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Evaluation of degree of phosphorus saturation (DPS) and easily soluble P in top- and subsoil as a predictor for P-leaching

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Abstract

Eutrophication has long been an environmental problem and the effects from fertilizing arable land is a known source. The degree of phosphorus saturation (DPS) and easily soluble phosphorus (P) are different variables used in different countries to try to predict the leaching of P from soils. The purpose of this master thesis was to investigate what variable can be used as a predictor for leached P from soils and to investigate what extent P leaching from the topsoil can be readsorbed in the subsoil. The extraction method used for DPS was the Swedish standard lactate extraction and for easily soluble P were distilled water, CaCl₂-solution and artificial rainwater with recipe from SMHI used. Three different soils in Sweden were used, two arable soils with different chemical properties and one forest soil. The soils were filtered (2 mm) and put into columns since the texture was of interest rather than the structure. CaCl₂-solution was used to saturate the samples and artificial rainwater was used for irrigation. The results showed that easily soluble P is a reasonable indicator for leached P and that the subsoil affects the total leaching of P. However, no significant correlation was found between DPS and leached P, indicating that it may not be a suitable indicator of leaching. On the other hand, DPS can be seen as a reasonable indicator for easily soluble P since the correlation test showed almost significant correlation. The results also showed that the electric conductivity in the leachate correlates to the leached P for the arable soils. For a more reliable result, more soils should be analyzed during more days.

Keywords: Degree of phosphorus saturation, dissolved reactive phosphorus, easily soluble P, column experiment, lactate extracted P

Referat

Övergödning är ett välkänt miljöproblem där den antropogena påverkan från jordbruk och åkermark har en betydande effekt. Fosformättnadsgrad och lättlösligt fosfor (P) är några variabler som används i olika länder för att försöka förutsäga risken för fosforläckaget från jordar. Huvudsyftet med den här studien var att undersöka om fosformättnadsgrad, bestämd utifrån svensk standardmetod för markkartering (AL-metoden) och analyser av lättlösligt P kan användas som indikatorer för P-läckagekänslighet i matjord respektive alvjord. Dessutom undersöktes alvens inverkan på läckaget från en profil med både matjord och alv. Experimentet genomfördes som ett kolonnförsök där utlakningen av fosfat från tre olika jordar med stora skillnader i innehåll av fosfor, aluminium och järn. Olika extraktionsmetoder användes, den svenska standarden för agronomisk markkartering som innebär extraktion med en ammoniumlaktatlösning (AL) användes för bestämning av förråd av relativt lättlösligt P (P-AL: normalt benämnt växttillgängligt P) och aluminium och järn. Förrådet av aluminium- och järn(hydr)oxider anses ha störst betydelse för hur P binds i ej kalkrika jordar. Lättlösligt P analyserades genom extraktion med destillerat vatten, CaCl₂-lösning samt artificiellt regnvatten med recept från SMHI. Resultatet visade att lättlösligt P är en rimlig indikator för P-läckage och att alven påverkar den totala utlakade fosformängden. Korrelationstestet visade däremot ingen significant korrelation mellan fosformättnadsgraden och P-läckaget och kan baserat på resultaten i den här studien inte anses vara en rimlig indikator för P-läckage. Korrelationen mellan fosformättnadsgraden och det lättlösliga P var mer tydlig och fosformättnadsgraden bedöms därför kunna och ses som en rimlig indikator för lättlösligt P. Resultatet visade också att den elektroniska konduktiviteten i P-läckaget korrelerar signifikant med den utlakade fosformängden för de två åkerjordarna. Fler jordar och en längre försöksperiod med fler provtagningsdagar behövs för att få ett mer tillförlitligt resultat.

Nyckelord: Fosformättnadsgrad, fosforläckage, lättlösligt fosfor, kolonnexperiment, laktat extraherat fosfor

Preface

This 30 credits master thesis is the last challenge in the Master Programme in Environmental and Water Engineering at Uppsala University and the Swedish University of Agricultural Sciences. The study is part of the European Union financed LIFE IP-project Rich Waters with supervision from both Karin Blombäck at the Department of Soil and Environment at SLU and from the co-supervisor Harald Cederlund at the Department of Molecular Sciences at SLU. Thanks to their genuine support and patience this thesis has reached the finish line and I am truly grateful for having not only one but both of them during the whole project.

I am also grateful for my academic supervisor Lars Bergström at the Department of Soil and Environment at SLU who has always been available for feedback of the entire work or just for a quick question. I would like to give a special thank to Kathrin Hesse who helped me during my six weeks in the laboratory, for teaching me during the first week about the machines and how everything works and also for supporting me the last week when I accidentally flushed one of the machines with water.

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Skam den som ger sig.

Jessie Torpner
Uppsala, August 2018

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Populärvetenskaplig sammanfattning

Övergödning är ett av de mest påtagliga miljöproblem Sverige upplever i nuläget, med bland annat algbloomning i olika vattenmiljöer, inte minst i Östersjön. Algbloomningen kan leda till syrebrist och bottendöd, vilket är ett stort problem i vissa delar av Östersjön. Fiskbestånden kan i sin tur påverkas så pass att vissa arter dör ut. Vissa alger är även giftiga och kan därmed påverka vattenkvaliteten negativt. Anledningen till att miljöproblemet är svårt att åtgärda och därmed har blivit en av de största utmaningarna är att det är svårt och tar lång tid att stoppa tillförseln av näringsämnen kväve och fosfor som driver på övergödningen till vattenmiljöerna. Kväve och fosfor kommer från så kallade diffusa källor som skogsmark och jordbruksmark, och från så kallade punktkällor som reningsverk, industrier och enskilda avlopp. Att Östersjön har drabbats så hårt har en förklaring i att det är många och folkrika länder som gränsar mot och har en avrinning till havet. Ett av Sveriges 16 miljömål är därför *Ingen Övergödning* och man har även åtagit sig att minska utsläppen fram till 2021 i enlighet med havsmiljökonventionen Helcoms aktionsplan för Östersjön.

Syftet med den här studien är att få mer vetskap om hur fosfor rör sig i olika jordar i Sverige och vilka variabler som kan indikera om en jord har goda chanser att binda fosfor eller om det finns risk för att fosfor kan utlakas från jorden. Mer specifikt undersöktes variabeln fosformättnadsgrad, Degree of Phosphorus Saturation (DPS), för att se om det är en bra variabel att använda för att förutsäga huruvida jordarna kommer binda eller släppa ifrån sig fosfor vid till exempel gödsling. Påverkan av alven, den delen av jorden som ligger under matjorden, vid ca 30 cm djup undersöktes särskilt då dess påverkan på det totala fosforläckaget ofta inte är fastställt. Studien är kopplad till EU-projektet LIFE IP Rich Waters.

Våra resultat visade inte någon signifikant korrelation mellan DPS och utlakat fosfor vilket indikerar att DPS inte är en lämplig variabel att använda för att förutsäga risken för utlakning hos jordar i Sverige. Det ska dock noteras att endast ett litet antal jordar användes och att endast sex olika prover ingick i korrelationstestet vilket genererar en låg styrka för resultatet. Alvens betydelse på det totala läckaget av fosfor visade sig däremot ha en avgörande påverkan. För de två undersökta åkerjordarna var det tydligt medan resultatet för skogsjorden var svårtolkat i och med den låga fosforhalten. Resultaten visade även att konduktiviteten i fosforläckaget var signifikant korrelerad med den utlakade fosformängden. Det vill säga beroende på jonstyrkan i markvätskan påverkas den mängd fosfor som lakas ut från jorden. Detta blev mer påtagligt för resultaten eftersom två olika lösningar med olika jonstyrka användes för att fukta jorden.

Studien gjordes i kolonner med bevattning av artificiellt regnvatten. Varje jord var uppdelad i ett matjordprov (0-25 cm djup) och ett alvprov (40-60 cm djup). Alla

analyser och experiment gjordes på tre replikat. Läckagevatten samlades från varje kolonn under två veckor (totalt 5 prov).

Abbreviations

PSC	Phosphorus sorption capacity
DPS	Degree of phosphorus saturation
DRP	Dissolved reactive phosphorus
P-AL	Ammonium lactate-extracted P
P-leach	Leached P from the column experiment
P-CaCl ₂	CaCl ₂ -extracted P from samples that have been shaken for 24 hours
P-H ₂ O	H ₂ O-extracted P from samples that have been shaken for 24 hours
P-AW	P extracted with artificial rainwater, also shaken for 24 hours
E C	Electric conductivity

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1 Background and Aims

One of the sixteen environmental objectives set by the Swedish Environmental Protection Agency is Zero Eutrophication (Naturvårdsverket, 2018). This study, which is hoped to help fulfill that objective, is part of the European-Union financed LIFE IP-project Rich Waters. Understanding how the chemical properties of soil affect the leaching of phosphorus can aid in the development of cheaper risk-analysis methods and ultimately aid in the regulation of anthropogenic input.

1.1 Eutrophication

Eutrophication is the term used for an increase in nutrients in a body of water mainly due to human practices. This kind of anthropogenic input has affected nutrient fluxes to aquatic and terrestrial ecosystems for decades. Increased biomass of phytoplankton, shifts in phytoplankton to bloom-forming species that may be toxic, and reduced water clarity are examples of effects of eutrophication in lakes, rivers, reservoirs and marine waters. Water treatment problems and unpleasant taste and odor are also some examples as is loss of fish species (Smith, 1998). The external loads of nitrogen (N) and phosphorus (P) can be traced to both point- and nonpoint sources through groundwater, fluvial and atmospheric inputs (Smith et al., 1999). Runoff and leachate from waste disposal, wastewater treatment plants, industries and runoff, as well as infiltration from animal feedlots, are examples of point sources which are localized and more easily controlled. Runoff from fields, pastures and rangelands, on the other hand, are examples of nonpoint sources which are diffuse and more difficult to regulate (Novotny and Olem, 1994).

