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Research Paper

Particle size determines the short-term phosphorus availability in biochar produced from digestate solids

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ABSTRACT

Biochar pellets produced from the solid fraction of manure-based digestates are rich in phosphorus (P) and may represent a P source that is easy to handle and suitable for transport to P-deficient regions. However, the effect of feedstock composition and particle size on P availability in this type of biochar remains unexplored.

To evaluate the effect of particle size on the short-term P availability in biochars derived from manure digestate solids, an incubation experiment was carried out, in which four biochars produced from digestate solids in powder and pellet form were incubated with three soils of low P content. The recovery of P in bicarbonate and water extracts was measured after 7 and 70 days of incubation. A subsequent pot experiment with barley on two of the soils was aimed at examining early crop recovery of P, comparing the effects of coarse and fine biochar particles.

Biochars from digestate solids had total P contents ranging from 12 to 63 g kg^{-1}. In all three soils, the recovery of P in water and bicarbonate extracts was lower after incubation with biochar pellets compared to powder, and P recovery remained constant or decreased slightly over time. Early shoot biomass and P recovery in barley were also higher when fine biochar particles were applied. The results suggest that particle size reduction improves the immediate availability of P in biochars produced from manure-based digestate solids.

1. Introduction

Large differences in phosphorus (P) surpluses exist at the subnational scale in northwestern European countries, with a surplus of P dominating in regions with intensive livestock farming and P deficits in regions with mainly cereal production (Klinglmair et al., 2015; Einarsson et al., 2020; Panagos et al., 2022). Thus, there is an urgent need to redistribute P in animal manure from regions with excess amounts of P relative to crop demand to regions with P deficits to level out the current imbalances of P supply to agricultural soils.

Transporting raw animal manure over long-distances is, however, energy- and cost-intensive (Liu and Wang, 2020), and it is therefore necessary to apply appropriate technologies to obtain a more transportable source rich in P. Mechanical separation of anaerobically digested animal manure concentrates P in the solid fraction (Tambone et al., 2017; Guilayn et al., 2019; Mazzini et al., 2020). Once dried, the solid digestate fraction can be used as an input material for pyrolysis (Peng et al., 2020). This process produces pyrolysis gas and/or bio-oil, which can be used for energy purposes, and biochar. The resulting biochar is rich in stable carbon (C) and can act as a long-term C sink (Matovic, 2011). The biochar produced from digestate also contains other elements, most notably P. In fact, the P content has been found to be higher in biochar than in the dried digestate (Tsai et al., 2018). This means that P can be effectively concentrated in the biochar, making it a suitable material for transport to regions in need of additional P inputs. However, during the pyrolysis process, P can be converted to less available forms than in the original feedstock material (Uchimiya and Hiradate, 2014; Dai et al., 2015). This highlights the need for management strategies that improve P availability in biochar, with the aim of promoting the transition towards a circular economy for P.

Particle size management of the biochar can be of considerable importance for its P fertilizer potential. Several challenges are associated with fine-textured biochar, including high dust emissions during field operations (Gelardi et al., 2019), and mass loss during transportation,

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storage and soil application (Mohammadi, 2021). The use of biochar pellets may therefore ensure safer handling and reduced losses. However, lower agronomic efficacy with increasing particle size has been found for struvite (Degryse et al., 2017) and rock phosphate (e.g. Alston and Chin, 1974), but very little attention has been paid to the role of the biochar particle size and the interaction with the feedstock characteristics on P availability. Grinding of biochar ensures homogenization and a more even distribution in the soil, and this may be the reason why studies on the P availability of biochars produced from manure-based digestate solids have mainly been carried out on powdered material (e.g. Christel et al., 2014; Bruun et al., 2017; Rose et al., 2019; Alotaibi et al., 2021; Brod et al., 2022; Duboc et al., 2022). Only a few studies have investigated the effect of particle size on P availability for biochar derived from sewage sludge (Müller-Stöver et al., 2021) and dairy carcass (Ma and Matsunaka, 2013). To our knowledge, no studies have investigated this for biochar produced from digestate solids. Thus, it is necessary to thoroughly investigate the effect of particle size on the availability of P in this type of biochar to ensure efficient recycling of manure-derived P.

Apart from particle size management, there may be other options for treating biochar materials to further improve their P availability. Acidification of finely ground biochar has recently been shown to improve plant growth and P uptake, attributed to solubilization of P associated with Ca (Kopp et al., 2023), but there is still uncertainty as to whether this also applies to P extractability from biochar pellets.

The objective of this study was to investigate the short-term availability of P in biochars produced from solid fractions of animal manure-based digestates as affected by particle size and feedstock composition. This was explored in an incubation study (up to 70 days) with three soils of low P concentration but different textural composition, and in a subsequent pot study with spring barley on two of the three soils to test effects on early growth. We hypothesized that short-term P availability would be lower in soils amended with coarse biochar particles than in soils with fine particles.

2. Materials and methods

2.1. Biochars

Four biochars produced from the solid fraction of digestates deriving from animal manure-based anaerobic digestion processes supplied with various co-substrates at three different biogas plants in Denmark were included in the initial characterization. Different separation technologies of the digested biomass were used (Table 1). The digestate solids were pelletized and then pyrolyzed at process temperatures between 560–600 °C in a large-scale pilot plant or in a smaller test plant in Denmark. A subsample of the biochar based on the solid fraction from Vesthimmerlands Biogas (BC_Vesth) was acidified with 1.5 *M* sulphuric acid (H_2SO_4) and used in the incubation experiment. Two biochars obtained from the pyrolysis of sewage sludge were included for comparison. The biochars were oven-dried at 40 °C to enable storage.

2.2. Characterization of the biochars

The dry matter (DM) content of the biochar was determined after drying at 105 °C.

The pH of the biochar samples was measured with a glass electrode after suspending 10 mL of air-dried, crushed product in 25 mL of a 0.01 *M* $CaCl_2$ solution for 1 h. For all other analyses performed directly on the biochars, the products were subjected to ball milling before analysis.

Total P contents were determined by digesting 0.1 g of biochar with 1 ml of concentrated H_2SO_4 and $HClO_4$ at 250 °C. Ortho-P concentration in the digested solutions was determined by spectrophotometry using the molybdate-blue reactive method for water samples after appropriate dilution (ISO, 2004), following the principle originally described in Murphy and Riley (1962). Total iron (Fe), aluminum (Al), calcium (Ca), potassium (K), magnesium (Mg), manganese (Mn) and sodium (Na) in the digestates were measured by ICP-OES (Thermo Fisher Scientific iCAP 6000, Waltham, MA). Biochar total nitrogen (N) and C contents were determined on 200 mg samples using dry combustion at 950 °C for 11 min with an elemental analyzer (vario MAX cube, Elementar Analysensysteme, GmbH, Hanau, Germany).

