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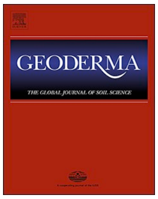

Comparing measures for determination of phosphorus saturation as a method to estimate dissolved P in soil solution

Karin Blombäck[a,*], Carl H. Bolster[b], Anders Lindsjö[a], Kathrin Hesse[a], Helena Linefur[a], Mohammed Masud Parvage[a,c]

[a] Swedish University of Agricultural Sciences, Department of Soil and Environment, P.O. Box 7014, SE-750 07 Uppsala, Sweden
[b] USDA-ARS, Food Animal Environment Systems Research Unit, 2413 Nashville Rd. B-5, Bowling Green, KY 42101, USA
[c] Uppsala University, Department of Earth Sciences, Villavägen 16, SE-752 36 Uppsala, Sweden

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ABSTRACT

In response to concerns over the translocation of P from soils to P-sensitive water bodies, there is high demand for developing simple indicators for evaluating a soil's risk of releasing P into solution. Many studies have shown that the degree of soil phosphorus saturation (DPS), calculated as the ratio of soil P concentration to soil P sorption capacity (PSC), is good predictor of a soil's risk of releasing P in solution. In this study we compared four different DPS indices in how well they predicted dissolved P following extraction with either a 0.01 M CaCl2 (P_{CaCl2}) solution or deionized water (P_W). The first two indices were calculated from the ratio of extractable P to extractable Al and Fe using either acid ammonium oxalate (Al_{OX} + Fe_{OX}) or ammonium lactate solutions (Al_{AL} + Fe_{AL}). The second two DPS indices were calculated from the ratio of either Olsen-extractable P or AL-extractable P with sorption capacity estimated from the single point P sorption index (PSI). On a subset of 11 soils, we compared the different methods for estimating PSC with fitted Langmuir sorption maximum (Smax) using data from complete sorption isotherms. Both (Al_{OX} + Fe_{OX}) and PSI were well correlated with Smax and hence regarded as good estimates for P sorption capacity. Conversely, (Al_{AL} + Fe_{AL}) was not significantly correlated with Smax. P saturation calculated from PSI together with P_{AL} or P_{Ols} predicted P_{CaCl2} and P_W best, whereas P saturation calculated from ammonium oxalate predicted P_{CaCl2} and P_W the least. We did not find notable improvements in the regression models when we added a second explanatory variable (clay content, pH or total carbon) to the models. Our results show that multiple measures of P saturation provide similar predictions of a soiĺs potential for releasing dissolved P into soil solution. This provides flexibility in how P saturation indices are calculated to identify leaching prone hotspots.

1. Introduction

Eutrophication caused by phosphorus (P) losses from land to surface waters is a worldwide problem (Sharpley et al., 2015). In Sweden, diffuse P loss from agricultural fields is the largest contributor to P loadings to surface waters (Boesch et al., 2006; Ejhed et al., 2016). As a result, there is significant pressure to identify leaching prone hotspots and suggest best management practices to reduce P loads from agricultural fields. Data on leaching losses of P from more than 30 observational sites in Sweden (from catchments and drained fields) show that the ratio of the load of dissolved P to total P in drain water (kg ha^{-1} $year^{-1}$) ranges between 0 and 100%, with a mean of approximately 30% (Linefur et al., 2018a,b). This indicates that losses of dissolved P can be substantial in certain areas and from certain fields. In Sweden, as in many other countries, a soil's ability to release dissolved P in runoff or with subsurface drainage is estimated based on agronomic soil P tests that originally were developed to assess plant available P. A disadvantage with this approach to estimate the risk for P losses is that the relationship between soil P tests and dissolved P in soil solution is soil specific because it not only depends on soil P quantity but also the capacity of soils to retain P (Sharpley, 1995; Maguire and Sims, 2002b; Nawara et al., 2017; Zehetner et al., 2018). The degree of phosphorus saturation (DPS) takes into account both the soil P concentration and the P sorption capacity (PSC) of the soil and has been shown to improve predictions of a soil's risk of releasing P in solution during runoff or leaching events compared to soil P test (Sharpley,

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[*] Corresponding author.
E-mail addresses: Karin.Blomback@slu.se (K. Blombäck), Carl.Bolster@usda.gov (C.H. Bolster), Anders.Lindsjo@slu.se (A. Lindsjö), Kathrin.Hesse@slu.se (K. Hesse), Helena.Linefur@slu.se (H. Linefur), masud.parvage@geo.uu.se (M.M. Parvage).

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1995; Maguire and Sims, 2002b; McDowell et al., 2002; Nair et al., 2004; Nair, 2014; Wang et al., 2012; Kleinman, 2017).

The degree of P saturation was originally defined as the molar ratio of P to Al and Fe as determined from ammonium oxalate extractions (van der Zee et al., 1987; Breeuwsma and Silva, 1992). This formulation was based on studies indicating that PSC in acid sandy soils was predominately due to P sorption to amorphous Al and Fe hydroxides (Beek and van Riemsdijk, 1979; van Riemsdijk and van der Linden, 1984; Breeuwsma and Silva, 1992). In many countries, however, oxalate extraction is not commonly used in standard soil tests; therefore, researchers have investigated alternative approaches for calculating DPS using data collected from routine soil tests. For instance, several authors have found strong relationships between DPS calculated from ammonium oxalate (OX) extracts and DPS calculated from Mehlich-3 extracts of P, Al and Fe (Khiari et al., 2000; Kleinman and Sharpley, 2002; Maguire and Sims, 2002a; Sims et al., 2002; Nair et al., 2004; Wang et al., 2012). In addition to estimating PSC from extractable concentrations of Al and Fe, estimates of PSC have been calculated from the single point sorption index (PSI) developed by Bache and Williams (1971) (Börling et al., 2004; Andersson et al., 2013; Eriksson et al., 2013). While originally intended to be a direct measure of buffer capacity rather than P sorption capacity, PSI has been shown to be correlated with maximum P sorption capacity as determined from the Langmuir sorption equation (Bache and Williams, 1971; Börling et al., 2001; Bolster et al., 2020).

A more direct method to estimate P solubility would be to measure dissolved P in soil solution. Prediction of dissolved P extracted by the weak salt solution of $CaCl_2$ (P_{CaCl2}) is often used as an estimate of P concentration in soil solution (Schofield, 1995; McDowell and Sharpley, 2001), whereas prediction of dissolved P extracted by distilled or deionized water (P_W) is used as an estimate of P dissolved by rain or snowmelt (e.g Pote et al., 1996; McDowell and Sharpley, 2001). A disadvantage with using such a direct measure of dissolved P such as P_{CaCl2} and P_W as an indicator for P leaching prone hot spots is that it only gives a current value on the intensity of dissolved P in the soil solution and no information about the capacity of the soil to retain additional P. A risk assessment tool for identification of leaching prone soils or to be used as a guidance for fertilization recommendations needs to include an indication of the additional storage capacity of P in the soil (Nair and Harris, 2014; Kleinman, 2017).

