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KNOWLEDGE REPORT

Solubility and plant-availability of P in manure

By Kari Ylivainio and Eila Turtola

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BALTIC MANURE
BUSINESS OPPORTUNITIES

Baltic Manure WP4 Standardisation of Manure Types with Focus on Phosphorus

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Natural Resources Institute (Luke) is a research and development organization established in 2015 by merging MTT Agrifood Research Finland, the Finnish Forest Research Institute (Metla), the Finnish Game and Fisheries Research Institute (RKTL) and the statistical services of the information Centre of the Ministry of Agriculture and Forestry (Tike). This report was published in 2013, http://balticmanure.eu/download/Reports/solubility_and_availability_of_p_in_manure_web.pdf, and this is a reprint of that version.

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1 Introduction

Phosphorus (P) is an essential plant nutrient with soil reserves, mineral P fertilizers and manure serving as main sources for agricultural crops. Phosphorus is also the key element in enhancing eutrophication of surface waters, originating mainly as diffuse loads from agricultural fields. In order to minimize P leaching to surface waters, P fertilization should be adjusted according to plant needs, avoiding over-fertilization. In Finland since 1990's the use of mineral P fertilizers has decreased substantially due to high P reserves in soil and concern of environmental side-effects. Unlike mineral P fertilizers, total content of P in manure (kg ha^{-1}) has remained at a constant level since the 1990´s and it is currently more than that used as mineral P fertilizers (Ylivainio et al. 2013). Compared to mineral fertilizers, the plant availability of P in different manure types has been under a debate. Therefore, in order to use manure P efficiently from both environmental and plant production point of views, P solubility and availability for plants needs to be verified.

In this study we analyzed P solubility in different types of manure and feces by using a modified Hedley fractionation scheme (Sharpley and Moyer, 2000). Availability of P for barley crops from these materials was further tested in a pot experiment.

2 Materials and methods

2.1 Manure samples

Manure samples consisted of dairy cow, pig, poultry and fox manures. Dairy cow slurry originated from MTT's experimental barn at Maaninka, including slurry samples prior and after anaerobic digestion and after post digestion. Separation of slurry to solid and liquid fractions prior to anaerobic digestion was carried out to examine P solubility in the solid fraction. Composted (about 1 year) dairy cow manure included straw and peat as bedding materials and originated from a local farm. Feces of dairy cow were collected straight from the animals in the course of two different feeding trials conducted at MTT: The diet contained either no additional protein feed or additions of either rapeseed expeller or soybean expeller (Tuori et al. 2006). Other feces samples originated from a feeding trial where the diet contained either low or high levels of concentrate feeds.

Pig slurry originated from MTT's experimental barn at Hyvinkää. Pig feces were obtained similarly to cows from a feeding trial where the diet was supplemented either with mineral P or phytase (500 PPU kg^{-1}). Poultry manures from two feeding trials (chicken and broiler) were included also; the diet contained either mineral P or phytase (150 PPU kg^{-1}) addition (chicken manure) or only mineral P (broiler). Fox manure originated from a local farm. Solubility of P in the above mentioned manures and feces was compared to that of mineral P fertilizer (superphosphate).

2.2. Solubility of P

Solubility of P in manures was analyzed according to a modified Hedley fractionation scheme (Sharpley and Moyer 2000). Air-dried and finely ground manure sub-samples (1 g) were first extracted twice with water at a 1:60 (w/v) ratio and then sequentially with each of the following: 0.5 M $NaHCO_3$, 0.1 M NaOH and 1 M HCl. Extraction times were 16 h, except for the first water extraction (4 h). After the extraction, the samples were centrifuged (3000 g, 15 min) and inorganic P (Pi) [supernatants filtered through a 0.2 µm nucleopore membrane (Whatman, Maidstone, UK)] and total P (unfiltered supernatant digested at 120 °C with sulfuric acid and peroxodisulfate) concentrations in the supernatants were analyzed according to Murphy and Riley (1962). The difference between total P and Pi was assumed to represent organic P (Po).

