

Evaluating Morgan's Phosphorus Test as an Environmental Indicator

END OF PROJECT REPORT

RMIS 4976

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This project was part-funded by the EPA RTDI programme

April 2005

ISBN No. 1 84170 387 7



Teagasc Headquarters, Oak Park, Carlow

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Summary

Phosphorus loss from soil to water is a major component of surface water pollution in Ireland and water quality reports from the EPA have shown an increasing upward trend in slight and moderate pollution often attributed to agricultural phosphorus (P). There is a need therefore for indicators of P loss from agriculture that might predict areas vulnerable to P loss. Soil test P is a typical parameter often used in risk assessment and catchment modeling and although originally designed as an agronomic indicator of P requirements for crop growth, it is often used to indicate risk of P loss from agricultural areas.

Soil phosphorus (P) testing in Ireland uses Morgan's reagent from samples taken to 10 cm depth for agronomic recommendations, however, its suitability as an environmental indicator has been questioned in terms of sample depth and extraction solution. In this study, seven grassland sites were sampled to 2, 5 and 10 cm depths and extracted for Morgan's P, the standard agronomic test, as well as, iron-oxide impregnated paper strip P (FeO-P), calcium chloride extractable P (CaCl₂-P) and water soluble P (WSP), all proposed as environmental soil tests. Extractable soil P increased with decreasing sample depth, as did variances in each test, such that, 2 cm samples had highest concentrations and variances. The current standard sample depth (10 cm) was linearly correlated with corresponding data from samples taken to 2 and 5 cm, indicating that surface soil P can be consistently estimated from the current standard depth. When soil tests were compared with Dissolved reactive P (DRP) in overland flow collected from field studies, certain soil tests were better indicators of P loss than others. The relative difference in Morgan's P values at the standard sample depth (10 cm) was reflected in the relative difference in P loss between the two sites. Values of DRP collected from two sites ranged from 0.032-0.67 mg/l at the low P site and 0.261-0.620 at the high P site. Average DRP values from the high P site and maximum DRP values from the low P site were simulated using water-soluble P extracts. In this study, Morgan's P to 10 cm gave a good indication of the relative difference in DRP loss between the two sites.

Introduction

Diffuse losses of agricultural phosphorus (P) are a major component of surface water pollution. Phosphorus in overland flow originating from grassland soils can exist in dissolved reactive and particulate form, with the former being more dominant. Losses to overland flow are typically measured by edge of field monitoring of the dissolved fraction. These are attributed to desorption of P from soil and losses of recently applied fertilizer and manure P, or incidental losses. Relating soil P to losses in overland flow relies on a measure of soil P concentration as an indicator of potential loss usually employing soil P tests. These were originally designed as an indicator of plant available P for crop growth and fertilizer recommendations. This has been a useful approach thus far and has proved successful in correlating edge-of-field losses to soil P level (Pote et al., 1999). However, recently soil P testing in environmental studies have focussed on methods that simulate the process of P desorption in soils (Pote et al., 1996). This is the process whereby solid phase P bound to sorption sites within the soil are solubilised or desorbed to solution, often into the soil solution for plant uptake but also to overland flow water during runoff events. Phosphorus desorption is usually measured with procedures that simulate P transfer into solution such as water extractable P (Van der Paauw, 1971), calcium chloride extractable P (Houba et al., 1994) and more long term desorption techniques such as FeO-P test (Menon et al., 1988).

The overall objective from this study was to attempt to relate field losses of P to laboratory extractable P taking into consideration soil sample depth, sample pre-treatment and extraction technique. Soil sample depth has been shown to affect soil test P values with higher P concentrations found at the surface 5cm, which is also assumed to be the effective depth of interaction with rainfall water (Gartley and Sims, 1994). The current study considered the effect that shallow sampling might have on the results obtained from various environmental soil P tests, and compared these data to soil sampled to 10cm. The effect of soil drying and re-wetting was examined by extracting soil samples under field moist and oven-dried conditions to gauge the effect this type of sample pre-treatment might have on water-extractable P. The effect of water to soil ratio on water extractable P was explored at different depths and also to simulate a range of extract P concentrations that might be mirror observed values of DRP in overland flow. Soil test P data from field sites were compared with loss of P from those sites to test whether Morgan's P was a good indicator of risk of P loss in overland flow.

