



OPEN Sorption site competition determines phosphorus availability of agrifood residues

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Phosphorus (P) extractability, and thus plant-availability, in processed agrifood residues incorporated in soil is not directly related to the extractability before the incorporation. However, the release of P has not been demonstrated over seasons, and the mechanisms involved are not well understood. We identified the fate of P fractions in manure and sewage sludge processed with current methods after incubation for two weeks, 2.5 months and 12 months in sandy loam or clay, using a modified Hedley fractionation scheme. We also identified the sorption of P to soil, and the determinants. The changes in readily extractable P since the incorporation ranged in sandy loam from – 23% to + 88% and in clay from – 49% to + 31%. Incorporation of manure and synthetic fertilizer led in soil to a clearly lower P extractability than sewage sludge. Precipitation of P in sewage with molar Fe/P 1.6 increased P extractability relative to biological precipitation (Fe/P 0.2) and molar Fe/P 9.0. Readily extractable P increased most with sewage sludge hygienized by acid and oxidizer, and next by anaerobic digestion only in clay but not with manure. The differences in P extractability were explained by competition for P sorption of varying capacity and intensity. The amount of added organic matter in residues, rather than of iron oxides and hydroxides, appeared as the consequently explaining factor to P sorption, increasing the sorption irrespective of the processing method.

Keywords Phosphorus, Acid and oxidizer, Manure, Sewage sludge, Soil sorption capacity, Soil

As the global population grows, so does the food demand. Agricultural production is the main utilizer of nutrients¹ and dependent on them. Conversion of inert phosphorus (P) to a reactive form already exceeds the carrying capacity of the Earth processes² and the pristine P resources are limited. Returning organic residues of agrifood systems to soil as a source of P for plant production and to enable carbon sequestration³ reduces further conversion of inert edaphic P as well as land degradation and thus strengthens food security. Processing of agrifood residues is under rapid development but the impact on the ultimate plant-availability of P when incorporated in soil is not known.

Organic agrifood residues such as manure and sewage sludge often contain P in sufficient amounts to compensate for the P exported in the harvest from a region^{1,4–6} and even provide an excess to be exported⁷. Currently, one of the main bottle necks for efficient utilization of P from organic residues is the unknown release rate of P, especially in the long term. The rate of P release varies among different organic residues where sewage sludge and manure are considered to represent the low and high extremes⁸. In addition, the release rate may depend on the means of P precipitation in sewage and on the energy recovery and residue hygienization processes in various agrifood residues^{9,10}. In sewage sludge P is precipitated using Fe coagulants or biologically. The Fe/P varies, but 1.5 to 4 is globally common. Sewage sludge is commonly hygienized using anaerobic digestion (AD), composting (Comp), lime-stabilizing (Lime) or oxidizer and acid (Kem). Manure is mostly used untreated or composted, but AD is becoming more common for energy recovery.

The release rate of P from organic residues after application into the soil is determined by (1) the readily extractable, i.e., labile, organic P of the residue, (2) microbial breakdown of the organic matter that contains P, and (3) the reactions of the organic residues with soil¹¹ depending on soil pH and other sorption properties. The sorption of P readily in the inorganic form or released from organic residues onto soil particles through ligand exchange is a relatively fast reaction, and the exchange equilibrium strongly favors sorption of P onto the solid phase over P release into soil water. Consequently, large concentrations of soluble P can be quickly retained, or alternatively leached if the sorption capacity or plant uptake is not sufficient. A continuous release of smaller

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quantities from mineralizable nutrients allows a greater window for plant uptake, as noted by earlier research for both nitrogen (N)¹² and P¹³. Organic matter also provides P sorption sites. On the other hand, dissolved organic carbon and humic and fulvic acids may compete with P for the soil sorption sites hindering P sorption¹¹ and even cause a net desorption^{14,15}.

While composting and anaerobically digesting (AD) sewage sludge, iron (Fe) or aluminum (Al) added into the sewage to precipitate P may be chelated by organic substances. Contrary to manure, P in sewage sludges is bound in organomineral complexes in addition to organic matter. Low redox potential¹¹, as well as very high or very low pH in the residue¹⁵, increase the extractability of P from Fe hydroxides. For manures the microbial breakdown of organic matter is the main route of release, regulated by available nutrients, oxygen and moisture. Sorption capacity and intensity are enhanced by greater surface area of soils with fine texture, and by high occurrence of Fe and Al hydroxides.

The impact of the current processing methods of organic agrifood residues on P availability to plants has been recently studied in a growth chamber¹¹ and in the field for one season¹⁶, indicating that the residue-soil interaction is of primary importance for P release. However, the longer-term role and mechanisms of this interaction are poorly understood¹⁷ and have not been directly determined. Consequently, we investigated the release and soil sorption of P from organic residues processed with methods currently in use two contrasting field soil types during incubation of varying lengths reaching one year to demonstrate a duration beyond one season. We posed the following research question: How do temporal changes of soil P fractions after residue incorporation depend on residue type, and processing method (precipitation and hygienization) in sandy loam and clay soil, and to what extent does soil sorption of P explain the dependence? The answers will enable the choice of processes which ensure the greatest P availability to crops in agrifood residues when incorporated in soil and thus reduce the use of pristine P sources and environmental burden.

Results

Two complementary experiments were conducted. First, the P release and retention from the residues when brought in interaction with soil were quantified in a long-term incubation experiment with no plants in sandy loam and clay. Second, the role of sorption as the potential mechanism determining the P release and retention in the interaction was quantified in a sorption experiment. P release was determined using a modified Hedley fractionation to observe the changes in P fractions during the incubation. Readily extractable, i.e., labile, P was extracted with H₂O and NaHCO₃, the moderately labile P with NaOH, and a hardly labile pool with HCl. The remaining residual P was considered stable.

Incubation experiment (exp. 1)

Temporal changes in soil phosphorus fractions

The greatest temporal changes of the P fractions took place during the first two weeks after the incorporation of the processed residues (Figs. 1, 2 and 3; Tables 1, 2 and 3). After that, in sandy loam, the changes in soil during the 12-month incubation were in the range of -10% to +15% for P_{H₂O+NaHCO₃} and P_{NaOH}, and in clay -10% to +7%. In sandy loam, the greatest increases in readily extractable P since the incorporation were noted for the sewage sludge hygienized with acid and oxidizer, i.e., Kemicond method S1 ADKem (+88%), and for the untreated sewage sludge (S1) (+27%); and in clay for S1 ADKem (+31%) and for AD sewage sludge (S1 AD) (+7%) (for all abbreviations of the processes, see Table 1). The greatest lowering in sandy loam was noted for the lime-stabilized sewage sludge (S1 ADLime), untreated manure (Man) and sewage sludge directly precipitated with a high Fe/P 9 (S3 DirAD) (-23% to -19%); and in clay with Man, AD manure (Man AD) and S1 ADLime (-39% to -49%).

For manures, P_{H₂O+NaHCO₃} increased steadily during weeks 0 to 10 whereas for sewage sludges an increase was noted in sandy loam only for S1 ADKem and in clay for S1 AD (Figs. 1, 2 and 3). For both residues, the inorganic P_{NaOH} decreased while the corresponding organic fraction increased. This change was the greater, the smaller the initial organic fraction was, i.e., greatest for S3 DirAD (Fig. 1). In clay, the changes were clearly smaller than in sandy loam. An average decrease of pH in sandy loam was 1.8 units (1.4 to 2.2 units) and in clay 0.6 units (~0 to 1.5 units) until the 2.5-month sampling point.

Residue types

The same amount of readily extractable P (P_{H₂O} + P_{NaHCO₃}) was incorporated in soil in all the residues. After incubation, the readily extractable P and NaOH fractions were greater with unprocessed sewage sludge (S1) than the unprocessed Man (Figs. 1, 2 and 3; Tables 1, 2 and 3). Similarly, those fractions were greater in S1 AD than in Man AD. The difference was similar in both soils.

Processing methods

Regarding the precipitation methods of P in sewage, during the incubation in soil for one year, precipitation with a low molar Fe/P of 1.6 consequently increased the readily extractable P relative to biological precipitation (S2 BioAD) and S3 Dir with a high Fe/P in clay (Figs. 2 and 3; Table 1). S1 ADKem increased soil readily extractable P clearly most in sandy loam across the incubation. Also, S1 AD increased the fraction in clay, whereas in sandy loam, unprocessed sewage sludge (S1) had a greater readily extractable fraction than that with AD. In manure, AD had no effect on readily extractable P, which declined immediately after incorporation in soil and was after the first two weeks maintained at the low level of the references NPK and NK. Impacts on P_{H₂O} were mostly similar to readily extractable P (Table 1).

