Effects of different treatments of cattle slurry manure on water-extractable phosphorus

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Abstract

Cattle slurry manure applied to land increases the risk of phosphorus (P) movement to surface waters, which may lead to eutrophication. The water-extractable fraction of P in slurry manure is correlated with P concentration in runoff from soils amended with slurry smanure, and thus is an effective indicator of environmental P loss. We evaluated the water-extractable P (WEP) and readily soluble P (RSP; i.e., P extractable in a single water extract) contents in slurry manure from nine farms. On some farms, the additive Euromestmix® (MX) is used to complex N-compounds in the slurry manure, but the effect of MX on P in the slurry manure is not known. We also focused on methodological factors affecting the measurement of P in slurry manure. Drying the slurry manure before analysis decreased WEP and RSP contents. Dilution of slurry manure by varying the dry matter-to-distilled water ratio increased the water-extractable fractions. Analysis of calcium and magnesium contents in water showed that these minerals are involved in the release of P in water. Total RSP content of slurry manures from the nine farms ranged from 1.83 to 4.06 mg P per g dry matter. A substantial portion of total P in the slurry manure occurred in the water-extractable (46–71%) and the readily soluble P fractions (24–51%). The addition of MX tended to decrease the readily soluble inorganic P content of slurry manures.

Additional keywords: readily soluble phosphorus, Euromestmix®, dairy cows, nutrient management, eutrophication

Introduction

In the Netherlands, agriculture is the main contributor to nonpoint source pollution of surface waters. Dairy farming occupies 64% of Dutch farmland (Van Der Molen *et al.*, 1998), generating about 35% of the national phosphate surplus and 60% of the nitrogen (N) losses (Van Bruchem *et al.*, 1999). Long-term agricultural fertilization has increased the potential for phosphorus (P) enrichment of agricultural runoff, which affects water quality. From 1987 onward, the Dutch government has taken measures to reduce the nutrient losses and associated pollution (Neeteson, 2000). As a result, nutrient surplusses decreased, but nevertheless remained unacceptably high (Oenema & Roest, 1998).

Guidelines for farmers to improve nutrient management, taking into account farm objectives and farm conditions, can be developed through farming systems research (Aarts *et al.*, 2000). In the province of Friesland, in the northern part of the Netherlands, dairy farmers organized within the co-operatives 'Vereniging Eastmars Lânsdouwe' (VEL) and 'Vereniging Agrarisch Natuur en Landschapsbeheer Achtkarspelen' (VANLA) are collaborating with Wageningen University and Research Centre to reduce negative effects of farming on the environment. The focus is on the reduction of N-emissions. The protein content of the animal diet has been lowered to reduce N emissions and improve N dynamics within the farming operations. Dietary manipulation is one means to reduce N in cattle slurry manure and subsequent ammonia volatilization (James *et al.*, 1999). To further improve slurry manure quality some farmers add Euromestmix® (MX), a powder containing chalk and clay minerals, to the manure. It is hypothesized that MX affects the complexation of N- and some toxic compounds. There is increasing interest in post-excretion treatment to chemically bind or remove dissolved reactive N and P from slurry manure before it is applied to the field (Burton, 1999). In the present study, research emphasis is placed on P, as no information is currently available on how MX affects slurry manure P.

Many studies have examined methods that best correlate soil P levels to concentrations of P in runoff (e.g. Sharpley, 1995; Torrent & Delgado, 2001) and found an excellent correlation between water-extractable P in soil and dissolved reactive P concentrations in runoff. Moreover, the readily soluble P (RSP, i.e., soluble P in a single water extract) content in manure has been correlated with dissolved reactive P concentrations in the runoff (e.g. Sharpley & Moyer, 2000; Kleinman *et al.,* 2002a). Phosphorus analyses reported in the literature are often performed using dry manure, as wet manure storage requires refrigerating or freezing facilities. However, there is evidence that drying affects manure N and C mineralization characteristics, which are not affected by refrigeration or freezing (Van Kessel *et al.*, 1999). Methods of analysis for manure P also vary in sample-to-distilled water ratio. We evaluated the effects of selected analytical parameters on water-extractable P content of slurry manure by comparing oven-dried and refrigerated wet samples.