1.2 Phosphorus in Soil

Phosphorus cycling in soils depend on many things such as the inorganic and organic solid phases present, chemistry of the solution and environmental factors (Sims and Sharpley, 2005). For soils that have never been used by humans, the natural storage of P is present in the mineral apatite or as phosphate bound to iron- and aluminium (hydro)oxide compounds but for farmed soils the major source of soil P comes from organic material (Eriksson et al., 2011). The outputs of P from soils are crop removal, erosion, surface runoff and leaching (Sims and Sharpley, 2005). The concentration of dissolved P in soil solution equilibrates with P in bound forms through processes of dissolution-precipitation, sorption-desorption, mineralization-immobilization and oxidation-reduction (Sims and Sharpley, 2005). Dissolved P in soil solution is in the form of orthophosphate (Sims and Sharpley, 2005). Depending on pH the molecule will be differently charged due to different number of hydrogen ions bound to the molecule. At normal pH in soils H_2PO_4^- and $\text{H}_2\text{PO}_4^{2-}$ are the predominant forms (Sims and Sharpley, 2005).

The use of ammonium oxalate (OX) as an extraction method is more commonly employed in Europe for P, Al and Fe among others (Renneson et al., 2015). The standard extraction method in Sweden is ammonium lactate extraction (AL) which was used in this study. The main difference between the methods is that they extract different fractions of P, Al and Fe. Plant available P is P immediately usable by the plant and when extraction by ammonium lactate-solution it is called P-AL to estimate plant available P during a growing season (Eriksson et al., 2011). The amount of P-AL in the soil is mostly affected by fertilization and crop removal. The soil's buffer capacity is on the other hand a function of sorption strength which controls the rate of diffusion and desorption (Sims and Sharpley, 2005). Temperature and increasing ionic strength increase P adsorption and thereby also P-AL (Sims and Sharpley, 2005). P sorption capacity (PSC) is another variable that is determined by the concentration of phosphate in soil solution and the ability of the solid phase to refill phosphate into soil solution (Vymazal, 2007). Aluminum- and iron hydroxides are examples of molecules in the soil that P can fixate to through adsorption (Eriksson et al., 2011). The amount of clay, organic matter and calcium ions in the soil also affect the PSC depending on pH in the soil (Renneson et al., 2015). In this study PSC, is investigated if it can be determined by extraction of aluminum (Al)- and iron (Fe) hydroxides with ammonium lactate (Equation 1).

$$PSC_{AL} = Al_{AL} + Fe_{AL} \quad (1)$$

Degree of P saturation (DPS) describes a soil's capacity to retain additional P and the potential ability of a soil to desorb soil P. P-AL is used to represent the amount of P in soil and PSC_{AL} to represent desorbed P (Equation 2) (Renneson et al., 2015).

$$DPS_{AL} = 100 * \frac{P_{AL}}{Al_{AL} + Fe_{AL}} = 100 * \frac{P_{AL}}{PSC_{AL}} \quad (2)$$

DPS gets an AL-subscript since both P-AL and PSC are extracted with ammonium lactate.

It is becoming more interesting to measure both top- and subsoil when analyzing risk of P leaching from soils (Andersson, 2016). The subsoil has been shown to act both as a source and a sink for leaching of P (Andersson, 2016). Knowledge about chemical properties such as pH, Al- and Fe hydroxides and P-AL from both top- and subsoil are therefore necessary to be able to use PSC and DPS as risk indicators for P leaching from different soils.

1.3 Objectives and Research Questions

The aim of the study was to determine the correlation between DPS and easily soluble P and leaching of P, respectively, in three different Swedish soils. The work was focused on laboratory column studies and chemical analyses of soil P. This thesis investigated the following four research questions:

- Is DPS a reasonable indicator of easily soluble P (P-H₂O, P-CaCl₂ and P-AW) in soils?
- Is DPS a reasonable indicator of leaching losses of P?
- Are measures of easily soluble P reasonable indicators of leaching losses of P?
- Does the subsoil affect the total leaching of P?

2 Materials and Methods

Three soils were used in this study and the topsoil and the subsoil from each soil were analyzed separately and together in the column equipment. CaCl₂ solution was used to mimic soil solution and artificial rainwater to mimic precipitation. The non-parametric method Kendall's tau was used to investigate the correlations.

2.1 The soils

Soils from three different places, two arable soils and one forest soil, were used in the experiment. Both top- and subsoil were collected. The soils were selected to represent a wide range in chemical properties.

The soil from 3M is from the county Skåne, in Sweden. It is one out of thirteen fields that belong to the national monitoring sites to follow up of nutrient leaching from arable fields which SLU is responsible to observe (Linefur et al., 2017). The soil is a loamy sand soil, and the soil is frequently fertilized with manure from beef cattle (Linefur et al., 2017).

The second sandy soil is from Mellby and is from the county Halland, in Sweden (Henriksson et al., 2016). The topsoil is a sandy soil with sparing clay content and the subsoil is also a sandy soil but with almost no clay content (Henriksson et al., 2016). The soil sample used in this study was taken approximately 1-2 meters from an observations field in southern Sweden (field number 14) (Aronsson and Torstensson, 2009).

The third soil, Julmyra, is a forest soil from Heby, 5 km northwest of Uppsala in Sweden. The soil is a sandy moraine (Anttila and Hagström Yamamoto, 2007). Less details are known about this soil since it is neither an observation field nor an experimental site studied for many years, but a new site within the Life IP project.

After sampling, the soils were dried at 30 degrees Celsius and sieved at 2 mm mesh size. Only the Julmyra soil had soil particles that was larger than 2 mm, but only the fraction that was less than 2 mm was used in this study.

2.2 Degree of P saturation

DPS was calculated as described in section 1.2 *Phosphorus in Soil*. Plant available P was determined from ammonium lactate-extraction (P-AL) as well as Al-AL and Fe-AL (Egnér et al., 1960). Three replicates were made for each top- and subsoil from the soils.

2.3 Easily soluble P

Easily soluble P, is in this study P extracted with CaCl_2 (P- CaCl_2), with artificial rain-water (P-AW) and with distilled water (P- H_2O). These samples were created using 6 g of air dried soil added with 18 ml solution and thereafter shaken for 24 hours.

The method of Murphy and Riley (1962) was used to prepare the leachate for the measurement with the spectrofotometer. The method is based on the fact that potassium antimonyl tartrate solution creates a blue color when added to a phosphate solution (Murphy and Riley, 1962), which can be measured in the spectrophotometer. This method determines the molybdate reactive dissolved ortho-phosphate (DRP). Sulfuric acid, ammonium molybdate, ascorbic acid and potassium antimonyl tartrate are the components in the reagent (Murphy and Riley, 1962).

2.4 Top- and subsoil

To minimize the influence of soil structure as a variable, the study was conducted using repacked soil columns. Two other studies have used exactly this column equipment (Cederlund et al., 2017; Riddle et al., 2018).

12 glass columns with a length of 37 cm and a diameter of 4 cm were used for the experiment. Each column was filled with 125 g air dried soil. One glass sinter plate was placed in the bottom and one on the top of the soil column to avoid erosion from the bottom and damage to the soil surface from the irrigation droplets. A peristaltic pump, IPC 12 was connected to the columns to irrigate with a constant rate. The height of the soil columns

differed since they have different particle sizes and therefore got different volumes. The columns were placed randomly for every replicate run since the velocity from the pump differed between the tubes.

Riddle et al. (2018b) has also made another study where it was concluded that using the same irrigation solution for the topsoil and the subsoil is not representative. The leaching solution from the topsoil should be used as the irrigation solution for the subsoil, to mimic reality. Top- and subsoil were therefore tested both separately, and in combination in this study. The combination was created by having one column filled with the topsoil connected to another column filled with the subsoil and the drainage from the topsoil was thereby used as irrigation for the subsoil (Figure 1). The columns that only contained the topsoil are called 1-samples, the columns that contained only the subsoil are called 2-samples and for the columns that contained both the top- and the subsoil are called 12-samples. Three replicates were used for each sample at different times so in total there were 27 samples used during the experiment.

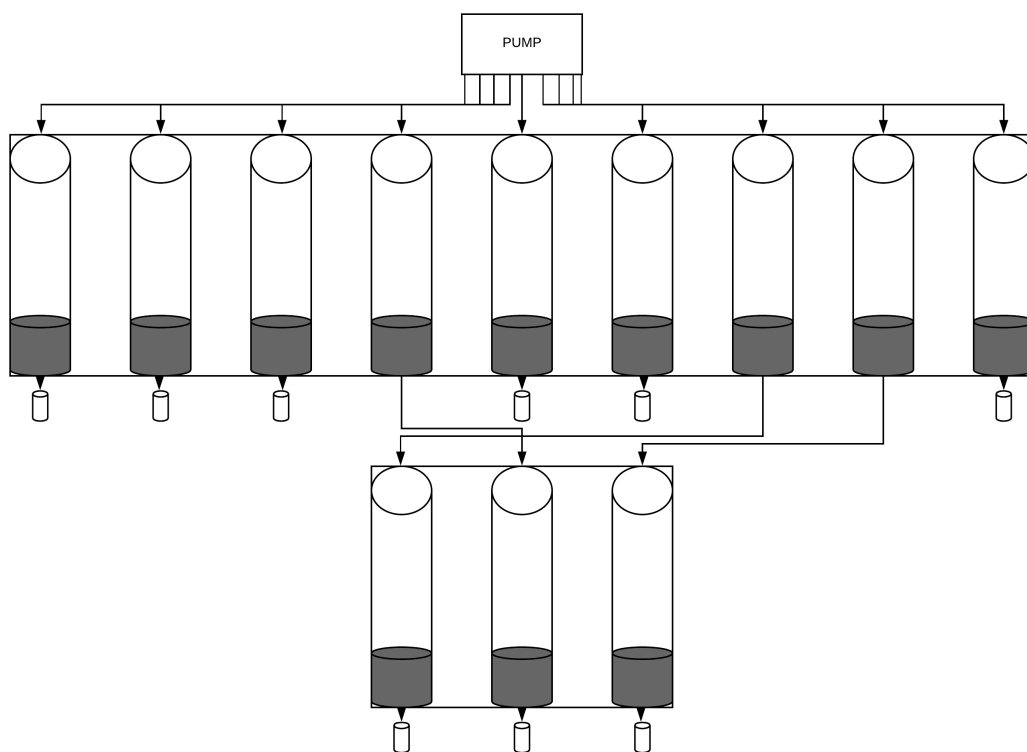


Figure 1: An example of the column experiment that illustrates the 1-, 2- and 12-samples placed randomly.