In Sweden, ammonium lactate extract (AL; Egnér et al., 1960) is routinely used for determining soil P concentrations. The method is also commonly used in Belgium, Norway, Lithuania, Germany, Poland, Hungary and Slovenia (Ulen, 2006; Jordan-Meille et al., 2012). As also Al and Fe can be analyzed, estimates of P sorption capacity as the sum of Al_{AL} and Fe_{AL}, and degree of P saturation can be calculated from the AL analysis. The degree of P saturation based on the AL analysis (DPS_{AL}) has been suggested as a simple risk assessment for losses of dissolved P from agricultural fields in Sweden (Ulen, 2006). However, while studies have shown that Mehlich-3 extracts can produce similar DPS estimates as ammonium oxalate extracts (DPS_{OX}) (Khiari et al., 2000; Maguire and Sims, 2002a), there are no results on how well DPS calculated from AL-extracts correlates with DPS_{OX} and whether DPS_{AL} is a good predictor of dissolved P in soil solution. There are also only few results (Ulen, 2006; Eriksson et al., 2013) on how well the P sorption capacity estimated from the AL extraction correlates with estimates based on ammonium oxalate extractions, PSI and sorption isotherms.

The overall objective of this study was to investigate the accuracy of different P saturation indices to predict concentrations of easily dissolved P in different Swedish arable soils, representing a wide range in terms of soil texture, soil P content and P sorption capacity. Specifically, our objectives were to: 1) determine the correlation between different soil P extractions, estimates of P sorption capacities and measures of P saturation, and 2) evaluate different P saturation indices as predictors of dissolved P in soil solution. Our goal was to find an inexpensive and readily available method to predict easily dissolved P suitable for arable soils young in geological origin, as in Sweden.

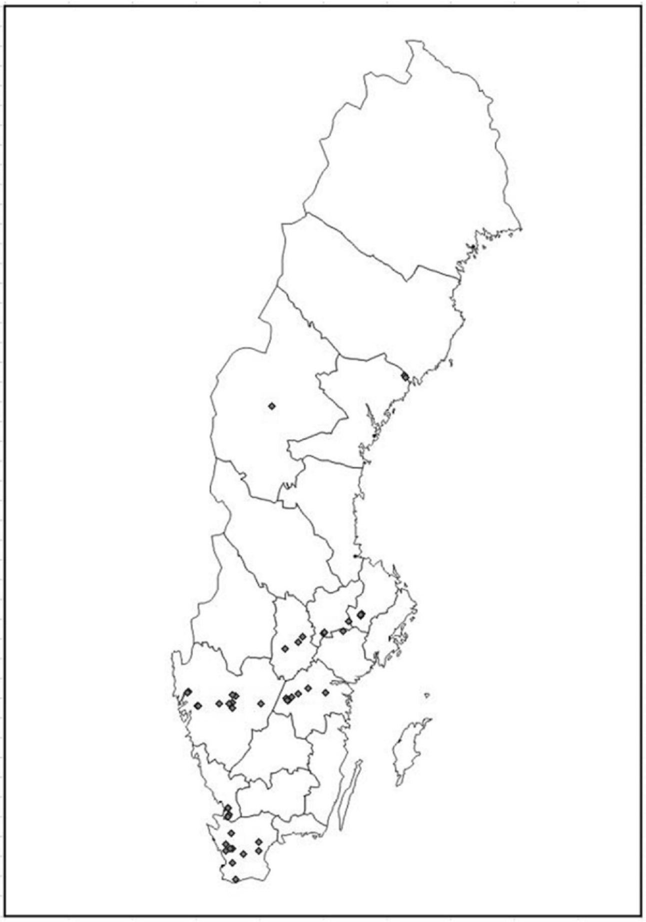

Fig. 1. Map showing soil sampling locations.

2. Material and methods

2.1. Soils

We collected 62 topsoil samples (0–20 cm) from 45 arable fields throughout Sweden (Fig. 1). The fields were selected from different field experiment locations and long-term monitoring sites to represent the range in texture and chemical properties typical for Swedish agricultural soils (Eriksson et al., 2010). Eight of the sites are used for long-term soil fertility experiments with different fertilization treatments in the experimental set-up (i.e. 'No P added', 'Replacement of harvested P', 'Replacement + 20 kg P' and 'Replacement + 30 kg P'). Only treatments without manure were sampled. At these sites, samples were taken from different P fertilization treatments to include both high and low soil P concentrations from the same soils and each treatment was regarded as a unique soil and not a replicate (no 3, 4, 9–12, 14, 16, 18, 20, 22, 23, 28, 29, 36, 39, 43, 51 and 55 in Table A1). At three of the fields, two soil samples were collected to capture within field differences in clay content (soil no. 17 and 30, 25 and 40, respectively in Table A1) and in soil P content (soil no. 19 and 45 in Table A1) as indicated by the farmers. Also these samples were regarded as unique samples and not as replicates. Eleven of the soils (soils no. 2, 7, 8, 12, 14, 23, 24, 32, 37, 59 and 62 in Table A1) were selected for additional characterization using P sorption isotherm studies. The 11 soils were selected to give a broad range in clay content in combination with ranges in PSC and P saturation indices (Table A2). Samples were collected at least 6 months after any P applications to avoid confounding effects of recent P application on extractable P concentrations. Samples were collected with a soil auger. Several soil cores were collected within a radius of 0.5 m and mixed together to a composite sample. All samples were air dried at 30 °C, crushed and sieved (2 mm).

Soil texture was determined with the pipette sedimentation procedure according to Ljung (1987) and classified according to the USDA soil classification (Soil survey division staff, 1993). Soil pH was measured in deionized water solution by the standard SIS method (SS-ISO 10390; Swedish Standard Institute). Total soil carbon (C_{tot}) was analysed by dry combustion using a LECO CNS2000 analyser (LECO Corporation, 2003).

Swedish arable soils are of very young age with little soil development and are usually categorized and considered in terms of texture classes rather than soil groups. According to Eriksson et al. (2017) it can be assumed that the majority of soils developed in fine to medium-textured material should be classified as Cambisols, while those in silty materials or glacial layered parent materials may also be Regosols (Fig. A1). The soils having sand or loamy sand in the topsoil can maybe qualify as Arenosols, but a depth of at least 100 cm of the sandy layer could not be verified in this study.

2.2. Extractions of P, Al and Fe

For each soil the following soil P extractions were performed. Acid ammonium oxalate solution (0.2 M ammonium oxalate + 0.2 M oxalic acid solution, pH 3) as described by Schwertmann (1964) was used to determine the pool of adsorbed phosphorus (P_{OX}). Ammonium lactate solution (0.1 M NH_4-lactate + 0.4 M HOAc, adjusted to pH 3.75; Egnér et al. (1960) was used in accordance with the Swedish standard to determine plant available P (P_{AL}). Olsen P (0.5 M $NaHCO_3$, pH 8.5; Olsen et al., 1954) was determined (P_{Ols}) as it has recently been advocated as an alternative soil P test for plant available P in soils with high pH in Sweden (Swedish Board of Agriculture, 2018). A 2 M hydrochloric acid solution was used in accordance with Swedish standard to determine storage P, i.e. plant available P over a longer period of time (KLS, 1965; P_{HCl}). Easily dissolved P was determined using two extractions: deionized water (P_W) or 0.01 M $CaCl_2$ (P_{CaCl2}) (soil:extract ratio 1:3) according to Börling et al. (2004) but with a longer equilibration time (20 h instead of 1 h). Concentrations of extractable Al and Fe were determined from the ammonium oxalate (Al_{ox}, Fe_{ox}) and ammonium lactate extracts (Al_{AL}, Fe_{AL}).