Methodology

Soil sampling and analysis

Soil samples were taken from seven grassland sites on the Johnstown Castle Estate, south-east of Ireland (latitude 52 °N; mean annual rainfall 1022 mm and mean annual temperature of 9.6 °C). The seven sites were selected to provide a range of soil test P levels and were taken from study areas within the Johnstown Castle Estate known locally as the Warren (sites 1 and 2), Cowlands (site 3 and 4) and the Dairy (sites 5 to 7). The sites were part of on-going beef and dairy trials and were used in this study primarily due to the range of soil P levels that ranged from below optimum to excessively high. The soil type at the Warren is described as a poorly-drained gley soil with sandy loam topsoil over a layer of sandy loam and loam, with heavy gleying evident at all horizons (Kurz et al., 2004). At the Cowlands, the soil is described as a poorly-drained gley with a sandy loam topsoil overlying a loam with a sandy loam lens imbedded in the loam (Kurz et al., 2004). At the Dairy the soils are moderately well to imperfectly drained with a loam to sandy loam surface soil over a loam at 50 cm (Ryan et al., 1998). Soil samples were taken on a field-by-field basis to sample depths, 2, 5 and 10 cm, with approximately 30-40 soil cores bulked to provide a single composite sample for each depth at each field. The sampling procedure was repeated in triplicate at each site for each sample depth. The agronomic 10 cm sample was taken using the conventional bucket sampler. The shallow samples were taken using a soil corer, slicing cores to provide samples to 2 and 5 cm depths. All soil samples were dried over-night at 40 °C, ground and sieved through a 2 mm mesh and stored in boxes at room temperature prior to analysis.

Triplicate soil samples at each sample depth were analysed for Morgan's P, Iron-oxide paper strip P, water-soluble P and calcium chloride P at Johnstown Castle Laboratories, Wexford. Agronomic P as determined by Morgan's P was measured on 6.5 ml of soil using a buffered (pH = 4.8) acetate-acetic acid reagent in a 1:5 (v/v) soil to solution ratio extracting for 30 min (Peech and English, 1944; Byrne, 1979). The iron-oxide paper strip P test was used as a 'P sink' method for estimates of labile inorganic P in soils according to the method of Menon et al., (1988). One iron-oxide impregnated paper strip (10 x 2 cm) was added to each sample of soil (1g) suspended in 40ml of 0.01 M CaCl₂ solution and shaken for sixteen hours on an end-over-end shaker. The paper strips were then eluted with 0.1 M H₂SO₄ for one hour and the P concentration in the acidic solution determined colorimetrically. Water soluble P (WSP) was measured using the method of Van der Paauw et al., (1971) using 1g soil suspended in 40 ml of distilled water shaken on an end-over-end shaker

for one hour. Extracts of soil were filtered using Watmann no. 2 filter paper before colorimetric analyses. Readily extractable P ($\text{CaCl}_2\text{-P}$) was determined using 0.01 M CaCl_2 in a 1:10 soil to solution ratio, extracted for 1 hour (Houba et al., 1994) on an end-over-end shaker. Extracts of soil were filtered using Watmann no. 2 filter paper before colorimetric analyses. Phosphorus concentration in filtered extracts was determined using the modified ascorbic acid method for soils by John (1970).

Mehlich3 extraction was carried out at the Department of Plant and Soil Science at the University of Delaware using the modified Mehlich test (Mehlich, 1984) to extract P, Al and Fe at a 1:10 soil solution ratio using Mehlich-3 reagent (0.2 M CH_3COOH + 0.25 M NH_4NO_3 + 0.015 M NH_4F + 0.13 M HNO_3 + 0.001 M EDTA). A Sample of each triplicate soil sample taken at the three depths was composited to represent soil at each depth and samples were shipped to the US for analysis. Percentage organic matter (%OM) in soil samples was measured by loss on ignition and the bulk density of soils estimate from %OM by the method of Jeffrey (1970).