The columns were first saturated with 0.1 M CaCl_2 solution for one hour, to mimic the soil solution, and thereafter freely drained during the night to reach field capacity before starting the leaching experiment. The CaCl_2 solution was added to the columns with

pipette and the amount solution that had drained during the night was weighed. To estimate the pore volume at drainage equilibrium the amount of drained CaCl_2 solution was subtracted from the added CaCl_2 solution (Table 1).

Table 1: *The estimated pore volume for 1- and 2-samples.*

Sample	Pore volume [ml]
3M1	47.90
3M2	44.74
Mellby1	46.0
Mellby2	35.38
Julmyra1	50.87
Julmyra2	36.37

Rain simulation with a precipitation rate natural for Swedish conditions was used to mimic the reality. A total amount of 20 ml was added to the columns each sample-day at a precipitation rate of 3.98 mm/h which is classified as moderate rain in Sweden (SMHI, 2015). Artificial rainwater blended according to a recipe from the Swedish Meteorological and Hydrological Institute (SMHI) was used for the experimental irrigation to mimic natural precipitation (Table 2) (SMHI, 2016).

Table 2: *Recipe from SMHI for the artificial rainwater (SMHI, 2016).*

Compound	mM
NaCl	0.01
$(\text{NH}_4)_2\text{SO}_4$	0.0053
NaNO_3	0.0059
CaCl_2	0.0039
HCl	0.0115

Leachate was sampled from each column daily for four days to test how P concentration in leachate changed over time when exposed to frequent rain events. A fifth sample was made after the columns were left for three days to test how the soil solution for the different soils would buffer. Five days of sampling were therefore made for each replicate and thereby 15 days in total.

The leached P (P-leach) from the columns was analyzed according to the Murphy and Riley method (Murphy and Riley, 1962) using a spectrofotometer (UV-1201V; SHIMADZU) in the same way as easily soluble P as described above. The EC was measured with a digital conductivity meter (PW 9527; PHILIPS).

2.5 Statistical analyses

To investigate correlations from the data the method Kendall's tau was used (Helsel and Hirsch, 2002). Simple linear regression was used to illustrate the relationship between the P-leach and the EC in the leachate. Rstudio was used to do the calculations.

2.5.1 Kendall's Tau

Kendall's tau test is a rank-based method that measures if two variables varies in the same direction but not at the same rate, so called monotonic relationship. It is a non-parametric method, resistant to outliers and is suited to analyze small amounts of samples just as much as large amounts (Helsel and Hirsch, 2002). This method was used because of the few samples and that the data was not normally distributed. The value of tau is generally smaller than the usual R-value from linear regression, a R-value of 0.9 corresponds to a tau-value of 0.7. This is only because of the methods are different not that linear regression is more sensitive (Helsel and Hirsch, 2002).

2.5.2 Linear regression

Linear regression or Ordinary Least Squares (OLS) describes the covariation between two or more variables. Response variable is the variable of interest and the explanatory variable is the other variable. To use linear regression, five assumptions must be fulfilled to test the hypotheses (Helsel and Hirsch, 2002):

1. y is linearly related to x
2. data used to fit the model are representative data of interest
3. variance of the residuals is constant
4. the residuals are independent
5. the residuals are normally distributed

A regression line should always be seen as a sample estimate of the true linear relationship. It is important to analyze possible patterns of the residuals to determine if the result from the linear regression is accurate (Helsel and Hirsch, 2002). Because of these assumptions and the limited amount of data in this study linear regression was only used when analyzing 15 samples and not when analyzing only 6.

3 Results

The study showed that there is a possible correlation between DPS and easily soluble P but not between DPS and P-leach. Significant correlation was on the other hand shown between easily soluble P and P-leach. The study did also show that the subsoils have an

impact on the P-leach for the arable soils. Chemical properties from the soils are shown in Table 3.

Table 3: *Al, Fe and P measurements extracted with ammonium lactate, the average value from three replicates for P-CaCl₂, P-H₂O and P-AW with the unit mg/100 g air dry soil. All negative values were transformed to zero.*

Sample	Al-AL	Fe-AL	P-AL	P-CaCl ₂	P-H ₂ O	P-AW	pH-CaCl ₂
3M1	20	22	47	0.72	1.95	1.98	6.5
3M2	5.4	32	21	0	0.051	0.0264	6.8
Mellby1	42	20	18	0	0	0.00003	5.4
Mellby2	11	19	<2.0	0.0054	0	0	5.4
Julmyra1	200	80	<2.0	0.048	0.318	0.291	4.1
Julmyra2	99	<10	<2.0	0	0.03	0.0189	4.6

There is a clear difference between the forest soil Julmyra and the two agricultural soils 3M and Mellby. The agricultural soils have much higher P-AL contents, whereas the Al-AL and Fe-AL contents of Julmyra1 is several times higher than for 3M1 and Mellby1. None of the soils was calcareous since all pH values were below 7. Julmyra had the lowest pH as could be expected from the very coarse texture. Differences in easily soluble P between the soils were not as clear, and especially for some of the agricultural soils the measured data were surprisingly low. Easily soluble P was on the other hand surprisingly high for the Julmyra topsoil in comparison with the very low P-AL values.

The contents of Al, Fe and the different forms of extracted P were in general higher for the topsoils compared to the subsoils. The only exception are a higher content of Fe-AL in the subsoil of 3M and of P-CaCl₂ in the subsoil from Mellby compared to the respective topsoil.

3.1 Degree of P saturation and easily soluble P

To answer the first two research questions, the average cumulative P-leach from the 1- and 2-samples were compared with the different forms of easily soluble P from Table 3 and the DPS values calculated from equation 2 (Table 4). The average cumulative P-leach from the 1- and 2-samples, that is only 6 samples that were used when applying Kendall's tau (Table 4).

Table 4: *DPS and average cumulative P-leach for the 1- and 2-samples.*

Sample	DPS [%]	P-leach [ppm]
3M1	133	24
3M2	88	0.79
Mellby 1	30	2.6
Mellby 2	4	0.037
Julmyra 1	0.37	0.054
Julmyra 2	0.86	0.005

The results show that the agricultural soils, 3M and Mellby, have higher DPS in their respective topsoil than the forest soil, Julmyra, with its low DPS in the topsoil (Table 4). This corresponds to the reality since the agricultural soils have been fertilized and the forest soil has not. The difference between 3M2 and Mellby2 from Table 4, which can also be seen studying their box plots (Figure 6 and 7), also correspond to the reality since they have subsoils with different chemical properties. Kendall's tau was used to investigate possible correlations between DPS, the average cumulative P-leach and the easily soluble P (Table 5). And the negative values were not transformed to zero when applying Kendall's tau but all the values that were less than a value, <10 for example, got a value half of that value, so the value 5 in this case. This was made so that all information from the column test could be shown, which can be seen as the distribution in the boxplots.

Table 5: *Correlation test with Kendall's tau between DPS and the average cumulative P-leach, plant available P and the easily soluble P, respectively.*

DPS	P-leach	P-AL	P-H2O	P-CaCl2	P-AW
Kendall's tau	0.60	0.89	0.73	0.73	0.73
p-value	0.14	0.016	0.056	0.056	0.056

The results showed that there were no significant correlations between easily soluble P and DPS but the p-value were on the hand close to the significant level and had a high tau-value (Table 5) so the relationship between DPS and easily soluble P are illustrated in Figure 2.

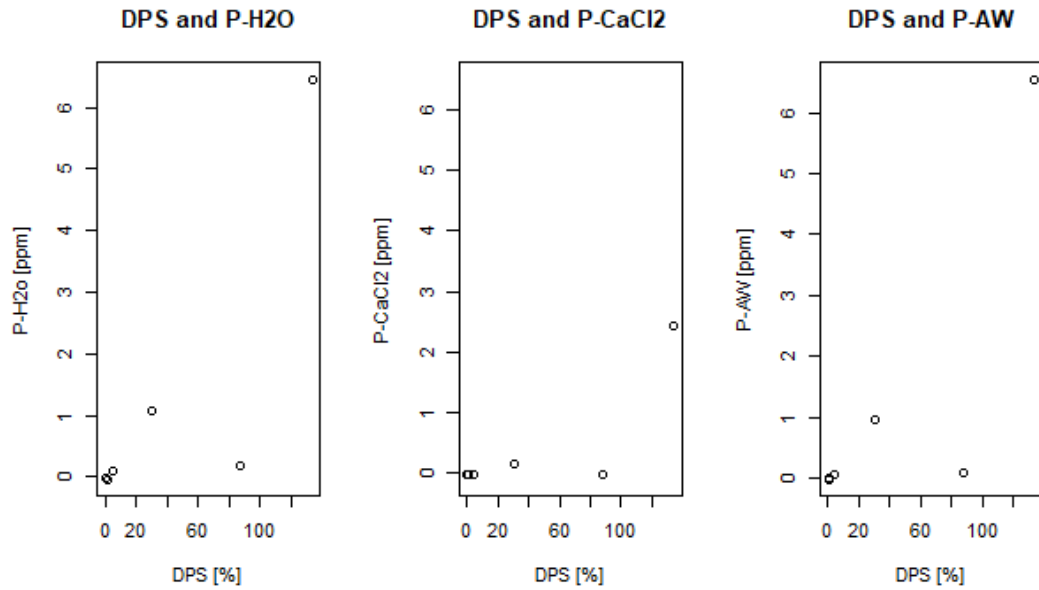


Figure 2: Relationship between DPS and P-H₂O, P-CaCl₂ and P-AW, respectively.