All extracts were filtered before analysis (0.2 µm). The OX, AL and HCl extracts were analysed for P, Al and Fe concentrations using inductively coupled plasma atomic emission spectroscopy (ICP-AES) according to Boumans (1979). Concentrations of dissolved reactive phosphate in deionized water and $CaCl_2$ extracts (P_W and P_{CaCl2}) were determined colorimetrically according to Murphy and Riley (1962).

2.3. Soil sorption studies

The single point P sorption index (PSI) of Bache and Williams (1971) was calculated for each soil. Two grams of soil were equilibrated with 19.4 mmol P kg^{-1} soil (KH_2PO_4) in 20 ml 0.01 M $CaCl_2$ on a shaker for 20 h before centrifugation at a relative centrifugal force of 2095 g for 10 min. The supernatants were filtered (0.2 µm) before the remaining P concentration in solution was measured colorimetrically according to Murphy and Riley (1962). The amount of P sorbed by the soil was calculated from the difference in concentration in the solution before and after equilibration and the solution to soil ratio (L kg^{-1}). The PSI (mmol kg^{-1}) was calculated as:

$$PSI = \frac{S}{\log(C_e)} \tag{1}$$

where S is the sorbed concentration of P (mmol kg^{-1}) and C_e is the equilibrium P concentration in solution (µmol L^{-1}).

We also conducted sorption isotherm studies following the procedure of Börling et al (2001) on a subset (n = 11) of the collected soils representing a range in soil texture, soil test P and OX-extractable Al and Fe. Briefly, two grams of air-dried soil was equilibrated with 20 ml of 0.01 M $CaCl_2$ containing one of 10 different concentrations of KH_2PO_4 ranging from 0 to 250 mg P L^{-1} with the exception of one soil which was equilibrated with an additional concentration of 500 mg P L^{-1}. The tubes were shaken end-over-end for 20 h. Following centrifugation at 3000 rpm for 10 min, solution equilibrium P concentration was analyzed colorimetrically according to Murphy and Riley (1962).

The sorption data were fit using the Langmuir equation modified to account for background P concentrations as follows (Zhou et al., 2005):

$$S = \frac{S_{max} \bullet k \bullet C_e}{1 + k \bullet C_e} - S_0 \tag{2}$$

where C_e is the P concentration (mmol L^{-1}) in solution following equilibration with soil, S is the net amount of P sorbed during the experiment (mmol kg^{-1}) calculated from the change in P concentration in solution following equilibration with the soil and the solution to soil ratio (L kg^{-1}), S_0 is the residual or background P on the soil (mmol kg^{-1}), S_{max} is the maximum sorption capacity of the soil (mmol kg^{-1}), and k is the Langmuir binding coefficient (L $mmol^{-1}$). S_0 was estimated from the solution concentration of P (C_0) following equilibration with an initially P-free solution (i.e. $C_0 = 0$):

$$S_0 = \frac{S_{max} \bullet k \bullet C_0}{1 + k \bullet C_0} + \frac{C_0 \bullet V}{M} \tag{3}$$

where V is the solution volume (L) and M is the mass of soil (kg) in the sample. Eqs. (2) and (3) were combined and fit to the sorption data using a modified version of the Excel spreadsheet of Bolster and Hornberger (2007).

2.4. P saturation indices

The degree of P saturation (DPS) is defined as the ratio of sorbed P to the P sorption capacity of the soil. DPS_{OX} was calculated as the ratio of the molar concentrations (mmol kg^{-1}) of P_{OX} to Al_{OX} and Fe_{OX} of the soil (van der Zee et al., 1987; Breeuwsma and Silva, 1992):

$$DPS_{OX} = \frac{P_{OX}}{\alpha (Al + Fe)_{OX}} \tag{4}$$

where $(Al + Fe)_{OX}$ is in mmol kg^{-1} and α is an empirical sorption factor that quantifies the fraction of Al and Fe involved in P sorption for P saturated soil. In studies where α was measured directly, the values generally ranged from 0.35 to 0.65 (van der Zee and van Riemsdijk, 1986, 1988; van der Zee et al., 1987; Pautler and Sims, 2000). In many studies, however, α is not measured but rather is assumed to be 0.5 (Lookman et al., 1995, 1996; Kleinman et al., 1999) or 1 (Pote et al., 1996; Kleinman and Sharpley, 2002). In this study, we assumed a value for α of 0.5.

DPS_{AL} was calculated from ammonium lactate (AL) extractable P, Al, and Fe

$$DPS_{AL} = \frac{P_{AL}}{(Al + Fe)_{AL}} \tag{5}$$

where $(Al + Fe)_{AL}$ is in mmol kg^{-1}.

P saturation indices were also determined from ratios of P_{AL} and P_{Ols} to PSI (i.e. P_{AL}/PSI and P_{Ols}/PSI) where P concentrations are in mg kg^{-1} (Börling et al., 2004; Eriksson et al., 2013). While PSI was initially developed to assess soil buffer capacity, it has been shown to be correlated with Langmuir S_{max} values for a wide range of soils (Bache and Williams, 1971; Bolster et al., 2020; Börling et al., 2001).

2.5. Data analysis

Correlations in Al, Fe, and P concentrations between different extraction methods were investigated as well as correlations between the different P saturation ratios. Ordinary least squares simple regression was performed to assess the relationship between dissolved P (P_{CaCl2} and P_W) and P saturation ratios (DPS_{OX}, DPS_{AL}, P_{Ols}/PSI, and P_{AL}/PSI). Because most of the data were not normally distributed, log-transformed data were used in the regression analysis. To determine whether accounting for soil properties could improve the prediction of dissolved P, we also performed 2-variable regressions with P saturation index as one explanatory variable and one of the following as a second explanatory variable: soil pH, total carbon, and fraction of clay, silt, or fine sand. Model fits were evaluated by comparing the coefficient of determination (r^2) and root mean square error (RMSE). Because models with more fitting parameters will almost always result in a better overall fit (i.e. increased r^2 and decreased RMSE values), we used Akaike's information criterion (AIC) to determine if regression models with two explanatory variables were an improvement over regression models with only one explanatory variable. Using this method, the best model fit is the model with the lowest (i.e., most negative) AIC value (Akaike, 1969).

3. Results

3.1. Soil composition and P contents

The soils collected showed great variation in composition (Table 1; Table A1). The most abundant soil textural classes were well represented in our study (Fig. A2). Clay content ranged between 4 and 57%. Total soil carbon content ranged from 1.1 to 3.3%, except for one soil that contained 9.4% total carbon. Soil pH values ranged from 5.3 to 7.7.

Soil P as measured by the different extraction methods varied considerably (Table 1; Table A1). In general, the amount of P extracted by each extraction method followed the pattern $P_{HCl} > P_{OX} > P_{AL} > P_{OLS} > P_W > P_{CaCl2}$. For the soils tested, we found a strong correlation between P_{OX} and P_{HCl} ($r = 0.90$; $p < 0.01$), whereas P_{OX} and P_{AL} were only moderately correlated ($r = 0.53$, $p < 0.05$) (Table 2).

The ratio of P_{OX}:P_{HCl} ranged from 0.42 to 0.88 (mean of 0.65), whereas the ratio between P_{OX}:P_{AL} ranged between 1.4 and 18.6 (mean of 7.0). We found P_{Ols} to be strongly correlated with P_{AL} ($r = 0.88$; Table 2). The ratio of P_{Ols}:P_{AL} ranged between 0.14 and 0.81 (average 0.32).