2.3. Plant availability of P

Availability of P in the same manure samples was estimated in a pot experiment. Spring barley (*Hordeum vulgare* var. Elmeri) was grown in a sandy soil (6.5 kg) that was P deficient [acid (pH 4.65) ammonium acetate extractable P was 1.9 mg l^{-1}], and slightly acidic (pH 5.8), outdoors under a glass roof at ambient temperature. Experimental soil was taken from plow layer, air dried and sieved through 14 mm sieve and weighed into Mitscherlich type pots. About 0.5 l of soil (< 4 mm) was removed for covering the seeds, after which a solution with plant nutrients was mixed in the rest of the soil volume. Experimental pots were supplemented with the following essential plant nutrients: nitrogen (N) as $Ca(NO_3)_2*4H_2O$ and NH_4NO_3 at a rate of 2000 mg pot^{-1}, potassium (K) as KCl (2000), magnesium (Mg) as $MgSO_4*6H_2O$ (300), calcium as $Ca(NO_3)_2*4H_2O$ (1431), iron (Fe) as $FeSO_4*7H_2O$ (20), zinc (Zn) as $ZnSO_4$ * 7 H_2O (20), manganese (Mn) as $MnSO_4$ * H_2O (20), copper (Cu) as $CuSO_4$ * 5 H_2O (10), boron (B) as H_3BO_3 (2) and molybdenum (Mo) as Na_2MoO_4 * 2 H_2O (2). Sulphur (S) was added as sulphate in the above mentioned compounds and as concentrated sulfuric acid (430). Additional N application (1000) was given when barley reached the booting stage.

Phosphorus fertilization originated from manures, feces or superphosphate. All manure samples were air-dried at room temperature, sieved (6 mm) and applied according to total P content to receive P application rates of 40 mg kg^{-1} soil, except 100 mg kg^{-1} as fox manure. Superphosphate P was applied as increasing application rates: 10, 50 and 100 mg kg^{-1} soil in order to obtain yield response curve for calculation of P availability in manures and feces. Availability of P for barley was calculated by comparing yield responses to that caused by superphosphate. The control treatment did not receive any P. All the pots received 25 barley seeds, were covered with soil, and after germination thinned to 20 shoots per pot. Pots were watered with deionized water. Due to the low temperature in the late autumn, experimental pots were transferred inside the greenhouse for the final ripening. All treatments were replicated four times.

Barley was harvested about 2 cm above the soil surface and dried at 65 °C. Samples from grain and straw yields were ground and about 1 g was digested with 10 ml of concentrated nitric acid (14.3 M) over night at room temperature after which temperature was raised up to 120-130 °C on a sand bath and the sample was evaporated until volume of nitric acid was decreased to about 2 ml. Total concentration of P was analyzed with ICP-AES (Thermo Jarrel Ash, Franklin, MA). Nitrogen concentration in grain was determined with NIR and in straw with the Kjeldahl method.

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3 Results and discussion

3.1 Solubility of P

Water extractable P concentration in dairy cow, pig and poultry manures was the main P fraction (55-91 %, Table 1, Fig. 1), whereas in fox manure it was only 19 %. In feces, water extractable P contents were at the range of manures, except in two dairy cow feces (18 and 27 %). Water soluble P is considered to give a reliable estimate for immediate leaching potential of manure P during rainfall (Sharpley and Moyer 2000). Anaerobic digestion of dairy slurry decreased the share of water extractable P from 71 % to 55 % of the total sum of P fractions (Fig. 1). This may lower P runoff from surface-applied manure, if heavy rainfall occurs before manure is incorporated into the soil. According to the Hedley fractionation scheme, water soluble P fraction in dairy slurry was less than in pig and poultry manures, shares being 66 and 74-83 %, respectively. In composted dairy manure and the solid fraction of dairy slurry, water extractable P share was at a higher level, 91 and 85 %, respectively, comparable to that in superphosphate (91 %).