Even though the p-value was 0.056 for the correlation test between DPS and easily soluble P and therefore not having a significant correlation it can still be a correlation between them. This can be seen in Figure 2 since the values seem to follow a pattern, almost a linear relationship, with the exception of the DPS value close to 85 % in the Figure.

The results also showed that there was no significant correlation between DPS and the P-leach with its high p-value of 0.14 but it is interesting to compare it to a significant correlation. Figure 3 shows two plots where the first one is the relationship between DPS and P-leach and the second one is the significant correlation between DPS and P-AL. The linear relationship for the second plot does not show as clear in the first one which correspond to Kendall's tau test. But it can also be seen that it might be a correlation for the first plot, the relation between DPS and P-leach, if more values would have been in the test.

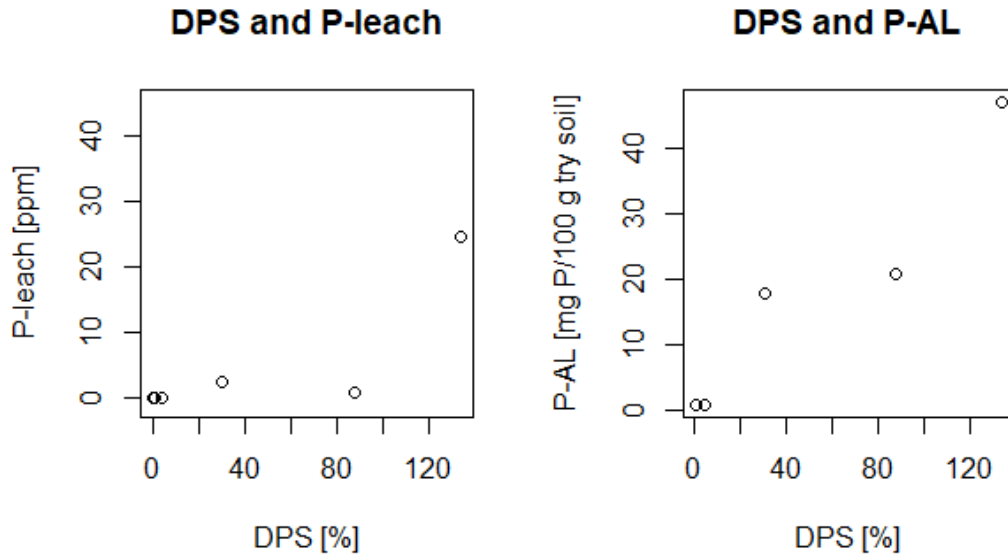


Figure 3: *Relationship between DPS and P-leach and the significant correlation between DPS and P-AL.*

To test if easily soluble P could work as a reasonable indicator for the risk for leaching of P a correlation test between leaching losses from each column sample, given as the mean of the three replicates, and all the different determined forms of easily soluble P was made. All forms of easily soluble P showed significant correlations with the P-leach (Table 6) which is illustrated in Figure 4.

Table 6: *Significant correlations between total P-leach and P-AW and P-CaCl₂, respectively.*

P-leach	P-H ₂ O	P-CaCl ₂	P-AW
Kendall's tau	0.87	0.87	0.87
p-value	0.017	0.017	0.017

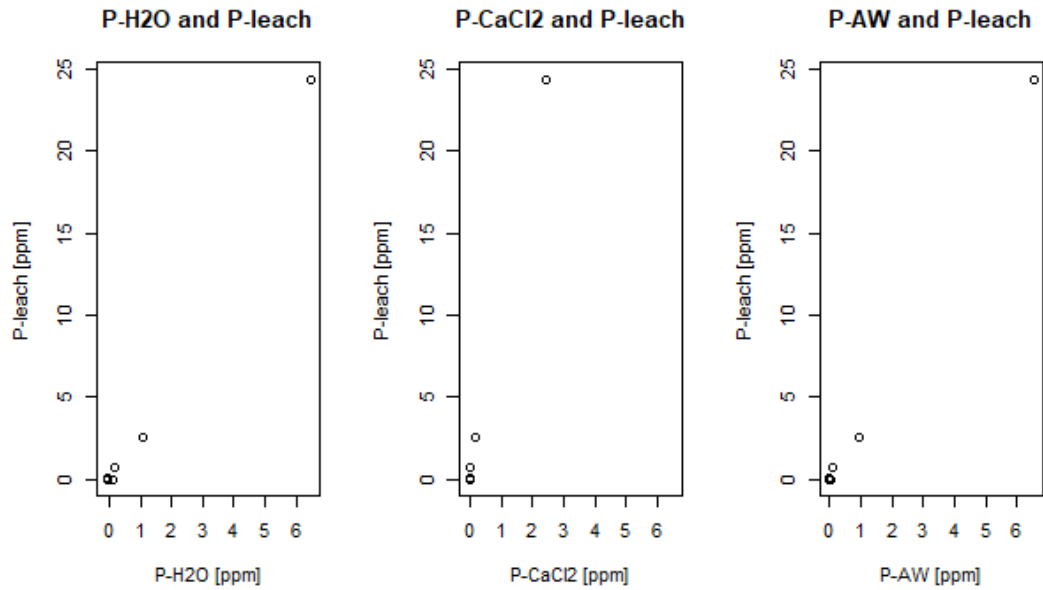


Figure 4: To illustrate the significant correlations between *P-leach* and *P-AW*, *P-CaCl2* and *P-H2O* respectively.

The differences between *P-H2O*, *P-CaCl2* and *P-AW* are small but can be seen in their plots in Figure 4 where their different values are shown. It can also be seen that there is a linear relationship between *P-leach* and easily soluble P since the values in all the plots follow a linear pattern. This linear pattern corresponds to the results from Kendall's tau test that showed significant correlations between *P-leach* and the different forms of easily soluble P.

3.2 Top- and subsoil

To investigate how the subsoil affects the total leaching of P, the average cumulative *P-leach* from the 1-, 2- and 12-samples were compared (Figure 5). The *P-leach* for the individual analyzed days are also illustrated in box plots where the data was not transformed or corrected in any way, hence negative values for some samples from Julmyra.

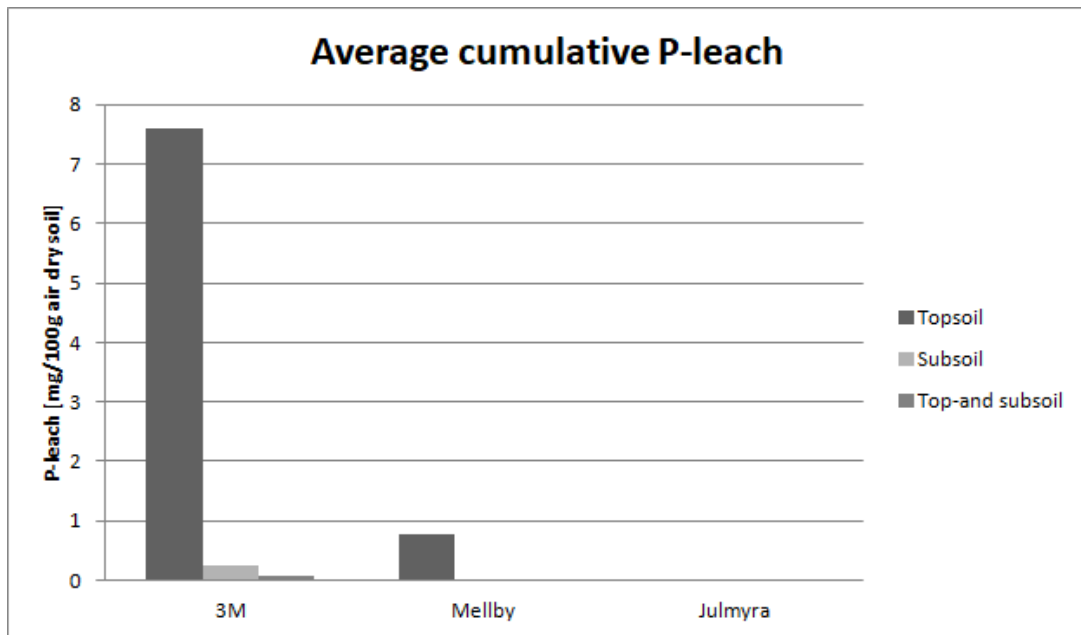


Figure 5: *The average cumulative P-leach from the 1-, 2- and 12-samples from 3M, Mellby and Julmyra during the analyzed days.*

In the agricultural soils, did the topsoils (3M1 and Mellby1) have distinctly higher amount of P-leach than the subsoils (3M2 and Mellby2), which correspond to their higher P-AL contents (Figure 5). The low amount of P-leach from Julmyra also corresponds to the P-AL values. The same outcome are for the DPS values for the soils which corresponds to the result from Kendall's tau test that showed that DPS and P-AL are significantly correlated. The average cumulative P-leach for the 12-samples have the same or a lower amount as the 2-samples for all the soils. This is a clear result that shows that the subsoil affect the total leaching and in this case the subsoil act as a sink. The daily leaching losses for each soil sample are shown in Figure 6 - 8.