The effect of water-to-soil ratio on P desorption was investigated using the method of Yli-Halla et al., (1995). Soil samples were weighed to give dry soil concentrations of 4, 5, 6.25, 10, 20 and 200 g/l in deionised water; representing water-to-soil ratios 250-5 l/kg. The soil extracts were filtered and P concentration in solution determined colorimetrically (John, 1970). The effect of sample pretreatment on water-soluble P was examined by carrying out extractions on field-moist and oven-dried (40 °C) samples. Soil sampled to 10 cm was collected from each site and the composite divided into two samples. Oven-dried samples were ground and sieved as previously described, whilst field-moist samples were hand sieved and percentage moisture determined. Both oven-dried and field-moist samples were weighed out to give equivalent dry soil concentrations and extracted at water-to-soil ratios in the range of 250-5 l/kg by the above method with water to soil ratios adjusted in moist samples to correspond to dry soil water-to-soil ratios.

Overland-flow analysis

Overland flow samples were collected from two of the seven sites used for soil sampling. These were a low P site (3.1 mg/kg; (2.8 mg/l)) at the Warren (Site 1) and a high P site (20.6 mg/kg (17.6 mg/l)) at the Cowlands (Site 3). These sites have been instrumented with discrete automatic sampler with integral data loggers (SIGMA 900). Overland flow was sampled from pipes before entering large tanks fitted with v-notch weirs and water level recorders. Both sites vary in size (0.5-1.54 ha) but have similar slope (3-4 °) and were hydrologically isolated from

surrounding sites by backfilling trenches about 25 cm deep with impermeable soil and building earth banks above to 20 cm re-seeded with grass.

Flow-related overland flow samples were collected from runoff events from December 2001 to March 2002. Samples were analysed for dissolved reactive P concentrations by filtering through 0.45 µm Sartorius filters and P concentration in solution measured using the ascorbic acid method (Murphy and Riley, 1962). In addition to overland flow collection, soil samples were taken across sites 1 and 3 on 13 sample dates from 4th December 2001 to 1st March 2002 to sample depths of 5 and 10 cm to correspond to overland flow events that occurred at these sites. Soil samples were oven-dried (40 °C) and sieved (2 mm mesh) prior to analysis of water soluble P extracting with water to soil ratios in the range of 250-5 l/kg using the method outlined above.

Statistical Methods

All chemical analyses were carried out in duplicate and an average value taken. The significance of R² values are denoted by the symbols *, ** and *** for significance levels $p < 0.05$, 0.01 and 0.001, respectively. Factorial analysis was carried out in Genstat and variance comparison was made using Bartlett's test. Morgan's P carried out at Johnstown Castle laboratories are quoted in mg/kg with mg/l in parentheses since the test is carried out on a volume basis and is compared with methods carried out on a weight basis. Water-soluble P extracts are quoted in mg/kg to describe quantity of P desorbed to solution while the term soil solution P concentrations (using the same method) refers to the concentration of water-soluble P in the soil-water extract in mg/l, following extraction.

Results

Effect of sample depth and extracting solution

The effect of sample depth on P concentration was examined using factorial analysis with four P tests, Morgan's, WSP, FeO-P and CaCl₂-P. Mehlich-3 was not included in this analysis because it represents a composite sample without replication, and the statistical analysis was therefore confined to P tests that included replication. Taking each method separately, a significant difference with sample depth was found for Morgan's P ($p=0.004$), CaCl₂-P ($p=0.004$), FeO-P ($p=0.003$) and WSP ($p=0.004$). In general soil P values were highest at the surface to 2 cm, and P concentrations decreased with increasing sample depth from 2 to 5 to 10 cm.

The variances at each sample depth were compared for each method. While there was no significant difference in the variances of individual soil test values between depths, the trend in variances suggested that as sample depth decreased the variance in the data increased (Table 1) and samples taken to 2 cm had highest variances in the data. Extractable P at each sample depth were significantly correlated with each other and the relationship between the standard agronomic sample depth to 10 cm and shallow sampling depths to 2 and 5 cm is presented in Figure 1 for