The sparingly soluble inorganic P_{NaOH} fraction was in sewage sludge greatest with S3 DirAD and smallest with biological precipitation and lime stabilization (Fig. 1; Table 3). AD of sewage sludge increased that fraction especially with composting. Where the inorganic P_{NaOH} fraction was smallest, the organic fraction was

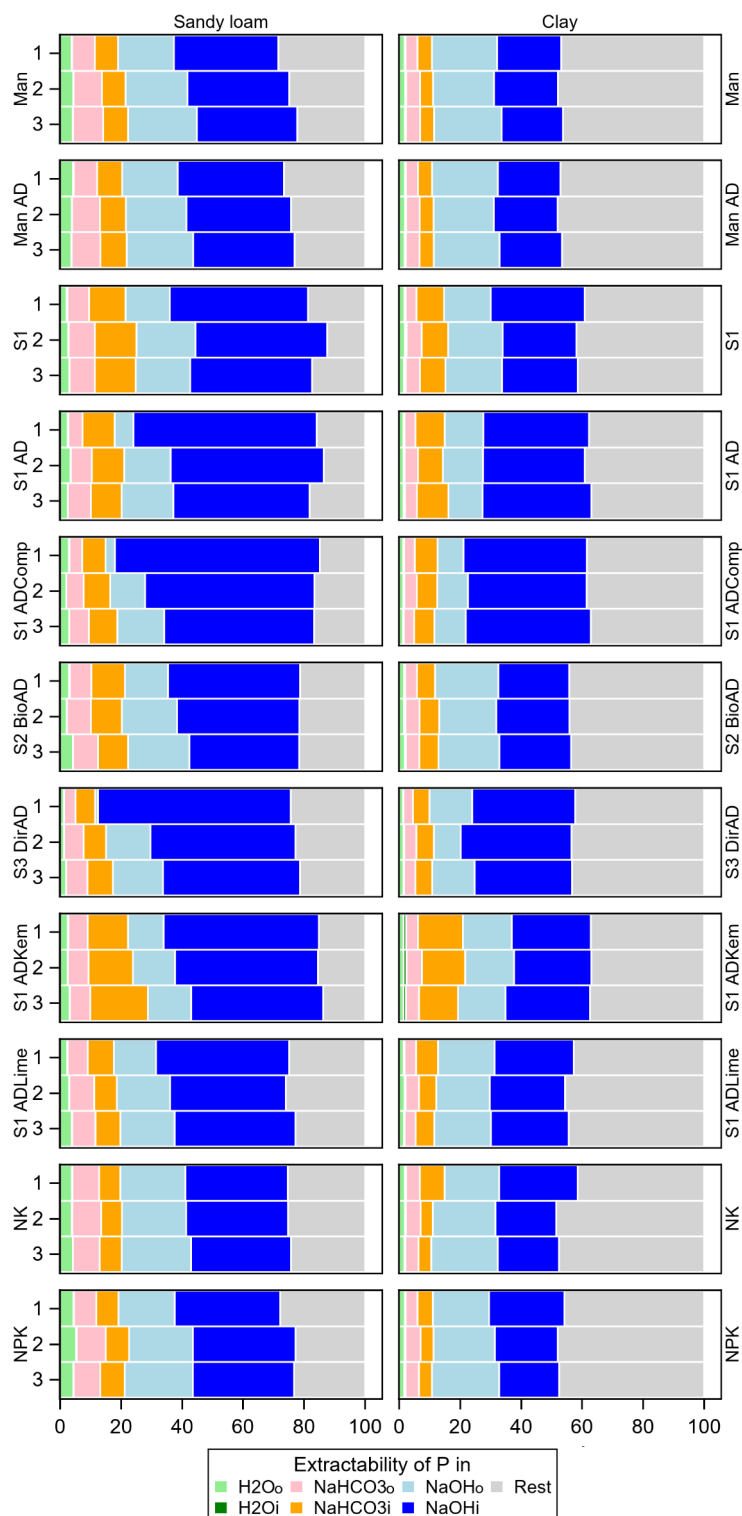


Fig. 1. Temporal development of soil phosphorus (P) fractions after 2 weeks (1), 2.5 months (2) and 12 months (3) of residue application. Initially (at the 0 week point), $0.01 \text{ g (kg dry weight)}^{-1}$ Olsen P (NaHCO_3 extractable P with no preceding H_2O extractions) was added to both soils in all the residues. For the abbreviations, see Table 1.

greatest. In manure, AD had no effect on P_{NaOH} either. The residual P fraction (P_{HCl}) tended to be greatest when incorporating sewage sludge with DirAD or BioAD or ADLime as well as with NK, NPK and manure (Fig. 3). Composting affected $P_{\text{H}_2\text{O}}$ in sewage sludge inconsequently but reduced readily extractable P and increased P_{NaOH} in clay after incubation (Tables 1, 2 and 3).

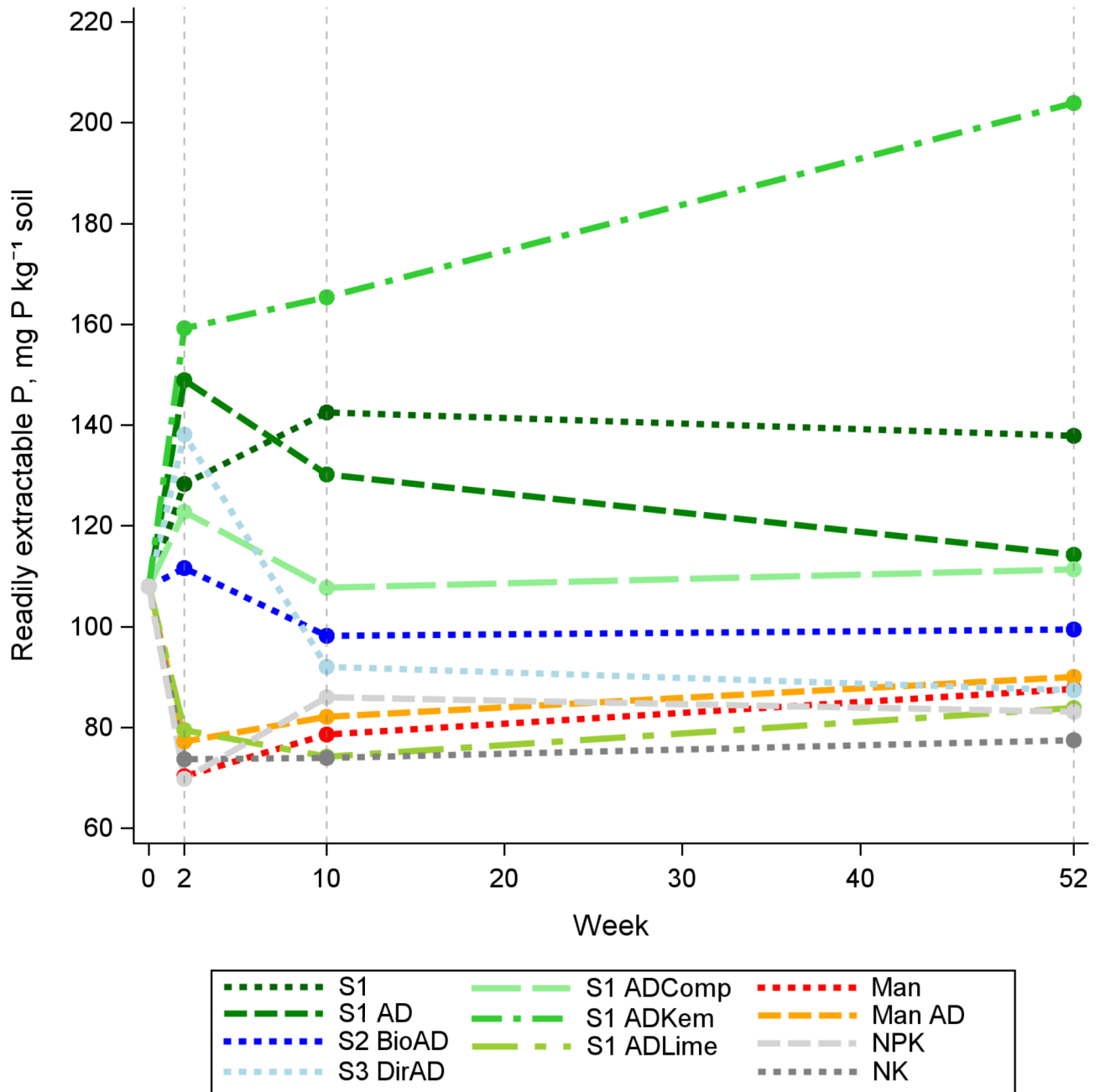


Fig. 2. Temporal development of readily extractable P in sandy loam. The sum of PH₂O and PNaHCO₃ during the incubation for 2 weeks, 2.5 months or 12 months. Initially (at the 0 week point), 0.01 g (kg dry weight)⁻¹ Olsen P (P extractable in NaHCO₃ with no preceding H₂O extractions) was added to soil with all the residues. For the abbreviations, see Table 1.

