

PHOSPHORUS SOLUBILITY IN POULTRY LITTERS AND GRANULATES: INFLUENCE OF LITTER TREATMENTS AND EXTRACTION RATIOS

G. S. Toor, B. E. Haggard, M. S. Reiter, T. C. Daniel, A. M. Donoghue

ABSTRACT. Phosphorus (P) loss from soils receiving manure has been strongly correlated to the water-extractable P (WEP) applied in the manure. Our main objective in this study was to assess the effects of different treatments (granulation alone and with urea, urea plus dicyandiamide, or hydrolyzed feathermeal) on WEP of poultry litter. We obtained poultry litters from two poultry farms located in the northwest Arkansas, and the selected litters were granulated in commercial granulating plants. During granulation, urea, urea plus dicyandiamide, and hydrolyzed feathermeal were added to poultry litters, which increased the total N:P ratio of litters up to 8.51. Results showed that granulated litters had greater amounts of WEP than raw and ground litters when measured at lower extraction ratio (<1:100). However, the WEP was similar for all litters (raw, ground, heated, granulated) at 1:200 or 1:250 extraction ratios. This suggests that granulation of poultry litter does not influence the total amount of WEP in poultry litters. The extraction ratio had the greatest effect on WEP in the litter, while filter paper and method of P determination had minor effects on litter WEP. Of all water-extractable elements, Mg was most strongly correlated ($R^2 \geq 0.75$) with P in these poultry litters and granulates, suggesting that Mg-P minerals might control aqueous P concentrations in litter extracts. Based on these results, we recommend that WEP in poultry litter should be determined by using a 1:200 extraction ratio, followed by the use of Whatman No. 40 or 0.45 μm filters, and the filtrates can be preferably analyzed using inductively coupled plasma – optical emission spectroscopy (ICP-OES). In conclusion, this study shows that (1) granulation of poultry litter does not increase WEP of the poultry litter as given by 1:200 or 1:250 extraction ratios, and (2) addition of urea during granulation made the poultry litter a balanced fertilizer (N:P = 8:1) compared with raw litter (N:P = 1.35:1), which would help decrease P surpluses in intensive animal production areas when litters are land applied.

Keywords. Granulation, Poultry litter, Water-extractable P, Water-extractable Al, Cu, Fe, Mg, Zn.

In intensive poultry production areas such as the southwestern Ozarks, more poultry litter is generated than needed to meet forage nitrogen (N) requirements within individual farms. The long-term application of poultry litter at N-based rates has increased soil phosphorus (P) contents in some pastures and hayfields above agronomic requirements and environmental thresholds (Kleinman et al., 2000; Sharpley et al., 1996), raising questions about the continued feasibility of land application, especially considering recent litigation concerning the Eucha-Spavinaw Basin in the Ozarks that focused on P loss from the landscape (DeLaune et al., 2006). Approximately 70% of the poultry litter pro-

duced (~82 Mg per annum) in the Eucha-Spavinaw watershed will need to be exported from poultry farms to users in other areas to meet P management practices as defined in the settlement agreement (DeLaune et al., 2006). The pending lawsuit concerning the Illinois River Basin in the Ozarks may result in the need to export similar proportions of poultry litter from farms. Therefore, a farm-level or a watershed-level P balance of inputs and outputs is needed to provide environmental and economic sustainability in the region. This may involve exporting excess poultry litter to agronomically P-deficient areas or developing off-farm uses of litter, such as in turf, lawn, and gardens.

To facilitate poultry litter export, the cost of litter transportation needs to be reduced and economic incentives from regulatory agencies are necessary. For example, under its Manure Transport Program, the Maryland Department of Agriculture provides cost-share assistance up to \$20 per ton to transport excess manure from producing farms to other farms or facilities that can use the manure (MDA, 2006). In addition to these incentives, a decrease in poultry litter mass and volume would help reduce the cost of transport. Poultry litter granulation is one potential option for litter reduction. In the granulation process, water is used as a binding material to form granules. The granules are then heated and dried, resulting in decreased moisture content and odor in the final product. Other materials such as commercial N fertilizer or feathermeal can be added during the granulation process to increase the overall total N content and the N:P ratio of the final product. However, Haggard et al. (2005b) reported that

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pelletizing of poultry litter, which also involves heating and drying, increased the water-extractable P (WEP) of pelleted litter (measured at a 1:10 extraction ratio of poultry litter to water). There are concerns that application of litters containing greater amounts of WEP may increase P loss to waters, because WEP application rates in surface-applied manures have been positively correlated with dissolved P concentrations in runoff (Eghball et al., 2002; Haggard et al., 2005a; Haggard et al., 2005b; Kleinman et al., 2004; Vadas et al., 2004).

Researchers employ a variety of methods with variable extraction ratios, filtrations techniques, and methods of P determination to determine WEP in manures. Studies have shown that WEP in animal manures (dairy, poultry, and swine) and biosolids varies from <10% to as high as 75% of total P (Angel et al., 2005; Applegate et al., 2003; Brandt et al., 2004; Dou et al., 2002; Kleinman et al., 2005; Toor et al., 2005; Toor et al., 2006). Vadas and Kleinman (2006) reported that WEP in dairy, poultry, and swine manures always increased with an increase in extraction ratio from 1:10 to 1:250. They attributed this trend to dissolution of P from manure or to greater P desorption from manure solid phase. Wolf et al. (2005) did not find any significant difference in WEP determined in poultry, dairy, and swine manure samples that were centrifuged or filtered through a Whatman No. 40 filter. The large variation reported in WEP studies is in part due to the use of different WEP methods (extraction ratio, filtration techniques, method of P determination, etc.). Fortunately, efforts are now underway to recommend a standard WEP test for various animal manures, biosolids, and other wastes in the U.S. (Kleinman et al., 2005; Wolf et al., 2005).

Our objectives in this study were: (1) to determine the contents of WEP in normal and granulated poultry litter products (granulated alone and with urea, urea plus dicyandiamide, or hydrolyzed feathermeal); (2) to investigate the effects of extraction ratio, filtration technique, and method of P determination on WEP of poultry litter; and (3) explore relationships between WEP and other water-extractable elements in the poultry litters. To our knowledge, no other study has evaluated the effect of poultry litter granulation on WEP in the final product. This information is vital to assess the effect of litter granulation on WEP so that this technology can be successfully adopted without any negative effects on the environment.