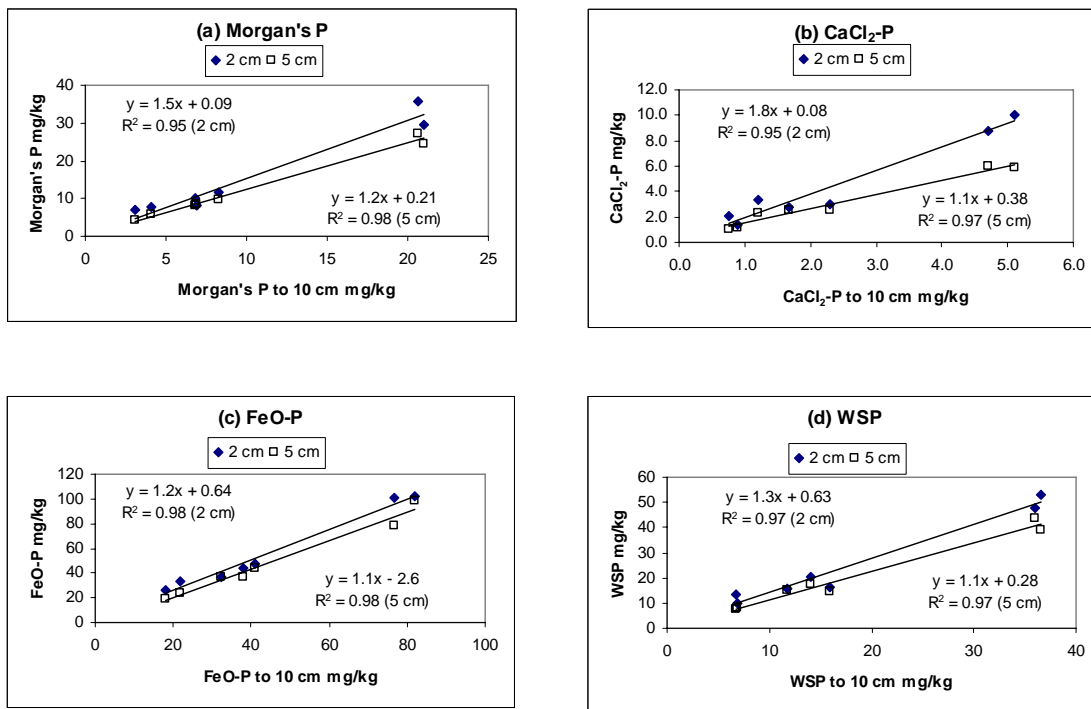


Figure 1 Relationship between P measured from 10 cm and 2 and 5 cm using (a) Morgan's P (b) CaCl₂-P (c) FeO-P and (d) WSP

(a) Morgan's P, (b) CaCl₂-P, (c) FeO-P and (d) WSP.

Table 1. The effect of sampling depth on soil P level (mg/kg) using four extractants across all seven sites.

P test	Sampling depth (cm)			s.e.d.* (df=12)	Chi-square (df=2)
	2	5	10		
Morgan's P Variance	15.6 138.8	12.6 85.8	10.1 56.6	1.28	1.32
CaCl₂-P Variance	4.5 11.8	3.1 4.3	2.4 3.2	0.51	3.29
FeO-P Variance	55.7 1006.4	48.2 863.9	44.3 640.4	2.59	0.34
WSP Variance	25.3 306.6	20.7 210.5	18.3 164.7	1.67	0.65

*s.e.d.= standard error of differences of means

Values of Morgan's P, CaCl₂-P, FeO-P, WSP and Mehlich3-P were compared and at similar depths, Mehlich-3 extracted the highest amount of P, followed by FeO-P, WSP, Morgan's P and CaCl₂-P and the difference in fertilizer application rates was reflected in soil P levels.

Effect of Water-to-Soil Ratio and sample pre-treatment

A factorial analysis of the data across the sites, of sample depth by water-to-soil ratio, showed a significant effect of both sample depth ($p < 0.001$) and water to soil ratio ($p < 0.001$) on WSP with no interaction between these factors. The effect of varying extract ratios on WSP, at each of the seven sites and at each sample depth, indicated that as water-to-soil ratios were widened, from 5 to 250 l/kg, the amount of extractable P (mg/kg) increased.