We observed a strong correlation between DRP extracted with 0.01 M $CaCl_2$ (P_{CaCl2}) solution and deionized water (P_W) ($r = 0.96$; Table 2). On average, deionized water extracted ~8.5 times more DRP than the 0.01 M $CaCl_2$ solution. Concentrations of P_W ranged between 0.039 and 13.05 mmol L^{-1} and P_{CaCl2} between 0.014 and 5.32 mmol L^{-1}. Both P_W and P_{CaCl2} were strongly correlated with P_{AL} ($r = 0.80$ and 0.77, respectively) and to a lesser extent with P_{Ols} ($r = 0.66$ and 0.58, respectively) (Table 2). P_W and P_{CaCl2} were not correlated with P_{OX} ($r < 0.27$) or P_{HCl} ($r < 0.35$).

Table 2
Correlation matrix for P extraction methods. Correlation coefficients > 0.25 are statistically significant at the $p = 0.05$ level. HCl solution (HCl), ammonium oxalate (OX), ammonium lactate (AL), $NaHCO_3$ (Olsen), deionized water (W), $CaCl_2$ solution (CaCl2).

	P_{HCl}	P_{OX}	P_{AL}	P_{Ols}	P_W	P_{CaCl2}
P_{HCl}	1					
P_{OX}	0.90	1				
P_{AL}	0.59	0.53	1			
P_{Ols}	0.61	0.66	0.88	1		
P_W	0.34	0.27	0.81	0.66	1	
P_{CaCl2}	0.29	0.21	0.77	0.59	0.96	1

3.2. Soil sorption properties

The sum of Al_{OX} and Fe_{OX} ranged from 41 to 247 mmol kg^{-1} with an average value of 117 mmol kg^{-1} (Table 1). The sum of Al_{AL} and Fe_{AL} ranged from 8.3 to 30 mmol kg^{-1} with an average value of 16 mmol kg^{-1}. Though statistically significant ($p < 0.05$), the sum of extractable Al and Fe for the two extraction methods was not strongly correlated ($r = 0.36$; Fig. A3). The sum of Al_{OX} and Fe_{OX} was correlated to the clay content ($r = 0.5$) and moderately correlated to pH ($r = -0.34$) total carbon ($r = 0.31$), whereas the sum of Al_{AL} and Fe_{AL} was only moderately correlated to pH ($r = -0.37$).

Moderate, yet statistically significant ($p < 0.05$) correlations between OX- and AL-extractable Al and Fe concentrations were observed with r values of 0.66 and 0.54, respectively (Table 3). The AL solution extracted proportionately less Fe than Al compared with the OX extractions. Fe_{AL} corresponded to on average 10% of Fe_{OX}, whereas Al_{AL} corresponded to 20% Al_{OX}. The ratio of Fe_{AL} to $[Al_{AL} + Fe_{AL}]$ ranged from 0.085 to 0.61 (mean of 0.38) whereas the ratio of Fe_{OX} to $[Al_{OX} + Fe_{OX}]$ ranged from 0.27 to 0.75 (mean of 0.53).

Values for the single point P sorption index (PSI) ranged from 1.5 to 7.6 mmol kg^{-1} with a mean value of 4.2 (Table 1; Table A1). PSI was significantly correlated with both $[Al + Fe]_{OX}$ and $[Al + Fe]_{AL}$, with r values of 0.67 and 0.56, respectively, though the relationship between PSI and $[Al + Fe]_{OX}$ was nonlinear (Fig. A4). PSI was moderately correlated to the clay content ($r = 0.44$), but not to pH or total carbon. Fitted values of S_{max} ranged from 9.7 to 54 mmol kg^{-1} for the 11 soils tested (Table1; Table A3). Our fitted S_{max} values were significantly

Table 1
Minimum, maximum, mean and median values for clay, total carbon, pH, extracted phosphorus, aluminum and iron, and single point P sorption index (PSI) of 62 top soils samples. Minimum, maximum, mean and median values for Langmuir S_{max} values were calculated for 11 of the soil samples.

	Min	Max	Mean	Median
Clay (%)	4.0	57	25.2	23.5
Total carbon (%)	1.1	9.4	2.2	2.0
pH	5.3	7.7	6.5	6.4
HCl extractable phosphorus (mmol kg^{-1})	11	42	24.8	24
Ammonium oxalate extractable phosphorus (mmol kg^{-1})	4.8	32	16	15
Ammonium lactate extractable phosphorus (mmol kg^{-1})	0.65	15	3.5	2.7
$NaHCO_3$ extractable phosphorus (Olsen P) (mmol kg^{-1})	0.15	2.9	1.0	0.82
$CaCl_2$ extractable phosphorus (mmol L^{-1})	0.01	5.23	0.27	0.083
Deionized water extractable phosphorus (mmol L^{-1})	0.04	13.05	1.17	0.64
Ammonium oxalate extractable aluminum (mmol kg^{-1})	20	89	52	52
Ammonium oxalate extractable iron (mmol kg^{-1})	20	130	65	56
Ammonium lactate extractable aluminum (mmol kg^{-1})	4.1	27	10	8.9
Ammonium lactate extractable iron (mmol kg^{-1})	1.4	15	6.1	5.6
PSI (mmol kg^{-1})	1.5	7.6	4.2	4.3
Langmuir S_{max} (mmol kg^{-1})	9.7	54	21.1	18.8

Table 3
Correlation matrix for Al, Fe, and Al + Fe content for ammonium lactate (AL) and ammonium oxalate (OX) extraction methods and with single point P sorption index (PSI), pH, total carbon and clay content. Values of $|r| > 0.25$ are statistically significant at $p = 0.05$ level.

	Al_{OX}	Fe_{OX}	$(Al + Fe)_{OX}$	Al_{AL}	Fe_{AL}	$(Al + Fe)_{AL}$	PSI	pH	totC	Clay
Al_{OX}	1									
Fe_{OX}	0.23	1								
$(Al + Fe)_{OX}$	0.57	0.93	1							
Al_{AL}	0.66	−0.14	0.12	1						
Fe_{AL}	−0.20	0.54	0.39	−0.22	1					
$(Al + Fe)_{AL}$	0.49	0.21	0.36	0.79	0.42	1				
PSI	0.57	0.54	0.67	0.36	0.35	0.55	1			
pH	−0.31	−0.27	−0.34	−0.31	−0.12	−0.37	−0.22	1		
totC	0.34	0.22	0.31	0.21	−0.05	0.17	0.21	−0.08	1	
Clay	0.27	0.48	0.50	−0.28	0.25	−0.11	0.44	0.23	0.02	1

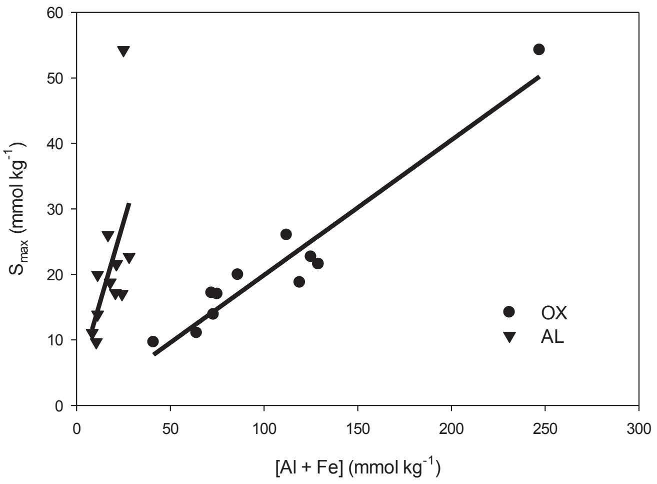

Fig. 2. Relationship between fitted Langmuir maximum sorption capacity (S_{max}) values and the sum of ammonium oxalate (OX)- or lactate (AL)-extractable Al and Fe concentrations (mmol kg^{-1}) for 11 Swedish soils. When the highest value is removed from the regression, the slope between S_{max} and oxalate-extractable Al and Fe changes slightly (0.18) while the strength of the overall relationship is noticeably reduced ($r^2 = 0.65$).