The aim of this paper is to determine the effect of slurry manure treatments (e.g. drying, water-to-slurry ratio) on the water-extractable P contents of slurry manure and finally to evaluate the effects of Euromestmix® addition on soluble P.

Materials and methods

Slurry manure sampling and analysis

Cattle slurry manure (C0) was collected from experimental farm De Ossekampen, a dairy research facility of Wageningen University and Research Centre. In addition, eight dairy farms in the province of Friesland in the northern part of the Netherlands were selected for slurry manure collection (Table 1). The dairy cattle were housed in cubicle buildings with slatted floors. Faeces and urine were mixed as slurry in the underfloor pit, which may also contain wash water from the milking parlour and some bedding material (sawdust). Four farmers added Euromestmix® (MX, Europlant BV, Appelscha, The Netherlands) to the slurry manure (MX1–MX4, with 2 kg MX per m^3 slurry manure per year over at least 7 years). No MX was added to the slurry manure produced at the other four farms and these manures were considered conventional slurries (C1–C4). The nine slurries were sampled between 13 March and 5 April 2002, just before the slurry was applied to the land. On each farm 10 slurry manure subsamples (ca. 300 cm^3 each) were collected from different locations in the in-ground pit storage and mixed in a plastic container from which 1 litre was taken as a representative sample (Crouse *et al.*, 2000). In the laboratory, slurries were mixed in a blender and stored at 4 °C in sealed plastic containers for 1 to 3 weeks before analysis. A 500-

Table 1. Characteristics of the cattle slurry manures from the selected farms.

Farm	Manure characteristics					
	Type[1]	pH	DM[2] (%)	Org. C	Total N[3]	Total P[3]
				- - - - - - - - (mg per g DM)	- - - - - - - -	
No Euromestmix® used						
'De Ossekampen', Wageningen	C0	7.11	9.3	486	26.1(0.07)b[4]	7.91(0.04)b
Mts. Boersma, Twijzel	C1	7.19	8.0	469	26.8(0.15)c	8.88(0.04)f
Mts. v/d Veen, Eastermar	C2	7.05	8.2	505	25.5(0.28)ab	8.28(0.11)d
Mts. Veenstra. Twijzel	C3	7.07	8.6	499	30.7(0.09)d	7.43(0.04)a
Mts. v/d Wal, Kootstertille	C4	6.98	9.6	504	26.0(0.47)b	7.48(0.05)a
Euromestmix® used						
Mts. Bloemhoff, Surhuisterveen	MX1	6.95	9.3	487	25.0(0.12)a	7.92(0.13)b
Mts. Douma, Gerkes-Klooster	MX2	6.81	9.5	468	25.6(0.33)ab	8.68(0.10)e
Mts. Hoeksma, Drogeham	MX3	6.87	12.3	471	26.3(0.38)bc	8.17(0.12)cd
Mts. Oosterhof, Eastermar	MX4	6.90	9.0	478	26.8(0.21)c	8.01(0.01)bc

[1] C = conventional slurry manure; MX = slurry manure to which Euromestmix® was added.
[2] DM = dry matter.
[3] Total N and total P as determined in acid digest. Standard deviations in parentheses.
[4] Values in a column, followed by the same letter are not statistically different ($P \leq 0.05$).

ml subsample was oven-dried at 60 to 65 °C, ground to pass a 1-mm mesh (Knowlton & Herbein, 2002), and stored in sealed containers at room temperature until further laboratory use.

Dry matter (DM) content of all slurry manures was determined by drying at 60 to 65 °C for 48 hours in a gravity air oven (Self-Davis & Moore, 2000; Dou *et al.*, 2001). Analyses were performed on 3 to 6 replicates of each well-mixed slurry manure sample. Variation between samples was less than 5%. Slurry manure was analysed for total N, total P and total organic C. Subsamples of 0.3 g dry slurry manure were digested in tubes with H_2SO_4, salicylic acid, H_2O_2 and selenium at 330 °C (Novozamsky *et al.*, 1983), and total N and P were determined in the acid digest using segmented-flow analysis. Total organic carbon was determined by sulfochromic oxidation (Walinga *et al.*, 1992) and measured spectrophotometrically using glucose as a standard C-compound.

