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## Knowledge status for structure liming in Norway

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*Summary:* Development of new mitigation methods to reduce phosphorus loss from agricultural areas is needed to improve water quality in Norway. In Sweden, structure liming has been of high interest the last years as a method to reduce phosphorus losses. This happens even though only a few studies have investigated their effectiveness. The expected effect of structure liming is due to its effect on aggregate stability and effect on solubility of phosphorus. This report summarizes the knowledge status on structure liming and describes a lab study on phosphorus solubility. In this study, no clear effects on soil phosphorus were found due to liming. The literature suggests an effect of structure liming only on soils with high clay content and a high soil phosphorus status and this may explain the missing effect of liming in our study.

*Sammendrag:*

Approved

  
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# Preface

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A literature review was undertaken to explore available information and data on liming and to identify knowledge gaps regarding the effect of liming on phosphorus (P) losses from soils, especially in Norway. This review report gives a theoretical description of liming materials and some practical examples of research results on effects of liming on P solubility, P losses by leaching and runoff and liming effects on soil structure. A pilot experiment on liming's effect on P binding was undertaken and the results are reported. This study was financially supported by the CATCHY project No. 8278.A6 as part of one of its projects components under mitigation measures.

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# 1. Introduction

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Phosphorus (P) runoff from agricultural land to surface waters (i.e. streams and lakes) is a major problem in Norway. Transport of P from agricultural land contributes to the eutrophication of surface waters. P is one of the limiting factors for algae growth in most of the eutrophic lakes in Norway. The main factors affecting P loss are soil P contents, P application (including rate, method, and time of application), soil erosion, surface runoff, subsurface runoff, and contributing distance (Bechmann *et al.*, 2005).

In Norway, the main mitigation measures to reduce P loss from agricultural land entails changes in soil tillage practices, more precise nutrient application rate, establishing vegetation buffer zones and construction of sedimentation ponds (Bechmann and Deelstra, 2013). Despite several mitigations measures applied in the different agricultural production systems across Norway by the National Agricultural Environmental Monitoring Program (JOVA) over the past 20 years, P losses from agricultural areas via leaching or run off have not been reduced significantly (Hauken *et al.*, 2013). There is a growing consensus among researchers, farmers and decision makers to look for improved and new mitigation method against P Losses from agricultural areas.

One new method that perhaps can be used to reduce P losses from agricultural areas is structure liming of soils. Structural liming, is a mitigation measure where lime (in the form of CaO or Ca(OH)<sub>2</sub>) is applied to soils to improve soil aggregate stability, and potentially reduce availability of P. Gypsum is not a liming material as such, but includes in the literature review because it is used in much the same way as structure liming. Improved aggregate stability of soils contributes to minimize erosion and P losses. Structural liming has not been tested in Norway so far, but there are positive results achieved in Sweden (Ulén *et al.* in press) and USA (Murphy 2012), in Finland there are positive results achieved with gypsum amendments (Alakukku and Aura, 2006; Muukkonen *et al.*, 2009; Ekholm 2012;). Studies in Sweden (Ulén *et al.*, in press) have been carried out on the effect of structural liming in heavy clay soils, but the knowledge and understanding of structural liming in soils with lower clay contents is limited.

To explore the available information and data on structural liming and help identify knowledge gaps on the effect of liming on P losses from agricultural soils, a literature review pertaining to the following topics were undertaken:

- Chemical effect of liming on P binding
- Structural effects of liming

Afterwards, a pilot study on liming effects on P binding under laboratory conditions was conducted.

## 2. Literature Review

### 2.1 Chemical effects of liming on P binding

#### 2.1.1 The importance of P binding for P loss

The risk of mobilisation and transport of P to nearby streams are determined by erosion of soil particles and the amount of P found in the soil solution. P is strongly adsorbed to soil particles and usually only a small amount is found in the soil solution. Hence, erosion of soil particles are the most important pathway for P loss in most soils. However, loss of dissolved P is also important, especially in areas with high P-AL status.