Bicarbonate extraction was performed on 0.1 g biochar in 20 ml 0.5 *M* $NaHCO_3$ (pH 8.5), shaken end-over-end for 30 min followed by centrifugation at 3000 rpm for 5 min. The supernatant was then filtered through 0.45 μm syringe filters. Two ml of the filtrate were added to 8 ml of H_2O and digested with 0.5 ml of sulphuric acid (H_2SO_4) and 1 ml of perchloric acid ($HClO_4$) for 1 h at 130 °C and for 8 min at 255 °C, before determination of the *ortho*-P-concentration in the filtrate by spectrophotometry as described above.

Water-extractable reactive P (WEP_r) was determined by pre-wetting 0.1 g biochar with 1 ml deionized water for 24 h. The sample was then mixed with 49 ml deionized water and shaken end-over-end for 1 h. The sample was centrifuged at 5000 rpm for 10 min and filtered (0.45 μm),

Table 1
Overview of the biochar products tested in the study.

Biochar	Pyrolysis plant in Denmark	Pyrolysis temperature	Retention time	Biogas plant	Feedstock and form	Pre-treatment
BC_Vesth	Large-scale pilot plant.	600 °C.	30 min, slow pyrolysis	Vesthimmerland Biogas.	Solid fraction of digested biomass consisting of mainly deep litter from dairy farming and straw from seed grass. Pellets.	Mechanical separation of digested biomass by screw press.
BC_Vesth_b	Test plant.	560–575 °C.	3–5 h, slow pyrolysis	Same as above.	Same as above.	Same as above.
BC_BB	Same as above.	560–575 °C.	3–5 h, slow pyrolysis	BB Bioenergi.	Solid fraction of digested biomass consisting of mainly pig slurry (36 %) deep litter from dairy farming (20 %); straw (23 %)*. Pellets.	Mechanical separation of digested biomass by screw press.
BC_Maabj	Same as above.	560–575 °C.	3–5 h, slow pyrolysis.	Maabjerg Energy Center.	Solid fraction of digested biomass mainly consisting of slurry and industry waste. Pellets.	Mechanical separation of digested biomass by decanting centrifuge.
BC_SS_1	Small-scale test plant.	630 °C.	Slow pyrolysis.	–	Sewage sludge from municipal wastewater.	The sewage sludge was chemically treated with Fe ($FeCl_3$ or $FeSO_4$). Before pyrolysis, the sewage sludge was dried at 250 °C by steam drying to a dry matter content of 95–98 %.
BC_SS_2	Test plant.	650 °C.	30 min, slow pyrolysis.	–	Sewage sludge from municipal wastewater.	The sewage sludge was chemically treated with aluminum chloride to precipitate P.

* The remaining constituents of the feedstock are: Olives (6%), beet roots (6%), beet shoots (1%), poultry litter (2%), potato pulp (4%), corn residues (2%).

before determination of the reactive P in the supernatant. A subsample of the supernatant was digested with acid persulphate in an autoclave (120 °C for 30 min) after Koroleff (1983) to determine the content of total dissolved water-extractable P (WEP_t). Water-extractable unreactive P (WEP_u) was determined as the difference between WEP_t and WEP_r.

Extraction with hydrochloric acid (HCl) was performed by shaking 50 ml 0.5 *M* HCl with 0.1 g biochar end-over-end for 1 h before centrifugation at 5000 rpm for 10 min and filtration (0.45 μm). The filtrate was mixed with 2 *M* NaOH and water for neutralization before determination of *ortho*-P-concentrations by spectrophotometry as described above.

2.3. Soils

The incubation experiment involved three soils and the pot experiment two of these soils (Table 2). The soils were collected from the plough layer (0–25 cm) in arable fields near Koege and Haslev, Zealand, and near Oestervraa, Northern Jutland, respectively. The soils from Koege and Haslev represent common agricultural soils in the eastern part of Denmark (sandy loam), and the soil from Oestervraa was selected to include a more light-textured soil (loamy sand), where a response to P fertilization had previously been observed. A subsample of the composite soil sample from each site was air-dried and sieved (<2 mm) for determination of soil texture, pH, Olsen-P content (Olsen et al., 1954) and soil organic C (Table 2). All three soils had Olsen-P contents below 22 mg P kg $^{-1}$ soil, indicating a low soil P status according to Jordan-Meille et al. (2012).

2.4. Incubation experiments

Four biochars produced from digestate solids (BC_Vesth, BC_Vesth_aci, BC_BB and BC_Maabj) were applied both as pellets (particle size between 2 and 6 mm) and as finely ball-milled powder (particle size < 0.125 mm) in the three soils (Table 2) to test the effect of biochar particle size on P recovery in extracts after incubation as an indicator for P availability in the biochar. Ball-milled triple super phosphate (TSP, 20.8 % P) was also incubated for comparison.

The incubation method was originally described in Christiansen et al. (2020). Briefly, each biochar product (as powder or pellets) was carefully mixed with 10 g air-dried and sieved (<2 mm) soil at a concentration of 80 mg P kg^{-1} soil in 50 ml tubes. Each tube containing the soil and biochar mixture was wetted with 2 ml demineralized water and incubated at 20 °C for 7 or 70 days, respectively. In the bicarbonate extraction test, 25 ml of 0.5 *M* sodium bicarbonate (pH 8.5) were then added to the tubes and shaken on an end-over-end shaker for 1 h. Similarly, in the water extraction test, 23 ml of demineralized water were added to the tubes and shaken end-over-end for 2 h. For both extraction tests, the mixtures were then centrifuged at 5000 rpm (5087 *g*) for 10 min and filtered (0.45 μm) before determination of the concentration of *ortho*-P in the filtrates by spectrophotometry using the molybdate-blue reactive method as described in section 2.2. All incubation extraction tests were carried out in triplicate.

Table 2
Properties of the three soils used in the incubation and in the pot experiments.