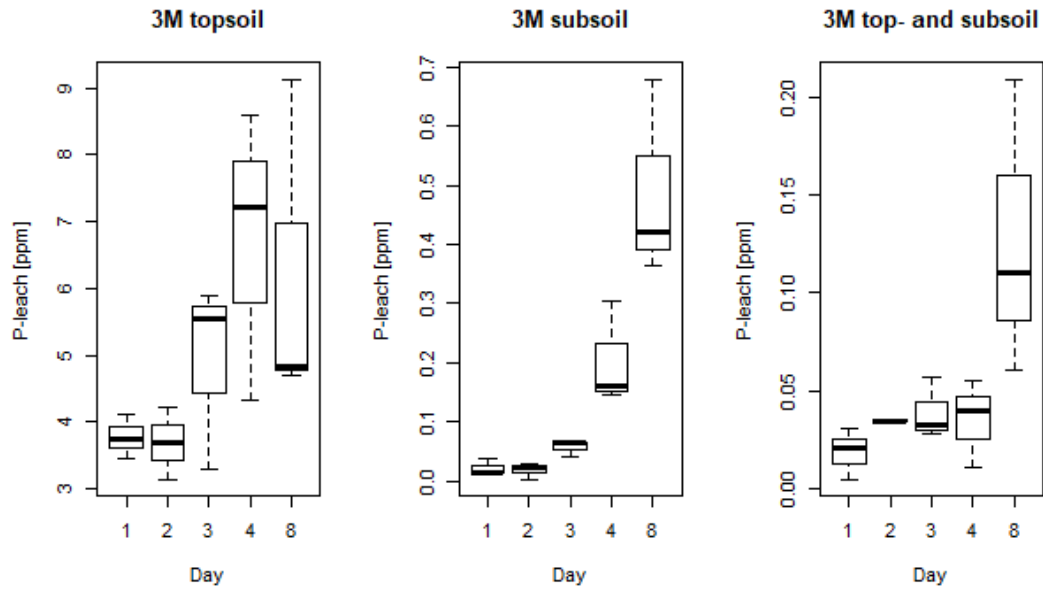


Figure 6: Daily P-leach from the soil 3M with all three replicates with the amount of 20 ml leachate each day. The boxplot shows the median which is the marked line and also the distribution which is the lines outside the boxes.

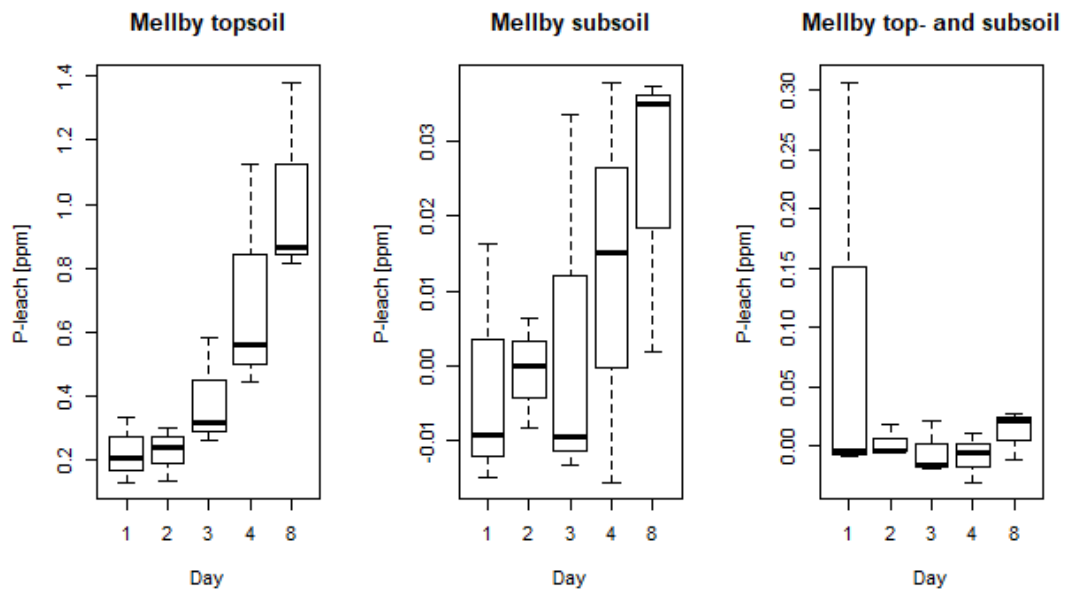


Figure 7: Daily P-leach from the soil Mellby with all three replicates with the amount of 20 ml leachate each day. The boxplot shows the median which is the marked line and also the distribution which is the lines outside the boxes.

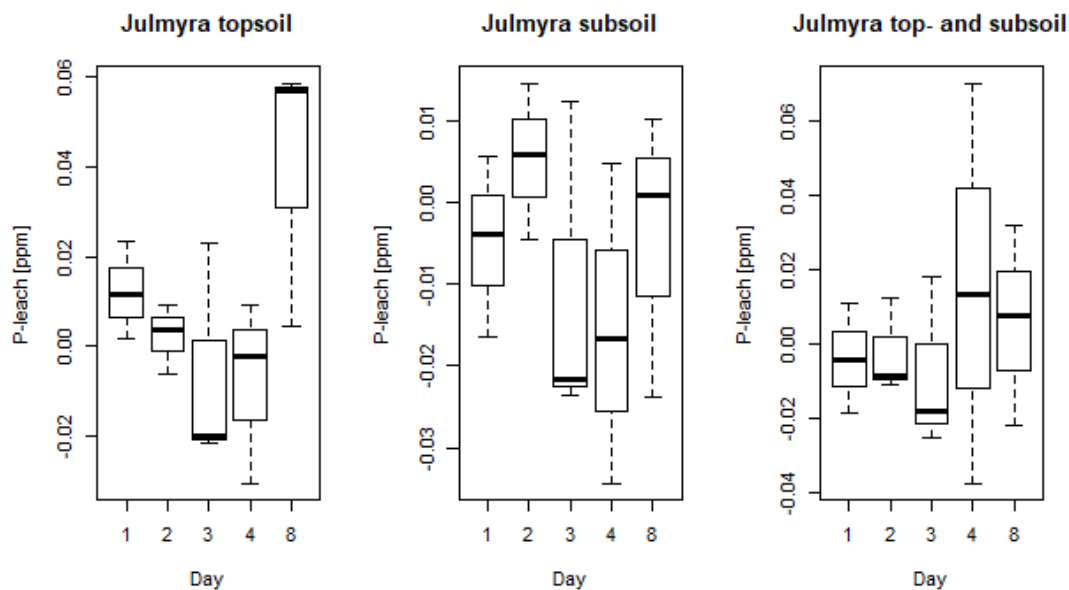


Figure 8: *Daily P-leach from the soil Julmyra with all three replicates with the amount of 20 ml leachate each day. The boxplot shows the median which is the marked line and also the distribution which is the lines outside the boxes.*

The P-leach from 3M1 seems to increase after the second day since the median for day 2 is close to 4 ppm and around 5.5 ppm for day 3. And for day 8, when the samples have been left for three days, the P-leach decreases again to the median close to 5 ppm. For the subsoil, 3M2, increases the P-leach for all the days, from 0.025 ppm for day 1 to 0.4 ppm for day 8. This pattern can also be found for 3M12 when both top- and subsoil are analyzed together. Another observation is that the distribution also increases during the days for all of the soil samples from 3M. For example is the boxplot for 3M2 for day 1 smaller than for the boxplot for day 8 which shows that the replicates from day 1 are closer to the median than the replicates from day 8.

The P-leach from Mellby1 show similar pattern as the P-leach from 3M2 which is that both the P-leach and the distribution increases during the days. For the subsoil, Mellby2, the P-leach increases also during the days but the distribution seems to be almost the same. The P-leach and the distribution from Mellby12 is also almost the same for the analyzed days with the exception for day 1 when the distribution is much higher.

The results from the soil samples from Julmyra do not show similar or as clear pattern as the results from the soil samples from 3M and Mellby. The P-leach for the soil samples are close to zero or have even negative values which reflect on the low amount of P-AL for Julmyra.

Since the P-leach changed during the days and the fact that CaCl_2 solution and artificial rainwater have different EC it is of interest to analyze the average EC for the 1-, 2-, and 12-samples (Figure 9).