The sum of water and 0.5 M $NaHCO_3$ –extractable P fractions is considered to form the labile, immediately plant-available P content. Although the anaerobic digestion of dairy slurry decreased the water-soluble P fraction, labile P contents were at the same level (86 %) compared to the undigested slurry (81 %). Composted dairy manure and superphosphate contained equal shares of labile P (92 %). Use of phytase in the poultry diet increased the labile P content in manure (Fig. 1) from 80 to 89 %. The labile P share in pig slurry (77 %) was equal to that in dairy cow slurry (81 %). Phosphorus in fox manure was the least soluble (27 %), corresponding to that found in an earlier study (Ylivainio et al. 2008). Moreover, fox manure contained 71 % of acid soluble P due to the diet with e.g. meat and bone meal, a source of low soluble P. Although P in fox manure is mostly acid soluble, after abundant use it can solubilize gradually and P can accumulate in subsurface layers (Uusitalo et al. 2007). Other manures contained from a few percent up to 16 % of acid soluble P (Fig. 1).

Organic P constituted at the most 45 % of the sum of P fractions in poultry manure (Table 1). When the diet contained phytase, the content of organic P decreased to 31 %. In dairy slurry the corresponding share of organic P was 18 % and anaerobic digestion decreased it to 13 % and post digestion further to 11 %. In feces, the organic P content (4-30 %) was depended on the diet. Fox manure had the lowest share of organic P (2 %).

Table 1. Concentration of inorganic (Pi) and organic (Po) phosphorus fractions in air-dried manures and feces according to Hedley fractionation scheme, g kg^{-1}.

Phosphorus sources	Water		0.5 M $NaHCO_3$		0.1 M NaOH		HCl	Σ Pi	Σ Po
	Pi	Po	Pi	Po	Pi	Po	Pi		
Dairy slurry	4.6	0.3	0.6	< 0.1	0.4	0.8	0.1	5.6	1.2
Anaerobically digested dairy slurry	4.3	0.4	1.6	0.1	0.3	0.5	0.3	6.5	1.0
Post digested dairy slurry	4.2	0.5	2.6	< 0.1	0.3	0.3	0.6	7.7	0.9
Solid fraction of dairy slurry	3.6	0.1	0.2	< 0.1	0.1	0.3	< 0.1	4.0	0.4
Composted dairy manure	2.8	0.2	< 0.1	< 0.1	< 0.1	0.2	< 0.1	2.9	0.5
Dairy feces, control (no additional protein feed in the diet)	3.2	0.6	0.4	0.2	0.1	0.8	< 0.1	3.8	1.6
Dairy feces, rape meal in the diet	4.4	0.6	0.6	0.2	0.3	1.3	0.3	5.6	2.1
Dairy feces, soybean meal in the diet	3.5	0.6	0.6	0.2	0.2	0.9	0.1	4.5	1.6
Dairy feces, low level of concentrate in the diet	1.2	0.4	4.6	< 0.1	0.8	0.7	1.9	8.5	0.4
Dairy feces, high level of concentrate in the diet	2.2	0.5	4.4	0.1	0.7	0.7	1.3	8.6	1.3
Pig slurry	8.5	0.3	1.4	0.1	0.3	0.6	2.1	12.3	1.0
Pig feces, without phytase (mineral P amendment in the diet)	7.8	0.3	1.4	0.3	0.7	0.8	2.6	12.5	1.3
Pig feces, phytase included in the diet	8.4	0.2	0.6	0.1	0.3	0.6	0.5	9.8	1.0
Chicken manure, no phytase in the diet	3.3	1.6	0.1	0.3	< 0.1	1.1	0.2	3.6	2.9
Chicken manure, phytase included in the diet	4.5	1.3	0.1	0.3	< 0.1	0.6	0.1	4.8	2.1
Chicken manure (broiler), mineral P amendment in the diet	10.8	2.5	0.2	0.3	0.2	1.3	1.2	12.4	4.1
Fox manure	7.2	0.2	2.8	0.3	0.2	0.3	27.4	27.7	0.8
Superphosphate	75.9		0.5		1.8		4.9	83.1	

