

Soil phosphorus fractionation in Icelandic long-term grassland field experiments

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ABSTRACT

Long-term fertiliser experiments on hayfields at Sámstaðir, South Iceland, and Hvanneyri, West Iceland, provided the basis for investigations on phosphorus fractions, the fate of P fertilisers and P sorption in Icelandic soils. Total (P_t), inorganic (P_i), organic (P_o), ammonium oxalate extractable (P_{ox}), ammonium lactate extractable (P_{AL}) and anion resin extractable (P_{an}) fractions were determined. The P sorption was measured and P sorption maximum (S_{max}) calculated. Ammonium oxalate extractable Si, Al and Fe were measured and the degree of P saturation (DPS) was calculated. We found all surplus applied P in the top 10 cm of the soil with the highest increase in the top 5 cm. While there was only a slight increase in P_o , most of the surplus P was inorganically bound, with a strong correlation between P_p , P_i and P_{ox} . Phosphorus saturation (S_{max}) was highly correlated with oxalate extractable Si and Al in the dry Silandic Andosol. However, in the Histic Andosol it was only highly correlated with Fe_{ox} and not to Al_{ox} , indicating a different behaviour relating to redox conditions. Available P (P_{AL}) increased with increasing P application mainly in the top 5 cm, but was not detectable at 10-20 cm depth. There was a good correlation between P_{AL} and the degree of phosphorus saturation which only reached critical level in the top 5 cm with the highest P application of 39 kg ha⁻¹ year⁻¹. Water soluble P_{an} was substantially higher than P_{AL} indicating that the phosphorus that had accumulated in the soil could be released and may be a useful source of P in future.

Keywords: Long-term experiments, P fractionation, P balance, P sorption, DPS

YFIRLIT

Binding fosförs í jarðvegi langtímatilrauna á túni

Jarðvegur úr langtímatilraunum á tünnum á Sámstöðum og á Hvanneyri var notaður til að kanna bindingu og afdrif áborins fosförs í íslenskum jarðvegi. Heildarmagn fosförs (P_t) í jarðveginum var mælt og skipting fosförsins í ólífræn sambönd (P_i) og lífræn (P_o). Einnig var mælt hversu mikið losnar af P í ammóníum oxalati (P_{ox}), ammóníum laktati (P_{AL}) og með anjóna resin (P_{an}). Binding fosförs var greind og hámarks aðsog (S_{max}) reiknað með Langmuir líkingunni. Si, Al og Fe voru mæld í ammóníum oxalati og mettunarstig fosförs (DPS) reiknað. Við fundum allan umframáborinn fosfór í efstu 10 cm jarðvegsins en mest var þó bundið í efstu 5 cm. Það varð einungis lítil aukning á lífrænum fosfór á tilraunátímanum, fosfórin var aðallega bundinn í ólífrænum samböndum og það er góð fylgni milli P_p , P_i og P_{ox} . Milli mettunarstigs fosförs (S_{max}) og Si_{ox} og Al_{ox} er góð fylgni í þurrum jarðvegi Silandic Andosol en í rökum Histic Andosol jarðvegi er einungis góð fylgni við Fe_{ox} en engin við Al_{ox} sem bendir til að oxunar og afoxunarstig hefur veruleg áhrif á það hvernig fosfórin

binnt. Nýtanlegur fosfór (P_{AL}) eykst með auknum P-áburði, aðallega í efstu 5 cm jarðvegsins en í 10-20 cm dýpt er engin aukning merkjanleg. Það var góð fylgni milli P_{AL} og mettunarstigs en það var einungis í efstu 5 cm þar sem hæsti áburðarskammturinn 39 kg P ha^{-1} var notaður sem fosfórin var við það að ná fullri metnun. Vatnsleysanlegur fosfór, P_{an} , var verulega meiri en P_{AL} sem gefur til kynna að fosfór sem safnast hefur fyrir geti losnað og nýst og verið mikilvægur fyrir framtíðina.

INTRODUCTION

In Iceland the main soil types in agricultural use are deep Silandic and Gleyic Andosols and Histosols (Arnalds & Oskarsson 2009). Andosols are known to adsorb phosphorus (P) and require more P fertilisation than other soils. The surplus P accumulates close to the surface, which may be a potential risk because of surface runoff or if this layer is lost through erosion upon tillage (Auxtero et al. 2008, Parfitt 2009, Mejías et al. 2013). Phosphorus has been applied to Icelandic agricultural soil in surplus quantities for decades and accumulates in the top few centimetres in permanent hay-fields (Gudmundsson & Sigvaldason 2000, Gudmundsson et al. 2005). In Andosols P is adsorbed by aluminium (Al) and iron (Fe) complexes (ligands), by allophane and by organic compounds (Dahlgren et al. 2004, Madeira et al. 2007, Parfitt 2009, Oburger et al. 2011). In fresh volcanic material appreciable amounts of P may be available due to rapid weathering of the primary P bearing mineral apatite (Dahlgren et al. 2004). As the Andosols develop active Al, Fe and the adsorption capacity of the soil tend to increase. Ammonium oxalate is used to extract poorly ordered Fe and Al oxides and hydroxides and it also extracts P bound by Fe and Al minerals and complexes which often are the main minerals responsible for P fixation. Ammonium oxalate extractable P has been used to calculate the degree of P saturation and to estimate the soil P fixing capacity of Andosols (Beauchemin & Simard 1999, Pinheiro et al. 2007, Mejías 2013).

Phosphorus fractions are frequently determined by sequential extractions where the method of Hedley et al. (1982) has been widely used and reviewed (Cross & Schlesinger 1995, Negassa & Leinweber 2009). Although

the Hedley fractionation is commonly used, this does not apply to Andosols. Negassa & Leinweber (2009) list over 100 studies on sequential P fractionation from different parts of the world and dealing with many different soil types. However, none of these studies were on Andosols. Otani & Ae (1999) used the Truog, Bray 2, Olsen and Citrate methods to estimate available P in Andosol and determined inorganically and organically bound P in the extracts. Pools of total, inorganic, ammonium oxalate extractable P and different methods for soil testing have been used to investigate the fate of P fertilisers, P retention characteristics of the soils, bio-available P and the soil phosphorus saturation degree (Beauchemin & Simard 1999, Takahashi & Anwar 2007, Auxtero et al. 2008, Mejías et al. 2013).