MATERIALS AND METHODS

POULTRY LITTER COLLECTION

Poultry litters (mixture of feces and bedding material) were collected from two poultry farms in northwest Arkansas and granulated at facilities located in Pennsylvania and Arkansas. Poultry litter from one farm near Decatur, Arkansas, was ground to pass through a 5.8 mm mesh screen and thoroughly mixed using a New Holland 352 feed mill mixer. The ground and mixed poultry litter was delivered to Mars Mineral, Inc. (Mars, Pa.) and placed in a holding bin. Feed-grade urea (Mosaic Co., Plymouth, Minn.) and dicyandiamide (DCD, Agrotain Intl., LLC, Collierville, Tenn.) were placed in an adjacent bin and used during the process to produce a portion of the granulated products. The poultry litter (and additives) was fed into a bench-scale granulator (12D54L pin mixer, Mars Mineral, Inc., Mars, Pa.) with vibrating screw

feeders (series 101 and 1015 volumetric screw feeders, Acrison, Inc., Moonachie, N.J.), and water was used as the binding agent in the granulation process. Granulates were moved to a vibrating fluid bed dryer that was kept at 232°C, and then granulates were dried at 121°C. Dried granulates having the size fraction of 0.85 to 4.75 mm were collected using mesh screens.

Five treatments resulted from this litter source: (1) raw poultry litter (raw litter no. 1), (2) ground poultry litter (ground litter no. 1), (3) granulated poultry litter (granulated no. 1), (4) a granulated mixture of poultry litter plus urea (granulated no. 1 with urea), and (5) a granulated mixture of poultry litter plus urea and DCD (granulated no. 1 with urea and DCD). Urea was used to increase the total N content of the granulated litter, which increased the total N:P ratio of the litter to approximately 8:1, a value similar to most crop requirements. Dicyandiamide (DCD) is a nitrification inhibitor often used in agricultural practices to reduce nitrate losses (Amberger, 1989). The raw poultry litter was heated at 180°C for 2 h (heated litter no. 1) at our laboratory.

A second poultry litter source was obtained from Organic-Gro, Inc., in northwest Arkansas. At this facility, poultry litter was passed through a 2.5 mm vibrating screen and then mixed with hydrolyzed feathermeal before granulation. Two treatments resulted from this litter source: (1) ground poultry litter (ground litter no. 2), and (2) granulated mixture of poultry litter and hydrolyzed feathermeal (granulated no. 2 with feathermeal). This facility dried granulates to less than 8% moisture to avoid composting during storage, because granulated no. 2 with feathermeal is a commercially available product from Organic-Gro, Inc. This product has been marketed as a lawn and turf fertilizer (Organic-Gro, 2004).

POULTRY LITTER EXTRACTION AND ANALYSES

Total P in the poultry litters was determined using concentrated HNO₃ and H₂O₂ digestion followed by inductively coupled plasma – optical emission spectroscopy (ICP-OES) analysis (Zarcinas et al., 1987). Poultry litters were analyzed for total N at the University of Arkansas Analytical Soils Laboratory using a dry combustion method (model 2000, Leco Corp., St. Joseph, Mich.). Poultry litter analyses for total P and total N were conducted in triplicate.

Water-extractable elements were measured by extracting poultry litters, in triplicate, at poultry litter (dry weight equivalent) to deionized water ratios of 1:10, 1:50, 1:100, 1:200, and 1:250. For example, the 1:10 ratio had 20 g dry weight equivalent of poultry litter mixed with 200 mL of water (including ambient moisture in the poultry litter), and this volume of water (200 mL) was used in all extracts. The mixture was shaken for 1 h in a reciprocating shaker followed by centrifugation at 2900 rpm for 20 min before filtration through a 0.45 µm nylon membrane or a Whatman No. 40 filter. The filtered aliquots were analyzed for P using the automated ascorbic acid reduction method (APHA, 1992) and are referred as P in the 0.45 µm filtrate (WEP_{COLORIMETRIC-0.45 µm}) or P in the Whatman No. 40 filtrate (WEP_{COLORIMETRIC-W40}). The 0.45 µm filtrate from the various ratios was also analyzed for water-extractable Al, As, B, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Mo, Ni, P, S, Se, Ti, and Zn by ICP-OES. The WEP measured in the 0.45 µm aliquot via ICP-OES was referred as total WEP (WEP_{ICP-0.45 µm}). The difference between WEP_{ICP-0.45 µm} and WEP_{COLORIMETRIC-0.45 µm} was assumed to represent water-ex-

tractable organic P (WEP_{ORGANIC-0.45 μm}). This article focuses on WEP and its relationship to several water-extractable elements measured via ICP-OES; it does not present data on the various elements that were not significantly correlated to WEP.

STATISTICAL ANALYSES

Descriptive statistical analyses, one-way and two-way ANOVA with means separation using the least significant difference (LSD), were performed by Genstat 4.2 (5th ed., Lawes Agricultural Trust, Rothamsted, U.K.) to calculate means and standard deviations, and to test for significant effects of treatments and extraction ratios on WEP in poultry litters. Simple linear regression was used to evaluate effect of filtration (0.45 μm membrane vs. Whatman No. 40) and method of determination (colorimetric vs. ICP-OES) at a significance level of 0.05. Stepwise linear regression using Statistix 8.0 (Tallahassee, Fla.) was performed to relate WEP_{ICP-0.45 μm} and WEP_{COLORIMETRIC-0.45 μm} concentrations with other water-extractable elemental contents (Al, As, B, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Mo, Ni, S, Se, Ti, and Zn).

RESULTS

DRY MATTER, TOTAL NITROGEN, AND TOTAL PHOSPHORUS IN POULTRY LITTERS

Dry matter content of raw and ground litters was 70% to 80%, which increased to 90% to 95% in the granulated litters (table 1). The two litter sources differed in total N and P contents (table 1). Raw litter no. 1 and ground litter no. 1 had total N:P ratios between 1.32 and 1.35. Granulated litter no. 1 had lower total P (21.9 g kg⁻¹) but higher total N (41.5 g kg⁻¹) than ground litter no. 1, resulting in a higher N:P ratio (1.89) than the raw and ground litters. The addition of urea and urea plus DCD to the granulated litter increased total N by 354% to 359% and decreased total P by 25% to 29% as compared to ground litter no. 1. This significantly increased the N:P ratio in these two granulated litters to 7.98 and 8.51, compared with an N:P ratio of 1.32 in ground litter no. 1. On the other hand, ground litter from the second source had an N:P ratio of 2.45. The addition of hydrolyzed feathermeal to ground lit-

ter no. 2 during granulation increased total N by 35% and decreased total P by 15%, resulting in a significantly increasing N:P ratio of 3.91. Overall, the granulation process did not alter total P contents in the poultry litter, except for the decreases associated with the addition of urea, urea plus DCD, and feathermeal during the granulation. Total Ca:P and Mg:P ratios are included in table 1.