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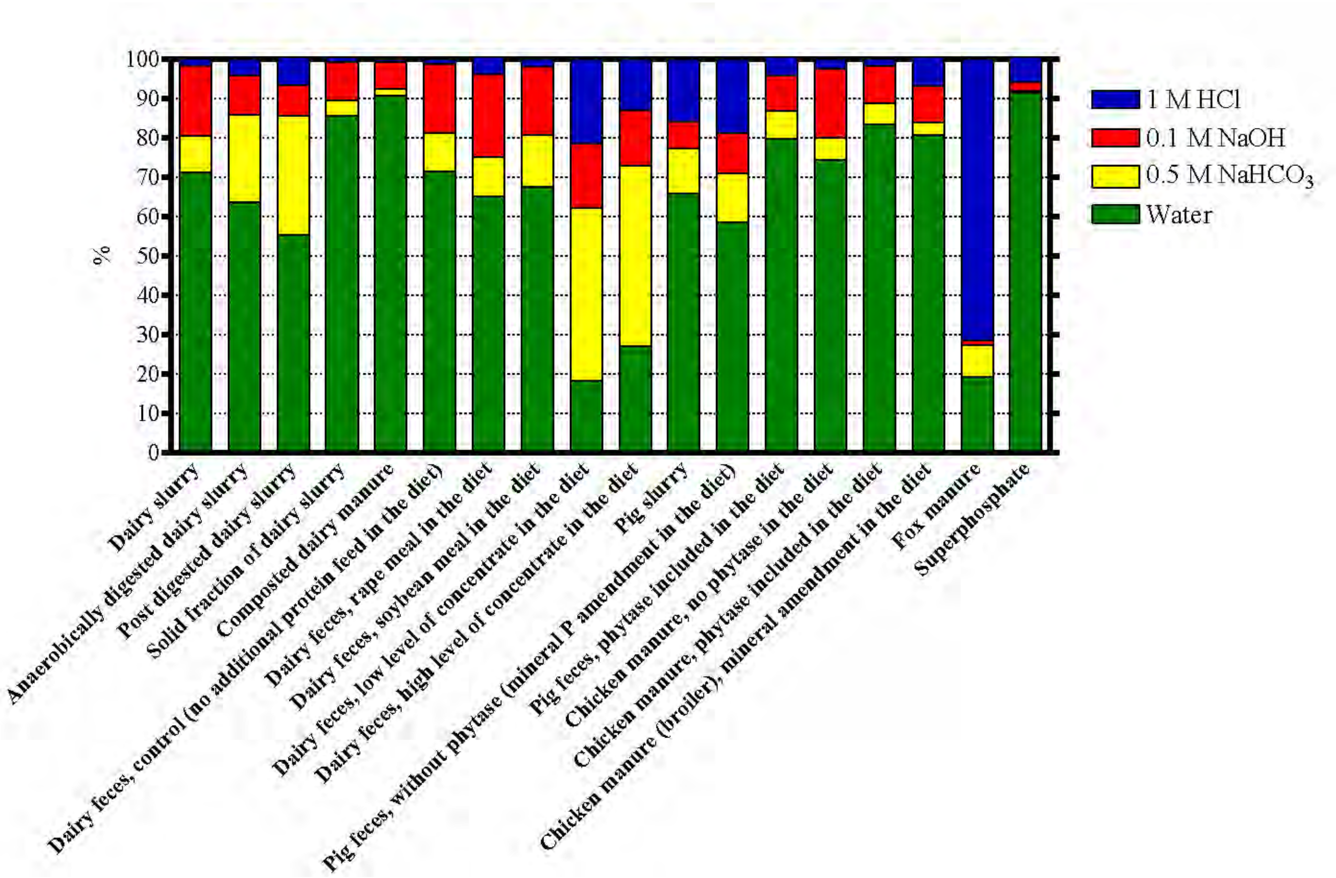

Figure 1. Share of P fractions in different manures, feces and superphosphate.