In the only study of P fractions in Icelandic soils so far, Helgason (2002) determined total, inorganic and organic P (P_t , P_i and P_o) using the method of Saunders & Williams (1955) modified by Olsen & Sommers (1982). This method differs from the Hedley method in that it is not a sequential extraction and the organic and inorganic fractions are not further characterised. The P fractions are determined by extracting ignited and not ignited soil with sulphuric acid. Because of the different methods results cannot easily be compared. In Helgason's (2002) study, the total P was in the range 440 to 2540 mg kg^{-1} in unfertilised areas and 1067 to 3129 mg kg^{-1} in fertilised fields. Organic P ranged from 0 to 1714 mg kg^{-1} and did not differ between fertilised fields and unfertilised areas. However P_i was lower in unfertilised soils with 207 to 1048 mg kg^{-1} compared with 996 to 1864 mg kg^{-1} in fertilised soils. This indicates that surplus fertiliser P is inorganically bound. This has also been reported for many other soils (Negassa &

Leinweber 2009, Pätzold et al. 2013) and specifically for Andosols (Otari & Ae 1999, Takahashi & Anwar 2007). In general, P_o was higher in West Iceland in the Helgason (2002) study, which is outside the country's active volcanic area. The method of Saunders & Williams (1955) for total P fails to dissolve P firmly held in primary silicate minerals. Gudmundsson et al. (2005) compared total P determined by melting the soil with lithium borate dissolved in HNO_3 and P dissolved in HCl in ignited samples for the "weatherable" fraction in a Gleyic Andosol. The "weatherable" fraction was 77 to 94% of the true total P without any obvious relation to depth or fertilisation.

According to Dahlgren et al. (2004) the most commonly used tests to estimate available P in Andosols are the Truog method, most commonly used in Japan, followed by Bray 2, Mehlich 3, Olsen and anion exchange resin methods, used in the USA and many other countries. Investigating European volcanic soils, Madeira et al. (2007) compared extractions of P with H_2O , $CaCl_2$, Mehlich 3, Olsen, Bray2 and Egner-Riehm methods and found large differences in extractable P between methods. In Iceland, soil tests for agriculture use ammonium lactate extraction, the AL-method of Egner & Rhiem (1960). For many years the Olsen method (Olsen et al 1954) was also used and Pálmason & Helgason (1990) compared P extracted by the Olsen and the AL-methods of Egner and Rhiem. They found that the AL-method extracted 2.53 (SE 0.18) and 1.78 (SE 0.18) times more P than the Olsen method in peat soils and freely drained soils, respectively. The coefficients of determination (R^2) of linear regression between the two methods were 0.63 for the peat soils and 0.26 for the Andosols. Madeira et al. (2007) looked at the relationship between all extracts they tested and obtained a coefficient of correlation (R) of 0.74 by comparing the Olsen and the Egner Riehm methods for European Andosols, which was slightly better than Pálmason & Helgason found for Icelandic Andosols.

Iceland has large areas of unfertilised soils where phosphorus deficiency is apparent (Óskarsson & Brynjólfsson 2000) whereas hayfields have often received surplus P through fertilisation for many decades. The aim of this study was to investigate P fractions in fertilised hayfields and furthermore to investigate the fate of surplus P and to establish if it is organically or inorganically bound. Using the results we set out to see if there is a correlation between the various P fractions and the pedogenic oxides. Our final aim was to estimate the P sorption capacity and the degree of P saturation of the soils.

MATERIALS AND METHODS

Soil samples from three long-term fertiliser experiments on permanent hayfields were used. Two were at Sámstaðir in South Iceland and one at Hvanneyri in West Iceland (Table 1). The oldest experiment started in 1938 (Sámstaðir 1-49, Silandic Andosol) one in 1950 (Sámstaðir 9-50, Histic Andosol) and one in 1970 (Hvanneyri 299-70, Hemic Histosol). The experiments at Sámstaðir were stopped at the time of sampling; the one in Hvanneyri is still ongoing. In addition samples were collected just outside the experiments at Sámstaðir as a reference for soils without fertilisation. The experimental areas were usually harvested twice a year, yield weight determined and samples taken for analysis. The grass was then removed from the site, including the unfertilised area. The grass samples were dried at 70° C and finely ground for analysis. Samples were digested by boiling in concentrated HNO_3 and P determined using molybdenum yellow at 440 nm.

Soil samples were collected from each plot using a 25 cm long conical core sampler, 3.1 cm in diameter. The cores were divided into 0-5, 5-10 and 10-20 cm segments at Sámstaðir and to 0-5 and 5-15 cm depths at Hvanneyri. From each plot 15 to 20 cores were collected. The samples were mixed, dried at 35 °C and sieved through a 2 mm sieve. Subsamples for the C and N analysis, H_2SO_4 and NH_4 oxalate

Table 1. Location, soil type, fertilisers applied annually, sampling depths and year of sampling. 1-49 was sampled in 2005, duration 67 years, 4 replicates; 9-50 was sampled in 2004, duration 54 years, 4 replicates. 299-70 was sampled in 2006, duration 36 years, 4 replicates. Soil types according to WRB 2006.

Treatment	Annually applied kg ha ⁻¹			Sampling depths cm		
	N	P	K			
	Sámsstaðir 1-49 Silandic Andosol.					
nf ¹⁾	0	0	0	0-5	5-10	10-20
0P ₃₈	70	0.0	62.3	0-5	5-10	10-20
0P ₄₉	70	0.0	62.3	0-5	5-10	10-20
26P	70	26.2	62.3	0-5	5-10	10-20
	Sámsstaðir 9-50 Histic Andosol					
nf ¹⁾	0	0	0	0-5	5-10	10-20
0P	70	0.0	74.7	0-5	5-10	10-20
13P	70	13.1	74.7	0-5	5-10	10-20
22P	70	21.9	74.7	0-5	5-10	10-20
31P	70	30.6	74.7	0-5	5-10	10-20
39P	70	39.3	74.7	0-5	5-10	10-20
	Hvanneyri 299-70 Hemic Histosol					
0P	100	0.0	100	0-5	5-15	
30P	100	30	100	0-5	5-15	

extractions were ground in a ceramic mortar prior to analysis.