		Haslev	Koege	Oestervraa
Soil type		Sandy loam	Sandy loam	Loamy sand
Clay (<2 μm)	g 100 g^{-1}	15.6	14.9	9.3
Silt (2-20 μm)	g 100 g^{-1}	10.5	11.6	9.8
Fine sand (20-200 μm)	g 100 g^{-1}	49.8	46.8	72.3
Coarse sand (200-2000 μm)	g 100 g^{-1}	24.1	26.7	8.7
Soil organic carbon	g 100 g^{-1}	2.0	1.7	2.5
pH ($CaCl_2$)		7.3	5.6	5.5
Olsen P	mg kg^{-1}	10	5	22
P_{ox}	mmol kg^{-1}	13	6.4	14
Al_{ox}	mmol kg^{-1}	40	32	77
Fe_{ox}	mmol kg^{-1}	28	30	51
P sorption capacity[A]	mmol kg^{-1}	34	31	64
P saturation degree[A]	%	38	21	22
Incubation study		x	x	x
Pot study			x	x

[A] Phosphorus sorption capacity determined as 0.5(Al_{ox} + Fe_{ox}) after Van Der Zee and Van Riemsdijk (1986) and P saturation degree as P_{ox}/PSC.

2.5. Pot experiments

A pot experiment with spring barley grown on two of the soils, i.e. Koege and Oestervraa, was performed under climate-controlled conditions to determine the short-term plant availability of the biochar P as affected by particle size. Three biochars produced from digestate solids (BC_Vesth, BC_BB and BC_Maabj) were used in the experiment. The biochars were crushed in a mortar and sieved to divide them into two fractions: a coarse one (particles between 0.25–2 mm) and a fine fraction (particles < 0.3 mm). Subsequently, the particle size distribution of the two fractions for each biochar was characterized by laser diffraction (Malvern Mastersizer 3000+, Table S1). For the fine fractions of the three biochars (BC_Vesth_fine, BC_BB_fine and BC_Maabj_fine), 50 % of the particles (D50) were smaller than 45, 107 and 130 μm, respectively. The coarse fractions (BC_Vesth_coarse, BC_BB_coarse and BC_Maabj_coarse) had DD50 values of 536, 531 and 535 μm, respectively (Table S1).

To reduce the number of samples to handle at sowing and harvest, the two soils were prepared and sown 14 days apart in the same growth chamber. Each soil was sieved to < 4 mm and mixed with sand (0.4–0.9 mm) purchased from Dansand Silkeborg A/S at a 2:1 soil:sand ratio to increase porosity and avoid crust-formation in the pots. The water holding capacity (WHC) was determined according to Wilke (2005) and the soil:sand mixtures were wetted to 30 % WHC. The following nutrients were added in solution to 1.2 kg soil:sand mixture for each pot: 200 mg N kg^{-1} (NH_4NO_3), 50 mg Ca kg^{-1} ($CaCl_2$), 150 mg K kg^{-1} (66.6 % KCl and 33.3 % K_2SO_4), 40 mg Mg kg^{-1} ($MgSO_4 \cdot 7H_2O$), 1.5 mg Cu kg^{-1} ($CuSO_4 \cdot 5H_2O$), 1.2 mg Zn kg^{-1} ($ZnSO_4 \bullet 7(H_2O)$), 0.1 mg Mo kg^{-1} ($Na_2MoO_4 \bullet 2(H_2O)$), 3 mg Fe kg^{-1} (Fe-EDTA), 0.3 mg B kg^{-1} (H_3BO_3) and 3 mg Mn kg^{-1} ($MnSO_4 \cdot H_2O$). The soil was stored in open plastic bags for one week before P fertilizer addition. The coarse and the fine fractions of the three biochars (BC_Vesth, BC_BB and BC_Maabj) were then applied based on their total P content. For the BC_BB and BC_Maabj biochars, the P application rate was 60 mg P kg^{-1} soil, whereas BC_Vesth biochar was erroneously added at 120 mg P kg^{-1}. As a positive control treatment, 75 mg P kg^{-1} were added as ground TSP (18 % P). A treatment without P fertilization was included as a negative control. In each type of test, the biochars and mineral P fertilizer were mixed homogeneously into the entire soil volume by shaking in plastic bags before transferring to 1 L pots (12.5 cm inner diameter) and increasing the water content to 60 % WHC. For each type of treatment tested, four replicate pots were prepared. Three spring barley seeds (*Hordeum vulgare* L., cv. Prospect) were sown in each pot and thinned to 2 plants pot^{-1} after one week. Pots were rotated weekly and watered three times per week to weight to maintain a water content of 60 % of WHC. Growth chamber settings were 16 h daytime at 21 °C and a light intensity of 300 μE, and 8 h of darkness at 16 °C. After 35 days, the above-ground biomass was harvested and weighed after drying for 72 h at 65 °C. The biomass was ground using a zirconium ball mill and the P concentration was determined by the dry ashing method of Saunders and Williams (1955) followed by the molybdate-blue method (Murphy and Riley, 1962) using flow injection analysis (FIAstar 5000 analyser, Foss, Hillerød, Denmark). After shoot harvest, the pots were emptied onto a tray and a soil sample was collected from the bulk soil, and pH was measured in $1{:}5_{w/v}$ 0.01 M $CaCl_2$.

2.6. Calculations and statistical analyses

Recovery of P in extraction tests with water or bicarbonate after incubation was calculated as:

$$\text{Recovery of P, \%} = \frac{\text{P extracted} - \text{P extracted in unfert. control}}{\text{P applied with biochar/TSP}} \text{x} 100 \quad (1)$$

To assess the short-term plant P availability in biochar, crop recovery efficiency (RE) of P from added biochar was calculated using the difference method (Johnston et al., 2014):

$$RE, \ \% = \frac{PU_{fertilized} - PU_{unfertilized}}{Amount\ of\ P\ applied} x100 \quad (2)$$

where PU is the P uptake in the barley shoot.

For the statistical analysis, the R-project software package Version 4.2.1 (R Development Core Team, 2021) was employed. A three-way analysis of variance (ANOVA) was performed to test the effect of soil type, incubation time and biochar type on the recovery of P extracted with bicarbonate and water after incubation. A one-way ANOVA was also applied to study the effect of fertilizer treatments on biomass, plant P recovery and pH for each soil type in the pot experiment. To perform multiple comparisons, Tukey's honestly significant difference (HSD) test was used. The assumption of homogeneity of variances and normality of residuals was tested using plots of residuals against fitted values and histograms of residuals.

Significance was declared at the $p \leq 0.05$ level of error probability.