3.2 Plant availability of P

The soil in the pot experiment had a very low content of plant available P (STP 1.9 mg l^{-1}), which caused a clear yield response after mineral P application (Fig. 2). According to Valkama et al. (2011), P fertilization gives yield response in coarse textured mineral soil when STP value is below 10 mg l^{-1}. Phosphorus originating from dairy cow and pig slurries increased barley yield more than the comparable amount of superphosphate P (Table 2). Pig slurry provided the highest grain yield (61.3 g), whereas the grain yield with an equal amount of superphosphate P (40 mg kg^{-1}) was only 45.6 g (Fig. 2). These results confirm the earlier findings of plant availability of P in dairy cow manure in both pot (Ylivainio et al. 2008) and field experiments (Ylivainio and Turtola 2009). It is unlikely that lower yields with superphosphate were caused by the deficiency of other nutrients than P, as they were abundantly provided. For example, nitrogen concentration in barley grains in the superphosphate (100 mg P kg^{-1}) and pig slurry (40 mg P kg^{-1}) treatments were 23.1 and 23.7 mg g^{-1}, respectively (Table 3).

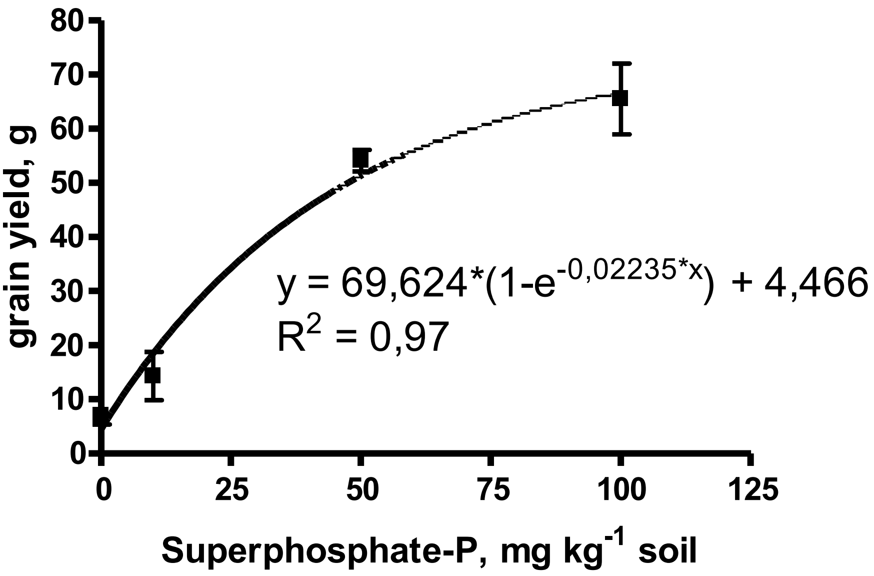

Figure 2. Yield response curve of barley due to superphosphate additions (±SD) in a pot experiment.

High availability of P in dairy cow and pig manure is probably related to the depressed P sorption strength of soil particles (Holford et al. 1997) as organic acids originating from manure may compete for the same binding sites as P (Øgaard 1996; Haynes and Mokolobate 2001).

Availability of P in dairy cow and pig feces were at a lower level compared to slurries (Table 2). Although the Hedley fractionation scheme indicated a comparable share of labile P content in manures and feces (Fig. 1), the growth experiment gave contradictory results. Phosphorus availability in dairy cow and pig feces ranged between 32-59 and 52-59 %, respectively (Table 2). Addition of phytase in the diet did not seem to improve P availability in pig feces, whereas opposite was observed with poultry manure (Table 2). In the study of Toor et al. (2005), dairy cow manure contained a lower proportion of phytic acid compared to feces, probably due to the hydrolyzation of phytic acid during manure handling and storage, increasing the share of inorganic orthophosphate in manures. Higher share of phytic acid in feces may depress P availability due to its strong binding in soil (Evans, 1985).