Bulk density was determined by using 100 cm³ cylinders for the Histosol in Hvanneyri and for the Histic Andosol at Sámsstaðir. For the Silandic Andosol at Sámsstaðir undisturbed bulk density samples were not taken from each plot. Instead it was measured by weighing 200 cm³ air dried and sieved samples and correcting to oven dry weight. Total C and N were determined by dry combustion using Elementar vario MAX CN (Elementar Analysensysteme GmbH). The pH was measured using a glass electrode in a 1:2 soil/water suspension using a glass electrode.

The methods of P fractionation used in this study are outlined in Table 2. The determination of total P (P_t), inorganic P (P_i) and organic P (P_o) followed the method of Saunders &

Table 2. The P fractions and the methods of extraction used in this study.

Fraction	Extraction
P _t = total P ¹⁾	0.5 M H ₂ SO ₄ after ignition
P _i = inorganic P ¹⁾	0.5 M H ₂ SO ₄
P _o = organic P ¹⁾	P _t - P _i
P _{ox} = acid oxalate P ²⁾	ammonium oxalate
P _{AL} = ammonium lactate P ³⁾	NH ₄ -lactate + acetic acid
P _{an} = anion resin P ⁴⁾	deionised water with anion resin bags

1) Saunders and Williams 1954, Olsen and Sommers 1982. 2) Schwertmann 1964, 3) Egner & Rhiem 1960, 4) Sibsen 1978, Burt 2004.

Williams (1955) as described by Olsen & Sommers (1982). This involves weighing 1.0 g of soil into a crucible, ignite at 550°C for 2 hours, transfer to a polyethylene bottle, add 50 ml 0.5 M H₂SO₄ and shake for 16 hours at room temperature. Parallel to this the same amount of soil was weighed into a polyethylene bottle and shaken in the same amount of sulphuric acid. The suspensions were sieved through Whatman No 42 filter paper and P in the solutions determined using molybdenum ascorbic acid blue at 880 nm. P in the ignited and not ignited sample is taken as P_t and P_i, respectively. Then P_o = P_t - P_i.

Available phosphorus (P_{AL}) was extracted using the ammonium lactate method after Egner et al. (1960) by shaking 5g of soil in 100 ml of 0.1 M ammonium lactate and 0.4 M acetic acid for 2 hours and measuring P in the filtrate.

Water soluble P fixed by anion resin (P_{an}) was extracted by the method of Sibbesen (1977 & 1978) as described by Burt (2004). In this method 4 g of soil and 4 g of resin (DOWEX, Matathon, type II, 510-610 µm spherical beds) in a Nitex nylon fabric bag with 300 µm pores were shaken in 100 ml of deionised water for one hour followed by shaking the sample with another bag for 23 hours and the third bag for 24 hours. The P was released from the resin by shaking the bags in 80 ml of 0.5 M HCl for 30 min after CO₂ release had subsided. P was determined using molybdenum ascorbic acid blue at 880 nm.

The acid oxalate extraction (P_{ox}) was adapted from Schwertmann (1964) and Burt (2004) where 0.5 g of soil and 100 ml of acid ammonium oxalate added were shaken in the dark for 4 hours, filtered and Fe, Al, Si and Mn determined by ICP in Spectroflame D produced by Spectro, Germany. P was determined using molybdenum ascorbic acid blue at 880 nm.

The determination of the P sorption isotherm is based on a method described by Graetz & Nair (2000) which is an adaptation of the method of Holford et al. (1974). One gram of soil was placed in a 250 ml polypropylene bottle with 50 ml of 0.01 M CaCl₂ containing a concentration of P added as KH₂PO₄ of 0, 50, 100, 150, 200 and 300 mg P L⁻¹. Two to four drops of chloroform were added in each bottle to inhibit microbial activity. The bottles were shaken for 24 hours at room temperature. The soil suspension was filtered through Whatman no. 42 filter paper. P in the solution was determined by the molybdenum blue colour at 880 nm. The linear Langmuir equation was used to calculate the sorption maximum, S_{max} (Holford et al. 1974). The Langmuir equation can be written as:

$$C/S = (1/kS_{\max}) + (C/S_{\max}) \quad (1)$$

where

S = S' + S₀, the total amount of P retained, S' = P retained by the solid phase, S₀ = P originally sorbed on the solid phase, mg kg⁻¹

C = concentration of P after 24 h equilibration, mg L⁻¹

S_{max} = P sorption maximum, mg kg⁻¹

k = Constant related to the bonding energy, L mg P⁻¹

The degree of phosphorus saturation (DPS) was calculated as oxalate extractable P, Al and Fe using the formula of Schoumans (2000):

$$DPS = P_{\text{ox}} / 0.5(Al_{\text{ox}} + Fe_{\text{ox}}) \times 100. \quad (2)$$

where P_{ox}, Al_{ox} and Fe_{ox} are in mol kg⁻¹.

The pools of P in soil were calculated using the amounts of P (mg kg⁻¹) in all fractions and the bulk densities of each treatment and adding the pools in 0-5 and 5-10 cm at Sámstaðir and in the top 15 cm at Hvanneyri.

All analyses of variance were conducted with the ANOVA procedure in SAS (9.4; SAS Institute Inc., Cary, NC, USA). Two-way ANOVA were used on each of the P fractions, mg kg⁻¹ (treatment + replicate), but one-way ANOVA for the total amount of P in the soil. The Tukey-Kramer test was used to test differences between each of the fertiliser treatments. Regression analyses were performed with the REG procedure in SAS.