Soil types

Extractability of P was higher and the differences between the incubation periods were greater in sandy loam than in clay. However, relative differences between residues and processes were similar in both soils (Figs. 1, 2 and 3; Tables 1, 2 and 3).

In sandy loam, the incorporation of the residues led to an immediate varied effect during the two first weeks depending on the process. For sludges, the effect varied from an 88% increase in the Hedley fractions with S1 ADKem to a 23% lowering with S1 ADLime. For manure a reduction by 18% and for NPK by 23% was observed. After the first two weeks of the incubation, a steady but small increase followed for the rest of the incubation. Effects similar to those for sandy loam were.

noted with clay, apart from less increase of extractable P with S1 ADKem (by 31%) and greater lowering with manures and NPK (by 49 to 53%). The efficiency of the Hedley fractionation (the proportion of Hedley fractions from the total extraction of P including aqua regia) was in sandy loam 65 to 74% and in clay 80 to 90%.

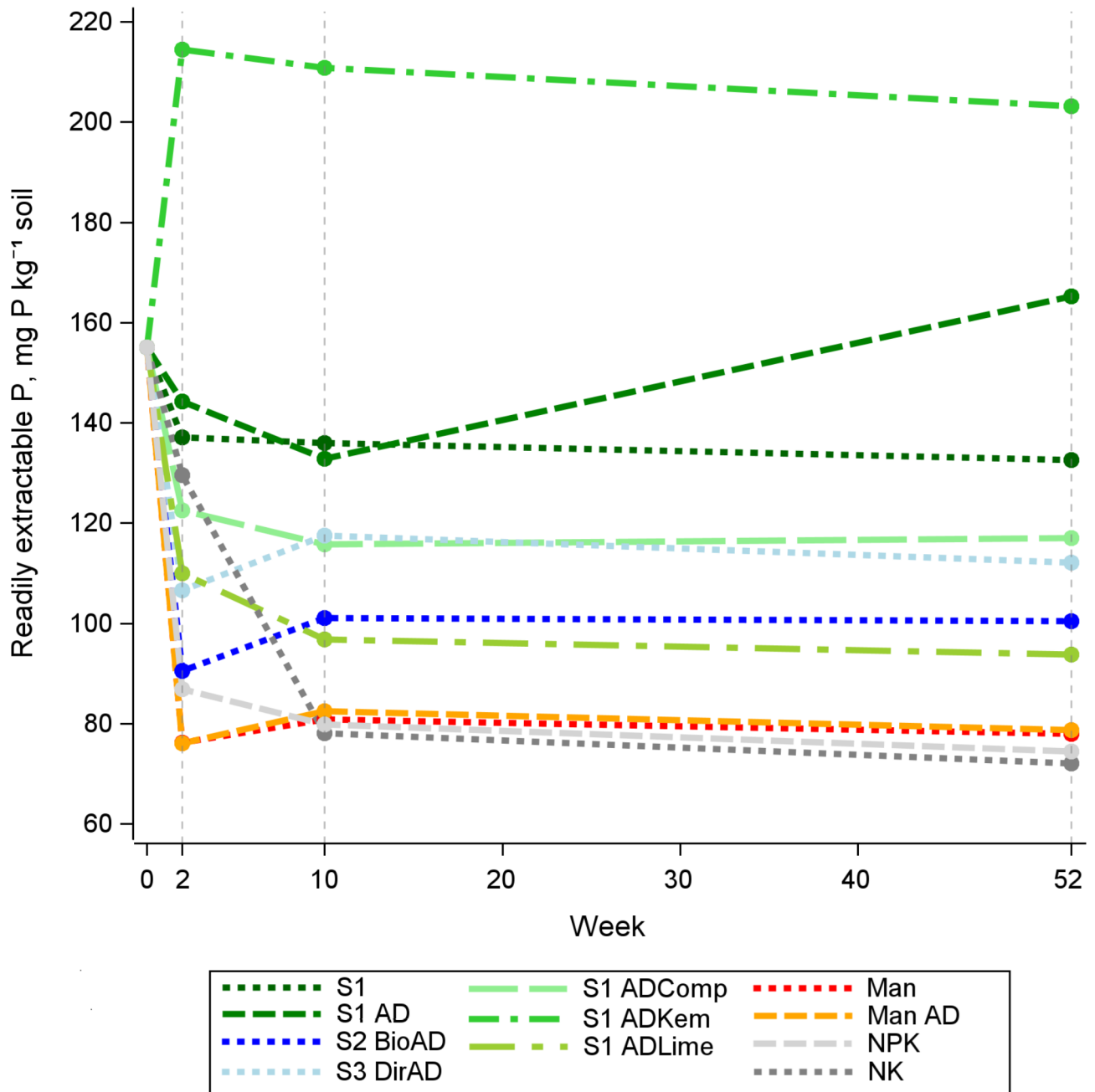


Fig. 3. Temporal development of readily extractable P in clay soil. The sum of PH₂O and PNaHCO₃ during the incubation for 2 weeks, 2.5 months or 12 months. Initially (at the 0 week point), 0.01 g (kg dry weight)⁻¹ Olsen P (P extractable in NaHCO₃ with no preceding H₂O extractions) was added to soil with all the residues. For the abbreviations, see Table 1.

Sorption experiment (exp. 2)

Processing impact on P sorption

There were differences in soil P sorption among the processing methods only with the higher application rates of the processed residues when soil P_{NaOH} was used as a covariate (for the difference among the rates $p \leq 0.004$), apart from S1 AD with no dependence on the rate ($p = 0.427$). S1 ADLime, and S3 DirAD with Fe/P 9.0, led to a higher soil P sorption than S1 AD with Fe/P 1.6 ($p \leq 0.005$), the sorption for S1 ADLime being greatest (Table 4). There were no other statistically significant differences in the sorption among the processes of sewage sludges.

Determinants of P sorption

Increase in the amount of added organic matter increased soil P sorption (180 mg kg⁻¹ dry soil, $\beta = 2.65$) consequently irrespective of the processed residue (Table 4). An increase in soil P_{NaOH} reflecting P fixed by iron oxides and hydroxides also increased soil P sorption (120 mg kg⁻¹ dry soil, $\beta = 0.12$) but the effect depended on