Values of WSP from oven-dry samples were significantly higher than corresponding values from field-moist samples ($p < 0.001$) and oven-drying significantly increased WSP by 27-175% over the range of water-to-soil ratios, compared to field-moist samples ($p < 0.001$). The data was averaged over the seven sites and WSP from oven-dry soil at the 5 and

250 l/kg ratios were 4.2 and 64 mg/kg respectively, while values measured from field-moist soil at corresponding extract ratios were 3.3 and 23.3 mg/kg, respectively. Taking average values across the seven sites, the relationships between WSP and water-to-soil ratio for field-moist ($R^2=0.94^{***}$) and oven-dry samples ($R^2=0.98^{***}$) are plotted in Figure 2. There was an interaction ($p<0.001$) between extract ratio and sample pretreatment with the oven-dried samples showing a higher rate of increase in WSP than the field moist-samples as the extract ratio widened.

Dissolved reactive P in overland flow and soil P data

Overland flow data were defined into discrete events and summary statistics on DRP concentration, flow volume and DRP loss (kg/ha) are presented in Table 2 for sites 1 and 3. The average DRP concentrations

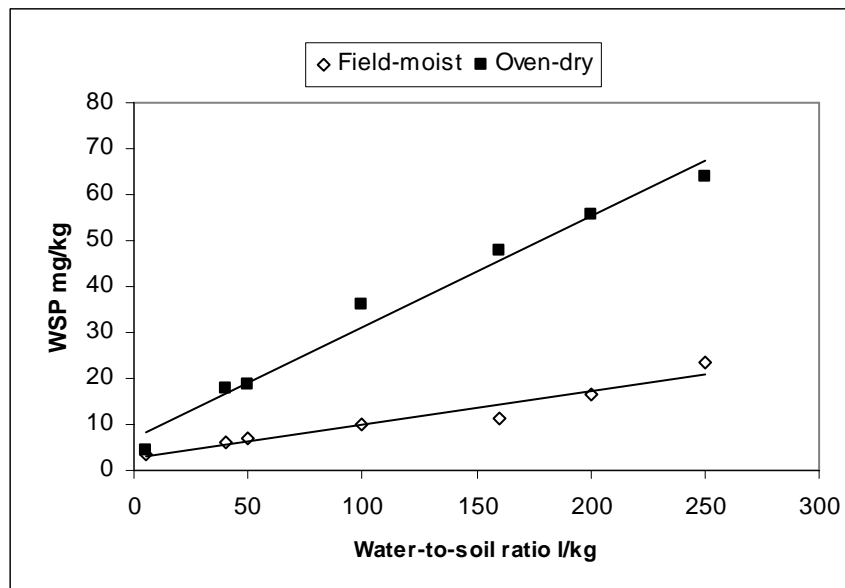


Figure 2 The effect of widening extract ratios on WSP in field-moist and oven-dried samples

from each event ranged from 0.032-0.067 mg/l from site 1, and 0.261-0.620 mg/l from site 3. The maximum values of DRP measured during each event ranged from 0.069-0.314 mg/l and 0.312-0.730 mg/l from sites 1 and 3, respectively. Minimum values of DRP recorded during events ranged from <0.005-0.027 mg/l from site 1 and 0.037-0.356 mg/l from site 3. The total volume of overland flow collected from the sites was calculated for each event and at site 1 flow volumes ranged from 42,949-1179,019 l while at site 3, from corresponding events, volumes

ranged from 3,832-413,376 l. The DRP loss from each site was calculated for each event and values ranged from <0.005-0.05 kg/ha P from the low P site, and from 0.002-0.37 kg/ha P for the high P site. The total loss from the sites during the sampling period, December 2001 to March 2002 were calculated as 0.065 and 0.457 kg/ha P from sites 1 and 3, respectively, reflecting a seven-fold difference between the sites.

Soil samples taken to 10 cm during overland flow events were extracted with distilled water at a range of water-to-soil ratios ranging (5-250 l/kg). In this instance, P concentration in soil-water extracts (mg/l) at each water:soil ratio is presented, where previously quantities of P desorbed (mg/kg) were used. These values are referred to here as extract P concentrations (mg/l). Average values of extract P concentration ranged from 0.13-0.63 mg/l and 0.27-1.01 mg/l from soil sampled to 10 cm from sites 1 and 3, respectively, reflecting the difference in soil test P levels between the sites.