RESULTS

The soils

Some soil properties at the experimental sites are shown in Table 3. The Silandic Andosol (1-49) at Sámstaðir had about 20 mg Al_{ox} kg⁻¹. Total soil organic carbon (C) decreased from 109 to 66 mg kg⁻¹ and bulk density increased from 0.54 to 0.75 kg L⁻¹ at 0-5 and 10-20 cm depths, respectively. The pH was in the range of 5.5 to 6.0.

The Histic Andosol (9-50) at Sámstaðir overlies more than a 3 m thick deposit of organic and mineral material with 20 to 54% organic matter and a few distinct volcanic ash layers. The soil had a Gleyic colour pattern to the top and the organic material was fibrous. In

Table 3. Soil properties at the experimental sites. Values are means of all treatments, the untreated adjacent area and replications.

Depth cm	Bd kg L ⁻¹	pH H ₂ O	C g kg ⁻¹	N g kg ⁻¹	C/N	Si _{ox} g kg ⁻¹	Al _{ox} g kg ⁻¹	Fe _{ox} g kg ⁻¹	Mn _{ox} g kg ⁻¹
Sámstaðir 1-49 Silandic Andosol									
0-5 cm	0.54	5.5	109	8.3	13.2	10	19	33	0.65
5-10 cm	0.66	5.7	85	7.0	12.1	11	19	36	0.63
10-20 cm	0.75	6.0	66	5.7	11.6	13	20	37	0.69
Sámstaðir 9-50 Histic Andosol									
0-5 cm	0.34	5.2	144	10.8	13.4	7	15	41	1.02
5-10 cm	0.45	5.5	84	7.1	12.0	15	22	74	0.64
10-20 cm	0.54	5.8	64	5.4	12.1	17	23	51	0.70
Hvanneyri 299-70 Hemic Histosol									
0-5 cm	0.31	4.5	286	18.0	15.9	nd	nd	nd	nd
5-15 cm	0.33	4.4	226	15.2	17.4	nd	nd	nd	nd

nd = not determined

the top 20 cm the C content decreased from 144 to 64 mg kg⁻¹ with depth and the C/N ratio was 12 to 13 (Table 3), which is low for poorly

decomposed organic material but can be explained by the long-term N fertilisation. Corresponding to the decrease in organic C, the bulk

Table 4. Phosphorus fractions (mg kg⁻¹), S_{max} (g kg⁻¹) and DPS % molar ratio in the three long-term experiments. Total (P_t), inorganic (P_i), organic (P_o), acid oxalate (P_{ox}), ammonium oxalate (P_{AL}) and anion exchange resin (P_{an}). The treatments were compared at each site, depth and extraction using Anova and the Tukey-Kramer test. Levels not connected by same letter were significantly different. (p≤0.05). Root MSE (RMSE) from the analysis are shown as well as phosphorus values from unfertilised area close to the experiments (NF).

Phosphorus fertiliser	Depth	P _t	P _i	P _o	P _{ox}	P _{AL}	P _{an}	S _{max}	DPS %
Sámsstaðir 1-49. Duration 67 and 56 years. Silandic Andosol.									
0P ₃₈	0-5	2360 a	747 a	1613 a	982 a	6 a	32 a	9.6 a	4.8 a
0P ₄₉	0-5	1738 b	809 a	929 b	977 a	2 a			5.0 a
26P	0-5	3839 c	2288 b	1552 a	2476 b	76 b	235 b	7.8 a	12.6 B
RMSE		216	120	181	175	11	31	0.5	0.6
0P ₃₈	5-10	2925 a	907 a	2018 a	987 a	3 a	16 a	10.7 a	4.7 a
0P ₄₉	5-10	1658 b	805 a	853 a	1064 a	4 a			4.9 a
26P	5-10	3025 a	1538 c	1487 a	1924 b	29 b	118 a	9.0 a	9.0 b
RMSE		501	138	634	109	6	31	1.4	0.4
0P ₃₈	10-20	2060 a	824 a	1235 a	1070 a	2			4.7 a
0P ₄₉	10-20	2027 a	729 a	1298 a	1045 a	7			4.6 a
26P	10-20	2200 a	1231 b	969 a	1277 a	9			6.2 b
RMSE		434	185	350	132	6			0.5
NF ¹⁾	0-5	1902	960	942	1297	5	41	8.9	6.5
NF ¹⁾	5-10	1840	907	933	1245	8	29	8.7	6.5
NF ¹⁾	10-20	1671	827	845	1161	5			5.7
Sámsstaðir 9-50. Duration 54 years. Histic Andosol									
0 P	0-5	1838 a	658 a	1287 a	1180 a	14 a	46 a	9.1 a	5.9 a
13 P	0-5	4008 ab	1569 a	2439 a	2012 a	39 ab			11.4 a
22 P	0-5	5137 b	2887 b	2250 a	3238 b	66 bc	312 a	8.2 a	14.9 ab
31 P	0-5	5942 bc	3658 b	2284 a	4148 b	108 c			21.8 bc
39 P	0-5	7504 c	5301 c	2203 a	6022 c	168 d	977 a	6.9 a	27.2 c
RMSE		958	584	663	473	17	451	1.6	3.9
0 P	5-10	1960 a	905 a	1055 a	1176 a	9 a	21 a	14.0 a	3.7 a
13 P	5-10	2725 a	1166 ab	1559 a	1453 ab	13 a			4.6 a
22 P	5-10	2497 ab	1441 ab	1055 a	1193 a	13 a	39 a	17.8 a	3.0 a
31 P	5-10	2740 a	1704 b	1035 a	1640 ab	18 a			5.3 a
39 P	5-10	3401 b	1872 b	1529 a	1976 b	32 a	127 a	13.0 a	6.1 a
RMSE		386	277	663	269	16	46	3.0	1.3
0 P	10-20	1883 a	822 a	1061 a	1022 a	10 a	22 a	8.7 a	3.8 ab
13 P	10-20	1883 a	938 a	945 a	1180 a	12 a			4.4 ab
22 P	10-20	1614 a	1066 a	707 a	973 a	13 a	37 a	11.1 a	3.2 a
31 P	10-20	2086 a	1185 a	899 a	1216 a	13 a			4.6 b
39 P	10-20	1990 a	1187 a	945 a	1184 a	13 a	38 a	10.5 a	4.4 ab
RMSE		256	270	195	168	5	8	2.0	0.6
NF ¹⁾	0-5	3440	1822	1618	2557	58	221	7.5	15.4
NF	5-10	1929	1165	764	1426	15	59	10.8	6.1
NF ¹⁾	10-20	1227	969	258	837	5	29	9.9	3.6
Hvanneyri 299-70. Duration 36 years. Hemic Histosol									
0 P	0-5	1003 a	163 a	840 a		25 a	35 a	8.5 a	
30 P	0-5	2887 b	1808 b	1079 b		231 b	445 b	10.9 a	
RMSE		108	118	71		19	70	1.4	
0 P	5-15	965 a	122 a	843 a		11 a	11 a	14.0 a	
30 P	5-15	1345 b	320 b	1025 b		29 b	41 b	24.0 b	
RMSE		54	38	66		4	4	1.5	

