{ab} (30)	3565^{bc} (219)
Struvite2	40	8.3 ^a (1.9)	3.7 ^{bcd} (0.2)	3.3 ^{ef} (0.9)	15.2^{bc} (2.4)	1922 ^a (205)	1100 ^{abc} (204)	646 ^{de} (112)	3667^{ab} (269)
Struvite3	40	7.4 ^a (0.8)	4.7 ^{ab} (0.4)	4.0 ^{de} (0.4)	16.1^{ab} (1.6)	1875 ^{ab} (211)	1117 ^{abc} (218)	846 ^{ef} (149)	3838^{ab} (252)
Struvite4	40	3.0 ^{cd} (0.5)	2.7 ^{def} (0.3)	1.4 ^f (0.8)	7.0^f (0.5)	1166 ^{cd} (282)	821 ^{abc} (167)	412 ^{ef} (134)	2399^d (186)
Ash	40	3.5 ^{cd} (0.5)	2.8 ^{def} (0.4)	3.0 ^{ef} (0.1)	8.3^{ef} (2.1)	891 ^{cd} (208)	812 ^{bc} (101)	235 ^f (32)	1938^d (193)
HC1	40	4.8 ^{bcd} (1.0)	4.9 ^{ab} (0.6)	7.4 ^{abc} (1.4)	17.1^{ab} (1.0)	1169 ^{cd} (210)	1179 ^{ab} (236)	983 ^{abc} (95)	3332^{bc} (350)
HC2	40	3.5 ^{cd} (0.1)	3.3 ^{cdef} (0.3)	6.3 ^{bcd} (0.7)	12.5^{bc} (0.3)	992 ^{cd} (266)	947 ^{abc} (78)	737 ^{cd} (109)	2536^{cd} (532)
HC3	40	4.0 ^{bcd} (1.2)	4.3 ^{abc} (0.5)	7.8 ^{ab} (1.0)	16.1^{ab} (1.6)	992 ^{cd} (329)	969 ^{abc} (281)	849 ^{bc} (23)	2856^{cd} (460)
Fe-DPS	40	4.0 ^{bcd} (1.0)	3.8 ^{bcd} (0.7)	4.3 ^{de} (0.4)	12.1^{cd} (1.3)	1084 ^{cd} (236)	920 ^{abc} (254)	453 ^{ef} (144)	2457^d (283)

Spring wheat

		Chuff + Straw	Grain	Chuff + Straw	Grain
Control	0	19.2 ^{ab} (2.0)	13.6 ^{ab} (2.4)	522 ^{ab} (100)	2995 ^{ab} (135)
SP	50	17.6 ^{ab} (4.8)	10.9 ^{ab} (1.6)	816 ^a (632)	4016 ^a (946)
Struvite 1	50	20.8 ^a (2.3)	14.4 ^a (1.4)	703 ^{ab} (85)	3879 ^a (252)
Struvite2	50	20.6 ^a (3.5)	14.5 ^a (5.4)	1089 ^a (534)	3948 ^a (927)
Struvite3	50	19.5 ^a (2.5)	11.7 ^{ab} (3.3)	983 ^{ab} (515)	3766 ^a (490)
Ash	50	11.9 ^c (3.6)	6.7 ^b (1.0)	272 ^b (67)	1225 ^d (215)
HC1	50	18.8 ^{ab} (2.8)	10.9 ^{ab} (3.2)	386 ^{ab} (124)	2496 ^{bcd} (283)
HC2	50	17.0 ^{abc} (3.1)	11.1 ^{ab} (4.6)	476 ^{ab} (282)	2518 ^{bc} (705)
HC3	50	16.4 ^{abc} (0.2)	11.8 ^{ab} (2.5)	408 ^{ab} (52)	2314 ^{bcd} (712)
Fe-DPS	50	14.2 ^{bc} (3.3)	8.0 ^b (2.8)	749 ^{ab} (432)	1724 ^{cd} (624)

Mean comparison by Fisher's Least Significant Difference (LSD) test ($p \leq 0.05$); Within columns shared letters denote no difference ($p > 0.05$), and unshared letters denote a statistical difference ($p \leq 0.05$); Values indicated in brackets are standard deviations ($n = 3$). Abbreviations used in table: SP=super phosphate; HC=hydrochar; DPS=dairy processing sludge.

* Three cuts of ryegrass.

Table 4. Ryegrass and wheat grain pot trial results for dairy processing sludge and derived STRUBIAS, rate applied in pot trial and % of mineral fertiliser equivalent value.

	P rate	APR from Eqn. 1	P-MFE from Eqn. 2
	kg ha⁻¹	%	%
		<i>Ryegrass</i>	
SP	40	30.4	100.0
Struvite 1	40	20.3	66.8
Struvite 2	40	21.4	70.5
Struvite 3	40	23.3	76.7
Struvite 4 ¹	40	7.5	24.5
Ash	40	2.4	7.8
HC1	40	17.7	58.3
HC2	40	10.7	35.1
HC3	40	12.5	41.1
Fe-DPS	40	8.1	26.6
		<i>Wheat grain</i>	
SP	50	9.0	100.0
Struvite1	50	7.8	87.0

Struvite2	50	8.4	93.5	531
Struvite3	50	6.8	77.9	532
Ash	50	-15.6	-151.3	533
HC1	50	-4.4	-35.8	533
HC2	50	-4.2	-34.5	
HC3	50	-6.0	-50.2	
Fe-DPS	50	-11.2	-106.4	

¹Struvite 4 was not used in the spring wheat trial.

Abbreviations used in table: APR=apparent phosphorus recovery; P-MFE=phosphorus mineral fertiliser equivalent value; HC=hydrochar; DPS=dairy processing sludge