Comparisons of effect	2 weeks				2.5 months				12 months			
	Estimate	DF	t-value	p-value	Estimate	DF	t-value	p-value	Estimate	DF	t-value	p-value
Sandy loam												
Manure vs. Sewage sludge												
Man vs. S1	-0.59	96	-6.00	<0.001	-0.59	96	-8.53	<0.001	-0.45	96	-13.95	<0.001
Man AD vs. S1 AD	-0.64	96	-6.47	<0.001	-0.45	96	-6.52	<0.001	-0.24	96	-7.28	<0.001
Precipitation												
S1 AD vs. S2 BioAD	0.29	96	2.93	0.004	0.28	96	4.03	<0.001	0.14	96	4.37	<0.001
S1 AD vs. S3 DirAD	0.07	96	0.69	0.49	0.34	96	4.92	<0.001	0.27	96	8.26	<0.001
Hygienization												
Man AD vs. Man	0.09	96	0.94	0.35	0.04	96	0.64	0.52	0.03	96	0.82	0.41
S1 AD vs. S1	0.14	96	1.42	0.16	-0.09	96	-1.36	0.177	-0.19	96	-5.86	<0.001
S1 AD vs. S1 ADComp	0.18	96	1.80	0.08	0.19	96	2.72	0.01	0.03	96	0.83	0.41
S1 AD vs. S1 ADKem	-0.08	96	-0.81	0.42	-0.24	96	-3.51	0.001	-0.58	96	-17.91	<0.001
S1 AD vs. S1 ADLime	0.62	96	6.26	<0.001	0.56	96	8.02	<0.001	0.31	96	9.47	<0.001
Clay												
Manure vs. sewage sludge												
Man vs. S1	-0.57	96	-5.77	<0.001	-0.51	96	-7.39	<0.001	-0.53	96	-16.19	<0.001
Man AD vs. S1 AD	-0.63	96	-6.32	<0.001	-0.45	96	-6.44	<0.001	-0.73	96	-22.64	<0.001
Precipitation												
S1 AD vs. S2 BioAD	0.46	96	4.62	<0.001	0.26	96	3.69	0.00	0.50	96	15.25	<0.001
S1 AD vs. S3 DirAD	0.29	96	2.96	0.004	0.10	96	1.45	0.15	0.39	96	11.86	<0.001
Hygienization												
Man AD vs. Man	0,00	96	0.00	1.00	0.02	96	0.29	0.77	0.01	96	0.29	0.77
S1 AD vs. S1	0.06	96	0.56	0.58	-0.05	96	-0.67	0.51	0.22	96	6.74	<0.001
S1 AD vs. S1 ADComp	0.16	96	1.58	0.12	0.11	96	1.65	0.10	0.34	96	10.58	<0.001
S1 AD vs. S1 ADKem	-0.4	96	-4.05	<0.001	-0.48	96	-6.95	<0.001	-0.21	96	-6.32	<0.001
S1 AD vs. S1 ADLime	0.28	96	2.85	0.01	0.32	96	4.65	<0.001	0.56	96	17.31	<0.001

Table 1. Temporal development of the readily extractable P fraction (sum of P_{H_2O} and P_{NaHCO_3}) in soils for the residues, processes and soil types with the same amount of readily extractable OlsenP (P extractable in $NaHCO_3$ with no preceding H_2O extractions) incorporated. ‘Estimate’ represents the difference of means in log-scale. A positive value indicates a greater P fraction in the first mentioned residue/process, while a negative value indicates a smaller P fraction. DF = degrees of freedom. Man = manure, s = sewage sludge, S1-S3 = sewage sludge from separate plants with Fe/P 1.6 (1), 0.2 (2) and 9 (3), ad = anaerobic digestion, bio = biological precipitation, dir = direct precipitation with Fe/P 9, comp = composting, kem = kemicond method, lime = lime stabilization, p = phosphorus. The residues and processes are described in more detail in Table 5.

the processed residue. Soil electric conductivity (EC) did not explain soil sorption of P after accounting for other covariates ($p=0.112$). The other potential continuous predictors of soil P sorption by the incorporation of the processed residues tested, i.e., added Fe and soil pH, were no major factors of soil P sorption.

Discussion

Our findings showed that soil P fractions changed significantly over one year of incubation. Changes ranged from a decrease of 49% to an increase of 88%, depending on the fraction, residue, process, and soil type. Readily extractable P in soil increased most with sewage sludge, with P precipitation by a low molar Fe/P rather than biologically or by a high Fe/P, and when hygienized by acid and oxidizer. Manure lowered P extractability, not affected by anaerobic digesting, and so did NPK. Only the increase in the amount of organic matter added, consequently determined soil P sorption irrespective of residues and processing methods.

The proportion of the residual P fraction (P_{HCl}) in soil tended to follow the proportions in the processed residues themselves, apart from those for manure, NPK and NK, the incorporation of which led to rapid fixation of soluble P to soil in sparingly soluble form. The greater observed extractability of P in sewage sludge compared to manure, NPK and NK, was explained by the greater organic matter content of sewage sludge.

The impact of NK was due to the increased microbial activity. The organic matter provided sorption sites with lower intensity than those provided by the Fe and Al oxides and hydroxides in soil. Further, the organic matter added in residues competed with P for sorption sites of soil oxides and hydroxides and thus increased P sorption. The exceptionally high molar Fe/P (9) in P precipitation of the Norwegian sewage sludge appeared to be required to counteract the increased P extractability by the abundant organic matter incorporated.

While the sludge treated with combined acid and oxidizer was consistently superior in maintaining P readily extractable in soil, the superiority of anaerobically digested sewage sludge relative to the undigested one observed by Stutter¹⁸ appeared here in clay only. This is explained by the reduction of iron oxides, and the consequent P

Comparisons of effect	2 weeks				2.5 months				12 months			
	Estimate	DF	t-value	p-value	Estimate	DF	t-value	p-value	Estimate	DF	t-value	p-value
Sandy loam												
Manure vs. Sewage sludge												
Man vs. S1	0.25	76	0.16	0.88	0.02	94	0.01	0.99	-3.28	93	-2.03	0.05
Man AD vs. S1 AD	-5.1	76	-3.27	0.00	-7.21	94	-3.77	0.00	1.31	93	0.81	0.42
Precipitation												
S1 AD vs. S2 BioAD	5.55	76	3.56	0.00	11.16	94	5.83	<0.001	-4.77	93	-2.95	0.00
S1 AD vs. S3 DirAD	6.57	76	4.21	<0.001	14.11	94	7.37	<0.001	4.09	93	2.52	0.01
Hygienization												
Man AD vs. Man	2.33	76	1.49	0.14	-1.23	94	-0.64	0.52	-0.97	93	-0.60	0.55
S1 AD vs. S1	7.67	76	4.92	<0.001	6.01	94	3.14	0.002	-2.83	93	-1.75	0.08
S1 AD vs. S1 ADComp	-3.57	76	-2.29	0.02	8.84	94	4.62	<0.001	-3.63	93	-2.24	0.03
S1 AD vs. S1 ADKem	2.68	76	1.72	0.09	4.44	94	2.32	0.02	-8.38	93	-5.18	<0.001
S1 AD vs. S1 ADLime	10.97	76	7.04	<0.001	9.93	94	5.19	<0.001	-2.34	93	-1.44	0.15
Clay												
Manure vs. sewage sludge												
Man vs. S1	-4.54	76	-2.91	0.005	-3.79	94	-1.98	0.05	-0.54	93	-0.33	0.74
Man AD vs. S1 AD	-1.41	76	-0.91	0.37	0.43	94	0.22	0.82	-4.48	93	-2.77	0.01
Precipitation												
S1 AD vs. S2 BioAD	2.23	76	1.43	0.16	0.72	94	0.38	0.71	2.27	93	1.40	0.16
S1 AD vs. S3 DirAD	1.86	76	1.19	0.24	-0.54	94	-0.28	0.78	3.37	93	2.08	0.04
Hygienization												
Man AD vs. Man	0.95	76	0.61	0.54	-0.05	94	-0.03	0.98	-0.19	93	-0.12	0.91
S1 AD vs. S1	-2.17	76	-1.39	0.17	-4.27	94	-2.23	0.03	1.01	93	0.62	0.53
S1 AD vs. S1 ADComp	1.64	76	1.05	0.30	0.70	94	0.37	0.72	4.22	93	2.60	0.01
S1 AD vs. S1 ADKem	-6.52	76	-4.18	<0.001	-7.11	94	-3.71	<0.001	-3.89	93	-2.4	0.02
S1 AD vs. S1 ADLime	1.96	76	1.26	0.21	0.42	94	0.22	0.83	4.59	93	2.84	0.01

Table 2. Temporal development of the water-extractable (P_{H_2O}) fraction in soil for the residues, processes and soil types with the same amount of Olsen P (P extractable in $NaHCO_3$ with no preceding H_2O extractions) incorporated. ‘Estimate’ represents the difference of means. A positive value indicates a greater P fraction in the first mentioned residue/process, while a negative value indicates a smaller P fraction. For the abbreviations, see Table 1.

release, in low redox soil such as clay. Regarding manure, the proportion of readily extractable P was reduced by AD in pot¹¹ and field experiments¹⁶ with similar residues, but the impact of the process was obviously too small to be detected in this experiment. The marked increase of P sorption capacity found with the limed sewage sludge is probably explained by the formation of OM-Ca-P complexes¹⁹.