1) NF = not fertilised area adjacent to the experiment.

density increased from 0.34 to 0.54 kg L⁻¹ in the top 20 cm and pH increased from 5.2 to 5.8. The oxalate extractable Al, Si and Fe were higher than the minimum requirements for Andic properties and the soil was classified as an Andosol. The Fe_{ox} maximum at 5-10 cm depth and Mn_{ox} maximum at 0-5 cm depth was an indication of low redox potential and segregation of Fe and Mn close to the surface. Bulk density and pH were slightly lower than in the Silandic Andosol.

The Hemic Histosol (299-70) at Hvanneyri, West Iceland, had the lowest pH of 4.5 and 4.4 in the 0-5 and 0-15 cm layers and bulk densities of 0.31 and 0.33 at the same depths. The organic C was high, 286 and 226 g kg⁻¹ at the 0-5 and 5-15 cm depths respectively and the C/N ratio of 15.9 and 17.4 distinguished the Histosol clearly from the Andosols (Table 3). There were no significant differences in C, N or acid oxalate extractable Si, Al or Mn between treatments. Only Fe_{ox} showed high variability in 9-50, which was due to the variability in redox potential within the site and Fe segregation near the surface.

The P fractions

Total P in the unfertilised area (NF) and at 10-20 cm depth was an indication of the original or background P content (Table 4). In the Andosols P_t was mostly in the range 1700 to 2000 mg kg⁻¹ and around 1000 mg kg⁻¹ in the Histosol (Table 4). The main exception was the OP₃₈ fertilization treatment in the Silandic Andosol which had not received P fertilisation since 1938, but had a high P content in the top 10 cm. The P_t, P_i and P_{ox} content at the 0-5 and 5-10 cm depths increased with increasing P application. However, this did not apply to the P_o content, except in the Histosol. The increase in P_i and P_{ox} with increasing P fertilisation and the positive correlation between P_t and P_i (R² 0.89) and P_{ox} (R² = 0.83) and a correlation between P_t and P_{ox} (R² = 0.87) (Table 5) indicated that most of the applied P remaining in the soil was inorganically bound. The P_o values were in all cases higher in the fertilised

than in the OP treatments at all depths, with the exception of OP₃₈, which had a much higher P_o than other OP treatments. The coefficient of correlation between P_t and P_o (R² = 0.62) also indicated that some, but significantly less, of the surplus P was bound in the organic matter than was inorganically bound.

The P_{ox} fraction was measured in the two Andosols and followed more or less the same pattern as P_i and was on the whole 14% higher than P_i. This indicated that P_{ox} was not only extracting P from mineral surfaces but also a part of the P_o or P in organic-mineral complexes.

In the unfertilised area and in the OP treatment, the P_{AL} values ranged from 2 to 58 mg kg⁻¹ in the top 5 cm but decreased to 2 to 11 mg kg⁻¹ below the top 5 cm (Table 4). The main increase in P_{AL} with increasing P fertilisation was in the top 5 cm and to a lesser extent at a depth of 5-10 cm. At 10-20 cm depth there was practically no change, which was consistent with no or only a small increase in other fractions (Table 4). P_{an} was 2-3 times higher than P_{AL}, showing the ability of P to be extracted in water provided it is removed from the solution.

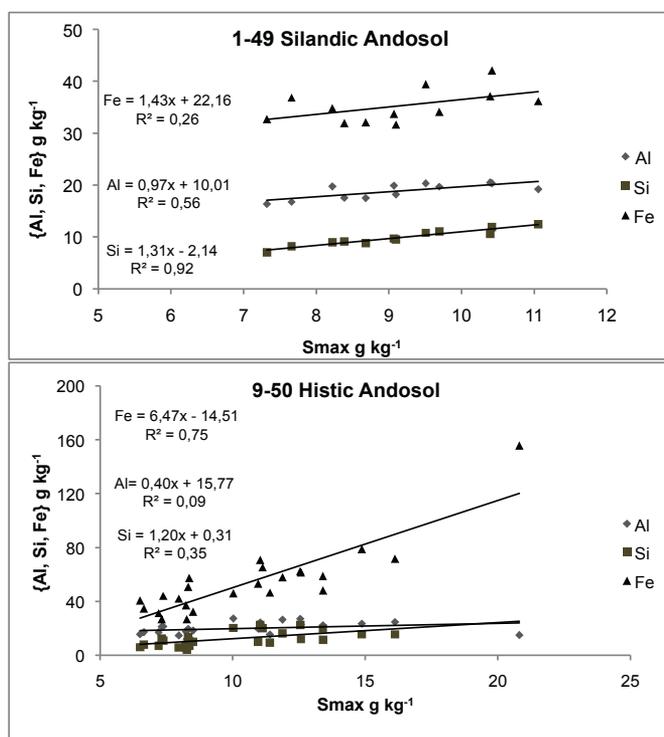
The high coefficient of determination (R² = 0.91) (Table 5) between P_{ox} and P_{AL} indicated that the soil test method of Egner et al. (1960) was extracting P that was bound in Fe and Al complexes. The correlation with P_{an} was weaker, possibly because of fewer samples analysed by the anion exchange resin. However, the high intercept at low P_{AL} and P_{an} values showed that the inorganic P and oxalate extractable P was partially too strongly bound to be accessible in the weaker extracts.