Effect of methodological variables on water-extractable P and readily soluble P in slurry manure

To assess the effect of drying on the water-extractable P content of the manure, six replicates of the slurry manure C0 were used in wet and dry forms with a slurry-to-distilled water ratio of 1:60 (w/v = g DM equivalent weight/ml). The procedure involved 0.5 g slurry manure (dry-weight equivalent) mixed with 30 ml distilled water and shaken end-over-end for 1 hour at room temperature (21 ± 3 °C). Kleinman *et al.* (2002b) showed that an extraction time of 1 hour provided the best relationships between water-extractable P in manure and dissolved reactive P concentration in runoff. The DM content (Table 1) was used to calculate the amount of wet manure to be extracted. Mixtures were centrifuged for 15 min at a relative centrifugal force (RCF) of 2000 × *g* and filtered through a medium-porosity filter paper (Whatman No. 2). Additionally, the pH was measured in the filtrates. It should be noted that we compared a RCF of 2000 × *g* with 5000 × *g* and found no statistically significant difference ($P > 0.05$) in P concentration (data not shown). The RCF of 2000 × *g* was preferred to 5000 × *g* in order to optimize the use of laboratory facilities. Aliquots of each filtrate were analysed for water-extractable inorganic P by the spectrophotometric method of Murphy & Riley as adapted by John (1970) and for total water-extractable P by inductively coupled plasma-atomic emission spectroscopy (ICP-AES). In addition, water-extractable Ca and Mg in the filtrates were determined by ICP-AES.

To assess the effect of the slurry-to-distilled water ratio, six samples of wet slurry manure C0 were shaken for 1 hour at slurry (dry wt basis)-to-distilled water ratios of 1:60 and 1:250. After centrifugation and filtration the P content was determined spectrophotometrically and by ICP-AES.

To investigate P release patterns, repeated extractions were conducted by shaking, centrifuging and filtering the same slurry manure samples several times in water and analysing the filtrates individually. Repeated shaking and filtering were performed until the P concentration in the last filtrate was negligible (i.e., after five repetitions). The sum of water-extractable total P and the sum of water-extractable inorganic P in the successive extractions are referred to as WEP_t and WEP_i, respectively. The amount of total P and inorganic P in the first extract is called the readily soluble total P (RSP_t) and the readily soluble inorganic P (RSP_i), respectively.

Effect of Euromestmix® on water-extractable P

Water-extractable P of the slurry manure was determined using a 1:60 slurry (dry wt basis)-to-distilled water ratio. Three 0.5-g samples of dry slurry manure were extracted by end-over-end shaking with 30 ml distilled water for 1 hour, followed by centrifugation and filtration. Repeated 1-hour shaking and filtration were performed until the P in the last filtrate was negligible. Extractable P was measured in individual filtrates by spectrophotometry and ICP-AES to assess water-extractable inorganic P and water-extractable total P, respectively.

Data analysis

Analyses of variance and means separation by Duncan's Multiple Range Test at a significance level of 5% were performed using STATISTICA (data analyses software system, version 6, StatSoft, Inc.). The mean contents are expressed on a dry matter basis (mg per g DM).

Results and discussion

Chemical composition of slurry manure

The DM content of the slurry manure ranged from 8.0 to 12.3% (Table 1). In the literature, manure is usually defined as slurry if DM content is within the range of 3–12% (Van Kessel & Reeves, 2000).