The extractability of P was notably lower in clay than in sandy loam, probably owing to the greater sorption capacity and intensity of clay. In clay there are more amorphous Fe oxides with higher affinity of sorption sites, and less mobile microbial P retention due to zones of low redox potential²⁰. Sandy loam with a lower buffering capacity than clay exhibited a greater lowering of pH during incubation, which would also strengthen the binding of P on oxide surfaces²¹, as it has been noted that that the minimum P concentration in solution is usually found between pH 5.5 and 7²².

The amount of added organic matter in residues appeared as the only consequently explaining factor to P sorption, increasing the sorption irrespective of the processing method. The higher extractability of P in sewage sludge than in manure, despite the increase in sorption by greatest addition of organic matter, can be explained by competition of P sorption between the sorption sites in organic matter and in iron oxides and hydroxides of soil. The sorption sites in organic matter maintain a greater P mobility than the sorption sites of iron oxides and hydroxides. Further, the organic matter added in residues competes with P for sorption sites of iron oxides and hydroxides and thus reduces P sorption. While Fe oxides and hydroxides in soil fix P more irreversibly than organic matter, the greater quantity of organic matter appears to determine P sorption from agrifood residues incorporated in soil. Consequently, organic matter reduces P sorption in oxides, and thus inhibits P precipitation as insoluble compounds, while increasing P sorption overall as also noted by Bueis et al.²³. Similarly, Debicka et al.²⁴ found, that the removal of soil organic matter increased both P desorption and soil P sorption capacity.

The differences among the processed residues were in the current incubation experiment with no plants similar to the earlier pot experiment¹¹ and field experiments¹⁶ which included plant P uptake. Therefore, the sorption mechanism rather than the slow decomposition of organic matter simultaneously with plant P uptake, appears to explain the P extractability in organic agrifood residues in soil. Similar results were noted by Debicka

Comparisons of effect	2 weeks				2.5 months				12 months			
	Estimate	DF	t-value	p-value	Estimate	DF	t-value	p-value	Estimate	DF	t-value	p-value
Sandy loam												
Manure vs. Sewage sludge												
Man vs. S1	-0.60	95	-7.36	<0.001	-0.59	96	-8.56	<0.001	-0.39	95	-10.5	<0.001
Man AD vs. S1 AD	-0.99	95	-12.1	<0.001	-0.67	96	-9.69	<0.001	-0.43	95	-11.5	<0.001
Precipitation												
S1 AD vs. S2 BioAD	0.61	95	7.42	<0.001	0.36	96	5.23	<0.001	0.33	95	8.97	<0.001
S1 AD vs. S3 DirAD	-0.35	95	-4.24	<0.001	0.06	96	0.88	0.38	0.12	95	3.23	<0.001
Hygienization												
Man AD vs. Man	0.03	95	0.43	0.67	0.05	96	0.71	0.48	0.04	95	1.05	0.30
S1 AD vs. S1	0.42	95	5.20	<0.001	0.13	96	1.84	0.07	0.08	95	2.13	0.04
S1 AD vs. S1 ADComp	-0.07	95	-0.80	0.42	-0.08	96	-1.19	0.24	0.00	95	0.12	0.91
S1 AD vs. S1 ADKem	0.20	95	2.41	0.02	-0.04	96	-0.60	0.55	-0.16	95	-4.22	<0.001
S1 AD vs. S1 ADLime	0.75	95	9.13	<0.001	0.60	96	8.74	<0.001	0.36	95	9.58	<0.001
Clay												
Manure vs. sewage sludge												
Man vs. S1	-0.34	95	-4.19	<0.001	-0.17	96	-2.51	0.01	-0.27	95	-7.25	<0.001
Man AD vs. S1 AD	-0.42	95	-5.15	<0.001	-0.35	96	-5.06	<0.001	-0.49	95	-13.2	<0.001
Precipitation												
S1 AD vs. S2 BioAD	0.29	95	3.61	0.00	0.26	96	3.73	0.00	0.35	95	9.27	<0.001
S1 AD vs. S3 DirAD	-0.12	95	-1.47	0.14	-0.09	96	-1.35	0.18	0.01	95	0.24	0.81
Hygienization												
Man AD vs. Man	-0.01	95	-0.12	0.90	0.00	96	-0.06	0.95	0.01	95	0.31	0.75
S1 AD vs. S1	0.07	95	0.83	0.41	0.17	96	2.49	0.01	0.23	95	6.26	<0.001
S1 AD vs. S1 ADComp	-0.04	95	-0.55	0.59	-0.07	96	-1.05	0.30	-0.08	95	-2.20	0.03
S1 AD vs. S1 ADKem	0.06	95	0.68	0.50	0.05	96	0.75	0.46	0.05	95	1.39	0.17
S1 AD vs. S1 ADLime	0.19	95	2.31	0.02	0.26	96	3.78	0.00	0.29	95	7.68	<0.001

Table 3. Temporal development of the NaOH-extractable P fraction (P_{NaOH}) in soil for the residues, processes and soil types with the same amount of the readily extractable Olsen P (P extractable in $NaHCO_3$ with no preceding H_2O extractions) incorporated. ‘Estimate’ represents the difference of means in log-scale. A positive value indicates a greater P fraction in the first mentioned residue/process, while a negative value indicates a smaller P fraction. For the abbreviations, see Table 1.

Fixed Effect	Both application rates with P_{NaOH} (N=80)	Low application rate (N=50)	High application rate (N=50)	Both application rates with OM (N=80)
	F value P value	F value P value	F value P value	F value P value
Processing method	$F_{9,59} = 9.6$ <0.001	$F_{9,36} = 2.5$ 0.025	$F_{5,24} = 10.2$ <0.001	$F_{9,64} = 4.0$ <0.001
Application rate	$F_{1,61} = 43.3$ <0.001			$F_{1,64} = 5.7$ 0.020
Processing method × Application rate	$F_{5,59} = 6.3$ <0.001			
Covariate of P_{NaOH}	$F_{1,62} = 14.6$ <0.001			
Covariate of organic matter				$F_{1,64} = 20.3$ <0.001
Differences in P sorption				
Precipitation	S3 DirAD > S1 AD (high rate)		S3 DirAD > S1 AD	
Hygienization	S1 AD _{Lime} > S1 AD (high rate)		S1 AD _{Lime} > S1 AD	S1 AD _{Lime} > S1 AD
Variance explained ($R^2_{marginal}$ / $R^2_{conditional}$)	85% / 87%		35% / 40%	68% / 68%
			68% / 68%	80% / 80%

Table 4. P sorption. Three models for sorption including all statistically significant fixed effects. Statistically significant differences (at a significance level of 0.05) between processing methods are given. For the abbreviations of the residues and processes, see Table 1.

et al.²⁵, who found that the addition of organic matter did not always reduce P sorption, but had a clear effect on impairing P fixation, which potentially could lead to greater P mobility and P availability in soil.

Since the proportions of the P fractions extracted from unprocessed or processed residues did not predict the proportions of P fractions after incorporation in soil, the initial extractability of P in residues cannot be used as a reliable criterion to predict P availability to crops but the interaction with soil properties is essential.

This confirms the former findings in a pot experiment¹¹ and in the field¹⁶ with the same materials. Regarding sewage sludges, not even molar Fe/P sufficiently predicts the effect of P capturing on extractability, as the critical threshold appeared not to be exceeded by the common practice of Fe/P 1.6 which ensured a greater extractability of P than biological precipitation of lower Fe/P.

The compared residues represent abundantly available agrifood residues with very high to the lowest extractability of P, the major precipitation methods of P in sewage as well as the most common hygienization (and energy recovery) processes. The range of textures of agricultural soils from sandy loam to clay was also covered. Temporal changes in the P fractions in controlled conditions are widely applicable and may represent changes in field conditions during more than one season. While the current results were similar to the pot¹¹ and field experiment¹⁶ with the same materials, long-term impacts deserve to be assessed in the field with repeated applications even if the differences among processed residues in soil fractions are thus more difficult to discern²⁶.

Residue type and processing method are of crucial importance for the release of plant available P fractions of agrifood residues in soil. We conclude that sorption site competition determines P availability of agrifood residues, when incorporated in soil. The amount of added organic matter most consequently predicts the differences in P sorption among processed residues. The P release potential of sewage sludges, when incorporated in soil, is high, and rapidly exploitable. This can be utilized in recycling of P, but caution is needed to account for the entire release potential when adjusting the application rate to plant uptake.