WATER-EXTRACTABLE P IN POULTRY LITTERS

The most important factor that influenced WEP in poultry litters was the extraction ratio. For example, with the exception of the heated litter, WEP_{COLORIMETRIC-0.45 μm} among all poultry litters was 959 to 3645 mg kg⁻¹ at 1:10 and then increased to 4228 to 6471 mg kg⁻¹ at 1:250 (table 2). For the heated litter, WEP_{COLORIMETRIC-0.45 μm} was 5756 mg kg⁻¹ at 1:10, as compared with 6301 to 6425 mg kg⁻¹ at 1:50 to 1:250 extraction ratios. At the 1:10 extraction ratio, WEP_{COLORIMETRIC-0.45 μm} was significantly (P < 0.05) greater in granulated and heated litters (2669 to 5756 mg kg⁻¹, 12% to 25% of total P) than that observed in the raw and ground litters (959 to 1135 mg kg⁻¹, 4% to 6% of total P) (table 2, fig. 1). From 1:50 to 1:100 extraction ratios, WEP_{COLORIMETRIC-0.45 μm} increased significantly (P < 0.05) for all poultry litters, with the exception of heated litter and granulated litter with urea (fig. 1). However, the difference in WEP_{COLORIMETRIC-0.45 μm} concentrations between raw or ground litters and granulated or heated litters were not as dramatic. For example, for the raw and ground litters, WEP_{COLORIMETRIC-0.45 μm} increased significantly (P < 0.05) by two-fold from 1:50 (10% to 15%) to 1:100 (16% to 22%), while for the granulated litters, WEP_{COLORIMETRIC-0.45 μm} was not significantly (P < 0.05) different at 1:50 (21% to 25%) and 1:100 (25% to 28%). The trend for increase in WEP for the raw and ground litters continued at 1:200 (25% to 26%) and 1:250 (26% to 28%) extraction ratios, whereas for the granulated and heated litters, WEP was not significantly (P < 0.05) different at 1:200 (25% to 29%) and 1:250 (26% to 29%), suggesting that these extraction ratios removed almost all of the WEP.

On the other hand, for most poultry litters the percentages of WEP_{ORGANIC-0.45 μm} were not significantly different between various extraction ratios: WEP_{ORGANIC-0.45 μm} was 0.2% to 3.8% at the 1:10 extraction ratio, 0.4% to 3.4% at

Table 1. Properties of poultry litters.^[a]

Poultry Litter Treatments	Litter Description	Dry Matter (%)	Total N (g kg ⁻¹)	Total P (g kg ⁻¹)	Total N:P	Total Ca:P	Total Mg:P
Source 1							
Raw litter no. 1	Unprocessed	69.5 a	30.6 a	22.7 de	1.35 a	1.40 a	0.331 a
Ground litter no. 1	Raw litter no. 1, sieved through a 5.8 mm sieve	69.5 a	31.1 a	23.6 e	1.32 a	1.41 a	0.327 a
Heated litter no. 1	Raw litter no. 1, heated at 180°C for 2 h	100.0 e	30.6 a	22.7 de	1.35 a	1.40 a	0.331 a
Granulated litter no. 1	Ground litter no. 1, granulated at 232°C, dried at 121°C	95.0 d	41.5 b	21.9 d	1.89 b	1.47 c	0.336 a
Granulated litter no. 1 with urea	Ground litter no. 1, granulated at 232°C, dried at 121°C, urea added	90.0 c	141.3 e	17.7 bc	7.98 d	1.45 b	0.326 a
Granulated litter no. 1 with urea and DCD	Ground litter no. 1, granulated at 232°C, dried at 121°C, urea and DCD added	90.3 c	142.9 e	16.8 b	8.51 d	1.51 d	0.333 a
Source 2							
Ground litter no. 2	Unprocessed litter sieved through a 5.8 mm sieve	80.1 b	44.9 c	18.3 c	2.45 b	1.58 e	0.317 a
Granulated litter no. 2 with feathermeal	Ground litter no. 2, granulated, hydrolyzed feathermeal added	94.6 d	60.6 d	15.5 a	3.91 c	1.62 f	0.316 a

^[a] Values followed by different letters in the same column are significantly different at P < 0.05.