3. Results

3.1. Biochar properties and P extractability

All products, except the acidified biochar, were alkaline (Table 3). The degree of alkalinity depended on the feedstock material, with the highest pH found in the biochars obtained from the treatment of manure-based digestate (pH between 9.4 and 9.9), whereas biochars from sewage sludge had a pH of 8. Phosphorus contents in biochars produced from digestate solid fractions varied from 12 to 63 g kg^{-1} (Table 3), with the highest P content in biochar BC_Maabj, where the digestate was separated by a decanting centrifuge. The two biochars produced from sewage sludge (BC_SS_1 and BC_SS_2) had higher contents of Al and Fe than the biochars produced from digestate solids. Carbon contents ranged from 30.8 to 62.5 g 100 g^{-1}, with the highest content in the biochar produced from digestate from Vesthimmerlands biogas (BC_Vesth).

The proportion of P extracted directly from the products varied depending on the extraction methods and biochar materials (Table 4). For biochars produced from the solid fraction of digestates, 4 to 40 % of total P was extracted with water, with acidified biochar produced from digested biomass at Vesthimmerlands Biogas (BC_Vesth_aci) having the highest proportion of water-extractable P. Biochars produced from sewage sludge had the lowest proportion of water-extractable P (below 2 %). Bicarbonate extracted 0.6 to 42 % of total P, with the highest extractability found for biochar produced from digestate solids at Vesthimmerlands Biogas (18.5–42.0 % of total P, Table 4). The HCl-P method extracted 35 to 87 % of the total P, with most of the P extracted in biochars produced from digestate solids from Vesthimmerlands Biogas (85–87 %). The digestate solids produced at Vesthimmerlands Biogas were pyrolyzed in two plants with slightly different pyrolysis temperatures (Table 1, BC_Vesth and BC_Vesth_b), but P extractability was similar for these two biochars.

Table 4
Extractable phosphorus measured in the biochars using different extraction methods (mean ± std, $n = 4$).

Biochar	Total P	P-water (reactive)	P-water (unreactive)	P-bicarb	P-HCl
	g kg^{-1} DM	% of total P	% of total P	% of total P	% of total P
BC_Vesth	22.8 ± 0.02	10.2 ± 0.53	0.2 ± 0.12	20.1 ± 0.1	86.9 ± 1.0
BC_Vesth_b	21.4 ± 0.03	11.9 ± 0.23	0.1 ± 0.09	18.5 ± 0.4	82.5 ± 0.8
BC_Vesth_aci	20.9 ± 0.01	40.1 ± 0.75	0.1 ± 0.73	42.0 ± 1.0	85.3 ± 0.3
BC_BB	12.0 ± 0.01	6.3 ± 0.04	0.0 ± 0.04	8.4 ± 0.2	55.6 ± 0.5
BC_Maabj	62.6 ± 0.05	4.0 ± 0.19	0.2 ± 0.06	11.1 ± 0.1	35.0 ± 2.6
BC_SS_1	49.1 ± 0.03	0.2 ± 0.01	0.0 ± 0.00	0.6 ± 0.4	45.1 ± 0.4
BC_SS_2	47.6 ± 0.04	2.1 ± 0.04	0.0 ± 0.04	2.9 ± 0.1	62.1 ± 3.3
TSP*	192.3	92.5	–	67.7	100

*data from Christiansen et al. (2020) included for comparison.

3.2. Recovery of P in bicarbonate and water extracts after incubation with three soils

The amount of P extracted with bicarbonate was affected by the soil type, with more P being recovered from the sandy loams (Haslev and Koege) than from the loamy sand (Oestervraa, Fig. 1). Recovery of P after water extraction was in general higher on the Koege soil as compared to the Haslev and Oestervraa soils, which presented very low recovery rates in many cases (Fig. 2).

For biochars BC_Vesth and BC_Maabj, P recovery with both water and bicarbonate extractions was affected by biochar particle size, with significantly lower recovery of P after incubation in pellets than in powder on all three soils. The highest P recovery rate was achieved with the acidified biochar (BC_Vesth_aci) applied as powder, with up to 12 % P recovered with bicarbonate on the Haslev soil after 7 days (Fig. 1).

Table 3
General characterization of the biochars ($n = 4$, except for pH ($n = 3$) and total N and C ($n = 2$)). The concentrations of elements are expressed on a dry matter (DM) basis.

Biochar	pH ($CaCl_2$)	Total contents of C	P	N	Fe	Al	Ca	K	Mg	Mn	Na	Water content	Original form	Ca:P molar ratio	C:P molar ratio
		g 100 g^{-1} DM					g kg^{-1} DM					g 100 g^{-1}			
BC_Vesth	9.9	60.1	22.8	13.7	7.8	2.9	35.2	20.9	16.8	0.8	20.8	30	Pellets	1.2	68
BC_Vesth_b	9.4	62.5	21.4	19.2	8.5	3.1	37.4	13.2	17.3	0.8	14.7	31	Pellets	1.4	75
BC_Vesth_aci	6.0	56.6	20.9	12.8	7.3	2.7	32.6	19.6	15.5	0.8	20.0	1.2	Pellets	1.2	70
BC_BB	9.5	60.0	12.0	14.2	4.9	1.9	27.4	31.5	10.1	0.4	6.4	29	Pellets	1.8	129
BC_Maabj	9.5	43.6	62.6	22.9	12.6	2.5	76.4	19.2	46.8	1.0	7.4	22	Pellets	0.9	18
BC_SS_1	7.9	30.8	49.1	36.0	96.0	20.8	35.6	9.9	8.4	0.7	2.0	11	Crushed	0.6	16
BC_SS_2	8.0	31.6	47.6	31.3	26.6	53.0	52.4	12.8	11.9	0.4	3.8	28	Crushed	0.9	17