P retention (S_{max}) and P saturation degree (DPS)

The P sorption maximum (S_{max}) calculated from the adsorption isotherms were in the range of 6.9 to 24 g kg⁻¹ (Table 4). It was significantly higher in the 30P treatment than in the OP treatment in the Histosol at Hvanneyri, but in the Andosols at Sámstaðir there was no

Table 5. Regression of P fractions (mg kg⁻¹) and of DPS% of all plots and depths that were analysed.

Equations	R ²	p	n
P _t = 880 + 1.29 P _i	0.89	<0.0001	139
P _i = 36.5 + 1.97 P _{ox}	0.62	<0.0001	139
P _i = 792 + 1.18 P _{ox}	0.83	<0.0001	105
P _t = 2054 + 10.0 P _{AL}	0.33	<0.0001	119
P _t = 1941 + 4.53 P _{an}	0.57	<0.0001	44
P _i = 5.72 + 0.86 P _{ox}	0.87	<0.0001	107
P _i = 907 + 12 P _{AL}	0.39	<0.0001	121
P _i = 784 + 3.5 P _{an}	0.67	<0.0001	44
P _{ox} = 933 + 27 P _{AL}	0.91	<0.0001	105
P _{ox} = 1241 + 4.3 P _{an}	0.76	<0.0001	36
P _{an} = 20 + 2.2 P _{AL}	0.88	<0.0001	43
DPS % = 3.7 + 0.13 P _{AL} mg/kg	0.87	<0.0001	105
DPS % = 5.2 + 0.022 P _{an} mg/kg	0.79	<0.0001	35

**Figure 1.** Regression analysis of P retention (S_{\max}) in relation to acid oxalate soluble Al, Si and Fe in the Silandic- and the Histic Andosols.

apparent trend with increasing P fertilisation.

S_{\max} was very highly correlated with Si_{ox} ($R^2 = 0.92$) and highly correlated with Al_{ox} ($R^2 = 0.56$) in the Silandic Andosol (Figure 1). In the Histic Andosol S_{\max} was very highly correlated

with Fe_{ox} ($R^2 = 0.75$) but for Si_{ox} and Al_{ox} the R^2 was 0.35 and 0.09, respectively. Thus, the P sorption could be explained to a large extent by the pedogenic oxides where Si and Al were the main factors in the well oxidised soil and Fe in the reduced environment.

The P saturation index (DPS) was in the range of 3 to 27%, which indicates the P sorption capacity of the soil (Table 4). It was lowest in the unfertilised treatments and it was low below the top 5 cm. Only in the top 5 cm of the soil was there a significant change with increasing P fertilisation. The DPS was highly correlated with the most soluble P fractions, the P_{AL} and P_{an} (Table 5). The coefficients of determination for P_{AL} and P_{an} were 0.87 and 0.79, respectively, indicating that the ammonium lactate method can possibly be used to estimate the P saturation of the soil.

P pools and budget

In the top 10 cm of the soils there were 1096 and 1019 kg P ha⁻¹ in the unfertilised area of two Andosols (Table 6). However, in the 0P treatments the range was much higher, 753 kg ha⁻¹ in the Histic Andosol, compared to 990 and 1627 kg ha⁻¹ in the two 0P treatments in the Silandic Andosol. At Hvan-

neyri there was no unfertilised area and there was only 460 kg P ha⁻¹ in the top 10 cm of the 0P treatment. P_t increased to a maximum of 2058 kg ha⁻¹ in 26P treatment in the Silandic Andosol and to 2041 kg ha⁻¹ in the 39P treatment in the Histic Andosol. In the 0P treat-

ments the P_{AL} fraction was only 2 and 3 kg P ha⁻¹ in the Silandic Andosol, 5 kg ha⁻¹ in the Histic Andosol and 7 kg ha⁻¹ in the Histosol. With increasing fertilisation, from 13P to 39P, in the Histic Andosol there was a continuous and significant increase in P_{AL} from 10 to 29 kg P ha⁻¹ after 54 years. Applying 26P for 56 years had increased the P_{AL} stock to 31 kg ha⁻¹ in the Silandic Andosol and 30P application for 36 years had increased the stock to 50 kg ha⁻¹ in the Histosol. The P_{an} stocks were in all cases substantially higher than the P_{AL} stocks (Table 6).

The P budget was derived from the P balance (P applied during the experiments minus P removed with the harvested grass) and the P_t stock in the top soil (Table 7). In the 0P treatments 3.7 to 7.5 kg P were removed annually from the Andosols at Sámstaðir, which adds up to 207 to 414 kg P ha⁻¹ over the experimental period. However, in the Histosol at Hvanneyri only 1.0 kg P was removed annually or 34 kg ha⁻¹ in 36 years. In the Histic Andosol the average P balance for the 5 treatments was 1155 kg P ha⁻¹ (\pm 155 S.d.) (Table 7) and that corresponded well with the 1096 kg P ha⁻¹ in the unfertilised area outside the experiment (Table 6). Thus all applied phosphorus could be accounted for by P in the harvest and P_t in the top 10 cm of the soil. Similarly all P could be accounted for in the Histosol in Hvanneyri. It was more difficult to explain the situation in the Silandic Andosol in Sámstaðir because of the high P content in the 0P₃₈ treatment.

Table 6. Pools of phosphorus in the top 0-10 cm of 1-49 and 9-50 and top 0-15 cm of 299-70, kg ha⁻¹. Total (P_t), inorganic (P_i), organic (P_o), acid oxalate (P_{ox}), ammonium oxalate (P_{AL}) and anion exchange resin (P_{an}). Treatments were compared in each experiment and each extraction using Anova and the Tukey-Kramer test. Levels not connected by the same letter were significantly different. ($p \leq 0.05$). Root MSE (RMSE) from the variance analysis are also shown.