P solubility is highly affected by the pH in the soil (Figure 2.1). Below pH 5, P is usually fixed by hydrous oxides of Fe and Al. While above pH 7 direct chemical precipitation of  $\text{Ca}^+$  combined with orthophosphate may occur. Between pH 6 and 7, P is relatively available to plants (Brady and Weil, 2002). Availability of P is in Norway most commonly expressed as P-AL (Ammonium lactate-extraction) or water soluble P (P- $\text{CaCl}_2$ ). As the soil phosphorus status, measured as P-AL og P- $\text{CaCl}_2$ , increases, the risk of P loss increase (Sharpley et al., 2001).

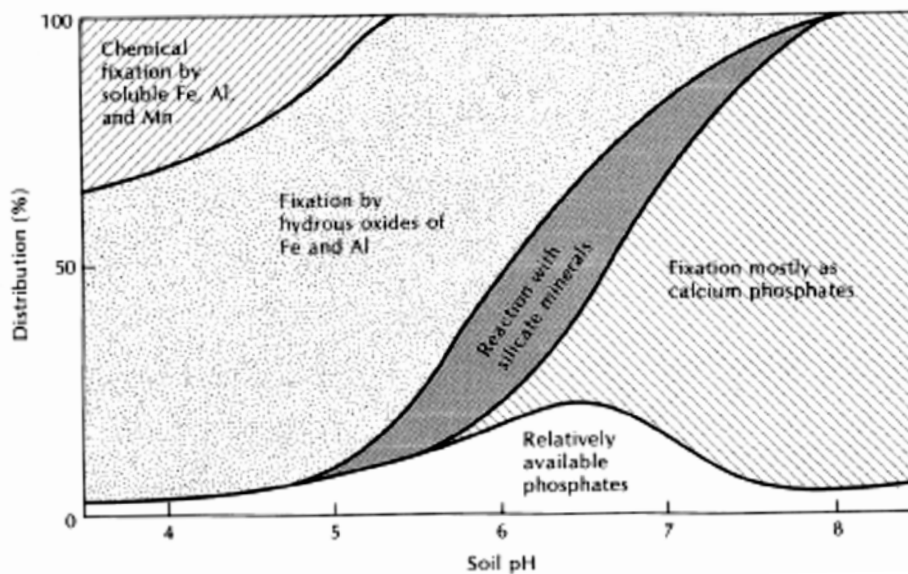


Figure 2.1. Inorganic fixation of added phosphates at various soil pH values. Average conditions are postulated and not necessarily true for a particular soil type (adapted from Brady and Weil, 2002).

#### 2.1.2 Type of liming materials

In general, liming materials fall into three main categories: carbonates, oxides, and hydroxides (Mahler, 1994). Gypsum is not a liming material as such, but used in much the same way as structure liming.

##### Carbonates

Carbonates ( $\text{CaCO}_3$ ,  $\text{MgCO}_3$ ) (Kalkstein) are the most widely available and most widely used liming materials. Carbonates are generally less expensive and easier to handle than other lime materials.

Ground high grade limestone or calcitic limestone is nearly pure calcium carbonate ( $\text{CaCO}_3$ ). The major advantage of  $\text{CaCO}_3$  (lime) is its low cost. Dolomitic limestone or dolomite ( $\text{MgCO}_3 + \text{CaCO}_3$ ) is also a commonly used source. Dolomite usually costs a little more than calcitic limestone and changes the soil pH more slowly, but it has the advantage of containing Mg as well as Ca (Mahler, 1994).

### Oxides

Oxide liming materials ( $\text{CaO}$ ) (Brent kalk) are known by many names such as burned lime, unslaked lime, and quicklime. Oxides are made by baking crushed calcitic limestone or dolomitic limestone in an oven or furnace, thereby driving off carbon dioxide ( $\text{CO}_2$ ) to form a pure oxide as shown in Fig 2.2. This material has low molecular weight and reacts rapidly in the soil to raise the pH. Because  $\text{CO}_2$  is driven off in the roasting process, oxides are the most efficient of all liming materials. Oxides are powdery, so they are sold in bags. Oxides are also caustic; that is, they react with moisture and are difficult and dangerous to handle. In addition, their costs are high relative to carbonate materials because of the high energy requirement for  $\text{CO}_2$  removal.