Of the studied manure samples, P originating from poultry manure was the least available for barley, ranging between 28-48 % (Table 1). This is contradicting to the water soluble fraction of 74-83 % according to Hedley fractionation scheme. Phytase addition in the diet increased plant availability to 48 % and this may be due to the decreased share of organic P in manure (Table 1). Diet of poultry consists mainly of cereals, where P is stored as phytic acid, whereas phytase in the diet decreases this share and increases the share of inorganic P. Availability of P may also be depressed by the higher calcium content of poultry manure compared to other manures and feces. Limestone is normally added to the diet of poultry due to the high requirement of calcium for egg production.

Among the manures and feces, fox manure P was the least soluble according to Hedley fractionation scheme. Only 27 % was labile (Table 1, Fig. 1), but availability in the pot experiment was 71 %. This result is comparable to that found for ryegrass (Ylivainio et al. 2008). Acid soluble P of fox manure probably turned into a plant available form in the slightly acidic soils.

Table 2. Barley yields in a pot experiment when P fertilization originated from manures and feces. Availability of P was estimated from the growth response curve of superphosphate (see Fig. 2).

Phosphorus source	grain yield, g	P availability, %
Dairy cow slurry	53.7	> 100
Anaerobically digested dairy cow slurry	50.7	> 100
Post digested dairy cow slurry	49.1	> 100
Separated solid fraction of dairy cow slurry	45.2	98
Dairy manure composted with peat and straw	47.1	> 100
Dairy cow feces, no additional protein in the diet	25.7	41
Dairy cow feces, rape meal in the diet	22.0	32
Dairy cow feces, soybean meal in the diet	30.9	53
Dairy cow feces, low level of concentrate in the diet	33.2	59
Dairy cow feces, high level of concentrate in the diet	28.0	46
Pig slurry	61.3	> 100
Pig feces, diet without phytase (mineral P amendment)	32.9	59
Pig feces, diet with phytase	30.2	52
Chicken manure, no phytase in the diet	24.1	37
Chicken manure, phytase included in the diet	28.8	48
Chicken manure (Broiler), mineral P amendment in the diet	19.9	28
Fox manure	59.9	71

As P deficiency depressed barley growth, utilization of other nutrients, both from soil as well as those nutrients taken by the plants, were depressed. This is evident with higher content of N in grain and straw in those treatments suffering from P deficiency (Table 3). Highest concentration of N was found in the control treatment.

Table 3. Phosphorus (P) and nitrogen (N) concentrations in barley grains and straw, mg g^{-1} DW.

Treatment/P source	grain		straw	
	P	N	P	N
Control	2.8	40.3	1.1	29.9
Dairy cow slurry	2.3	26.8	0.7	16.9
Anaerobically digested dairy cow slurry	2.2	27.3	0.8	17.5
Post digested dairy cow slurry	2.0	25.2	0.6	18.9
Separated solid fraction of dairy cow slurry	2.4	26.9	0.9	17.0
Dairy manure composted with peat and straw	2.0	26.0	1.0	21.8
Dairy cow feces, no additional protein in the diet	2.9	32.0	1.0	21.6
Dairy cow feces, rape meal in the diet	3.0	32.9	0.9	23.0
Dairy cow feces, soybean meal in the diet	3.0	33.1	0.8	19.9
Dairy cow feces, low level of concentrate in the diet	2.9	31.7	0.9	18.8
Dairy cow feces, high level of concentrate in the diet	2.7	30.6	0.8	20.7
Pig slurry	2.0	23.7	0.8	15.7
Pig feces, diet without phytase (mineral P amendment)	2.9	33.3	0.7	19.0
Pig feces, diet with phytase	3.0	32.6	0.8	19.6
Poultry manure (chicken), diet without phytase	2.7	34.5	1.0	25.2
Poultry manure (chicken), diet with phytase	2.7	33.1	0.7	23.9
Poultry manure (broiler), diet with mineral P supplements	2.8	34.6	0.8	23.2
Fox manure	2.2	23.9	1.1	17.0
Superphosphate, 10 mg P kg^{-1}	2.3	32.6	0.7	27.2
Superphosphate, 50 mg P kg^{-1}	1.8	22.9	0.6	16.0
Superphosphate, 100 mg P kg^{-1}	2.1	23.1	0.8	12.8