Table 2. Event date and summary statistics of DRP recorded at sites 1 and 3.

Event dates	Event no.	No. samples	Flow (l)	DRP (mg/l)		P loss (kg /ha)
				Range	Mean	
Site 1						
3 Dec- 5 Dec 2001	1	16	180,394	0.025-0.085	0.059	0.008
6 Dec-13 Dec 2001	2	3	42,949	0.027-0.069	0.046	0.001
13 Jan-13 Feb 2002	3	77	1179,019	<0.005-0.314	0.067	0.050
24 Feb-2 Mar 2002	4	20	281,189	<0.005-0.084	0.032	0.006
Site 3						
3 Dec-6 Dec 2001	1	47	26,259	0.356-0.730	0.620	0.035
13 Jan-12 Feb 2002	3	151	413,376	0.037-0.695	0.411	0.37
25 Feb-25Feb 2002	4	6	3,832	0.207-0.312	0.261	0.002
28 Feb-2 Mar 2002	5	23	56,064	0.334-0.506	0.417	0.05

Dissolved reactive P concentrations in overland flow from sites 1 and 3 were compared with extract P concentrations from soil sampled to 10 and 5 cm, from the same sites, extracted using water-to-soil ratios ranging from 5 to 250 l/kg. At site 1, extract P concentrations ranged from 0.13-0.63 mg/l from samples taken to 10 cm which exceeded the average DRP values (0.032-0.067 mg/l) measured in overland flow. Only maximum values of DRP were comparable to extract P concentrations at this site. At site 3, extract P concentrations from samples taken to 10 cm (0.27-1.01 mg/l) at water-to-soil ratios 250-100 l/kg captured the average DRP values (0.261-0.411 mg/l) recorded during the events at this site. Maximum values of DRP were reflected in extract P concentrations using water-to-soil ratios from 5-250l/kg.

Soil test P data was compared with P loss in overland flow from the two sites monitored in this study (Table 3). The relative rates of P loss from site 1 and 3 were compared with the relative difference in soil test P levels using Morgan's P, CaCl₂-P, FeO-P, WSP, M3-P at both sites. Since Morgan's P is carried out on a routine basis using a volume of soil and other tests were done on a weight basis, both mg/kg and mg/l are reported in Table 3 (converting mg/kg to mg/l using bulk densities). There was a seven-fold difference in P loss between the two sites using the winter data in this study and the difference in Morgan's P values (6.6) was closest to the difference in P loss, compared with other P tests.

Table 3. Phosphorus loss (kg /ha) from site 1 and 3 and soil test P results, as mg/kg with mg/l in parentheses, comparing the ratio of P loss to the relative difference in soil test P values between the two sites.s

	P loss kg /ha	Morgan's P	CaCl ₂ -P	FeO-P	WSP	M3-P
Site 1	0.065	3.1 (2.8)	0.8 (0.8)	18.2 (16.5)	6.1 (5.6)	38.2 (35)
Site 3	0.457	20.6 (17.6)	4.7 (4)	82 (70.1)	29.5 (25.1)	124 (106)
Ratio of P loss and STP	7	6.6 (6.3)	5.9 (5)	4.5 (4.2)	4.8 (4.5)	3.2 (3)

Discussion

Effect of sample depth

In this work there was a statistically significant difference in soil P with sample depth from samples taken to 2, 5 and 10 cm for four soil P tests used across the seven sites as soil P decreased with increasing sample depth. The trend of decreasing soil P with increasing sample depth from 2 to 5 and 10 cm in each of the four P tests used here is also demonstrated in the correlations between sample depth for each test, indicating that the standard agronomic sample depth to 10 cm can be correlated to shallow sample depths (2 and 5 cm) using Morgan's P, CaCl₂-P, WSP and FeO-P. Recommendations to adopt shallow sample depths to 2 cm have been reported by Mulqueen et al., (2004) for situations in Ireland where soil sampling is used for environmental purposes. However, in our study, data from samples taken to 2 cm had higher variance than corresponding values from samples taken to 5 and 10 cm. This finding concurs with Torbert et al., (2002) who surmised that the greater variability in 2.5 cm samples was due to dung patches and problems in obtaining a consistent sample at these depths. Furthermore, the authors opined that whilst 5 cm samples gave the 'best fit' to DRP in rainfall simulated runoff, shallow sampling depths would be easier to falsify. This may be an important consideration if shallow sample depths were used for setting limits or environmental threshold values of soil P for agriculture. Therefore, it would be difficult to justify a change in sample depth when the standard agronomic depth has been

correlated to shallow depths in this and other studies; the same information can be gleaned from the standard sample depth.