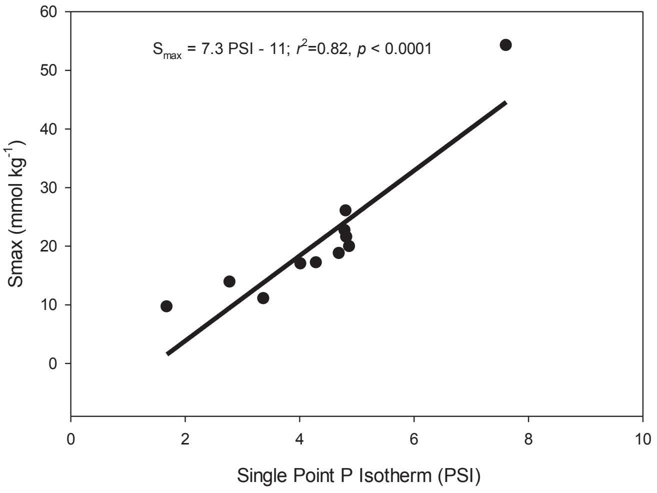

Fig. 3. Correlation between fitted Langmuir maximum sorption capacity (S_{max}) and the single point P sorption index (PSI) for 11 Swedish soils. Removal of the highest value resulted in a notable decrease in slope (4.2) but only a minor decrease in r^2 (0.76).

correlated ($r^2 = 0.91$) with $[Al + Fe]_{ox}$ (Fig. 2). This strong correlation was predominately a result of Fe_{OX} ($r^2 = 0.80$) compared with Al_{OX} ($r^2 = 0.33$) (Fig. A5). The ratio of S_{max} to $[Al + Fe]_{ox}$ ranged from 0.17 to 0.24, with an average value of 0.2. Conversely, the correlation between S_{max} and $[Al + Fe]_{AL}$ was not statistically significant ($r^2 = 0.30$, $p > 0.05$), though S_{max} was significantly correlated with Fe_{AL} ($r^2 = 0.37$, $p < 0.05$). S_{max} was correlated to clay content ($r = 0.73$, $p < 0.001$), but not to pH or total carbon ($p > 0.05$. The ratio between S_{max} and $[Al + Fe]_{AL}$ ranged from 0.7 to 2.2 with an average of 1.2. PSI was strongly correlated with S_{max} ($r^2 = 0.82$) (Fig. 3).

3.3. P saturation indices and correlation to easily dissolved P

Assuming a value of 0.5 for α, DPS_{OX} varied between 0.07 and 0.54, but only in two soils (2 and 6) did the value exceed 0.25. DPS_{AL} varied between 0.04 and 0.66 and P_{Ols}/PSI varied between 0.03 and 0.66, except for soils 2 and 6, where the values exceeded 1 for both soils. P_{AL}/PSI varied between 0.15 and 9.33, and 21 of the soils had values of 1 or higher. There were strong correlations between all P saturation indices with r values ranging from 0.84 to 0.98 (Table 4).

Based on linear regression analysis of the log-transformed data, P_{Ols}/PSI ($r^2 = 0.87$ and RMSE = 0.45) and P_{AL}/PSI (0.86 and 0.46) provided the best fit to the observed P_{CaCl2} data followed by DPS_{AL} (0.84 and 0.49) and DPS_{OX} (0.66 and 0.72) (Fig. 4). Results for P_W were similar with P_{Ols}/PSI providing the best fit and DPS_{OX} providing the poorest fit with r^2 values of 0.66 and 0.44 and RMSE values of 0.64 and 0.82, respectively (Fig. 5). All four P saturation ratios explained more of the observed variability in log-transformed P_{CaCl2} than P_W as evidenced by higher r^2 and lower RMSE values for the P_{CaCl2} data. Although statistically significant ($p < 0.001$) relationships were observed between log-transformed dissolved P concentrations (P_{CaCl2} and P_W) and all four log-transformed P saturation ratios, there still existed a significant amount of unexplained variability in P concentrations (Figs. 4 and 5).

In general, we did not find notable improvements in the regression models when we added a second explanatory variable to the model. For instance, r^2 values increased only slightly from 0.84 to 0.86 and RMSE values decreased slightly from 0.48 to 0.46 when log-transformed pH or total carbon was added to log-transformed DPS_{AL} to predict log-transformed P_{CaCl2} with AIC becoming slightly more negative from −85 to −90 (Table A4). This trend was similar for nearly all combinations of P saturation ratio and a second explanatory variable for both the log-

Table 4
Correlation matrix for easily dissolved P (P_{CaCl2} and P_W) and degree of P saturation (DPS) indices and P saturation ratios (P_{Ols}/PSI and P_{AL}/PSI). All correlations are statistically significant at $p = 0.001$ level.

	P_{CaCl2}	P_W	DPS_{OX}	DPS_{AL}	P_{Ols}/PSI	P_{AL}/PSI
P_{CaCl2}	1					
P_W	0.96	1				
DPS_{OX}	0.83	0.84	1			
DPS_{AL}	0.85	0.88	0.84	1		
P_{Ols}/PSI	0.89	0.92	0.91	0.93	1	
P_{AL}/PSI	0.90	0.90	0.87	0.98	0.95	1