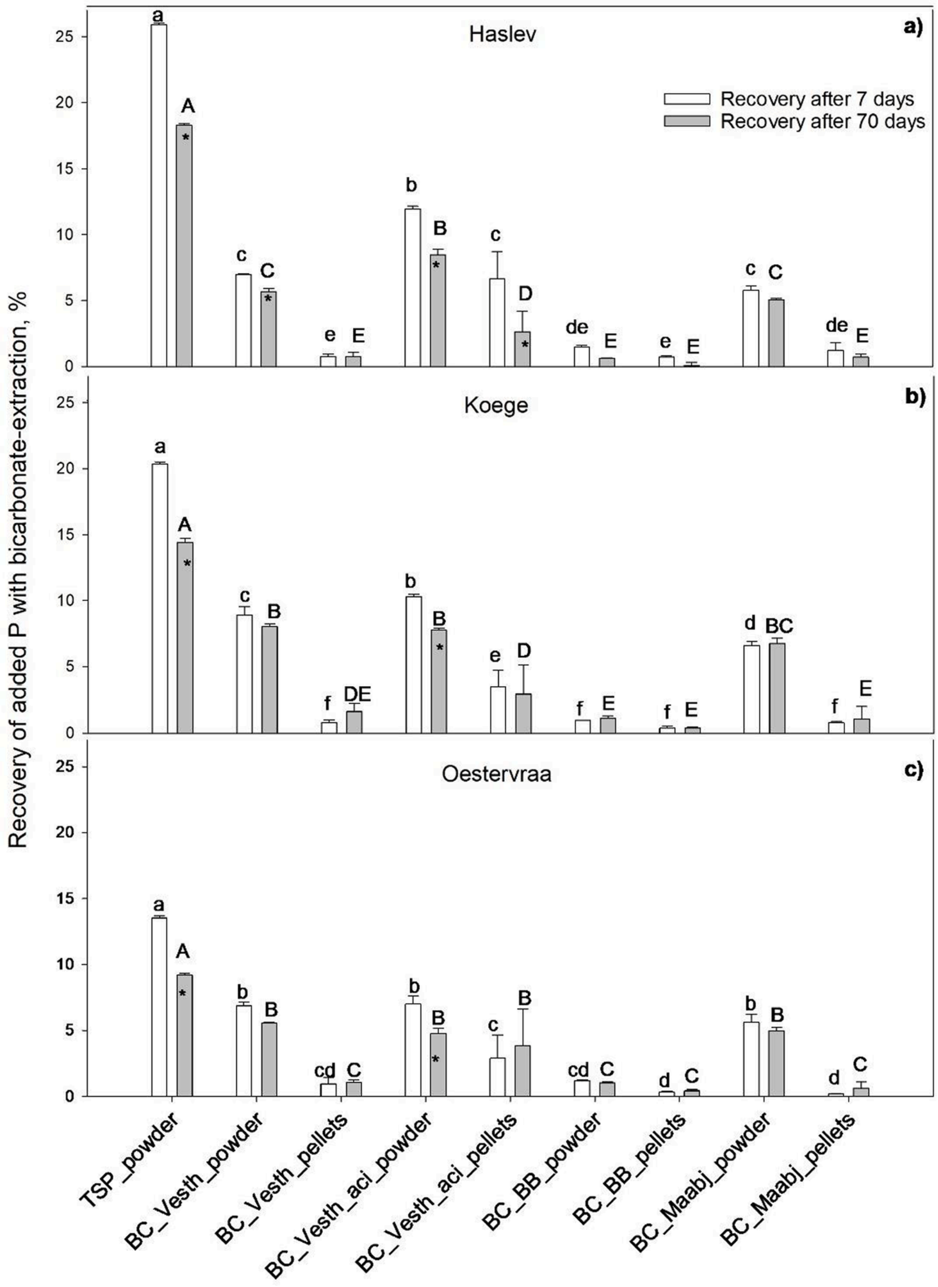

Fig. 1. Recovery of added biochar P after extraction with 0.5 *M* bicarbonate in three soils; a) Haslev, b) Koege and c) Oestervraa after 7 and 70 days of incubation. Significant differences between the biochars for each incubation time are indicated by different letters (lower case = 7 days incubations and upper case = 70 days incubations). Asterisks (*) indicate significant differences in P recovery for each biochar between 7 and 70 days of incubation. Error bars represent the standard deviations.

Biochar BC_BB presented the lowest recovery of P with water and bicarbonate on all three soils, and reducing the particle size did not improve P extractability for this biochar product.

For the biochars produced from the digestate solids and the TSP treatment, the recovery of P in bicarbonate and water extracts decreased with time (from 7 to 70 days) in most cases (Figs. 1 and 2).

3.3. *Early P recovery in barley and soil pH after biochar additions*

Plant growth in soil without added P was higher for the Oestervraa soil than for the Koege soil, but both soils responded to mineral P addition (TSP) with at least a doubling of the shoot biomass produced (Fig. 3). On the Koege soil (sandy loam), the addition of biochar from Vesthimmerland (BC_Vesth) at a rate of 120 mg P kg^{-1}soil resulted in a significant increase in shoot biomass compared to the treatment without P addition, and the finely ground material (BC_Vesth_fine) ensured the same biomass production as the reference treatment with TSP. The other two biochars with an application rate of 60 mg P kg^{-1} soil (BC_BB and BC_Maabj) only gave significantly higher biomass than the control without P addition when applied as finely ground material, and the small-sized biochar from Maabjerg (BC_Maabj_fine) performed as well as TSP. Among the biochar treatments, the shoot P recovery was highest when the two finely ground biochars (BC_Vesth_fine and BC_Maabj_fine) were applied (Fig. 3).

On the Oestervraa soil (loamy sand), biomass was significantly increased in all biochar treatments compared to the reference without P fertilization. The particle size of the biochars from digestates produced at Vesthimmerland and BB Bioenergi biogas plants (BC_Vesth and