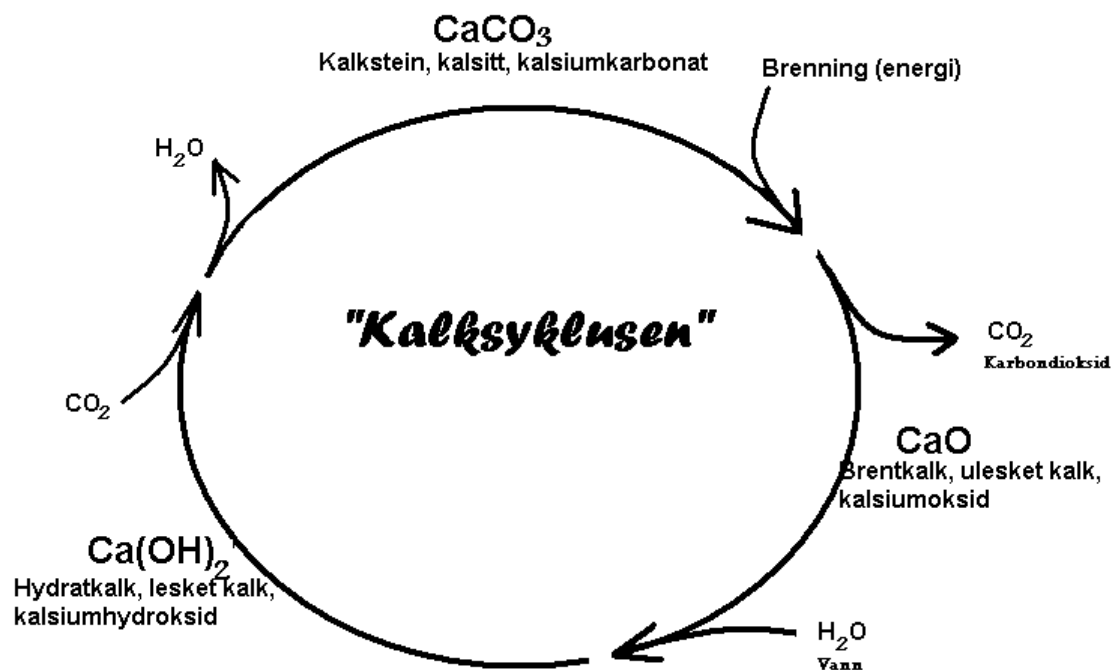


Figure 2.2. Lime production cycle (Source: Adapted from Franzefoss Minerals, 2003).

### Hydroxide

Hydroxide ( $\text{Ca(OH)}_2$ ) (Hydratkalk) is simply oxide materials with water added. They are also known as hydrated lime, slacked lime, or builders lime. These materials are similar to oxides because they are powdery and quick acting, and also unpleasant to handle. Hydroxides are also more expensive than carbonate materials.

### Gypsum

Gypsum ( $\text{CaSO}_4$ ) is a natural mineral found in sedimentary rocks. It has been used as a soil amendment, plaster and also for sculpturing.

### 2.1.3 Influence of chemical composition of liming materials

The effect of lime can be expressed in several ways:

- By content of elemental calcium and magnesium.
- The percentage of conventional oxide (CaO or MgO).
- CaO equivalent (the neutralizing ability expressed in terms of CaO).
- CaCO<sub>3</sub> equivalent (the neutralizing ability expressed in terms of CaCO<sub>3</sub>).
- Total carbonates (the sum of calcite and dolomite)

If we compare pure calcite limestone (molecular weight=100) with pure calcium oxide (molecular weight=56), we see that 56 g of calcium oxide will neutralize the same amount of acid as 100 g of calcite limestone (Table 2.1.) Hence, to neutralize the same acidity you need twice as much CaCO<sub>3</sub> as CaO. Burned lime (CaO) has around 20% more neutralizing power than hydrated lime (Ca(OH)<sub>2</sub>) (Table 2.1). The fineness of lime determines the efficiency of soil↔lime reactions and to a large extent, the rate of short-time neutralization power (Mahler, 1994). Burned and hydrated limes generally react quickly with soils and can induce changes in soil pH in only a few weeks, for limestone to react fully with the soil colloids it can take a full year (Brady and Weil, 2002).