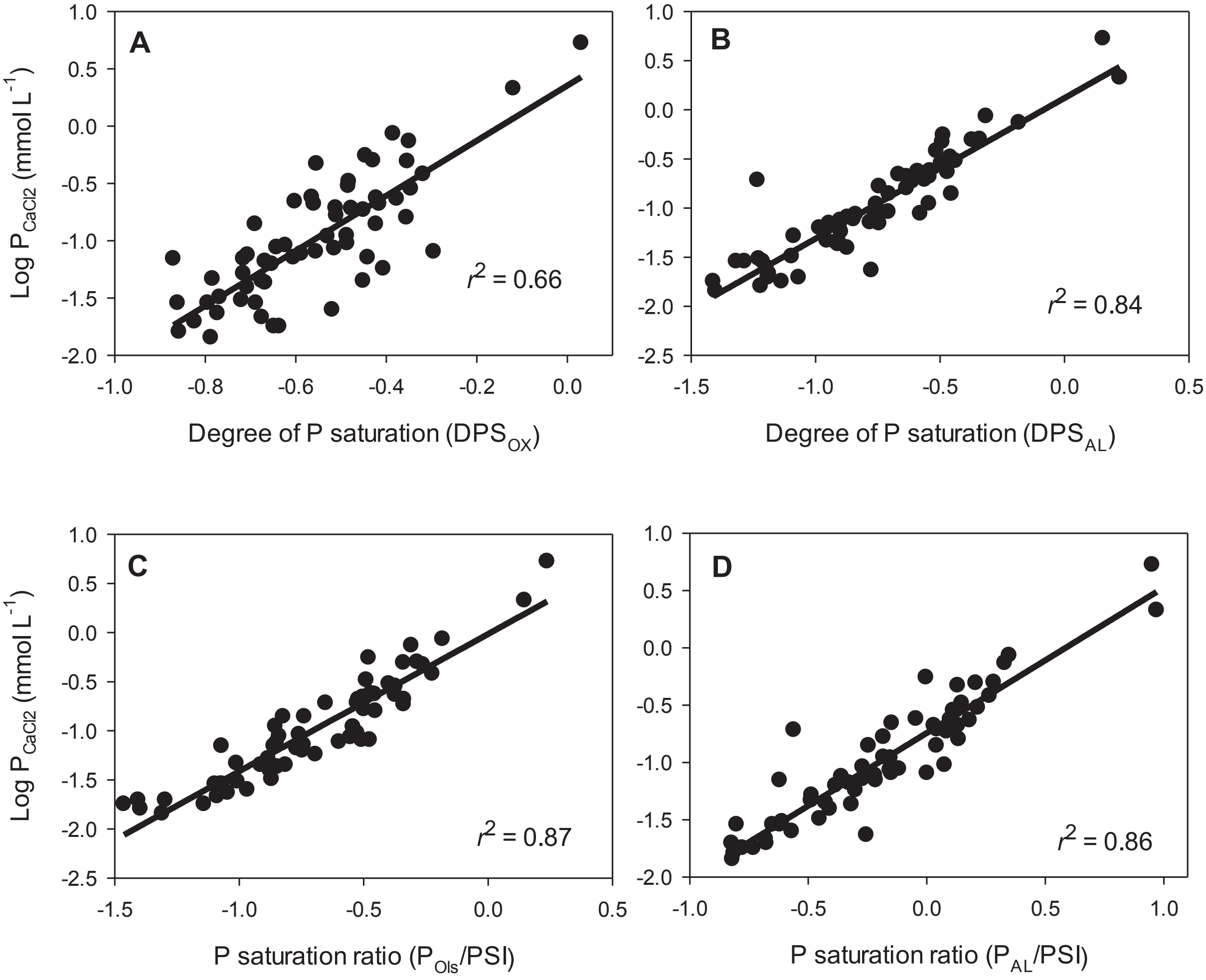

Fig. 4. Relationship between log-transformed $CaCl_2$-extractable (P_{CaCl2}) P concentrations and log-transformed A) DPS_{OX}, B) DPS_{AL}, C) P_{Ols}/PSI, and D) P_{AL}/PSI. Also included are best-fit regression line and r^2 values. Additional fit statistics can be found in Table A4.

transformed P_{CaCl2} and P_W data. The largest improvement for P_{CaCl2} was addition of pH and DPS_{OX} which resulted in r^2 increasing from 0.66 to 0.72, RMSE decreasing from 0.72 to 0.65, and AIC decreasing from −37 to −48. The second largest improvement in predicting P_{CaCl2} was the addition of pH to P_{Ols}/PSI which resulted in r^2 increasing from 0.87 to 0.91, RMSE decreasing from 0.45 to 0.36, and AIC decreasing from −95 to −117. For P_W, the addition of clay as a second explanatory variable with DPS_{OX} resulted in slight improvements in model fits with r^2 increasing from 0.44 to 0.50, RMSE decreasing from 0.82 to 0.78, and AIC decreasing from −20 to −25. When clay and P_{Ols}/PSI were used as explanatory variables, r^2 increased from 0.66 to 0.70, RMSE decreased from 0.64 to 0.60, and AIC decreased from −52 to −58 (Table A5).

4. Discussion

4.1. Soil P contents

The ability of the different P extraction methods to release P followed the expected order with HCl as the strongest extractant and $CaCl_2$ as the weakest. P extracted by HCl is regarded as the storage content of the soil and has been used to approximate the total inorganic P concentration in the soil whereas the OX extraction was developed to dissolve phosphorus adsorbed to less ordered crystalline structure forms of Al- and Fe- (hydro)oxides and hence only represent a fraction of the total P. The high correlation and the narrow ratio between the concentrations of P_{HCl} and P_{OX} suggests the two extraction methods to a large extent target the same chemical P pools in our soils. This is supported by findings from other studies reporting OX extraction to also dissolve Ca-phosphate, such as apatite and secondary, more amorphous forms of Ca-phosphate, and hence overestimate the sorbed P in young, less weathered glacial and postglacial soils such as those found in the Nordic countries (Uusitalo and Tuhkanen, 2000; Peltovuori et al., 2002: Börling et al., 2004; Schmieder et al., 2018). While the AL solution can also extract Ca-bound phosphate, it does so to a lesser extent than the OX solution due to the higher pH (3.75 compared with 3.0) (Börling et al., 2004).

The strong correlation and the ratio between P_{AL} and P_{Ols} were consistent with previous studies (e.g. Eriksson et al., 2013; Otabbong et al., 2009; Andersson et al., 2013). There was only a moderate correlation between pH and the P_{AL}:P_{Ols} ratio (r = 0.68). Based on this, it was not obvious from our data that the AL extract calcium phosphates to any large extent in our tested soils.

Both P_W and P_{CaCl2} were better correlated with P_{AL} than with $P_{Ols,}$ even though the AL extract is much more aggressive than the Olsen solution. Deionized water extracted on average 8.5 times more DRP than the $CaCl_2$ solution. This is consistent with findings from other studies showing greater P concentrations in water extracts compared with $CaCl_2$ extracts (Eriksson et al., 2013; McDowell and Sharpley, 2001; Maguire and Sims, 2002b; Nair et al., 2004; Sanchez-Alcala et al., 2014). Greater P concentrations in deionized or distilled water extracts are attributed to lower ionic strength and the effect of different soil solution composition on P solubility (Barrow and Shaw, 1979; Evans and Sorensen, 1986). There is also a greater likelihood for dispersion of