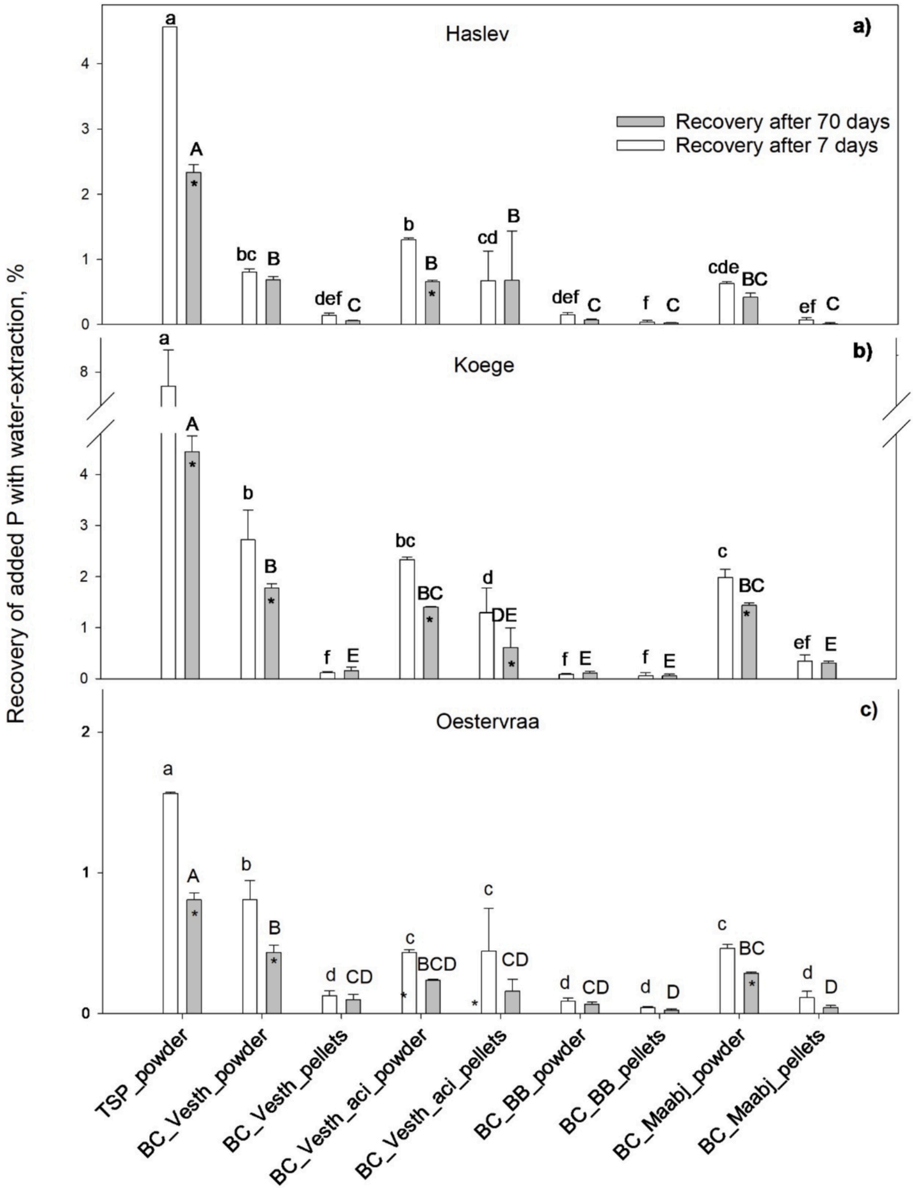

Fig. 2. Recovery of added biochar P after extraction with water in three soils; a) Haslev, b) Koege and c) Oestervraa after 7 and 70 days of incubation. Significant differences between the biochars for each incubation time are indicated by different letters (lower case = 7 days incubations and upper case = 70 days incubations). Asterisks (*) indicate significant differences in P recovery for each biochar between 7 and 70 days of incubation. Error bars represent the standard deviations.

BC_BB) did not influence the early shoot biomass. Particle size reduction of biochar from Maabjerg (BC_Maabj_fine) resulted in a significantly higher shoot biomass and enhanced P-recovery than the coarser biochar particles (BC_Maabj_coarse).

On both soils, P recoveries in shoots were low in response to the application of the biochar BC_BB (regardless of particle size) compared to the other two biochars.

On both the Koege and the Oestervraa soils, pH increased after plant harvest in the soil amended with the biochar produced from digestate solids from Vesthimmerlands Biogas (BC_Vesth) compared to the soil with TSP addition (Fig. 4). In general, the pH increases were more pronounced for the Koege soil (sandy loam) than for the Oestervraa soil (loamy sand), and the pH increased more after the application of finely ground biochar compared to the coarse one for the Koege soil.

4. Discussion

4.1. Short-term P availability as influenced by biochar particle size

Phosphorus contents varied from 12 to 63 g kg^{-1} in biochars produced from digestate solids. Thus, some of the biochars considered in the present study had a relatively low P content as compared to other biochars produced from digested manure solids studied by Kuligowski et al. (2010); Christel et al. (2014); Bruun et al. (2017), with total P contents ranging from 5.3 to 6.6 %. Pyrolysis of solids separated by a decanter centrifuge resulted in the highest biochar P content, confirming that the mechanical separation method applied to the digestate influences the P concentration in the solid fraction (Lyons et al., 2021).

The P extractability of biochars produced from manure-based digestate solids differed considerably despite similar pyrolysis temperatures. Water-extracted P represents dissolved P readily available for plant uptake, whereas P extracted with bicarbonate represents weakly adsorbed inorganic P and easily hydrolysable organic P compounds