Materials and methods

Experimental materials

The processes applied to manure and sewage sludge (Table 5) were selected to cover the practices currently used in Europe as well as the range of P fraction compositions (Fig. 4). These residues were used (1) in an unprocessed form, (2) anaerobically digested, (3) composted, (4) lime-stabilized (addition of high pH lime), and (5) treated with the $H_2SO_4 + H_2O_2$ acid oxidizer (the Kemicond method to hygienize sewage sludge, developed by Kemira Oyj). The hygienization methods affect the residue properties such as the humification of organic matter, redox potential, pH and nutrient solubility, which in turn affect soil biological processes when incorporated.

In all the sewage sludges P was precipitated to bind the available P into a less soluble form. Fe/P is essential to P availability to biological processes and plant uptake. In order to identify the possible effect of Fe on P availability in the sludge, a high Fe/P (9) (S3) precipitation commonly used in Norway, as well as the common methods of biological precipitation with a very low Fe/P (0.2) (S2) and the precipitation with a modest Fe/P (1.6) (S1) were included.

To allow the comparison of processing methods, each organic residue hygienized with various methods originated from a single source¹¹ for more details), the same materials were used for the incubation experiment (Exp. 1) and soil sorption experiment (Exp. 2). The residues did not contain stormwater drain inputs. The organic residues were supplemented with mineral N and potassium (K) to ensure that the shortage of these nutrients was not limiting microbial processes. Mineral NPK and NK treatments were used as references, and a control with no fertilization was also included (see 2.3).

Two soils with contrasting textures and sorption properties, a coarse mineral soil (sandy loam) and a clay soil, were compared as the incubation media (Table 6; Fig. 5). Both soils (Gleyic Stagnosols) had been under permanent grassland for more than five years before the experiment. The plough layer soils were collected from a depth of 5–25 cm and sieved through a 5 mm mesh for the incubation experiment. For the chemical analyses, the dried soil was ground and sieved through a 2 mm mesh. To understand the effect of residues and processing methods on soil sorption of P, a sorption experiment (Exp. 2) with the same processing methods as in the incubation experiment (Exp. 1) was organized. In the sorption experiment, the coarse mineral soil (sandy loam) was used. Since clay has a much greater natural P sorption capacity than sandy loam, the changes due to incorporation of residues are more difficult to discern in clay than in sandy loam.

Experimental design and management

Incubation experiment (Exp. 1)

The soils and residues were mixed in plastic bags, and the bags were set into uncovered 1 l (7 cm x 26 cm) cylinder pots for the incubation; a total of 0.500 kg of soil in dry weight (dw) was used for each pot. The pots were watered to bring the soil to 70% of water-holding capacity. The uncovered pots were circulated within the blocks and watered every three weeks to the original weight. The temperature of the growth chambers was set to 21 °C during the day and 15 °C during the night.

The pots in the experiment were organized in a randomized complete block design (RCBD) with five replicates per incubation period. The pots were placed in the growth chamber according to earlier records of environmental variation in and between the chambers so that any potential difference in conditions would appear among blocks rather than among treatments within a block.

The soils were sampled at two weeks (Sampling point 1), 2.5 months (Sampling point 2) and 12 months (Sampling point 3) after the establishment. These sampling points were chosen to represent the immediate effects after application, over the whole northern growing season, and to reach beyond a single growing season. The first sampling point represents the effect of the interaction between the residue and soil, prior to biological processes which are also involved in the second sampling point. The third sampling point represents the longer term impact on soil P availability.

The sampling was destructive; the sample pots were removed from the growth chamber permanently upon sampling, and the collected samples were stored in +4 °C. A total of 180 samples (90 per soil) were analyzed at each of the three sampling points.

Residue	Type	P precipitation	Hygienization	Exp. Rate	Residue added (g fw)/(kg dw) ⁻¹	Incorporated in soil with residue					C/N	C/P _{AR}	Fe/P mol	pH
						C	N	P _{AR}	P _{NaHCO3}	Fe _{AR}				
Man	Liquid dairy manure			1	37	0.09	0.01	0.02	0.01		8.1	43		8.32
				2. Low	120	5.03	0.05	0.09	0.06					
Man AD	Liquid dairy manure		AD, mesophilic process 37 °C, OLR 3 kgVS m ³ -1 d ⁻¹ , HRT 26 d.	1	47	2.05	0.01	0.03	0.01		20.1	84		8.55
				2. Low	190	3.05	0.06	0.08	0.06					
S1	Sewage, Plant 1	Fe-coagulant, Fe/P 1.6	dewatered	1	71	3.04	0.04	0.2	0.01	0.06	7.1	17	2.0	6.01
				2. Low	35	1.05	0.02	0.08	0.01	0.34				
S1 AD	Sewage, Plant 1	Fe-coagulant, Fe/P 1.6	AD, dewatered	2. High	157	6.09	0.09	0.36	0.02	1.54				
				1	47	3.06	0.06	0.41	0.01	1.17	6.1	9	2.0	8.18
S1 ADComp	Sewage, Plant 1	Fe-coagulant, Fe/P 1.6		2. Low	8	0.07	0.01	0.09	0.003	0.37				
				2. High	69	5.07	0.08	0.1	0.02	3				
S2 BioAD	Sewage, Plant 2	Biological P removal, Fe-coagulant, Fe/P 0.2	AD, dewatered	1	30	3.08	0.05	0.36	0.01	1.08	7.1	10	2.0	7.27
			AD, dewatered, 2 wk tunnel composting (bricks; peat, 1t:2m ³ ; 1m ³ w: v:v) 2 wk	2. Low	11	1	0.01	0.09	0.004	0.33				
S3 DirAD	Sewage, Plant 3	Fe-coagulant, Fe/P 9.8		2. High	76	6.09	0.09	0.58	0.03	2.21				
				1	14	1.01	0.02	0.1	0.01	0.33	5.1	11	0.06	7.68
S1 ADKem	Sewage, Plant 1	Fe-coagulant, Fe/P 1.6	AD, acid and oxidizer: H ₂ SO ₄ , H ₂ O ₂ , NaOH, dewatered w. centrifuge	2. Low	12	0.09	0.02	0.08	0.02	0.07				
				2. High	20	1.03	0.02	0.08	0.003	1.2				
S1 ADLime	Sewage, Plant 1	Fe-coagulant, Fe/P 1.6	AD, dewatered, CaO 80 kg t ⁻¹ , tractor mixed	1	95	3.03	0.06	0.34	0.01	0.06	5.1	10	1.1	5.06
				2. Low	19	0.09	0.01	0.09	0.004	0.4				
NPK, NK	Mineral fertilizer			2. High	67	3.03	0.05	0.31	0.01	1.4				
				1	15	1.02	0.02	0.13	0.01	0.06	7.08	9	2.04	12.53
				2. Low	9	1	0.01	0.08	0.01	0.36				
				2. High	131	13	1.04	1.17	0.08	5.02				
				1					0.01					
				2. Low					0.08	0.08				

Table 5. Residue processing methods And application rates in soil of the incubation (Exp 1.). And the sorption experiment (Exp 2.). P = phosphorus, c = carbon, n = nitrogen, fe = iron, ad = anaerobically digested, ar = aqua regia extraction, dw = dry weight, fw = fresh weight, (g fw)/(kg dw)⁻¹ = residue addition g of residue (fw) into 1 kg of soil (dw).