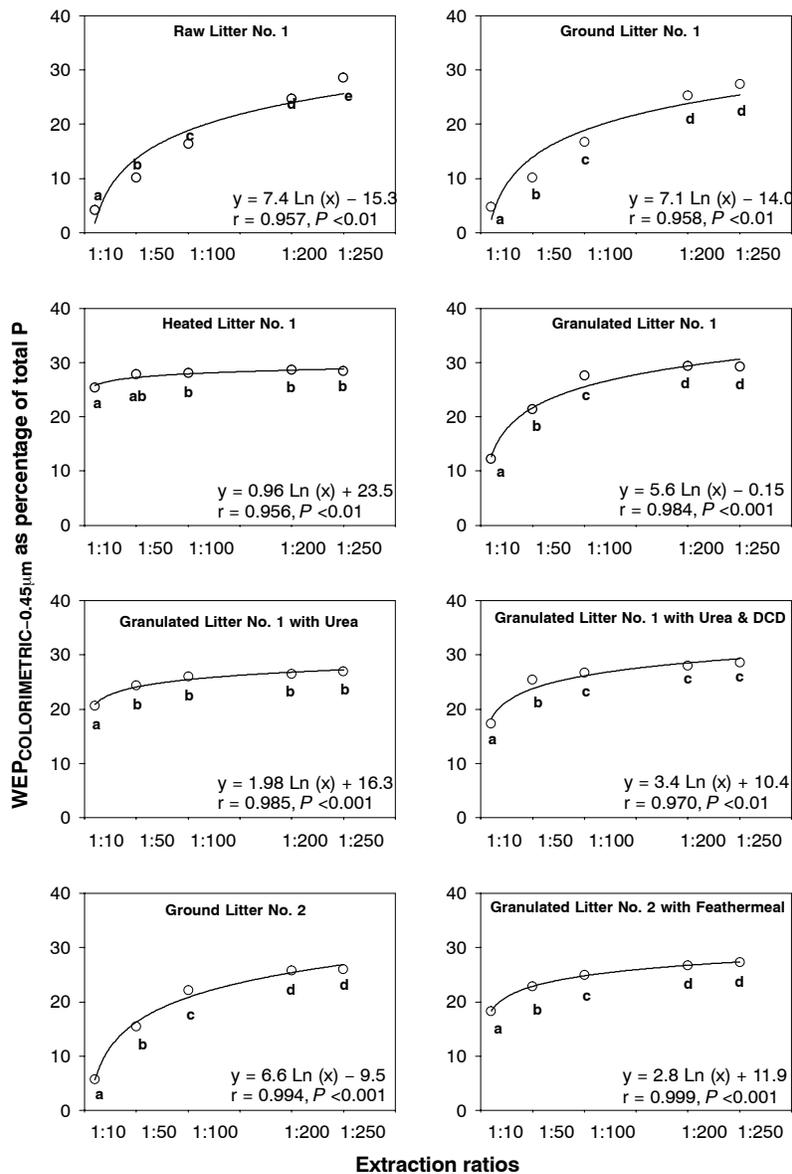


Figure 1. Water-extractable P (WEP_{COLORIMETRIC-0.45 μm}) as a percentage of total P at different extraction ratios for poultry litters. Values followed by different letters in the same graph are significantly different at $P < 0.05$.

1:50, 0.6% to 3.5% at 1:100, 0.8% to 4.6% at 1:200, and 1.2% to 4.9% at 1:250 for all poultry litters except heated litter. For the heated litter, WEP_{ORGANIC-0.45 μm} was greater than for the other litters and varied between 4.9% and 7.8% of total P from the 1:10 to 1:250 extraction ratios.

Contents of other water-extractable elements such as Ca, Fe, Mg, and Zn also increased from the 1:10 to 1:250 extraction ratios in all poultry litters. Figure 2 shows the relationship between WEP_{ICP-0.45 μm} and water-extractable Mg, Fe, Ca, and Zn. Similarly, a significant relationship was observed between WEP_{COLORIMETRIC-0.45 μm} and water-extractable Mg (data not shown). Of these water-extractable elements, Mg had highest correlation with P ($r \geq 0.87$). The stepwise linear regression of WEP_{COLORIMETRIC-0.45 μm} or WEP_{ICP-0.45 μm} with other water-extractable elements (table 3) confirmed that water-extractable Mg explained more than 75% of the variability in WEP concentrations, i.e., $R^2 \geq 0.75$. The other elements that significantly explained variation in

WEP were different for WEP_{COLORIMETRIC-0.45 μm} (Cu, S, Zn) and WEP_{ICP-0.45 μm} (Ca, Fe, Zn). Overall, these elements together explained more than 95% of the variability in WEP across the various extraction ratios (table 3).

In our samples, WEP determined by colorimetric analyses of the water extracts (WEP_{COLORIMETRIC-0.45 μm}) was linearly related with WEP determined by ICP-OES analyses of the water extracts (WEP_{ICP-0.45 μm}) with significant correlation coefficient of 0.98 and a slope of 0.87 (fig. 3), suggesting that on average WEP_{COLORIMETRIC-0.45 μm} was 87% of WEP_{ICP-0.45 μm} with the remainder being WEP_{ORGANIC-0.45 μm}. Our study also compared WEP determined in filtrates from different filters, i.e., 0.45 μm membranes and Whatman No. 40, where WEP_{COLORIMETRIC-0.45 μm} and WEP_{COLORIMETRIC-W40} were significantly correlated ($r = 0.99^{**}$; fig. 4), and the slope of this linear relationship showed that WEP_{COLORIMETRIC-0.45 μm} was approximately 98% of WEP_{COLORIMETRIC-W40}.

Table 2. Water-extractable P at different extraction ratios for poultry litters.^[a]

Poultry Litter Treatment	1:10 (mg kg ⁻¹)	1:50 (mg kg ⁻¹)	1:100 (mg kg ⁻¹)	1:200 (mg kg ⁻¹)	1:250 (mg kg ⁻¹)
WEP_{COLORIMETRIC-0.45µm}					
Raw litter no. 1	959 ±82 a	2280 ±36 a	3690 ±50 a	5588 ±168 c	6439 ±238 c
Ground litter no. 1	1135 ±101 a	2401 ±67 a	3935 ±61 b	5953 ±167 d	6471 ±213 c
Heated litter no. 1	5756 ±146 e	6301 ±199 f	6366 ±232 e	6473 ±234 e	6425 ±221 c
Granulated litter no. 1	2669 ±67 b	4684 ±61 e	6048 ±111 d	6440 ±111 e	6393 ±85 c
Granulated litter no. 1 with urea	3645 ±157 d	4328 ±161 d	4614 ±62 c	4691 ±102 b	4769 ±162 b
Granulated litter no. 1 with urea and DCD	2908 ±62 c	4290 ±112 d	4501 ±56 c	4707 ±138 b	4809 ±226 b
Ground litter no. 2	1035 ±6 a	2816 ±35 b	4042 ±57 b	4711 ±16 b	4758 ±133 b
Granulated litter no. 2 with feathermeal	2826 ±25 b	3552 ±53 c	3852 ±74 ab	4135 ±85 a	4228 ±69 a
WEP_{ORGANIC-0.45µm}					
Raw litter no. 1	318 ±81 ab	481 ±342 b	493 ±339 ab	841 ±706 bc	802 ±646 cd
Ground litter no. 1	429 ±21 b	737 ±54 b	714 ±68 b	1090 ±122 c	1166 ±98 d
Heated litter no. 1	1104 ±76 c	1769 ±97 c	1330 ±222 c	1556 ±252 d	1733 ±119 f
Granulated litter no. 1	342 ±25 ab	631 ±155 b	585 ±46 b	992 ±68 c	1077 ±376 e
Granulated litter no. 1 with urea	681 ±314 b	77 ±62 a	109 ±116 a	243 ±65 a	284 ±143 ab
Granulated litter no. 1 with urea and DCD	34 ±53 a	89 ±91 a	191 ±53 a	132 ±109 a	202 ±187 a
Ground litter no. 2	334 ±10 ab	552 ±25 b	634 ±64 b	580 ±94 b	576 ±53 bc
Granulated litter no. 2 with feathermeal	411 ±25 b	526 ±76 b	369 ±59 ab	407 ±38 ab	451 ±88 ab

[a] Means ± standard deviations. Values followed by different letters in the same column for WEP_{COLORIMETRIC-0.45µm} or WEP_{ORGANIC-0.45µm} are significantly different at P < 0.05.