P-fertiliser	Pt	Pi	Po	Pox	P _{AL}	P _{an}
Sámstaðir 1-49. Duration 67 and 56 years. Silandic Andosol.						
0P ₃₈	1627 a	513 a	1114 a	602 a	3 a	14 a
0P ₄₉	990 b	471 a	519 b	523 a	2 a	
26P	2058 a	1136 b	921 ab	1316 b	31 b	103 a
RMSE	242	70	240	54	4.7	21
NF ¹⁾	1096	550	546	744	5	20
Sámstaðir 9-50. Duration 54 years. Histic Andosol						
0 P	753 a	316 a	438 a	450 a	5 a	12 a
13 P	1294 b	529 ab	765 b	669 ab	10 a	
22 P	1435 b	815 b	620 ab	819 bc	14 ab	62 a
31 P	1627 bc	1005 c	621 ab	1074 c	18 bc	
39P	2041 c	1322 c	719 ab	1468 d	29 d	195 a
RMSE	202	170	132	124	6.0	87
NF ¹⁾	1019	572	447	756	13	51
Hvanneyri 299-70. Duration 36 years. Hemic Histosol						
0 P	462 a	64 a	398 a		7 a	8 a
30 P	949 b	418 b	531 b		50 b	90 b
RMSE	39	29	21		4.5	12

1) NF = not fertilised area adjacent to the experiment.

DISCUSSION

Fractions and pools of phosphorus

In the unfertilised area and in the lowest depths that were not affected by fertilisation P_t was in the range of about 1200 to 2000 mg kg⁻¹, which was in the same range as we found in an earlier study (Gudmundsson et al. 2005). These were substantially higher P_t values than Negassa & Leinweber (2009) report from various long term land use and management systems and soil types without P applications, where P_t was in the range of 207 to 828 mg kg⁻¹. In uncultivated and not fertilised Japanese Andosols P_t was in the range 246 to 1622 mg kg⁻¹ (Otani & Ae 1999, Takahashi & Anwar 2006). This indicates that the Icelandic soils are in the higher range in P_t , which is in line with a relatively high P content of Icelandic basalt (Jakobsson 1980). The P_t content increased with increasing fertilisation and the main increase was in the top 5 cm (Table 4), with a maximum of 7504 mg kg⁻¹ in the Histic Andosol (39P). These were high values compared with the fertilised soils investigated by Otani

& Ae (1999), Takahashi & Anwar (2006) and Negassa & Leinweber (2009), who were looking at bulk samples from the top 20 cm of arable soils. In the present study of permanent grasslands the high values were, however, only found in the top 5 cm and are thus not directly comparable with the other studies.

Phosphorus fertilisation has mainly affected the inorganic fractions (P_i and P_{ox}) and the available P_{AL} and P_{an} , but did not significantly influence P_o . That suggests that surplus mineral P fertiliser is basically inorganically bound as is often the case (Pätzold et al. 2013). Furthermore, the results were in this respect in accordance with the findings of Helgason (2002) on Icelandic soils and the results of Otani & Ae (1999) and Takahashi & Anwar (2007) on Japanese Andosols. They also separated P_i in Fe and Al complexes and found that in their Andosols the P_i was primarily fixed by Al, followed by Fe compounds. There was a good correlation between the P sorption capacity S_{max} and Si_{ox} and Al_{ox} in the Silandic Andosol, but in the Histic Andosol S_{max} was mainly correlated with Fe_{ox} and to a lesser extent with Si and Al (Figure 1). This indicated that the ability to fix fertilised P was associated with Al and Si in the well-drained Andosols but with Fe in the poorly drained soils.

A higher P_{ox} than P_i implied that P_{ox} not only extracted inorganically bound P but also some bound in organic substances. It is likely that

the P_{ox} was associated with stable Fe and Al organic complexes as has been observed in Japanese Andosols (Otani & Ae 1999, Takahashi & Anwar 2007).

P_o increased only significantly in the Histosol, where part of the surplus P was organically bound, although most of it was bound inorganically. Although P_o was not significantly higher in the treatments with P fertilisation in the Andosols the fertilised treatments were slightly higher in P_o , but increasing P application beyond 13 kg P in the Histic Andosol did not have any further effect. It may therefore be that low rates of P fertiliser application influence the turnover part of P_o , as suggested by Turner et al. (2005). Therefore it may be more useful to assess the turnover rates, rather than amounts in static pools, considering that about 2 to 5% of the organic P in cultivated soil is present in the soil microbial biomass (Stevenson & Cole 1999).

The high P_t content in $0P_{38}$, which was due to high P_o , remained unexplained and, despite the higher content than in $0P_{49}$, the P uptake was lower (Table 7). In $0P_{38}$ P_o was significantly higher than in both $0P_{49}$ and 26P. We do not have complete records of harvest and uptake for 1938 to 1949, but then $0P_{49}$ received 16P annually, which cannot explain the large differences in P_o . Even if the ignition method used in this study tends to overestimate organic P_o in most soils, by increasing the solubility

Table 7. Phosphorus budget at the three locations. The balance is the difference between applied- and harvested P. Total P (P_t) in the top 0-10 or 0-15 cm. All values in kg ha⁻¹.

Sámsstaðir 1-49. Duration 67 and 56 years. Silandic Andosol.					
	0P₃₈	0P₄₉	26P		
P _{annual harvest}	3.7	5.6	13.5		
P _{balance 1949-2005}	-207	-313	701		
P _{t 0-10 cm - P_{balance}}	1834	1302	1357		
Sámsstaðir 9-50. Duration 54 years. Histic Andosol					
	0P	13P	22P	31P	39P
P _{annual harvest}	7.5	14.9	17.3	19.9	20.3
P _{balance 1950-2004}	-414	-99	255	588	1044
P _{t 0-10 cm - P_{balance}}	1167	1393	1180	1039	997
Hvanneyri 299-70. Duration 36 years. Hemic Histosol					
	0P	30P			
P _{annual harvest}	1.0	16.3			
P _{balance 1970-2006}	-34	480			
P _{t 0-15 cm - P_{balance}}	496	469			

of inorganic P minerals following ignition (Turner et al. 2005), it does not explain the high P_o in $0P_{38}$ compared to the other treatments. We cannot exclude the possibility of excessive manure applications before records started in 1938. That might be the most probable reason.