Total P and N contents varied among slurry materials (Table 1) whereas total organic C contents were not significantly different ($P < 0.05$). The P and N contents were in the range of values reported by Van Kessel & Reeves (2000) for 64 samples of dairy slurries. Total P in slurry manure acid digests was on average 7.99 mg P per g DM (Table 1) for the conventional group (C0–C4) and 8.19 mg P per g DM for slurries amended with Euromestmix® (MX1–MX4). The addition of Euromestmix® did not significantly affect total P content of the slurry manure. Total P content in dairy manure can vary greatly as a function of animal diet, manure collection, treatment, and storage (Barnett, 1994; Eck & Stewart, 1995). Much of the P consumed by cows is excreted in the dung (Greaves *et al.*, 1999; Wu *et al.*, 2001). In the literature a close relationship between total P excretion and feed P intake has been reported (e.g. Moir *et al.*, 1960; Morse *et al.*, 1992; Knowlton & Herbein, 2002; Powell *et al.*, 2001; Dou *et al.*, 2002). We therefore expect the statistically significant farm-to-farm differences in total P (Table 1) to be due to differences in cow diets.

Effect of methodological variables on slurry manure water-extractable P and readily soluble P

During repeated 1-hour extractions of dry material, P release followed a general pattern of a rapid decrease with increasing number of repetitions (Figure 1). Water-extractable total P (WEP_t) was 4.44 mg P per g DM. WEP_t released from wet slurry manure using the same 1:60 slurry (dry wt basis)-to-water ratio was not significantly different from dried samples (4.67 mg P per g DM). However, the pattern of release differed: for the extractions of wet material P content in the first extraction was lower and in the second and the third extractions higher than in the extractions of dried slurry manure. Water-extractable Ca content in the first extract was higher in the wet slurry manure than in the dried material whereas Mg content was lower (Figures 1B and 1C). The extraction of wet slurry manure using a ratio of 1:250 resulted in a higher release of P in water (WEP_t = 6.72 mg P per g DM), whereas the general pattern of release was similar to that of dry material. Also Kleinman *et al.* (2002b) reported an increase of WEP_t with greater dilution ratio, probably because of the dissolution of calcium phosphates. Phosphorus solubility is controlled by cations that are more abundant than P in the solution and by pH conditions (Lindsay, 1979). Water-extractable total P of each extract was positively correlated with water-extractable Ca and Mg contents and pH, with the highest correlation coefficients for the dry samples (WEP_t & Ca: $R^2 = 0.95$; WEP_t & Mg: $R^2 = 0.96$; WEP_t & pH: $R^2 = 0.67$; with $P < 0.0001$). This could suggest that there are Ca and Mg phosphate minerals in the slurry manure that affect water-extractable P. Gerritse & Ekstren (1978) showed similar effects of Ca and Mg concentrations and pH on inorganic P concentration in a pig slurry solution. The inorganic mineral magnesium ammonium phosphate hexahydrate ($MgNH_4PO_4 \cdot 6H_2O$), more commonly known as struvite, is one of the minerals that can occur in animal wastes (Bril & Salomons, 1990). Salutsky *et al.* (1972) determined that a solution pH above 7 enhanced struvite formation. The critical low nutrient limit for mineral formation (i.e., crystallization process) was found by Westerman *et al.* (1985) to be 30 mg per litre of P and 15 mg per litre of Mg, which are values lower than those found in the present study. The dissolution of Ca- and Mg-phosphates itself is affected by the conditions during extraction (slurry-to-water ratio, dry versus wet sample) and will have influenced water-extractable Ca and Mg as well as P.

Water-extractable P and readily soluble P

Water-extractable total P (WEP_t) accounted for 46 to 71% of total P content of the slurry manure as determined in acid digest (Table 2). Figure 2 shows the water-extractable P contents in five successive water extractions of the nine slurries. The amount of P release declined dramatically between the first and the second extraction, followed by a more gradual decrease thereafter. Similar P release patterns have been reported by Dou *et al.* (2000; 2002) for dairy manure and faeces samples. The soluble total P content in the first filtrate, i.e., readily soluble total P (RSP_t), accounted for approximately 50 to 75% of the cumulative total P released over five extractions (i.e., WEP_t). The P content in the 2nd–5th extracts did not differ significantly among the slurry