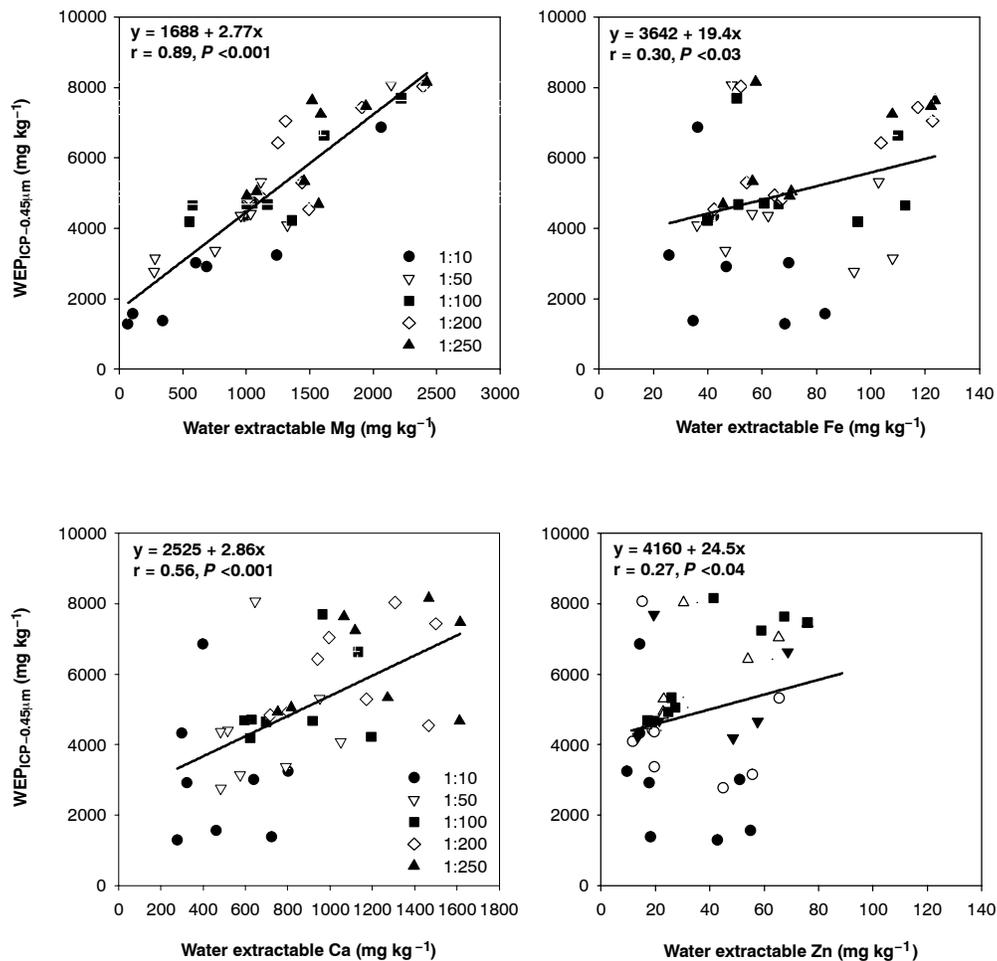


Figure 2. Relationship between water-extractable P (WEP_{ICP-0.45 µm}) and water-extractable Mg, Fe, Ca, and Zn analyzed in 0.45 µm filtrates at the different extraction ratios for all poultry litters.

Table 3. Stepwise linear regression analysis of WEP_{ICP-0.45 μm} or WEP_{COLORIMETRIC-0.45 μm} (mg kg⁻¹) and total water-extractable Al, As, B, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Mo, Ni, S, Se, Ti, and Zn (mg kg⁻¹) in the water extractions at the different ratios with raw, ground, heated, and granulated poultry litters.

Step No.	Stepwise Regression Equation ^[a]	R ²	P Value
WEP _{ICP-0.45μm} and other water-extractable elements			
1	1688.3 + 2.8Mg	0.7976	<0.01
2	-190.8 + 2.9Mg + 24.8Fe	0.9415	<0.01
3	-806.2 + 2.9Mg + 52.8Fe - 41.3Zn	0.9651	<0.01
4	-526.6 + 3.3Mg + 49.3Fe - 32.7Zn - 0.84Ca	0.9781	<0.01
Adjusted R ² for the final model = 0.9756, P < 0.01			
WEP _{COLORIMETRIC-0.45μm} and other water-extractable elements			
1	1683.6 + 2.3Mg	0.7538	<0.01
2	-118 + 2.5Mg + 9.9Cu	0.8981	<0.01
3	1594.4 + 2.6Mg + 11.4Cu - 0.3S	0.9382	<0.01
4	746.2 + 2.7Mg + 22.1Cu - 0.3S - 34.2Zn	0.9696	<0.01
Adjusted R ² for the final model = 0.9661, P < 0.01			

[a] All coefficients in various steps of regression analysis were significant at P < 0.01.

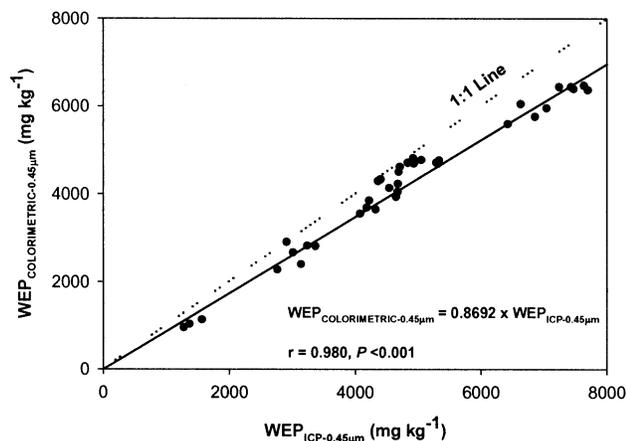


Figure 3. Relationship between water-extractable P in 0.45 μm filtrates analyzed via colorimetric (WEP_{COLORIMETRIC-0.45 μm}) and ICP-OES (WEP_{ICP-0.45 μm}) for all poultry litters. Regression was forced through the origin.