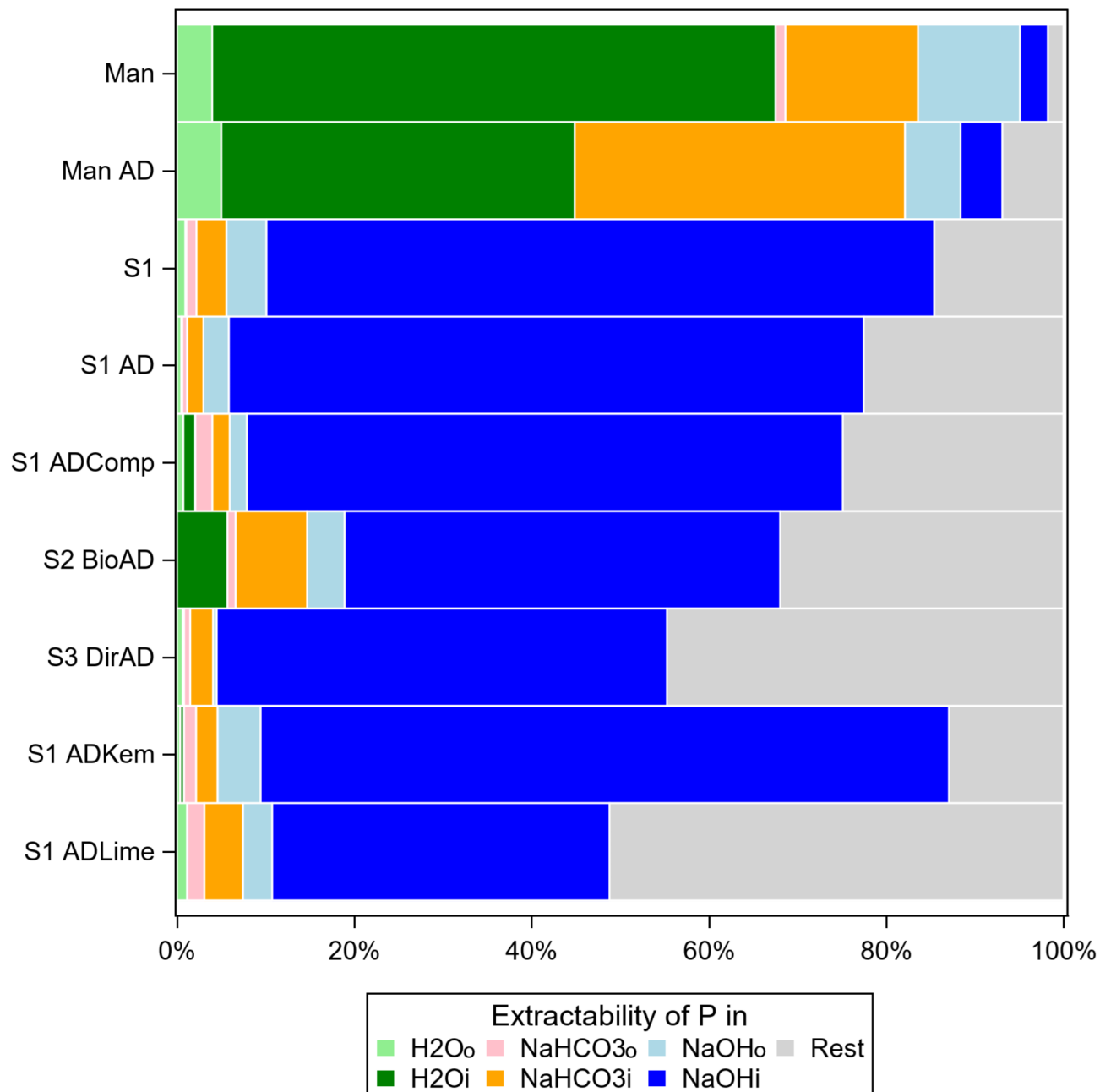


Fig. 4. Phosphorus (P) fractions of the processed residues before incorporation into the soil. The fraction denoted with ‘i’ refers to inorganic P extractable with a given extractant, while ‘o’ refers to the corresponding organic P fraction.

Analysis method	pH	N_{tot} mg (kg dw) ⁻¹	C_{tot} mg (kg dw) ⁻¹	P_{aq} mg (kg dw) ⁻¹	$P_{\text{H}_2\text{O}_1}$ 4 h (kg dw) ⁻¹	$P_{\text{H}_2\text{O}_1}$ 16 h (kg dw) ⁻¹	P_{NaHCO_3} (kg dw) ⁻¹	P_{NaOH_1} (kg dw) ⁻¹	P_{HCl} (kg dw) ⁻¹
Sandy loam	6.4	1300	26,200	466	8.3	16.1	74	270	364
Clay	6.7	2900	40,000	1080	10.1	16.4	119	410	762

Table 6. Chemical properties of the soils before the incorporation of the residues. The concentrations of H₂O, NaHCO₃ and NaOH extractable P in soil are presented as the sum of the organic and inorganic phosphorus (P) within each fraction of the Hedley procedure. N = nitrogen, c = calcium, dw = dry weight, ar = aqua regia extraction.

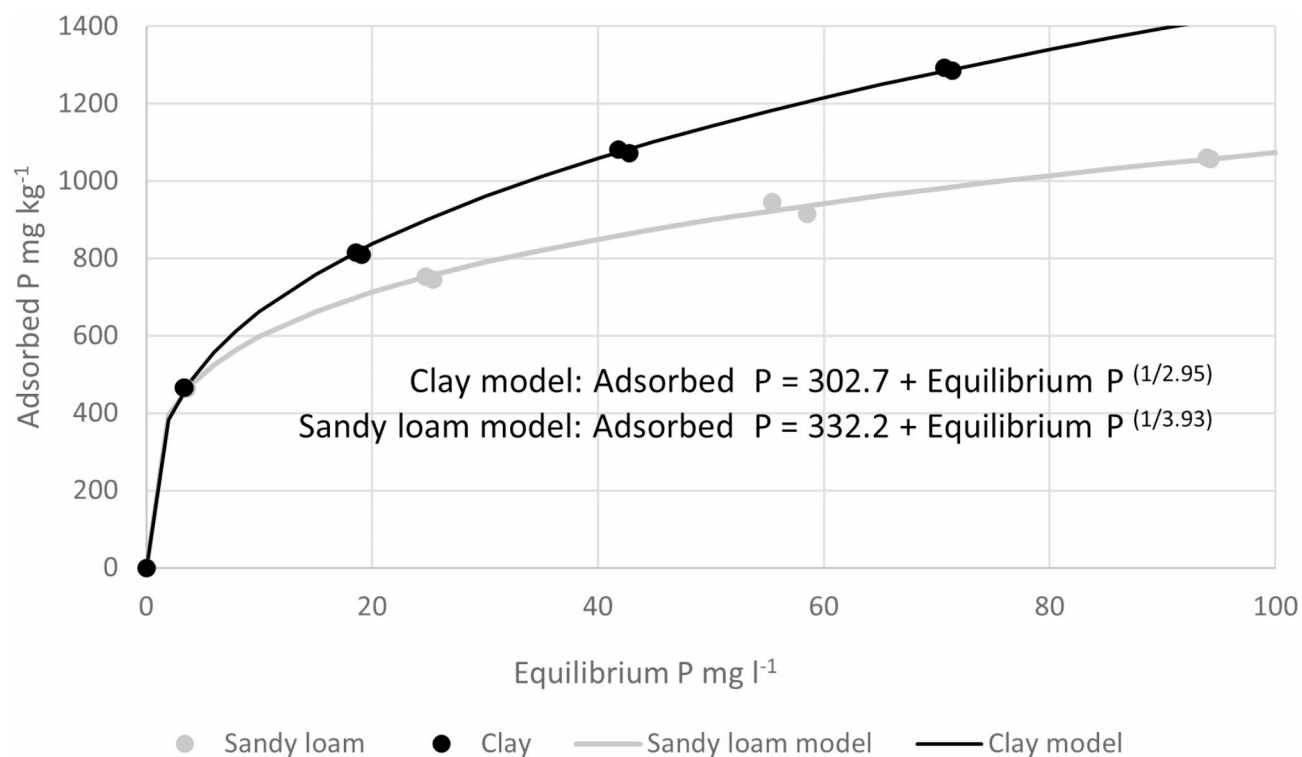


Fig. 5. Phosphorus sorption by the soils before the incorporation of the residues. The fit for sandy loam is $r^2=0.997$ and for clay: $r^2=0.999$.

Sorption experiment (Exp. 2)

The sorption experiment (Exp. 2) was conducted to determine the contribution of the sorption mechanism to the differences found among the processed residues in P extractability in the incubation experiment (Exp. 1). Soil samples from a pot experiment¹¹ with the same processed residue and sandy loam soil as well as experimental conditions as in the incubation experiment (Exp. 1) were used. Two equal total P input rates were applied apart from manures and S2 BioAD for which only the lower rate was used due to the clearly greater P extractability, to avoid the smaller dry matter rate potentially interfering with the results (Table 4). Soil was sampled in the pots after four weeks. The impact of nutrient uptake of the small plants on soil P concentrations was considered negligible. The sorption isotherms were determined similarly to the incubation experiment (Exp. 1) (2.2.1), but the P solution of 150 mg P l⁻¹ was used²⁷. When determining the contribution of soil sorption to the differences in P extractability among the residues and processing methods, the previously published results of the sorption experiment (Exp. 2) on extractability¹¹ were used.