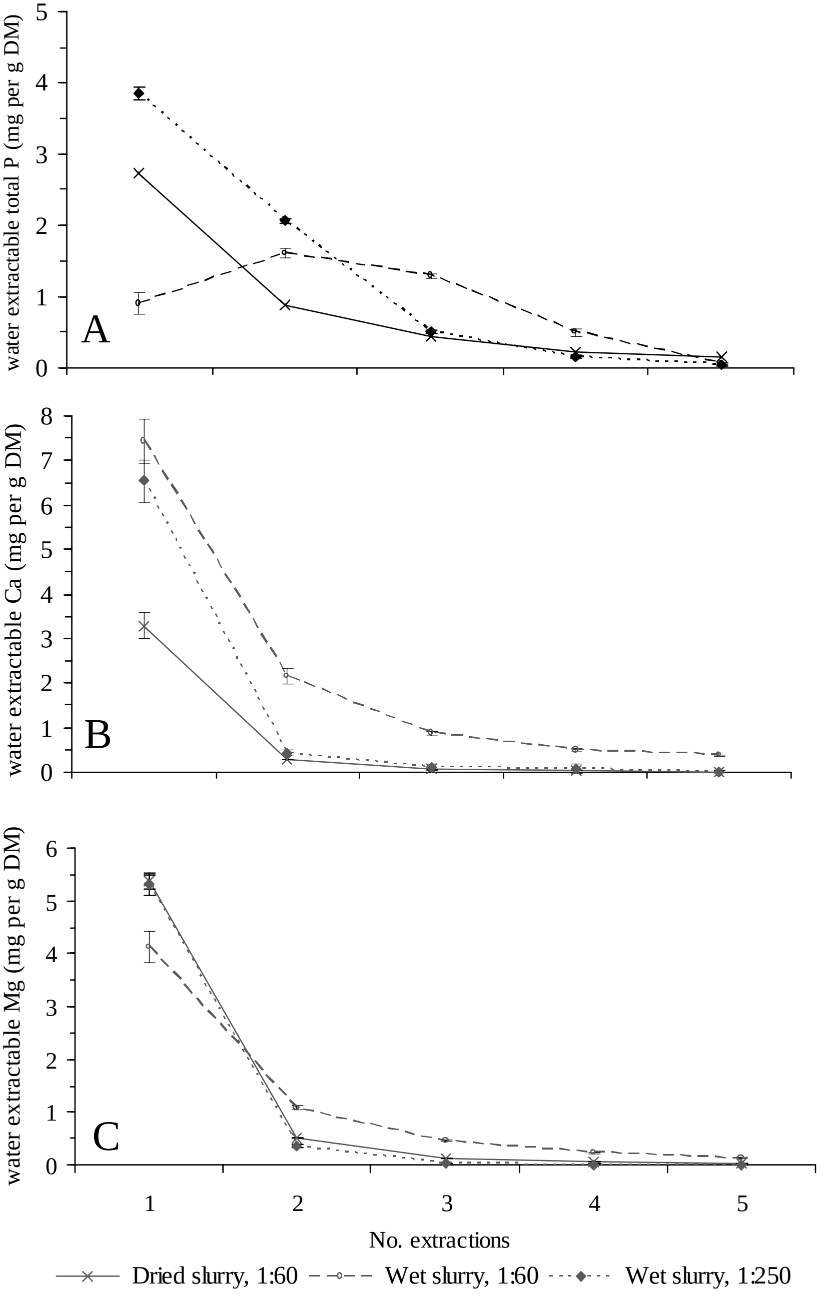

Figure 1. Water-extractable total phosphorus (A), calcium (B) and magnesium (C) release from Co cattle slurry manure in five successive 1-hour extractions as affected by nature of the sample (dried versus wet slurry manure) and the slurry (dry wt basis)-to-water ratio.

Table 2. Water extractable total P (WEP_t), readily soluble inorganic P (RSP_i) and readily soluble total P (RSP_t) contents of the cattle slurry manures collected at the nine farms.