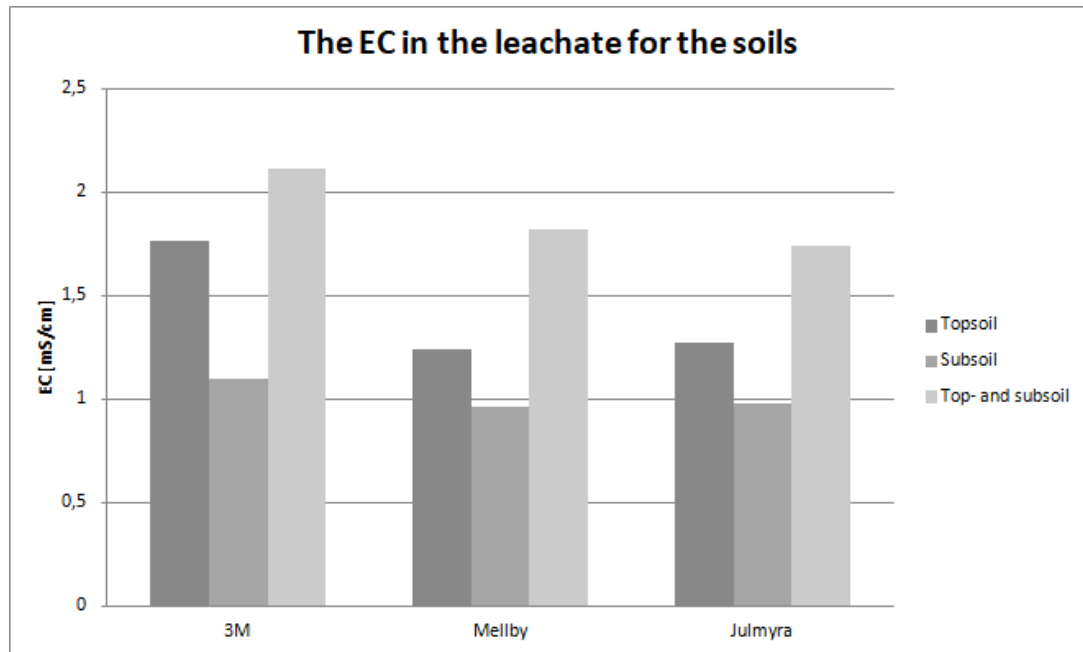


Figure 9: *The average EC for the soils and their 1-,2- and 12-samples.*

The average EC for the different soils follow a similar pattern, lowest for the 2-samples, higher for the 1-samples and highest for the 12-samples (Figure 9). Since the estimated pore volume for each sample was higher than the daily volume irrigated rainwater for each day, the composition of the soil solution became a blend of the initially added CaCl_2 solution and the irrigated artificial rainwater which changed from day to day to become more and more dominated by the rainwater and less influenced by the CaCl_2 solution. Due to this change in the blend of soil solution there was also a shift in the EC, with lower values the more dominated by the rainwater. For the 1- and 2-samples it was after the third day that the leachate consisted of only the artificial rainwater according to the calculations from the pore volume (Table 1). For the 12-samples on the other hand, where the experimental irrigation had to travel through both a topsoil and a subsoil column (Figure 1), it was only during the last day that the leachate consisted mostly of artificial rainwater. So the 12-samples had the double pore volume and the leachate therefore consisted of mostly CaCl_2 solution for the first four days and this explains why the 12-samples have the highest average EC. The EC for the soils for each day is illustrated in box plots (Figure 10 - 12).

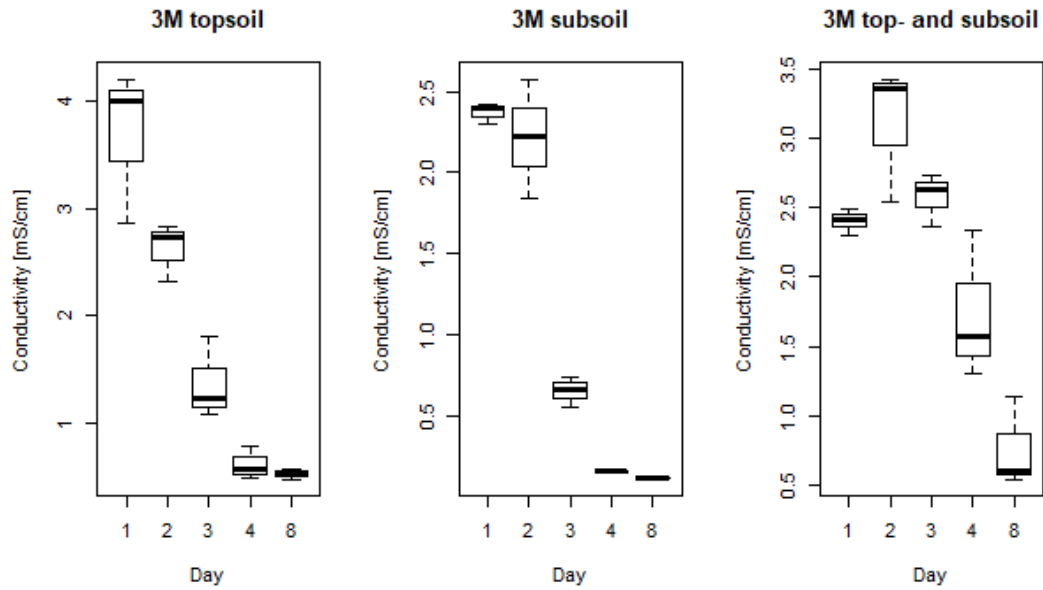


Figure 10: How the electrical conductivity in leachate for 3M changed through the days where the marked line in the boxes is the median and the distribution is the lines outside the boxes.

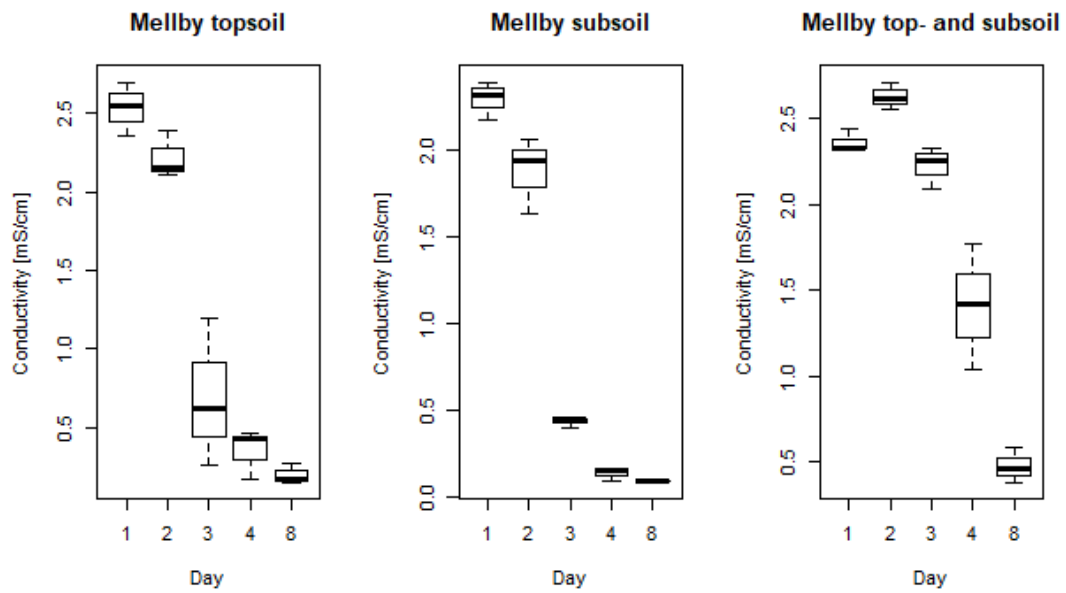


Figure 11: How the electrical conductivity in leachate for Mellby changed through the days where the marked line in the boxes is the median and the distribution is the lines outside the boxes.

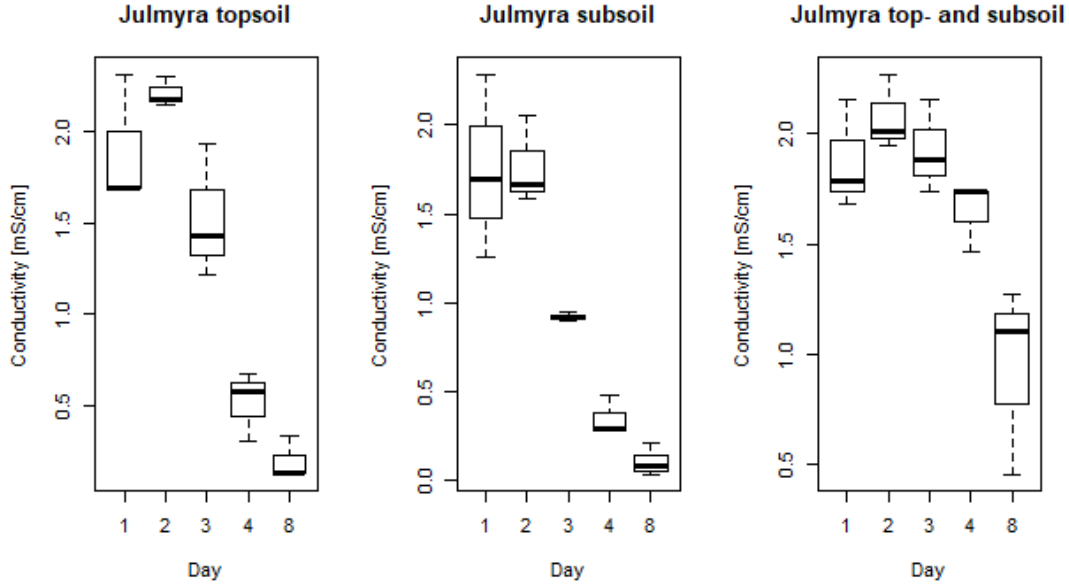


Figure 12: How the electrical conductivity in leachate for Julmyra changed through the days where the marked line in the boxes is the median and the distribution is the lines outside the boxes.

The EC in the leachate for all the soils follow a similar pattern in contrast to the pattern of P-leach in the leachate for the soils. Comparing EC for 3M2, Mellby2 and Julmyra2 (Figure 10 - 12) for example it seems that regardless if it is an arable soil or a forest soil the EC changes in a similar way.

3.2.1 Correlations between EC and the P-leach

Correlation test between P-leach and EC in the leachate was made since patterns between them were expected. All data from the different days and its replicates were used, that is 15 samples in total per treatment, and correlation was assessed for the non-parametric method Kendall's tau (Table 7).

Table 7: Analysis of correlation between P-leach and the EC in the leachate, $p < 0.05$ shows a 95 % probability of a correlation and is marked in the table.