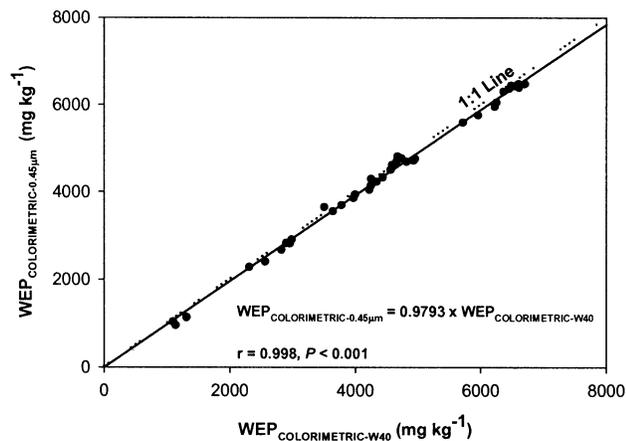


Figure 4. Relationship between water-extractable P in 0.45 μm membrane (WEP_{COLORIMETRIC-0.45 μm}) and Whatman No. 40 filtrates (WEP_{COLORIMETRIC-W40}) analyzed via colorimetric method for all poultry litters. Regression was forced through the origin.

DISCUSSION

INFLUENCE OF GRANULATION ON WEP IN POULTRY LITTERS

In this study, greater amounts of WEP were extracted at 1:10 to 1:100 ratios in the heated and granulated litters compared to raw and ground poultry litters, probably because the former litters were heated and dried. However, all litters had relatively similar WEP contents at 1:200 or 1:250 extraction ratios, suggesting that granulation does not increase the total amount of WEP (fig. 1, table 2). Perhaps the heating and drying during granulation precipitated aqueous Mg and P present in the poultry litter. As a result, when heated and granulated litters were extracted at lower extraction ratios (<1:100), the equilibrium between aqueous P and solid phase P was the factor driving increased dissolution of Mg-P compounds. Therefore, at a 1:10 to 1:100 extraction ratios, heated and granulated litters can release more P during water extraction, and the amount of P released is similar to the total amount of WEP. On the other hand, dissolution of P minerals is reduced in raw and ground litters at low extraction ratios due to the presence of aqueous Mg and P. While at higher extraction ratios (>1:200), the aqueous Mg and P concentrations in raw and ground litters are diluted, allowing the dissolution of Mg-P solid phase.

Therefore, we suggest that if poultry litters are heated and dried prior to water extractions or are extracted at 1:200 or 1:250 ratios, these poultry litters would have relatively similar measurements of WEP. Our results support the findings of Toor et al. (2005), who reported that water extraction not only removed soluble P from poultry litter, but also extracted P from solid-phase minerals. Other studies (e.g., Kleinman et al., 2005) have reported significant correlations between Mg and WEP for a range of animal manures, supporting our observations and theories on the possible association of Mg and P in poultry litters. Gungor and Karthikeyan (2005) observed that P solubility in water extracts of dairy manure was initially controlled by Mg-P minerals (struvite). Furthermore, Josan et al. (2005) and Nair et al. (2003) observed stronger correlations between Mg and P in soils that received dairy manure, suggesting that Mg-P minerals controlled P solubility in these soils. However, the effect of litter extractions and post-processing treatments on P species in litters may be better understood by using advanced spectroscopic techniques such as x-ray absorption near edge structure (XANES) spectroscopy for mineral P identification, and nuclear magnetic resonance (NMR) spectroscopy for identification of organic P forms (Toor et al., 2006).

INFLUENCE OF EXTRACTION RATIO, METHOD OF P DETERMINATION, AND FILTRATION ON WEP IN POULTRY LITTERS

Several factors, such as extraction ratio, method of P determination in filtrate (ICP-OES or colorimetric), and filtration (pore size of filter paper or membrane) can influence WEP in poultry litter, as WEP is a mixture of organic P (e.g., labile monoester P and diester P; Toor et al., 2003) and inorganic P (dissolved orthophosphates and P minerals; Toor et al., 2005). Of all these factors, extraction ratio was the most important factor that influenced WEP in poultry litters, followed by the method of P determination and filtration technique.

Table 4. Percentages of water-extractable Ca and Mg at different extraction ratios for various poultry litters.^[a]

Poultry Litter Treatment	1:10	1:50	1:100	1:200	1:250
Water-Extractable Ca (% of total Ca)					
Raw litter no. 1	0.9 ±0.2 a	1.5 ±0.4 a	2.0 ±0.4 a	3.0 ±0.8 a	3.6 ±0.7 a
Ground litter no. 1	1.4 ±0.4 a	1.7 ±0.1 ab	2.1 ±0.2 a	3.0 ±0.4 a	3.2 ±0.2 a
Heated litter no. 1	1.3 ±0.1 a	2.1 ±0.2 b	3.1 ±0.2 b	4.1 ±0.3 b	4.7 ±0.3 b
Granulated litter no. 1	2.0 ±0.2 b	3.0 ±0.2 c	3.5 ±0.5 b	4.7 ±0.6 c	5.0 ±0.6 b
Granulated litter no. 1 with urea	1.2 ±0.2 a	2.0 ±0.2 ab	2.5 ±0.3 a	3.1 ±0.4 a	3.2 ±0.4 a
Granulated litter no. 1 with urea and DCD	1.3 ±0.1 a	1.9 ±0.1 ab	2.4 ±0.1 a	2.9 ±0.1 a	3.0 ±0.1 a
Ground litter no. 2	2.5 ±0.1 b	2.7 ±0.1 c	3.2 ±0.1 b	4.1 ±0.2 b	4.4 ±0.4 b
Granulated litter no. 2 with feathermeal	3.2 ±0.2 c	4.2 ±0.4 d	4.8 ±0.5 c	5.9 ±0.6 d	6.5 ±0.9 c
Water-Extractable Mg (% of total Mg)					
Raw litter no. 1	0.9 ±0.1 a	3.7 ±0.4 a	7.4 ±0.7 a	16.7 ±2.4 a	21.2 ±1.3 c
Ground litter no. 1	1.4 ±0.6 a	3.6 ±0.1 a	7.5 ±0.3 a	16.9 ±1.3 ab	19.7 ±1.4 b
Heated litter no. 1	27.6 ±1.2 g	28.6 ±1.1 f	29.7 ±0.7 f	32.0 ±0.6 f	32.3 ±0.6 e
Granulated litter no. 1	8.2 ±0.2 c	15.1 ±0.5 c	21.9 ±0.6 d	25.9 ±0.7 d	26.4 ±0.3 d
Granulated litter no. 1 with urea	17.1 ±0.3 e	17.9 ±0.1 d	18.1 ±0.2 b	19.1 ±0.3 c	18.7 ±0.4 ab
Granulated litter no. 1 with urea and DCD	12.3 ±1.4 d	17.0 ±0.9 d	17.8 ±0.8 b	18.1 ±0.5 bc	17.9 ±1.0 a
Ground litter no. 2	5.9 ±0.2 b	13.0 ±0.2 b	20.2 ±0.5 c	24.9 ±0.8 d	25.2 ±1.6 d
Granulated litter no. 2 with feathermeal	25.3 ±0.7 f	27.0 ±0.4 e	27.7 ±0.5 e	30.5 ±0.7 e	32.0 ±1.0 e