The P_{AL} in 0-5 cm $0P$ treatment was in the range of 2 to 25 mg kg⁻¹ and increased steadily with increased P (Table 4). In Iceland 40 mg P_{AL} kg⁻¹ is used to distinguish low and medium P_{AL} level in the top 5 cm in Icelandic grassland soils (Sigvaldason 1996, Gudmundsson & Sigvaldason 2000). The $0P$ and soils below the top 5 cm remained below this level. If the P accumulating in the top 5 cm were mixed through soil tillage there would inevitably be less available P in the tilled layer.

Phosphorus extracted in water and removed from the solution by anion resin is an indicator of P release. The P_{an} pool in the top 10 cm of the soils were 381 and 123 kg P ha⁻¹ in the 39P treatment in the Histic Andosol and in the 30P treatment in the Histosol, respectively. As annual uptake of P is often in the range 12 to 20 kg ha⁻¹ (Óskarsson & Brynjólfsson 2000) it may be postulated that these soils would be able to sustain P availability where appreciable amounts of P have been build up. The results suggest that the P added to the soil was not so strongly bound that it is permanently unavailable.

As there were no significant changes in the P fractions below a 10 cm depth, the stock budgets were calculated for the top 10 cm layer. The stocks increased continually with increasing P fertilisation in the Histic Andosol and were significantly higher with P fertilisation in the Silandic Andosol and in the Histosol than in the $0P$ treatments. By comparing the stocks in the unfertilised areas in the Andosols at Sámstaðir with the treatments (Table 5), both P_i and P_o were lower in the $0P$ than in the NF area (except in $0P_{38}$ in the Silandic Andosol), indicating that P was taken up both from the organic and the inorganic pools. In all treatments only inorganic P was applied, resulting

in an increase in P_o . However, increasing the P application beyond 13P in the Histic Andosol did not result in a further increase in P_o . The continuous and significant increase in P_{AL} with increasing P application gradually increased available P and thus the soil fertility.

P retention and saturation

The maximum P retention (S_{max}) calculated by the Langmuir adsorption isotherms were mainly in the range of 7 to 15 g kg⁻¹ (Table 4, Figure 1), similar to the range Madeiera et al. (2007) reported for Andosols. However, they were lower than Auxtero et al. (2008) found in the Andosols of the Azores, where maximum retention values reach 47 g kg⁻¹ and are frequently >10 g kg⁻¹. The differences in the correlation of S_{max} and oxalate extractable Si, Al and Fe showed that the soil type played a major role. In the dry Silandic Andosol, S_{max} was associated with Si and Al, whereas in the Histic Andosol the P sorption could to a large extent be explained by Fe_{ox} , whereas Al_{ox} and Si_{ox} played a lesser role. Iron segregation and very high Fe_{ox} values are common near or at the surface in Icelandic Histosols and Histic Andosols (Gudmundsson 2009). García-Rodeja et al. (2004) found that phosphate retention is closely related to $Al_o + 1/2Fe_o$. Our results suggest that the soil type and redox conditions also need to be taken into account.

Large surpluses of P through fertilisation and excessive P contents in the soil may lead to the pollution of waterways and lakes and long-term P surpluses will eventually lead to a gradual saturation of the soil P-sorption capacity (Beuchemin & Simad 1999, Hooda et al. 2001). The molar ratio of $P_{ox}/0.5(Al_{ox} + Fe_{ox})$ is often used to assess the degree of phosphorus saturation (DPS). Values of >25 to 30% have been found to be a critical limit for P to avoid loss of P to the environment (Beauchemin & Simad 1999, Schoumans 2000). The highest DPS values in the present study were 22 and 27% in the 31P and 39P treatments in the Histic Andosol, respectively, with a surplus P of 588 and 1044 kg ha⁻¹. A surplus of 701 kg

ha⁻¹ in the Silandic Andosol had a DPS of 13%, indicating that the soil type may play a role in P saturation.

The results from the present study showed that P did not increase with depth and only after applying 600 to 1000 kg P ha⁻¹ over many decades may a critical DPS be reached in the top 5 cm of the soil. Pollution problems through leaching of P are not expected in these soils. However, surface run-off or erosion upon tillage could bring appreciable amounts of soluble P into waterways. There was a linear correlation between DPS and P_{AL} and in the fertilised treatments the P balance was in the range of about 500 to 1000 kg ha⁻¹ with a corresponding increase in P_{AL} values. According to the review of Beauchemin & Simard (1999) and Mejias et al. (2013) a linear relationship between soluble P and DPS is common up to a certain level of the saturation index, where there is a quick increase in soluble P. In the present study this level was apparently not reached.

CONCLUSIONS

The present study indicated that P uptake in treatments without P fertilisation can be both from the organic and inorganic forms. Surplus P was mainly found in the inorganic fraction in all soil types and all surplus application of P could be found in the top 10 cm of the soils. Thus, there was no or only a minimal loss of P to the environment. The increase in P_i increased the available P_{AL} and P_{an}, which were highly correlated with the degree of P saturation. This increased the soil fertility with regard to P and a stable base for future P management has been built up. Further investigations would be needed to establish for how long and at what rate the increased P will be released as available P. In spite of an increase in P stocks of between 500 and 1000 kg in the top 10 cm of the soils, which corresponds to a 50 to 100% increase, the DPS was still under or close to the maximum level before P is expected to cause pollution to the environment. However, care has to be taken not to lose the top few cm

where the excess P is found. S_{max} was correlated to Si and Al in the Silandic Andosol but to Fe and not to Si and Al in the Histic Andosol. This shows that in Iceland these soil types need to be considered separately when studying P dynamics and P management.

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