Method	3M1	3M2	3M12	Mell1	Mell2	3M12	Jul1	Jul2	Jul12
Kendall's tau	-0.49	-0.77	-0.43	-0.58	-0.28	-0.096	-0.22	0.10	-0.077
p-value	0.011	0.00001	0.028	0.0019	0.17	0.62	0.28	0.63	0.69

All the samples from 3M showed 95 % probability of correlation, also called significant correlation, between the P-leach and the EC in the leachate. They also showed a high tau value which indicates that the correlation is strong. It was only the topsoil for Mellby

that showed a significant correlation between the P-leach and the EC in the leachate and had also a high tau value. Non of the samples from Julmyra did show any significant correlations which is not that surprising since the data from the P-leach itself did not show any pattern, as it did for 3M and Mellby (Figure 8). The P-leach and the EC in the leachate for the samples from 3M are plotted in Figure 13 and it is apparent that the correlation is not linear which can also be seen for the topsoil from Mellby in Figure 14.

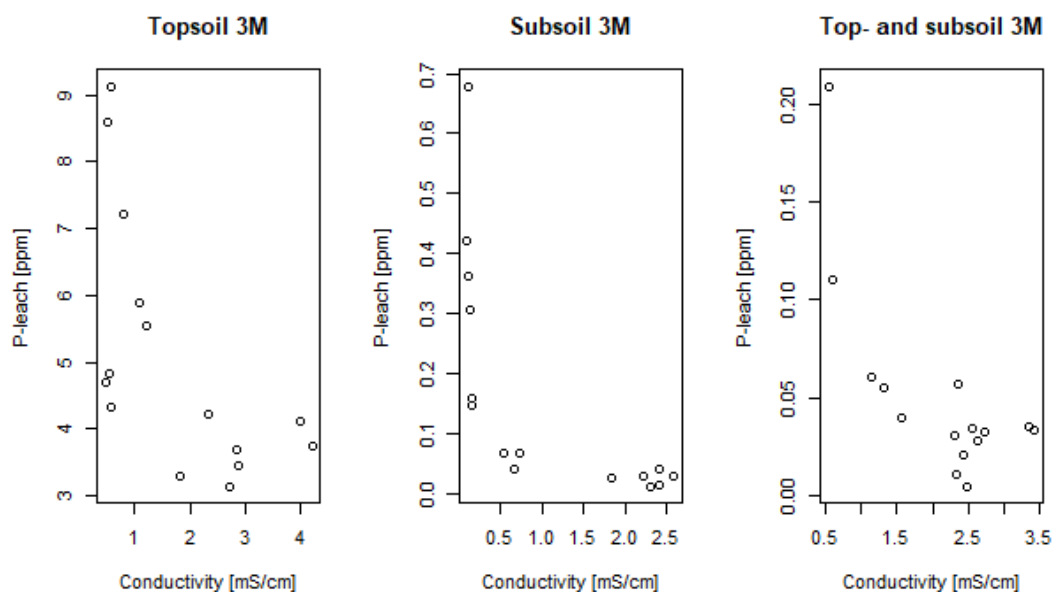


Figure 13: *The relationship between the P-leach and the conductivity of the leachate for the samples from 3M.*

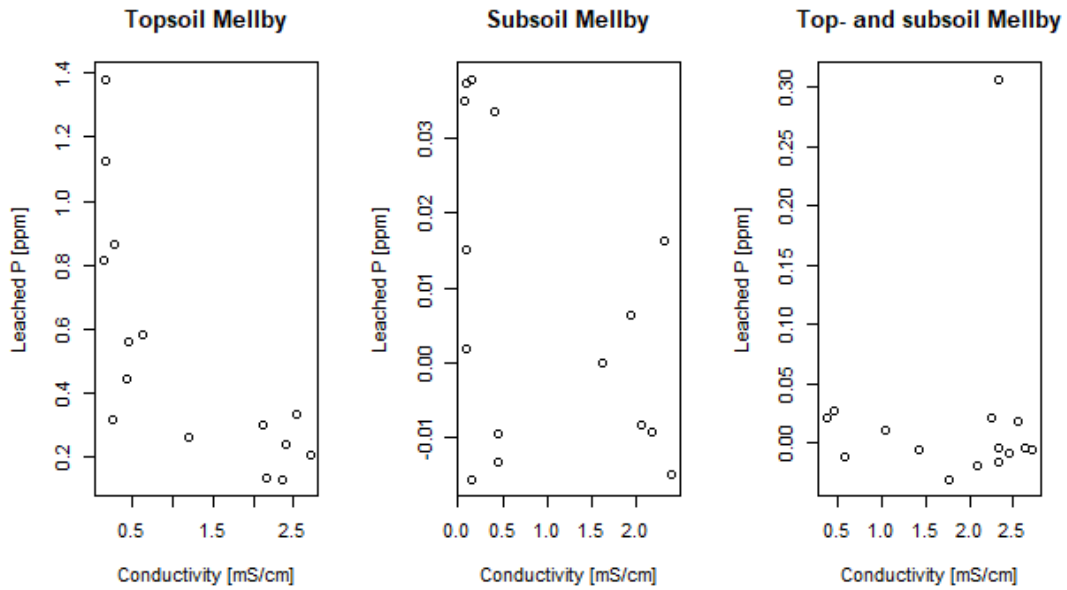


Figure 14: *The relationship between the leached P and the conductivity of the leachate for Mellby.*

The relationship between the P-leach and the EC in the leachate looks exponential (Figure 13) so the data was transformed with a log-function and that relationship showed more of a linear appearance (Figure 15). The none significant correlations for all the samples from Julmyra can be seen in Appendix.

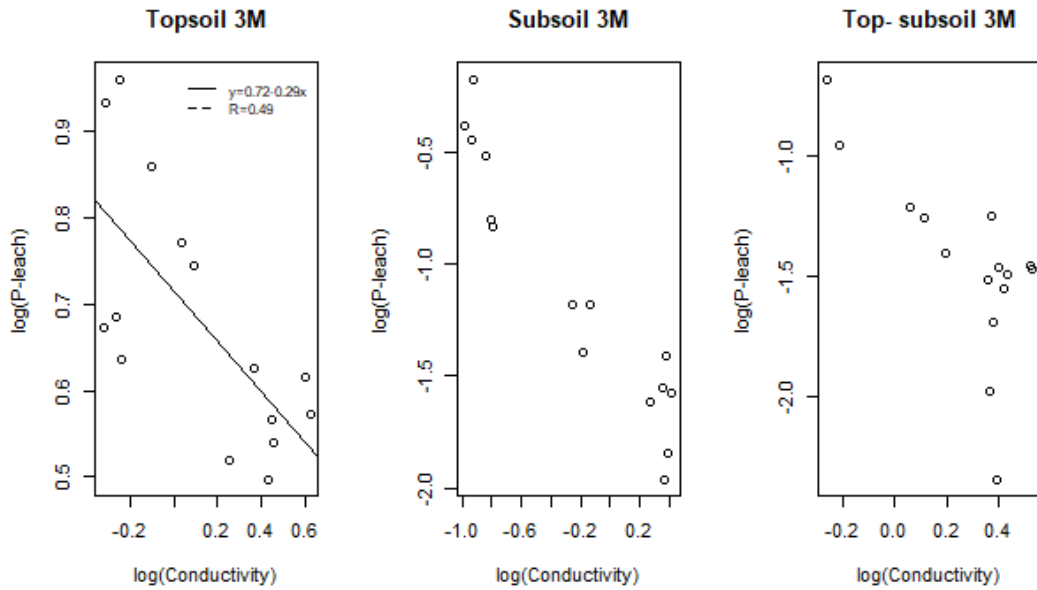


Figure 15: *The correlation between the P-leach and the EC of the leachate for 3M when the data is log-transformed and linear regression lines are plotted.*

Both the data for the P-leach and the EC in the leachate from 3M1 was normally distributed with the log-transformation and a linear regression could be applied (Figure 15). For 3M2 and 3M12 could linear regression not be used since the data for the EC in the leachate could not be transformed so that the data got normally distributed. When plotting the log-transformation for the P-leach and the EC in the leachate for 3M2 and 3M12 however, it indicates that it might be exponential since the log-transformation shows a linear pattern, which reveals that the relationship between the P-leach and the EC is not linear but more exponential (Figure 15).

4 Discussion

The first research question was if DPS is a reasonable indicator for easily soluble P and the result showed that there is a correlation between them with almost 95 % probability. The second research question that investigated if DPS is a reasonable indicator for leached P showed, however, no significant correlation. Investigating easily soluble P as a reasonable indicator for leached P also showed significant correlations, which answers research question three. The last question was if the subsoil affect the leached P and for the arable soils, 3M and Mellby, it was apparent that it did.

4.1 Degree of P saturation and easily soluble P

Figure 2 illustrates the relationship between DPS and easily soluble P and it can be seen that there is a correlation between them and Kendall's tau test confirms the relationship with its p-value of 0.056 which is close to the significant limit of 0.05. The value of tau was 0.73 which is high and the test indicates thereby also a strong relationship. DPS is therefore seen as a reasonable indicator for easily soluble P. A study made in Florida used oxalate extraction instead of lactate extraction and a linear relationship was found between water-soluble P (P-AW and P-CaCl₂) and DPS when four different fields were investigated (Nair et al., 2004). It is interesting that two different extraction methods show similar results.