^[a] Means ± standard deviations. Values followed by different letters in the same column for water-extractable Ca or Mg are significantly different at $P < 0.05$.

The effect of extraction ratio was more predominant in the raw and ground litters than in the heated and granulated litters (table 2, fig. 1). For the raw and ground litters, it seems that a significant increase in WEP with an increase in each extraction ratio (i.e., from 1:10 to 1:250) is probably related to the increased dissolution of P compounds. Therefore, WEP should display some relationship with other water-extractable elements across these extraction ratios. Of these elements, water-extractable Mg had the highest correlation coefficient with WEP ($r \geq 0.87$); water-extractable Mg increased from <6% at 1:10 to >20% at 1:250 for raw and ground litters (table 4). Other water-extractable elements such as Ca, Fe, and Zn only exhibited small increases with increases in the extraction ratio (fig. 2). For instance, water-extractable Ca for all litters was 0.9% to 3.2% at 1:10 and 3.0% to 6.5% at 1:250, indicating that the Ca in these litters was present in relatively less soluble forms (table 4). In the heated and granulated litters, the heating and drying increased the solubility of Mg and P. As a result, water-extractable Mg was significantly ($P < 0.05$) greater for the heated and granulated litters (8.2% to 27.6%) than for the raw and ground litters (0.9% to 5.9%) at the 1:10 extraction ratio (table 4). Ajiboye et al. (2004) also observed greater WEP contents in oven-dried (105 °C) dairy and swine manure compared to fresh manure. Lindsay (1979) suggested that Mg–P minerals, such as newberryrite and struvite, are more soluble compared to Ca–P, Fe–P, and Al–P minerals in the neutral pH range; therefore, the increases in WEP with increases in the extraction ratio may be due to the dissolution of some of these Mg–P minerals in raw and ground litters and increased solubility in heated and granulated litters prior to extractions.

The effect of extraction ratios on WEP_{ORGANIC-0.45 µm} of poultry litters was not substantial, indicating that dissolution of an Mg mineral phase is the predominant mechanism of P release. In addition, granulation of poultry litter had no clear effect on either the increased solubility of WEP_{ORGANIC-0.45 µm} or its breakdown. However, it is likely that heating and drying during granulation may have hydrolyzed labile forms of P to inorganic P (Turner et al., 2002), which were measured as WEP_{COLORIMETRIC-0.45 µm}.

The method of P determination in the filtrate affected WEP contents in the various poultry litters and granulates. For instance, WEP determined by ICP-OES was approximately 13% greater than that measured using the colorimetric method. This is because WEP determined by ICP-OES includes dissolved organic and inorganic P. The variation in WEP by method of P determination would obviously depend upon the proportion of dissolved organic and inorganic P present in poultry litters and any changes caused by post-processing of poultry litter on P forms. Haggard et al. (2005b) reported that WEP determined by ICP-OES and colorimetric method was similar in alum-treated poultry litters, while WEP determined by ICP-OES was approximately 12% greater than that determined by the colorimetric method in raw and pelleted poultry litters. Similarly, Sims et al. (2000) observed that WEP determined by ICP-OES was 4% to 28% greater than that determined by the colorimetric method in poultry litters generated from low phytic acid corn and phytase-amended diets.

The obvious reason for slightly greater (~2%) WEP contents in the Whatman No. 40 filtrates than in the 0.45 µm membrane filtrates is the greater pore size (1.0 µm compared to 0.45 µm, respectively); the larger pore size allows additional particles containing P to pass through and increase WEP measurements. The similar amounts of WEP in 0.45 µm and Whatman No. 40 filtrates suggest that the difference between these filters should not present comparison problems, and the selection of filters and pore size should be made to fit into typical individual laboratory activities. In contrast, Sims et al. (2000) observed that WEP was 62% to 78% greater in centrifuged extracts compared to extracts filtered through 0.45 µm membranes because of the presence of particulate P in the centrifuged extracts. This suggests that some standardization among laboratories is needed regarding the use of centrifugation and/or filtering in WEP determination.

PREDICTING RUNOFF PHOSPHORUS CONCENTRATIONS FROM LABORATORY EXTRACTIONS

The variability observed in WEP contents in our study suggests a need to standardize the WEP extraction and

Table 5. Addition of total N, plant-available N, and water-extractable P from application of poultry litters at 50 kg total P ha⁻¹.

Poultry Litter	Manure (dry weight, kg ha ⁻¹)	Total N (kg ha ⁻¹)	Plant-Available N (kg ha ⁻¹)	N from Fertilizer (kg ha ⁻¹)	WEP _{COLORIMETRIC-0.45µm} (1:250) (kg ha ⁻¹)
Raw litter no. 1	2119	66	17 ^[a]	274 ^[b]	13.7
Granulated litter no. 1 with urea	2825	400	320 ^[c]	–	13.5
Granulated litter no. 1 with urea and DCD	2976	425	340 ^[c]	–	14.3

^[a] Assuming 25% of total N is plant-available to rice (Golden et al., 2006).

^[b] Assuming 80% of total N from granulated poultry litter products is plant-available (Cooperband and Good, 2002).