According to Berglund (1971) the burned and hydrated lime can dissolve up to 1000 mg Ca per liter water and increase the pH up to 12. Limestone can only dissolve about 6 mg Ca per liter water and increase the pH to a maximum of 8.2.

Table 2.1. Common liming materials and their corresponding neutralizing power and values.

Liming material	Neutralizing power expressed as CaO (%)	Neutralizing power expressed as CaCO <sub>3</sub> (%)
Calcitic lime (CaCO <sub>3</sub> )	40-56	71-100
Dolomitic lime (CaCO <sub>3</sub> MgCO <sub>3</sub> )	50-62	89-111
Burned lime (CaO)	75-100	134-178
Hydrated lime Ca(OH) <sub>2</sub>	66-80	118-143

### 2.1.4 Liming effects on P solubility in soils

The primary reason for liming soils is its effects on raising soil pH and increase the solubility of most plant nutrients (like N, P, S, Mo, K, Mg, Ca), making most of them more available (pH 6-7) to the plant and a few (like B, Mn, Cu, Zn) less available (Filipek, 2011).

According to Sanchez and Uehara (1980), liming increases phosphate availability through raising soil pH. Increased pH increases the negative charge on soil particles and forces P into the soil solutions (Murphy 2007). Liming promotes the decomposition and/or mineralization of soil organic matter (SOM) and consequently, it increases the mobilization of plant nutrients from the organic matter as well. On the other hand, increasing pH can also lead to the formation of Ca, Al or Fe complexes with P, making it less available to plants. The addition of large amounts of Ca ions increases the ionic strength by suppressing the diffuse double layer of the soil particles (Murphy, 2007) thereby increasing P adsorption in the soil particles.

According to Murphy (2007) «Lime has been reported to increase, decrease, or have no effect on P solubility, plant availability or sorption». The effects of liming on P availability are highly dependent on soil type and its pH level. The effect of soil phosphorus status has not been investigated.

#### Liming effects on losses of P by leaching

Laboratory leaching experiments with lime have been conducted in several countries to investigate the effects of liming on P losses via leaching. For example, in Korea CaCO<sub>3</sub>, Ca(OH)<sub>2</sub> and gypsum (CaSO<sub>4</sub>) were used to reduce P solubility in the soil. Among the selected liming materials, Ca(OH)<sub>2</sub>



was the most effective to decrease the water-soluble P concentration of soil (by 69%), then followed by gypsum (53%) and CaCO<sub>3</sub> (20%) (Lee *et al.*, 2011).

In Florida (USA), on sandy soil, total reactive P loss was reduced by 36% through amendments of CaCl<sub>2</sub>, by 17.5% through CaCO<sub>3</sub> and 40% by CaCl<sub>2</sub>+CaCO<sub>3</sub> when compared with chemical fertilizer alone (Yang *et al.*, 2007). The corresponding values for dissolved reactive P were 70.8%, by additions of CaCl<sub>2</sub> 71.9 by CaCO<sub>3</sub> and % by CaCl<sub>2</sub>+CaCO<sub>3</sub>. The results indicated that the use of CaCl<sub>2</sub>, CaCO<sub>3</sub>, or their combination significantly could reduce P leaching from sandy soils.