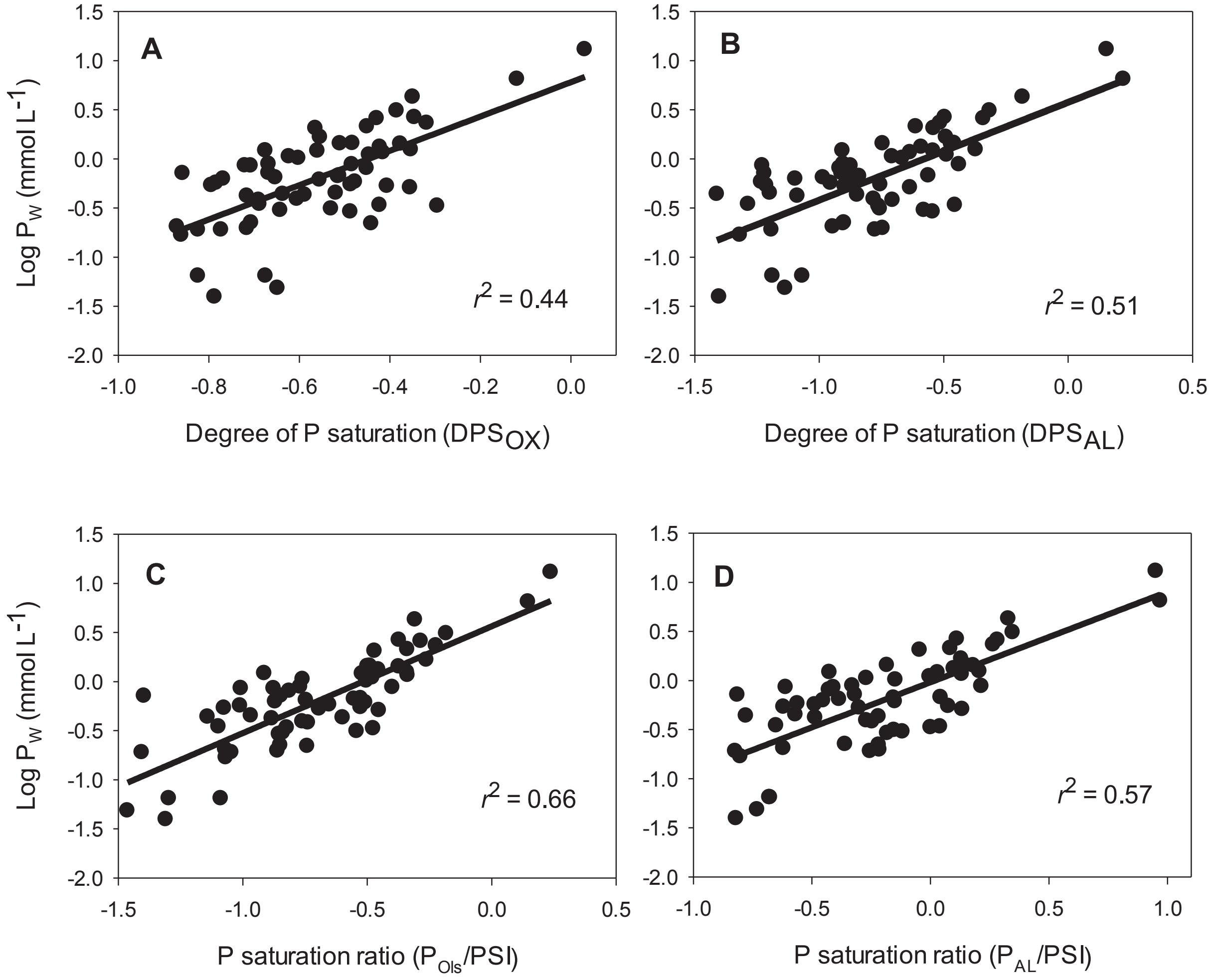

Fig. 5. Relationship between log-transformed water-extractable (P_W) P concentrations and log-transformed A) DPS_{OX}, B) DPS_{AL}, C) P_{Ols}/PSI, and D) P_{AL}/PSI. Also included are best-fit regression line and r^2 values. Additional fit statistics can be found in Table A5.

clay colloids when using an extract with low ionic strength (Sinaj et al., 1998; Koopmans et al., 2005) and thus P bound to colloids less than 0.45 μm in diameter may contribute to P_w. This may explain why the correlations of P sorption indices with P_w are not as strong as with P_{CaCl2}.

4.2. Soil sorption properties

The values of $[Al + Fe]_{OX}$ for our soils were notably higher than values reported in other studies. For instance, Lookman et al (1995) reported a range of 39 to 118 mmol kg^{-1} in the upper 30 cm for 300 Belgium soils whereas Nair et al. (2004) reported a range of 15 to 40 mmol kg^{-1} in sandy soils from Florida. Koopmans et al. (2006) reported a range between 14 and 133 mmol kg^{-1} in different mineral soil types in the Netherlands. Our values are, however, in the same range as values reported for soils in the some of the Nordic countries. Rubæk et al. (2013) reported values for 229 soils in Denmark between 17 and 272 mmol kg^{-1} and a mean of 81 mmol kg^{-1}. Börling et al. (2001) reported values between 70 and 203 mmol kg^{-1} for 10 Swedish arable soils. Uusitalo and Tuhkanen (2000) reported even higher values for 10 Finnish arable soils ranging between 142 and 408 mmol kg^{-1}. The reason for the higher concentrations reported by Börling et al. (2001) and Uusitalo and Tuhkanen (2000) were explained by the fact that the soils developed after the latest glacial period and thus are relatively young compared to unglaciated soils in Europe and North America. In the younger soils, Al and Fe oxides have less ordered crystalline structure forms and are thus to a higher degree extracted by ammonium oxalate (Uusitalo and Tuhkanen, 2000).

Our values for the sum of Al_{AL} and Fe_{AL} were within the range found in the limited amount of literature reporting on AL-extractable Al and Fe. Ulen (2006) reported values between 2.6 and 20 mmol kg^{-1}, while Eriksson et al. (2013) reported values between 10 and 17 mmol kg^{-1}. In a Swedish national inventory on 12,600 sites made by the Swedish Board of Agriculture in 2011–2012 (Paulsson et al., 2015), the sum of Al_{AL} and Fe_{AL} varied between 3.9 and 274 mmol kg^{-1} with a median of 24.2 (average 29.1). Ulen (2006) reported higher correlations between the OX and the AL extraction methods for Al and Fe compared with our study (r = 0.947 and 0.891 for Al and Fe, respectively). Although the study by Ulen (2006) was based on 40 soil samples, they only represented 10 different sites with samples from four different experimental P-fertilizer treatments at each site. The higher correlation in that study may thus be a result of less variation in soil characteristics compared to our study based on 62 soil samples from 45 different sites.

Since the OX solution extracted proportionally more Fe than the AL solution, Fe was a more important component of the sorption capacity determined from OX than from AL. This is consistent with the findings of Eriksson et al. (2013). The difference between the two extraction methods is mainly explained by the stronger complexation of Fe-hydroxides than of Al-hydroxides by the oxalate at pH 3 compared to the lactate at pH 3.75.

The relatively strong correlation between our fitted S_{max} values and $[Al + Fe]_{OX}$ suggests that for the soils tested, $[Al + Fe]_{OX}$ is a good approximation for the P sorption capacity of the soil, at least for quickly sorbed P (24 hrs). For the ratio of S_{max} to $[Al + Fe]_{OX}$ referred to as β (van der Zee et al., 1987), we observed values between 0.17 and 0.24. These values are consistent with findings by Khiari et al. (2000) who observed average value of 0.22 and to van der Zee et al. (1987) who estimated a value of 0.22 based on amount of P desorbed from soils and an assumed value of α of 0.6. Conversely, the correlation between S_{max} and $[Al + Fe]_{AL}$ was not statistically significant. Given the lower values of $[Al + Fe]_{AL}$ compared with $[Al + Fe]_{OX}$, the ratio of S_{max} to $[Al + Fe]_{AL}$ was notably greater, ranging from 0.70 to 2.2. Values exceeding 1 indicate soils in which AL did not extract all of the Fe and Al which contributed to P sorption capacity of the soil, which occurred in 5 out of the 11 soils tested for S_{max}. The development of DPS was based on the assumption that P sorption is primarily controlled by two mechanisms: relatively fast and reversible sorption onto the solid phase (primarily Al and Fe oxides) and slow and mostly irreversible diffusion-precipitation processes with amorphous Al and Fe oxides resulting in metal phosphate compounds (van der Zee and van Riemsdijk, 1986; van der Zee et al., 1987). Thus, the total inorganic P content of P saturated soils is the summation of the adsorption maximum and the maximal sorption due to slow reactions. Because studies have shown that sorption of P can continue up to 250 days following P application (Lexmond et al., 1982; De Willigen et al., 1982), S_{max} obtained from short-term experiments will not adequately quantify the maximum sorption capacity of the soil. Long-term P sorption can be estimated by extrapolating results from short-term sorption experiments (van Riemsdijk and van der Linden, 1984). Van der Zee et al. (1987) found sorption to increase log-linearly with time and recommended multiplying the short-term P sorption capacity by 1.8 to estimate the maximum P sorption of the soil. Assuming our fitted S_{max} values are good approximations of the maximum short-term reversible P sorption capacity, we can estimate the maximal sorption factor, α, by (Pautler and Sims, 2000);