To quantify the P sorption of the soils used in the study, P sorption curves were determined on the soils according to Lajtha et al.²⁷. P solutions of 0, 50, 100, 150 and 200 mg P l⁻¹ were prepared in 0.01 M KCl from a stock solution containing 1000 mg P l⁻¹ KH₂PO₄, and 30 ml of P solution was mixed with 3 g of air-dry soil. Soil was run in duplicate samples. The mixtures were shaken for 24 h at room temperature and then centrifuged for 30 min at 13 000 g. PO₄-P concentration was measured from decanted supernatant with Lachat autoanalyzer with an ammonium heptamolybdate blue method using ascorbic acid as the reducing agent. Sorption curves were plotted as adsorbed P (mg P kg⁻¹ soil = (mg P l⁻¹ in original working solution - mg P l⁻¹ in equilibrium solution) × ml working solution per g dry soil, i.e. 30 ml per 3 g dry soil) against equilibrium concentration (mg P l⁻¹) (Fig. 5).

Nutrient application rates

Incubation experiment

In the incubation experiment, due to the different processing methods and origins of the agrifood residues, a single widely used extraction method was chosen to be used to harmonize the P additions. Therefore, P corresponding to 10 mg NaHCO₃ extractable P kg dw⁻¹ of soil was applied either in organic residues or as a mineral fertilizer using potassium phosphate (KH₂PO₄) (Table 4). 10 mg NaHCO₃ extractable P kg dw⁻¹ equates to a commonly used application of 20 kg P ha⁻¹ in the field, assuming a 1 kg dm⁻³ dry bulk density, and a 20 cm tilling depth. Additional mineral N and K were mixed into the pots as solutions, N as ammonium nitrate (NH₄NO₃), and K as potassium chloride KCl (100 mg kg⁻¹ of N, 93 mg kg⁻¹ K for sandy loam, and 139 mg kg⁻¹ N, 66 mg kg⁻¹ K for clay) (Table 4).

Sorption experiment

In the sorption experiment, the soil was fertilized with the processed residues using rates equal to total P addition of 200 mg in 2.5 kg_{dw} soil, and with additional high rates of sludges with P precipitated with Fe coagulant where total P application was from 780 to 2900 mg in 2.5 kg_{dw} soil (Table 4). Treatments without any fertilizer application were included as controls.

The rate of residues and fertilizers incorporated was adjusted to ensure an equal supply of P for all the fertilized treatments. Extraction with NaHCO₃ is internationally one of the most used methods to illustrate plant-available P. It is not suitable for the most acidic soils, but since the pH of residues used was close to neutral or alkaline and the field soil pH was also close to neutral, the method was deemed appropriate. The chemical properties of the fertilizers were analyzed upon incorporation and the realized rates of C, N and P added were calculated (Table 4).

Mechanical and chemical analyses of soil and processed residues

The dry matter content of the soils and organic fertilizers was determined by drying them in 105 °C until no weight change occurred. Total N and C concentrations were determined with the Dumas method (ISO 13878:1998, LECO CN-2000) from 2 g air-dry samples. The residues' total Fe, Al and P concentrations were determined with an aqua regia (3:1 HCl: HNO₃) extraction (EN 13346 for sludges and EN13650 for soil improvers and growing media).

Phosphorus was extracted with the Hedley method²⁸, in a modified form previously used for agrifood residues^{20,29}. The extraction was performed in room temperature with five sequential steps, i.e., two times with water (4 h and 16 h; 1:60 w/w), 0.5 M NaHCO₃ (1:60 w/w), 0.1 M NaOH (1:60 w/w), and finally 1 M HCl (1:60 w/w). Both inorganic phosphorus (P_i) and organic phosphorus (P_o) concentrations were determined from the solution, except from the HCl extracts, which were analyzed only for P_i. For P_i, the samples were filtered through a 0.2 μm membrane filter, and for P_o, the unfiltered samples were digested with persulfate upon autoclaving for 30 min at 120 °C to decompose P_o, allowing the determination of the total P of the extract. The difference in the total P and the P_i concentrations was assumed to represent the concentration of P_o. The sum of the most readily extractable fractions; extracted by H₂O and NaHCO₃, was bundled together as the readily extractable or labile P. The total concentration of P was extracted from soils and agrifood residues with an aqua regia (3:1 HCl: HNO₃) extraction (EN 13346 for sludges, and EN13650 for soil improvers and growing media), which was then compared to the sum of the Hedley fractions to ascertain the efficiency of extraction of the Hedley fractionation.

Statistical analysis

Generalized linear mixed models with logarithm and gamma distribution, with an identity and a log link, respectively, were used in the analysis of the dependence of the soil P fractions and their change rates in the incubation experiment (Exp. 1). This approach was chosen due to highly skewed distributions of some response variables and to account for differences in variances between residues. A Gaussian distribution with an identity link was used for other response variables. The models were fitted by using the residual maximum likelihood (REML) for Gaussian models, while the more suitable residual pseudo-likelihood (RSPL) estimation method was used for non-Gaussian (gamma) models. Fixed effects included residue/fertilizer, soil texture (*clay/sandy loam*), and sampling point. All the interactions between fixed effects were also included in the models when possible; the analysis of HCl extractable P was simplified by analyzing the soils separately.

The repeated measures' design (three sampling points) was accounted for through unstructured (UN) and heterogeneous first-order autoregressive (ARH₁) covariance structures. The model can be expressed in the following equation form:

$$Y_{ijkl} = \mu + F_i + T_j + S_k + SR_{kl} + FT_{ij} + FS_{ik} + TS_{jk} + FTS_{ijk} + TSR_{jkl} + \varepsilon_{ijkl}$$

Where μ is the overall mean, F_i , T_j and S_k are the fixed effects of the residue/fertilizer, sampling point, and soil texture type, respectively. Two- and three-factor interactions of fixed effects were also included in the model; interaction of the R_l (replicate) term with fixed effects SR_{kl} and TSR_{jkl} represent the random effects, and ε_{ijkl} is the residual error. The random effects were assumed independent and normally distributed. The terms in the model regarding soil texture were removed from the analysis of Hedley P_{HCl} because the soils were analyzed separately regarding the total P concentration. Only the pairwise comparisons pre-determined according to the research questions were performed within the application rates and between the rates to avoid multiple testing.

The sorption experiment (Exp. 2) was analyzed with the same methods as the incubation experiment (Exp. 1) having treatment (residue and processing method), application rate, and their interaction as fixed effects. In addition, the significance of continuous predictors (readily extractable soil P, NaOH extractable soil P, added organic matter, added Fe, soil pH, and soil electrical conductivity EC) was tested in the models, and statistically significant predictors were kept in the models. Block and block \times application rate was used as random effects.

The first model consisted of the effects of ten processing methods and two application rates of the processed residues and their interaction (Table 5). The concentration of P_{NaOH} was included as a covariate. The differences among the ten and six processing methods, respectively, were analyzed in a model with a low rate and a high rate of the processed residues. Due to the random effects, two R² values are presented, where the first one accounts for the fixed effects and the latter one for random effects as well. The appropriateness of the models was studied by residual analyses. The residuals were checked for normality using a boxplot and normal probability plot³⁰. The residuals were also plotted against the fitted values. These plots indicated that the assumptions of the models were adequate. In the case of a pairwise comparison of means, the Holm-Tukey or the Tukey-Kramer post hoc test was used. A significance level of $\alpha = 0.05$ was used in all analyses. Degrees of freedom were calculated using the Kenward-Roger method. The analyses were performed using the MIXED and GLIMMIX procedures of the SAS Enterprise Guide 8.3 (SAS Institute Inc., Cary, NC, USA).

Data availability

The datasets generated during and/or analyzed during the current study are available from the corresponding author on reasonable request.

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Author contributions

All authors contributed to the study conception and design. Material preparation, data collection and analysis were performed by JJM, TS, MK, JK and HK. Statistical analyzes were done by JK, with contributions by J JM, TS and HK. The original experiment planning and supervision of research and experiments were done by HK. The first draft of the manuscript was written by JJM, and all authors commented on previous versions of the manuscript. All authors read and approved the final manuscript.

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Declarations

Competing interests

The authors declare no competing interests.

Additional information

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