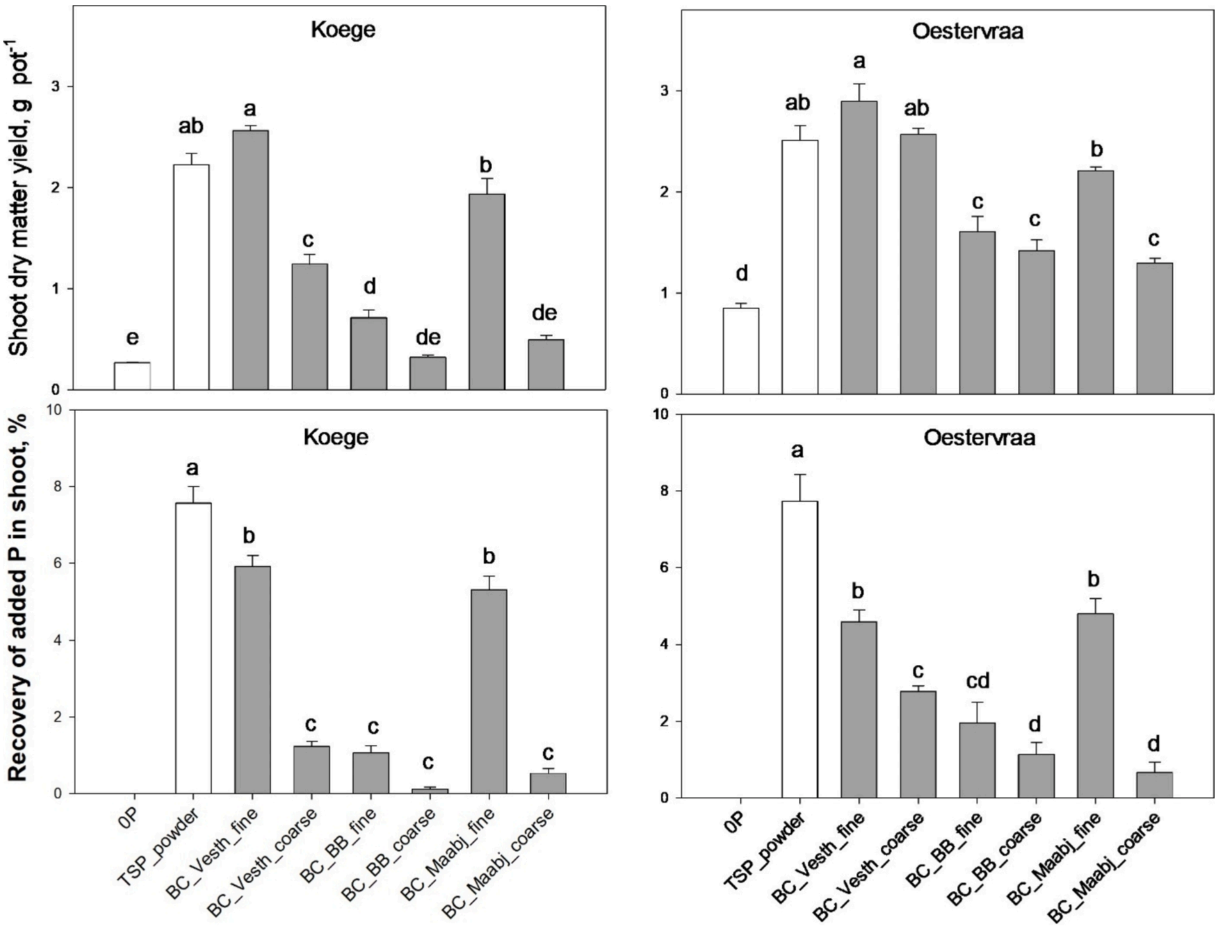

Fig. 3. Shoot dry matter yield (g pot^{-1}) and P recovery (%) in spring barley on two different soils (Koege and Oestervraa) after application of biochars produced from solid fraction of animal manure-based digestates as either coarse or fine particles. References with mineral P (TSP) and without P were included for comparison. Significant differences between treatments are indicated by different letters (Tukeýs HDS, $p < 0.05$).

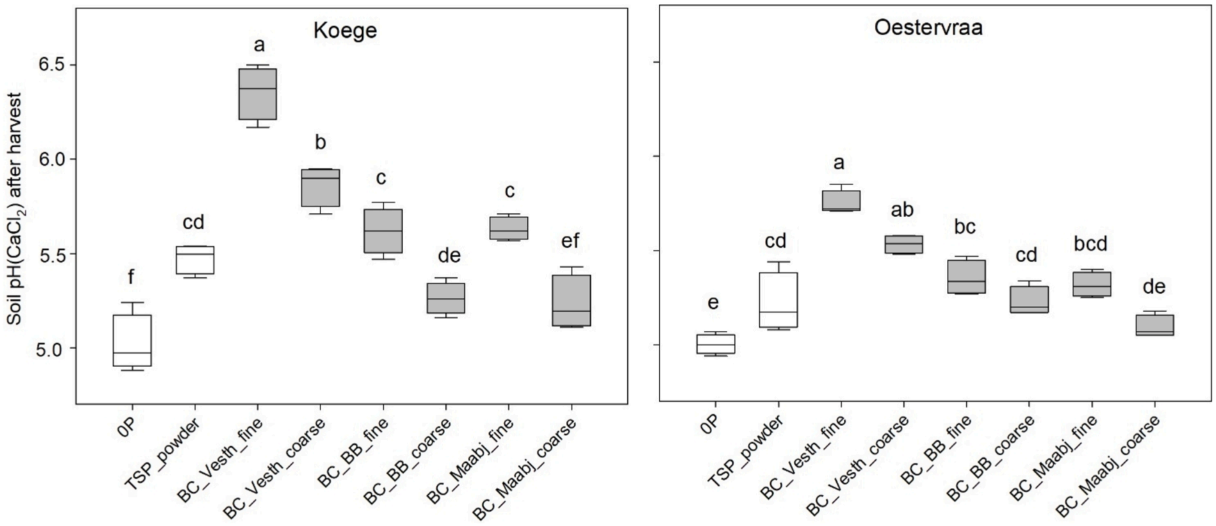

Fig. 4. Soil pH($CaCl_2$) measured after plant growth on the Koege and Oestervraa soils as affected by application of biochars produced from the solid fraction of animal manure-based digestates as either coarse or fine particles. References with mineral P (TSP) and without P (0P) were included for comparison. Significant differences between treatments are indicated by different letters (Tukeýs HDS, $p < 0.05$).

(Kratz et al., 2019). Biochar BC_Vesth had a high proportion of both water (>10 %) and bicarbonate (>20 %) extractable P, which was further increased by acidification with sulfuric acid (Table 4). Kopp et al. (2023) also report an increase in plant available P following acidification of P-rich digestate biochar, which was mainly ascribed to solubilization of hydroxyapatite.

Some studies have suggested that the proportion of total P extracted with water (Rose et al., 2019) or bicarbonate (Brod et al., 2022; Duboc et al., 2022) directly from the material provides the best prediction of its mineral fertilizer replacement value. However, our results are not in line with these observations. For example, biochar BC_BB contained more water extractable P than biochar BC_Maabj measured directly on the material, but the plant P availability of the two materials was very different and higher for biochar BC_Maabj (Fig. 3). Similarly, the incubation study revealed that the recovery of P with bicarbonate and water extraction was significantly reduced in all three soils when pellets were applied, even for biochar BC_Vesth (both untreated and acidified) with high contents of both water- and bicarbonate-extractable P measured directly on the product. This suggests that the biochar particle size had a major impact on short-term P availability. The importance of particle size was also reflected in the early growth of barley, where the addition of fine biochar particles improved plant biomass and P recovery in most cases; whereas early growth was less positively affected when biochars with larger particle sizes were applied (Fig. 3). The degree of contact between soil and biochar P is reduced with increasing particle size (Degryse et al., 2017). The current results confirm that the fertilizer-soil interface area is critical for P exchange. The distribution of fine biochar particles over a larger soil volume is also likely to result in a shorter distance between the biochar and the roots, thereby improving the plańts access to the nutrients in the biochar. Thus, to achieve the full initial P fertilizer potential, biochar should preferably be applied as a finely ground material.