Effect of water-to-soil ratio and sample pre-treatment on water soluble P

At each of the sites WSP in soils increased as water-to-soil ratios increased from 5-250 l/kg, which agrees with other findings that extract ratios have an important effect on P desorbed from soil to solution (Sharples, 1983). When the effect of sample pre-treatment was introduced, field-moist samples desorbed significantly less P to solution compared to oven-dried samples. This difference was evident at each site and could be attributed to effects of drying and rewetting of soils that have been cited in the literature, namely, release of P from soil microbial biomass following cell rupture (Turner and Haygarth, 2001), increased solubility of organic matter and soil P (Olsen and Court, 1982) and changes in soil structure. Whilst oven-drying soil samples prior to analysis affects P desorption, the standard agronomic soil P test in Ireland uses oven-dried (40 °C) soil prior to extracting with Morgan's acetate-acid reagent. However, Olsen and Court (1981) reported that since acid and alkaline reagents alter the components and structure of soil, the effects of drying are less noticeable. Furthermore, Several Northeastern states in the U.S use Morgan's reagent, or a modified Morgan's reagent, for fertilizer recommendations and P runoff risk indices from soil samples that have been oven-dried to 55 °C prior to analysis (Ketterings et al., 2002).

Relating soil P data to DRP concentrations in overland flow

With only two field sites monitored in this study a statistical relationship between DRP loss and soil P data was not possible, and the relative difference in P loss and extractable soil P between the sites was considered in order to compare P loss in overland flow with soil test P data. Losses of P from sites 1 and 3 were 0.065 and 0.457 kg/ha, reflecting a seven-fold difference between the sites for the monitoring period December 2001 to March 2002. Kurz et al., (2004) monitored DRP concentrations from the same sites and computed annual losses of 0.698 and 4.764 kg P/ha from sites 1 and 3, respectively, showing a similar seven-fold difference between the sites. The total monitoring period reported by Kurz et al., (2004) was 493 days and total losses measured over that time were 0.778 and 5.299 kg/ha from sites 1 and 3, respectively, with significant losses in overland flow measured in summer

months from May to September, indicating significant losses during the summer months. Whilst we can only discuss the relative difference in DRP loss and soil P levels between these sites it is important to note that the relative difference in P loss is reflected in the relative difference in soil test P levels using the standard agronomic test (Morgan's P) to 10 cm. This observation concurs with Kurz et al., (2004) who monitored DRP losses from a total of three field sites at low (site 1 in this study), medium and high (site 3 in this study) soil test P levels, and found a pattern of increasing minimum, maximum and flow-weighted average DRP concentrations with a clear increase in P loss from sites at low to medium to high soil P levels, as measured by Morgan's P to 10 cm. Whilst absolute concentrations of DRP in overland flow were not easily simulated by laboratory extraction in this work, the standard agronomic soil P to 10 cm gave a good indication of the relative losses of P at edge-of-field scale. This concurs with other studies that have used Morgan's P as an indicator of P loss from soil to water.

Conclusion

There was a significant difference in extractable P with sample depth as soil P increased with decreasing sample depth from 10 to 5 to 2 cm, when extracted with Morgan's P, $\text{CaCl}_2\text{-P}$, WSP and FeO-P. Although extractable P was higher at the surface 2 cm in samples, there was more variation in the data at this depth. Since the standard agronomic sampling depth to 10 cm correlated with samples taken to 2 and 5 cm in this and other work reported in the literature, there is little justification for changing to a shallow sample depth (with greater variance) when the same information can be obtained from the standard agronomic sample. The standard agronomic P test, Morgan's P, gave a good indication of the relative loss of DRP in overland flow from high and low P sites, and whilst soil P testing is not a measure of absolute values of P loss it can provide a good indication of environmental losses in addition to agronomic status.

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