Manure[1]	WEP_t[2]		RSP_i[3]	RSP_t[3]		RSP_i/RSP_t
	mg P per g DM	%TP[4]	mg P per g DM	mg P per g DM	%TP	
C0	4.44	56.1	2.34(0.01)c[5]	2.73(0.09)c	34.5	0.86(0.03)c
C1	5.19	58.4	2.86(0.04)d	3.29(0.06)d	37.0	0.87(0.03)c
C2	5.58	67.4	3.87(0.12)f	3.93(0.06)f	47.5	0.98(0.02)d
C3	4.97	66.9	3.64(0.09)e	3.80(0.07)e	51.1	0.95(0.02)d
C4	3.44	46.0	1.53(0.10)b	1.83(0.03)a	24.5	0.83(0.05)c
MX1	4.52	57.1	1.66(0.10)b	2.38(0.09)b	30.1	0.69(0.02)b
MX2	6.18	71.2	3.82(0.13)f	4.06(0.08)g	46.8	0.94(0.04)d
MX3	4.07	49.8	1.54(0.07)b	2.32(0.04)b	28.4	0.66(0.02)ab
MX4	3.84	47.9	1.24(0.09)a	1.92(0.11)a	24.0	0.64(0.02)a

1 C = conventional slurry manure; MX = slurry manure to which Euromestmix® was added. The figures 0 to 4 refer to the farms where the slurry was collected (see Table 1).

2 WEP_t = sum of soluble P contents of each repeated extraction.

3 RSP_i and RSP_t determined in the first of the series of water extracts (using spectrophotometry for RSP_i and ICP-AES for RSP_t); they are equivalent to P measured in a single water extract. Standard devitions in parentheses.

4 TP = total P in acid digest (see Table 1 for values) recovered in the first series of water extracts, i.e., as readily soluble P.

5 Values in a column, followed by the same letter are not statistically different ($P \leq 0.05$).

manures: 0.74 to 1.04 mg per g DM in the second, 0.27 to 0.49 mg per g DM in the third, 0.11 to 0.50 mg per g DM in the fourth and 0.06 to 0.27 mg per g DM in the fifth extract. So the differences in WEP_t among the tested slurry manures (Table 2) were due to differences in P content determined in the first water extraction (Figure 2). This supports the use of RSP as an indicator of the available and mobile P fraction of slurry manure. The total readily soluble P content ranged from 1.83 to 4.06 mg P per g DM and accounted for a substantial portion of total P in the slurry manure as determined in acid digest (Table 2). The range of water-extractable P contents was similar to those found in other studies using comparable conditions of extraction (Dou *et al.*, 2000; Sharpley & Moyer, 2000). For the nine slurry manures in this study, water-extractable P was strongly related to the Ca and Mg contents of the extracts ($R^2 > 0.76$; $P < 0.0001$).

Effect of Euromestmix® on slurry manure P contents

The slurry manures amended with Euromestmix® (MX1–MX4) had a readily soluble total P content statistically lower (average RSP_t = 2.67 mg P per g DM) than the