^[c] Assuming plant-available N requirements of 291 kg ha⁻¹ for rice (134 kg ha⁻¹) – wheat (157 kg ha⁻¹) – soybean (0 kg ha⁻¹) rotation (Chapman et al., 2001; Lanny, 2001; Slaton, 2001).

analysis procedure. Although Kleinman et al. (2005) and Wolf et al. (2005) have proposed methods to extract WEP in manures, there is need to bring uniformity to WEP analysis of manures. This would help to accurately determine WEP in manures and allow comparisons among different studies. Importantly, the extraction of greater amounts of WEP from poultry litters and granulated products with increases in the extraction ratio has important implications regarding the release of WEP from poultry litter into surface runoff. For example, Vadas et al. (2005) reported that runoff P concentrations from small boxes and field plots fertilized with poultry litter were more accurately predicted using a manure P extractability coefficient, which could be based on a linear or nonlinear relationship between WEP concentration and the extraction ratio. Based on these findings, the 1:10 to 1:100 extraction ratios represents the amount of WEP that would be released from poultry litters into water infiltrating the soil surface and into the surface runoff that occurs during initial precipitation events following land application. Thus, the WEP application rates based on the 1:10 to 1:100 extraction ratios for poultry litters may explain a slightly larger amount of the variability in runoff P concentrations during rainfall simulations and small box or field plot studies (e.g., Haggard et al., 2005a), because these studies most often evaluate runoff P losses in one or two rainfall simulations following applications. On the other hand, the 1:200 or 1:250 extraction ratios better represent the total amount of WEP that could be released from poultry litters during multiple rainfall events that occur after land application. Perhaps Vadas et al. (2005) has the best approach to predicting runoff P concentrations, because this approach predicts the amount of P released from poultry litter when runoff is occurring based on data representing a range of extraction ratios.

BENEFITS OF POULTRY LITTER GRANULATION

The addition of urea, urea plus DCD, and hydrolyzed feathermeal during poultry litter granulation made the poultry litter a better fertilizer by bringing the total N:P ratio closer to most crop and forage needs. For example, the granulated litters with urea alone and urea plus DCD had total N:P ratios of approximately 8, a value similar to most grain and forage requirements (Sharpley et al., 1998). While addition of hydrolyzed feathermeal to litter during granulation increased the N:P ratio from 2.5 to 3.9, the most significant improvement in N:P ratio was achieved with the addition of urea to the poultry litter. An increased N:P ratio in poultry litter would help to reduce buildup of P in intensive animal production areas when granulated litter is land-applied to meet crop and forage nutrient requirements. Moreover, the use of granulated litter products with urea or feathermeal would reduce

the additional need for commercial N fertilizer under P-based nutrient management strategies.

For example, if poultry litter is applied at 50 kg total P ha⁻¹, the raw litter will add 17 kg plant-available N ha⁻¹, while granulated litters with urea and urea plus DCD will add 320 to 340 kg plant-available N ha⁻¹ (table 5). Assuming plant-available N requirements of 291 kg ha⁻¹ for a rice-wheat-soybean rotation (Chapman et al., 2001; Lanny, 2001; Slaton, 2001), an additional 274 kg total N ha⁻¹ from commercial N fertilizer needs to be added to meet crop N requirement in soils that receive raw litter. In comparison, no commercial N fertilizer will be needed for soils amended with granulated litter with urea or urea plus DCD. At the same time, addition of these raw and granulated poultry litters will add similar amounts of WEP_{COLORIMETRIC-0.45 µm} (13.5 to 14.3 kg ha⁻¹ at the 1:250 extraction ratio) to the soil. Thus, soil P availability and potential of P loss should be similar for the raw and granulated litter products. Another significant benefit of granulation of poultry litter is increased dry matter content (>90%) of the granulated products, which would prevent the decomposition of litter during storage and reduce transport and application costs.

SUMMARY AND CONCLUSIONS

Our study has shown that extraction ratio is the most critical factor influencing WEP amounts in poultry litters. For example, the 1:10 extraction ratio showed several significant differences between raw or ground litters and granulated or heated litters, whereas the 1:200 and 1:250 ratios showed that the total amount of WEP was similar among all poultry litters. So, one might conclude that granulated poultry litters have a higher potential for P transport in runoff based on WEP from the 1:10 extraction ratio. Importantly, the greater variability observed in WEP with the variation in extraction ratio suggests that WEP is not a fixed entity in poultry litter, and the WEP term should be used with caution or perhaps should be specified with other variable factors such as extraction ratio, method of P determination (i.e., colorimetric or ICP-OES), and filtration technique, which would reduce confusion and aid comparisons across various research studies. In this study, the method of P determination in filtrates (ICP-OES vs. colorimetric) had some impact on WEP, while the filtration through Whatman No. 40 or 0.45 µm filters had only minor affect on WEP.

Our results are in agreement with those of previous studies (Kleinman et al., 2005; Wolf et al., 2005), in which researchers showed that extraction ratio is the single most important factor determining WEP in manures. Based on the results of this study, we recommend that poultry litters should be extracted on a dry weight equivalent basis using a 1:200 extrac-

tion ratio. The method of P determination should be chosen to suit what would be easiest for commercial laboratories required to conduct WEP analysis, which would probably be ICP-OES analysis of filtrates with a filtrate from Whatman No. 40 or similar filters. However, if poultry litters are heated and dried (or granulated) prior to water extractions, then a 1:100 extraction ratio followed by P determination by ICP-OES in Whatman No. 40 filtrates would extract relatively similar amounts of WEP as a 1:200 extraction ratio. Despite what technique is used to evaluate WEP, this study showed that water-extractable Mg was strongly correlated to WEP and likely plays a role in the control of the dissolution of inorganic P into water. In summary, granulation of poultry litter does not increase WEP of litter when assessed at 1:200 or 1:250 extraction ratios. From the land application perspective, the granulated litter with urea or urea plus DCD may be the best product given its greater total N:P ratio (8:1) and similar amounts of WEP as compared to raw litter. The use of granulated litters in intensive animal production watersheds would help reduce the P surplus and P saturation of soils and decrease P losses to natural waters.

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NOMENCLATURE

DCD	= dicyandiamide.
ICP-OES	= inductively coupled plasma-optical emission spectroscopy.
WEP	= water-extractable P.
WEP _{ICP-0.45 μm}	= water-extractable total P determined in 0.45 μm filtered extract by ICP-OES.
WEP _{COLORIMETRIC-0.45 μm}	= water-extractable P determined in 0.45 μm filtered extract by colorimetric method.
WEP _{ORGANIC-0.45 μm}	= difference between WEP _{ICP-0.45 μm} and WEP _{COLORIMETRIC-0.45 μm} .
WEP _{COLORIMETRIC-W40}	= water-extractable P determined in Whatman No. 40 filtered extract by colorimetric method.