This study also showed that fine biochar particles provided a significantly higher initial liming effect than the coarser biochar particles on the Koege soil (sandy loam) for all three biochars. Biochar is generally considered to present a relevant liming value on soils with pH levels below 6.5. The liming effect is attributed to the dissolution of carbonates and alkaline oxides in the ash fraction contained in the biochar (Glaser and Lehr, 2019; Bolan et al., 2023). The observed differences in soil pH effects among the biochars tested may therefore be attributed to different contents of inorganic C. This was not determined in the present study, but previous research has demonstrated that inorganic C content can reach up to 2.8 % of dry weight in biochars derived from manure (Lataf et al., 2022). It is therefore essential that future studies include these measurements to elucidate the underlying mechanistic explanation for the different effects on soil pH among biochars produced from manure-based digestate solids. With regard to the influence of particle size on the liming effect, fine particles of calcitic liming materials (Heinrich et al., 2022) and biochar produced from spent mushroom substrate (Sarfraz et al., 2020) also demonstrate elevated liming potential, suggesting that small-sized particles may be more reactive, thus also more efficient at releasing alkaline ions than larger particles. The alteration of soil pH levels through biochar application can also indirectly affect the availability of P. For acid soils, a pH increase will weaken P retention as the variable charge mineral surfaces become less positive, (Barrow, 1983). However, P can also precipitate with Ca at high pH levels (Haynes, 1982), and the relative importance of the two mechanisms will depend on soil properties, the Ca content of the applied biochar, etc.

The biochar BC_BB presented the lowest recovery of P with water and bicarbonate after incubation with all three soils, regardless of particle size (Figs. 1 and 2). This biochar product also resulted in a lower shoot biomass production and P recovery in spring barley than biochar BC_Maabj, despite the same P application rate and similar contents of water-extractable P measured directly on the material. Phosphorus in biochars produced from digestate solids is mainly associated to Ca such as hydroxyapatite and di-and tricalcium phosphates (Bekiaris et al., 2016; Bruun et al., 2017; Huang et al., 2018). The BC_BB biochar had the highest molar Ca:P ratio (Table 3), which may indicate that the formation of stable Ca-phosphates was more dominant for this biochar. Biochar BC_BB also had the highest C:P ratio (Table 3), which may be related to the high proportion of straw in the input material to the biogas plant (Table 1). In some cases, aromatic C fractions, which tend to increase in pyrolyzed biomass, can incorporate a significant proportion of P thus limiting the physical access to the P (Robinson et al., 2018). This may also explain the very low P recovery in shoots and soil extracts of this biochar product (Fig. 3).

4.2. *Availability of P in biochar over time*

The incubation study showed that the recovery of P in soil extracts in the treatments with biochars produced from digestate solids and with TSP remained constant or decreased with time (from 7 to 70 days) for all three soils (Figs. 1 and 2). These results suggest that the P contained in the biochar may be sorbed onto the soil mineral phase over time in less-available pools that are not extractable with bicarbonate or water. Two of the three soils had a relatively low P saturation degree (Koege and Oestervraa, Table 2) in comparison to Danish agricultural soils, which have an average P saturation of 32 % (Rubæk et al., 2013). The low saturation degree of the Koege and Oestervraa soils may provide available sorption sites for P released from the biochar. The biochar itself can also have a considerable P sorption capacity (Rodríguez Alberto et al., 2021), and the decrease in P recovery over time suggests that slow sorption processes may occur onto both biochar and soil. A number of studies suggest that biochar can act as a slow-release P fertilizer (e.g. Wang et al., 2014; Glaser and Lehr, 2019; Li et al., 2020), but this may be highly dependent on interaction effects between soil properties and biochar. For instance, Bruun et al. (2017) report that P availability after biochar addition increased with time in a soil with a clay content of 11.5 %, whereas the P availability remained at a low level in a soil with a clay content of 23 %. The current study suggests that P contained in biochar can be converted into more firmly held forms in sandy loam and loamy sand soils with a low P saturation degree within a growing season (70 days). It is therefore important that plants can act as a sink for P released from biochar to avoid the build-up of biochar P in less available soil pools.

The present study assessed P availability in biochar within a relatively short growth and incubation period. The recovery rate of P from biochar after incubation was of up to 12 %, confirming that much of the applied P enters the less available soil pools. It is therefore important to complement the current findings with studies on the residual P effects of biochar as affected by particle size to determine its long-term fertilizer value. Furthermore, field studies on crop growth after biochar application over a full season are needed to assess the implications for final yields where P application rates with biochar represent typical agricultural practices.

4.3. *Implications for biochar management*

The current study emphasizes the importance of testing biochar not only as powder but also in its final pellet form to accurately ascertain its potential as a P fertilizer. Finely ground biochar had the greatest initial P availability. However, small biochar particles can be lost through macro pores in the soil matrix (Rumpel et al., 2015), and powdered biochar is less easy to handle during transport and field application processes (Gelardi et al., 2019). Thus, there may be a trade-off between maximizing initial P recovery from finely ground biochar and, on the other hand, minimizing loss of biochar particles and ensuring safe handling during field operations. Moistening the biochar powder could be an option to reduce the risk of mass loss through dust. It is also noteworthy that P in biochar pellets may become available at a later stage as the pellets disintegrate. It would therefore be relevant to study the factors affecting pellet disintegration, such as soil water content and tillage operations.

5. Conclusion

This study aimed to assess the initial P availability of biochars produced from the solid fraction of animal manure-based digestates as affected by particle size and feedstock composition. The chemical characterization performed directly on the products showed that the extractability of P differed considerably between biochars produced from digestate solids, with the highest proportion of water- and bicarbonate extractable P found in acidified biochar. However, the P characterization performed directly on the powdered material did not take into account the effect of particle size on P availability in biochar. Incubation of three soils with four of the biochars revealed that less P was recovered in bicarbonate and water extracts, when biochar was applied as pellets than as fine powder. This was also demonstrated for early growth of barley, where the application of small biochar particles resulted in higher biomass and P recovery in the shoots for most biochars. Therefore, to achieve the greatest immediate P fertilizer effect of biochar produced from digestate solids on soils with low P level, the preferred approach is to apply it as fine particles. Optimized particle size management will contribute to a more efficient recycling of biochar P produced from surplus manure when applied to P-deficient soils.

CRediT authorship contribution statement

Ingeborg F. Pedersen: Conceptualization, Formal analysis, Investigation, Visualization, Writing -original draft, review & editing. **Dorette S. Müller-Stöver:** Writing – review & editing, Conceptualization. **Camilla Lemming:** Writing – review & editing, Resources. **Klara Cecilia Gunnarsen:** Writing – review & editing, Visualization, Investigation, Formal analysis, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary material

Supplementary data to this article can be found online at https://doi.org/10.1016/j.wasman.2024.11.006.

Data availability

Data will be made available on request.

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