$$\alpha = \frac{(P_{ox} + 1.8S_{max})}{(Fe + Al)_{ox}} \tag{6}$$

Using this equation yielded α values ranging from 0.37 to 0.95 with a mean of 0.54. With the exception of our highest value of 0.95 (our second highest value was 0.64), these values fall well within the range reported by others (van der Zee and van Riemsdijk, 1986, 1988; van der Zee et al., 1987; Pautler and Sims, 2000). We compared the relationship between P_{CaCl2} or P_W with DPS for the 11 soils using either α_m estimated for each of the 11 soils individually or assumed that all 11 soils had a value of 0.5. We did not see a notable difference in the correlations for the two values of α_m thus supporting our use of 0.5 for the entire soil data set.

Our PSI values are similar to other studies using the same initial solution concentration where reported values of PSI ranged from 2.5 to 4.6 (Börling et al., 2001; Eriksson et al, 2013). Consistent with earlier findings (Bache and Williams, 1971; Bolster et al., 2020; Börling et al., 2001), PSI was strongly correlated with S_{max} (Fig. 3). PSI was initially developed as a simple tool to estimate the buffer capacity of the soil but has been used extensively to estimate P sorption capacity. Our results further confirm the utility of using PSI to rank the relative P sorption capacity among soils.

4.3. Degree of P saturation and correlation to easily dissolved P

Given the similar correlation between easily dissolved P (P_{CaCl2} and P_W) and the various DPS indices, our results suggest that no single measure of DPS is superior to the others and that there are multiple DPS metrics for evaluating a soil's potential for releasing dissolved P into solution. In Sweden, it is a great advantage of using AL extraction to calculate DPS_{AL} since it is already used as the standard agronomic soil P test. Thus, there is an established method at commercial labs, the analysis is inexpensive, and data can be used for both the purpose of evaluating soil fertility and the risk of P leaching. Traditionally, Al and Fe have not been included in routine soil analysis but can easily be included at a relatively low cost. A disadvantage with DPS_{AL} would be that estimation of the sorption capacity given by the sum of Al_{AL} and Fe_{AL} is not supported by the comparisons with S_{max} and the sum of Al_{OX} and Fe_{OX}, assuming they are good predictors of the P sorption capacity of the soils. Since PSI was very well correlated with both S_{max} ($r = 0.91$) and the sum of Al_{OX} and Fe_{OX} ($r = 0,67$), our results indicate that PSI maybe could be a more suitable predictor for the P sorption capacity. However, P saturation values > 1 when using PSI as a measure for the P sorption capacity and in combination with P_{AL} and P_{Ols} show that PSI underestimates maximum sorption capacity (Bolster et al. 2020). PSI is not currently included in the standard procedures at many commercial labs, but it is a simple procedure and would be relatively easy to include in a standard soil test package.

While the sum Al_{OX} and Fe_{OX} was the most correlated of our estimates of P sorption capacity with Langmuir S_{max}, the OX extraction likely overestimates the amount of sorbed P because calcium phosphates are dissolved (Börling at al. 2001). The AL extract is also supposed to dissolve calcium phosphates, but to a lesser extent than ammonium oxalate. In Sweden, the Olsen P extraction is sometimes included to complement the AL extraction on soils with high pH to avoid overestimation of plant available P due to dissolution of calcium phosphates. The narrow ratio between P_{AL} and P_{Ols} and the low correlation to soil pH values did not indicate a great dissolution of calcium phosphate.

While the sum of Al_{AL} and Fe_{AL} was a weak predictor of the sorption capacity, DPS_{AL} was a strong predictor for both P_{CaCl2} and P_W, suggesting DPS_{AL} may be a promising and more or less 'ready to use' empirical tool to predict soils with high concentrations of easily dissolved P (P_w and P_{CaCl2}). The best predictors for P_w and P_{CaCl2} were the P saturation indices using PSI as a measure of the sorption capacity, and hence suitable to be used in combination with P_{AL} as well as with P_{Ols} to calculate P saturation. Including other soil parameters such as clay content, pH or total carbon into the in the calculations had no or only minor effect on the predictions of P_w and P_{CaCl2}.

Many studies have shown that the relationship between DPS and dissolved P in soil solution or in leachate and runoff is best described using a two-segment line where the concentration of dissolved P increases slightly or remains relatively constant below a certain DPS value (e.g. Sims et al., 1998; Maguire and Sims, 2002a; Kleinman et al., 2000; Nair et al., 2004; Wang et al., 2012). Above this threshold, often referred to as the "change point", the concentrations of dissolved P increase rapidly with increasing DPS due to nonlinear desorption of P from soils (Koopmans et al., 2002). Estimates of the DPS change point normally correspond to values between ~0.1 and 0.5 depending on soil mineralogy and extraction method (Nair, 2014). In contrast to these studies, we did not find a distinct change point which is consistent with the findings of Ulen (2006), who did not identify any change point when comparing DPS_{AL} and DRP concentration in drainage water from tile-drained fields. One possible explanation for the absence of a change point in our study could be the lack of soils with high degree of P saturation among the soils we tested. With the exception of two extreme loamy sand soils (no 2 and 6), DPS_{OX} was less than 0.5, whereas DPS_{AL} was less than 0.5 in all soils except three. Further studies on soils with a wider range of P saturation values, and especially soils with low P sorption capacity in combination with high soil P contents, needs to be conducted to identify such threshold values. Results also need to be confirmed on a larger number of soils before being recommended to be used in the extension service.

5. Conclusions

Our results suggest that multiple DPS metrics provide similar predictions for evaluating a soil's potential for releasing dissolved P into solution. Taking into account the availability of data, DPS_{AL} established from standard soil tests of Swedish agricultural soils appears to be a promising and useful empirical tool to predict soils with high concentrations of easily dissolved P. However, PSI as an easily obtained measure for P sorption capacity, is better correlated to the Langmuir S_{max} and the sum of Al and Fe extracted by ammonium oxalate. Although only small differences between the different DPS metrics, P saturation calculated from PSI together with P_{AL} or P_{Ols} predicted P_{CaCl2} and P_W best. No distinct change point values where easily dissolved P concentrations start to increase rapidly could be identified for the different DPS metrics.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.geoderma.2020.114708.

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