4 Conclusions

Share of water soluble P fraction in dairy, pig and poultry manures ranged between 55-91 %, indicating different P leaching potential if rainfall occurs before manure is incorporated into soil. In the feces of dairy and pig, the share of water soluble P was more variable (18-80 %) compared to manures and was greatly influenced by the composition of animal's diet. Also the total content of P in feces was greatly affected by the diet. Therefore, adjusting P content in the diet according to animal's requirement decreases P content in manures and reduces P accumulation in soil after manure spreading. Changes in P solubility may also be an efficient way to mitigate immediate P leaching potential caused by manure spreading.

As such, the share of labile P (water + 0.5 M $NaHCO_3$ extractable P content) was a poor indicator for plant available P content as was evident with dairy cow and pig feces and poultry manure. Although the solubility of P in poultry manure was at the same level as in dairy cow and pig manures, plant-availability was at the most about 60 %. This inconsistency may be due to the higher share of phytic acid in feces and poultry manures. Compositions of fox and poultry diets have a strong effect on immediate P availability. While meat and bone meal is an important source of P in the diet of fur animals, thus depressing P solubility in manure, in slightly acid soils it turned into a plant available form. However, although poultry manure P was more soluble than that of fox manure, plant availability was less. This is probably related to speciation of P in manures, as poultry manure contained highest level of organic P among all manures and feces, probably as phytic acid that is strongly adsorbed in soil and thus immediately unavailable for plants. Nevertheless, further studies should be carried out to define the mechanisms controlling the plant-availability of poultry manure P. Phosphorus originating from dairy cow and pig slurry and composted dairy cow manure was comparable to mineral P fertilizer.

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This report in brief

Manure is a significant source of phosphorus (P), and its P content is nowadays more than what is sold as mineral fertilizer in Finland. Efficient utilization of manure based P, both from environmental and economical point of view requires knowledge of P solubility and availability. In this study we used Hedley fractionation to evaluate P solubility and pot experiment to estimate plant availability of P in different types of manures and feces. Barley (Hordeum vulgare var. Elmeri) was the test plant in the pot experiment.

The investigated manure types consisted of dairy cow, pig, poultry and fox manures. Samples of dairy cow and pig feces originated from feeding trials with different types of diets. Water-soluble P was the main P fraction in dairy cow, pig and poultry manures (55-91 %), but in fox manure only 19 %. Anaerobic digestion reduced the water-soluble P fraction in dairy cow slurry, but the labile P content (water + 0.5 M NaHCO3 –extractable P) was not affected. Use of phytase in the poultry diet increased labile P content in manure. Most of the P in fox manure was acid soluble (71 %). Poultry manure contained the highest share of organic P (45 %), while phytase in the diet reduced it down to 31 %.

This report was prepared as part of work package 4 on Standardisation of Manure Types with Focus on Phosphorus in the project Baltic Manure.

About the project

The Baltic Sea Region is an area of intensive agricultural production. Animal manure is often considered to be a waste product and an environmental problem.

The long-term strategic objective of the project Baltic Manure is to change the general perception of manure from a waste product to a resource. This is done through research and by identifying inherent business opportunities with the proper manure handling technologies and policy framework.

To achieve this objective, three interconnected manure forums has been established with the focus areas of Knowledge, Policy and Business.

Read more at www.balticmanure.eu.

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