The relationship between DPS and P-leach is illustrated in Figure 3 and even though it looks like there is a correlation between them, the correlation test Kendall's tau did not show significant correlation; and in contrast to easily soluble P the p-value was not even close to the significant level (Table 5). A study with 22 Nordic soils showed that DPS determination could be used as a risk assessment for acid soils (Ulén, 2006), and that DRP concentration in drainage water and DPS in clay topsoil and sandy subsoil was correlated using Pearson's method ($r=0.918$, $p=0.000$) (Ulén, 2006). Another study compared the topsoil from just Mellby and another sandy soil with lower P status and investigated the effects of application of pig slurry. It showed that both before and after slurry application in the laboratory the DRP concentrations in the leachate were significantly correlated with the DPS values in the topsoil (Liu et al., 2012). According to the results from Kendall's tau test on our results is DPS not a reasonable indicator for P-leach in soils. But due to the low number of samples ($n = 6$) the results must be considered as uncertain.

Our results showed significant correlations between P-leach and P-H₂O, P-CaCl₂ and P-AW, respectively (Table 6 and Figure 4). The test also showed that the correlations were strong since the tau-value was high which is a good result for only having 6 samples but it does not make the result less sensitive. Easily soluble P can therefore be seen as a reasonable indicator for leaching of P from soils. The correlation also means that these different extraction solutions for easily soluble P show similar results.

4.2 Top- and subsoil

It is apparent from our results that the subsoil influence the total leaching from the soil columns. The P-leach from 3M1 and Mellby1 have a distinctly higher amount than 3M12 and Mellby12. This indicates that the subsoil do affect the total leaching of P for the arable soils. For the forest soil, Julmyra, the amount of P-leach was too low that it is

difficult to analyze the difference between the samples. These results are less sensitive than the previous results since the data from all replicates are used so 15 samples in total instead of only 6.

All the data from the replicates were also used when investigating the correlation between P-leach and the EC in the leachate, therefore 15 samples in total instead of 6. The samples 3M1, 3M2, 3M12 and Mellby1 showed significant correlations between the P-leach and the EC in their corresponding leachate with Kendall's test (Table 7). Kendall's tau for the samples were also high which indicate a strong correlation. The correlations look more of a logarithmic relationship rather than a linear relationship, which is illustrated in Figure 13. After log-transforming the data it got clear for 3M1 that the relationship was logarithmic which can be seen in Figure 15 and since the data was normally distributed linear regression could be applied ($R=0.49$). In Figure 15 it can also be seen that the log-transformation for 3M2 and 3M12 indicate on logarithmic relationship but since their data were not normally distributed linear regression was not applied. This result matches with the fact that EC affect a soil's buffer capacity. Learning from this result it is important to use a solution with the same EC as irrigation during the whole experiment.

The 2-sample and the 12-samples for Mellby and all the samples from Julmyra showed no significant correlations as confirmed when studying their plots (Appendix). They contained only a small amount of P-AL and P-leach compared to the soils from 3M and this can be one explanation why no significant correlations were found.

Studying the mean EC for the 1-, 2- and 12-samples from the soils a distinct difference between them can be seen (Figure 9). The EC in the 2-samples was generally lower than for the 12-samples. This observation is logic, since CaCl_2 -solution has the EC of 2.175 mS/cm and artificial rainwater has the EC of 0.008617 mS/cm. This observation is logical since the pore volume for the columns were around 38-53 ml and only 20 ml was leached each day. This means that the first two days of leaching for the 1- and 2-samples with only one column filled with soil, CaCl_2 -solution was the dominate component in the leachate and first on day three the leachate had artificial rainwater as the dominant component. For the 12-samples, with two columns filled with soil, it was only on the last day that artificial rainwater was the dominate component in the leachate. This can be seen studying the EC for each day in the box plots (Figure 10, and 11), for example is the EC for the 12-samples only under 1 mS/cm the last day. Learning from this result the leached amount sample used each time should match the pore volume of the soil sample in the column.

4.3 Sources of Errors

As pointed out earlier only six values could be analyzed for the correlation test with the different P-samples. Therefore, the statistical power of the test is low. This makes interpretation of the result uncertain. DPS and P-AL showed significant correlation and DPS and P-leach did not but when comparing their plots it is more or less one data point that makes a big difference, which shows the high sensitivity in a test with few values (Figure 3). More sampling occasions as well as matching amount leachate corresponding to the pore volume should have enabled a more certain result. Since the mean EC is different for the 1-, 2- and 12-samples, it probably affected the result. Especially since the results showed significant correlations between the EC in the leachate and the P-leach for the arable soils.

The results from our study is valid for constructed sandy soil columns, showing only the effect of the soil texture and not taking into account any natural soil structure. In a natural situation, also the soil structure as well as flow pathways of water from the field must be accounted for when assessing the risks for P losses. It is interesting to observe how the distribution of the P-leach gets higher for each day, it is more distinct for 3M (Figure 6). And it is hard to say if it is because of the filtered soil, the changing EC in the leachate or just something unique for this study.

The chemical properties of the soil changes depending on where in the field the sample is collected. Comparing our Al, Fe and P-AL values from Mellby with another study with soil sampled from the same area show differences in the amount (Aronsson and Torstensson, 2009). In a natural situation, more samples from the field should thereby be collected to represent the situation in the field. The fact that the P status from three replicates for Mellby1 is zero for P-CaCl₂, P-H₂O and P-AW is suspicious since Mellby is a known arable soil.

The amount of leachate collected each day should have been weighted so that the pore volume could have been calculated more closely. The 20 ml leachate that were collected each day was only estimated by looking at a limit for each leachate-sample.

5 Conclusion

The few samples in the correlation tests makes it difficult to interpret the results because of high sensitivity to the inclusion of individual values, except between the P-leach and the EC in the leachate with its 15 samples. DPS can be seen as a reasonable indicator for easily soluble P and easily soluble P can in turn be seen as a reasonable indicator for

leached P in soils. DPS calculated from lactate extractable P, AL and Fe can, however, not be seen as a reasonable indicator for leached P in soils, based on the results in this study. The subsoil does affect the total leaching of P in soil.

The results also indicate that there is a significant correlation between the P-leach and the EC for sandy soils.

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Appendix

All data

All the results from the five analyzed days for the three analyzed soils are summarized in the following document:

<https://docs.google.com/spreadsheets/d/1YBmF70jp1IoTKHOrc3oiAk94QWzUCQ2B-XHF2qo9Iyk/edit?usp=sharing>

Transforming ppm

The unit from the spectrophotometer is ppm and the transformation to mg/ 100g air dried soil is presented. Firstly ppm is transformed to the amount of solution that was used, 18 ml. Since 6 g of air dried soil was used for the extraction (e.g. 6 g soil/18 ml solution) into the solution it means that 100/6 times the new value gives the unit mg/100g air dry soil.

$$P_{H_2O}[ppm] = P_{H_2O}[mg/Lsolution]$$

$$P_{H_2O}[mg/Lsolution] = \frac{P_{H_2O}}{1000} * 18[mg/18ml]$$

$$\frac{P_{H_2O}}{1000} * 18[mg/18ml] = \frac{P_{H_2O}}{1000} * 18 * \frac{100}{6}[mg/100gsoil]$$

Calculating DPS

DPS is calculated as P-AL divided by phosphorus sorption capacity (Al-AL + Fe-AL) all values given as mmol/100 g air dry soil.

$$n(Al) = \frac{m(Al)}{M(Al)} = \frac{Al[mg/100gairdrysoil]}{Al[g/mol]} = Al[mmol/100gairdrysoil]$$

$$DPS[mmol] = 100 * \frac{P[mmol]}{PSC[mmol]} = 100 * \frac{P[mmol]}{Al[mmol] + Fe[mmol]}$$

No significant correlation between P-leach and the EC in the leachate

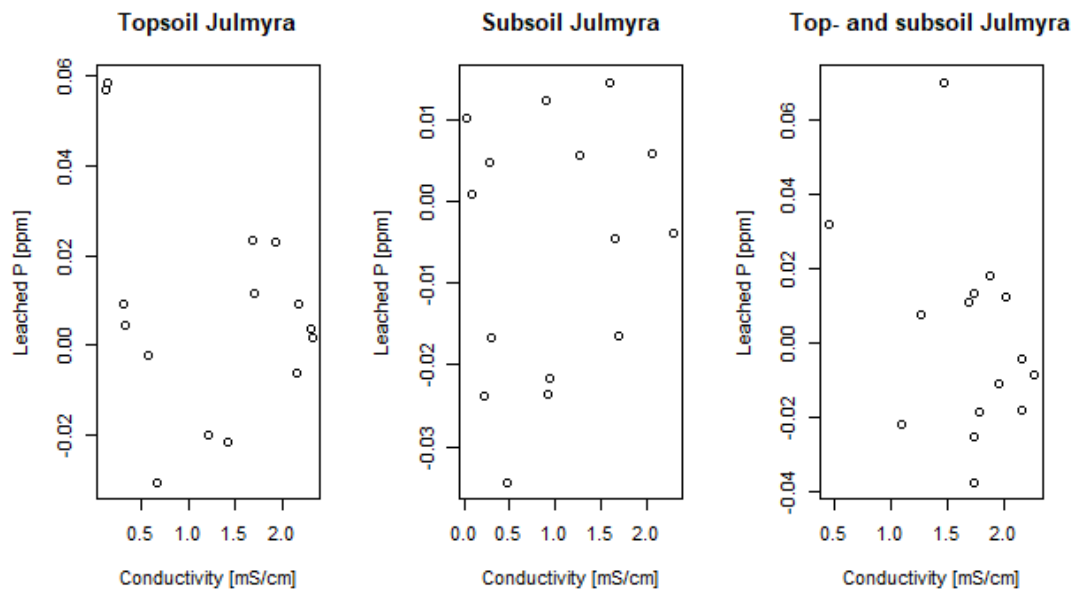


Figure 16: *The relationship between the leached P and the conductivity of the leachate for Julmyra.*