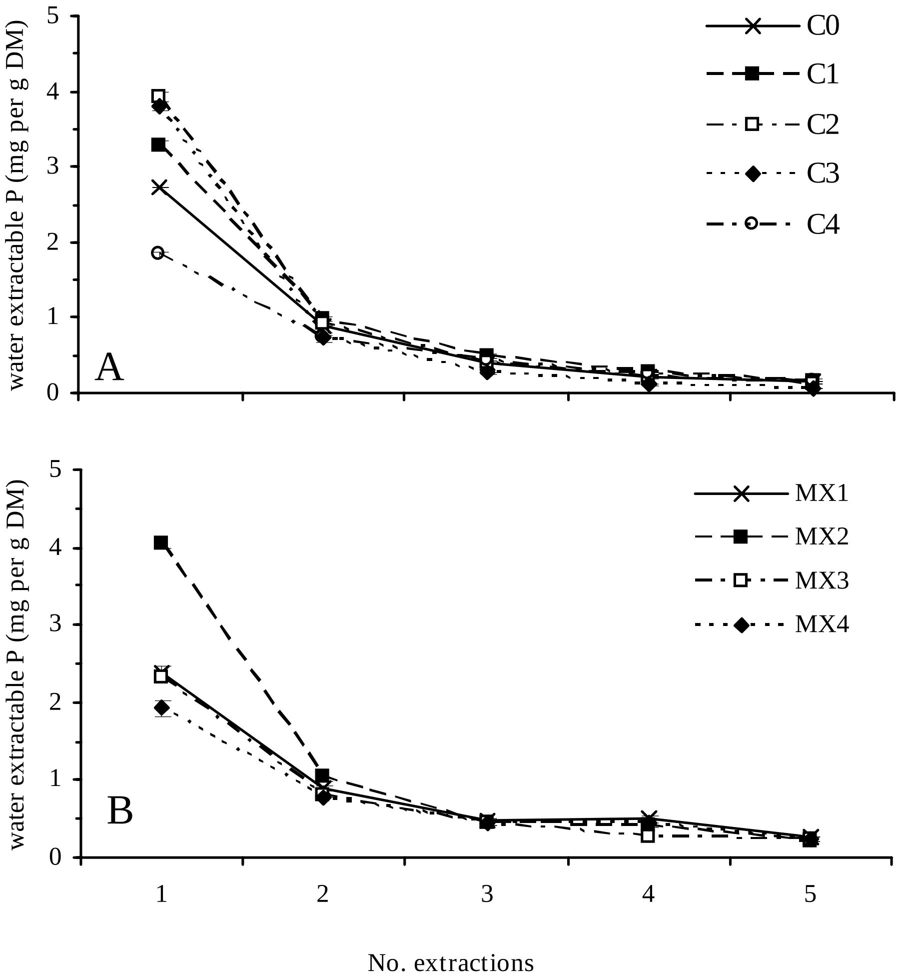

Figure 2. Release of water-extractable total phosphorus from dried cattle slurry manure samples in successive 1-hour extractions with water. Slurry manures were collected at farms that dit not add MX (A) and at farms that added MX to the slurry (B).

conventional slurries (C0–C4, 3.11 mg P per g DM). The ratio between the RSP_i and the RSP_t contents in the slurry manure (Table 2) was significantly ($P = 0.0002$) affected by the addition of MX (0.90 on average in controls versus 0.73 in slurry manure amended with MX). Readily soluble inorganic P (RSP_i) accounted for 65–98% of the RSP_t. This reflects the amount of slurry manure P that is most susceptible to potential losses once the manure is surface-applied (Sharpley & Moyer, 2000; Dou *et al.*, 2000; 2002). The MX tended to decrease the RSP_i content of the slurry manure (2.06 versus 2.85 mg P_i per g on average, $P = 0.0483$, one way ANOVA). Despite these statistically significant positive results, the difference between conventional and MX slurry manure can partly be related to cow diet as RSP is known to be potentially affected by cows' daily P intake (e.g. Powell *et al.,* 2001; Dou *et al.*, 2002).

Conclusions

The inorganic P content of slurry manure measured in a single water extract, i.e., readily soluble inorganic P (RSP_i), was used as an indicator of the amount of available and mobile P that is most vulnerable to runoff-loss after surface application of slurry manure. Results demonstrated that RSP_i was greatly affected by the slurry (dry wt basis)-to-water ratio and the nature of the material (dry versus fresh manure). Calcium and magnesium phosphates were involved in the P release in water. Drying the sample reduced the water-extractable fraction of slurry manure P, probably by reducing the solubility of minerals as revealed by the higher correlation coefficients between water-extractable P, Ca and Mg in dry sample extracts. Further research should focus on crystallization and dissolution processes of the minerals in the slurry manure. Varying the dry matter-to-water ratio showed that adding water to wet slurry manure increased the water-extractable P. Farmers often add water to slurry manure so that it can be agitated into a liquid consistency and handled with manure pumps and tankwagon spreaders. Farmers should be informed of the potential effect of such practices on the availability of P in slurry manure.

Euromestmix® (MX) had a statistically significant effect on the readily soluble inorganic P content of the slurries. Despite the small number of samples (9 farms) and the lack of data on the cows' P diet – which makes our findings on a MX-effect less conclusive – this could indicate a more general pattern. The treatment of slurry manure with MX may reduce the soluble inorganic P content, which can affect surface water quality after land-application of slurry manure. However, efforts should also focus on other alternatives, such as dietary P manipulation.

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