In Finland, methods where Ca is applied with lime or other soil amendments (e.g. board mill sludge or kiln dust) have proved to be effective in reducing P even 5 years after application (Alakukku and Aura, 2006). According to Muukkonen *et al.* (2007) liming and higher Ca concentrations in the soils might be the cause for lower percolated reactive P from the limed clay soil compared to the unlimed soil. Laboratory leaching experiments carried out in Finland showed that spreading fresh sludge (by products of liming) on the surface of soils column, improved the quality of the percolates in the rainfall simulation, turbidity, total P and particulate P diminished (Muukkonen *et al.*, 2009). The effect was related to the amount of Ca in solution. Addition of gypsum to soils has also shown to effectively reduce both particulate and dissolved P losses from clayey soils on a catchment scale in Finland (Ekholm *et al.* 2011). Gypsum increased the ionic strength of the soil solution, and, according to Ekholm *et al.* (2011), thereby decreased the detachment of these forms of P. The P fluxes from the catchment decreased after the gypsum amendment.

In 2012, Murphy investigated lime as a soil amendment to decrease P loss in runoff from two Delaware sandy loam soils, one high and one low in P. Lime showed potential to decrease P loss in runoff, but initial soil P status was more important than liming in determining P loss.

The latest study on effects of liming on P leaching was carried out by Ulén and Etana (in press) who studied two sites with different levels of soil P status (P-AL). In their study, liming affected leaching of phosphate in the site with the highest P-AL (9-14 mg/100g) throughout the subsoil. They suggest that formation of Ca-P complexes or Ca-precipitates at this site are formed presumably due to high concentration of dissolved reactive P in the soil water.

## 2.2 Structural effects of liming

### 2.2.1 Importance of soil structure for erosion and P losses

Structures of soils are often considered to be one of the most important properties affecting soil erosion. Well-structured soils with good aggregate stability allow the free movement of air and water through fissures (or cracks) between the structural units called aggregates and hence increase infiltration and reduces the risk of surface runoff. Stable aggregates also enhance infiltration during heavy rain and thereby reducing surface erosion (Ulén *et al.*, 2011).

Factors influencing soil aggregate stability are related to:

- (1) Binding actions of microorganisms through thread like filaments or fungi;
- (2) Intermediate products of microbial synthesis and decay, such as gums and certain polysaccharides; and
- (3) Stable humus components.

Moreover, di-valent and polyvalent inorganic cations such as Ca<sup>2+</sup>, Mg<sup>2+</sup>, Fe<sup>2+</sup>, Al<sup>3+</sup> that enhance flocculation, are thought to provide mutual attraction between organic matter and soil clay particles.

Aggregate breakdown is induced by several factors. When a dry soil is wetted, the entry of water into the soil may produce internal and external forces promoting aggregate breakdown. Internal forces in soil may arise from the release of entrapped air, differential swelling of clay minerals, slaking and dispersion of clay particles (Marshall *et al.*, 1996). External forces, which may cause aggregate breakdown, include the impact of raindrops and overland flow. Upon drying, the soil material may reorganize leading to shrinkage and cracking of clays, and coalescence of neighboring soil material. Small particles may clog soil pores and a crust can be formed on the soil surface, which could enhance erosion by reducing infiltration rate and increase runoff (Le Bissonnais *et al.*, 1993; Loch, 1994; Marshall *et al.*, 1996).

### *2.2.2 Influence of lime products on soil aggregate stability*

Liming (in quantities relevant in agriculture) may reduce P losses indirectly by improving the growth condition of plant (P is more efficiently taken by plants) and by improving the stability of soil aggregates, which reduces the surface runoff and, consequently, the transport of P dissolved in water or bound to particle surface (Helinä Hartikainen, personal communication, 2013).

Liming-induced increase in pH will enhance biological activity and production of organic compounds that stabilize the aggregate of soils. Organic substances such as gums and resins are produced which coat and stabilize mineral aggregates (McLean, 1971).

Liming promotes better soil structure through the addition of  $\text{Ca}^{2+}$ . According to Berglund (1971), the effect of lime happens in three steps. The first step is that cation exchange reactions decreases the hydration around clay minerals, the decreased hydration makes the clay minerals rearrange and aggregates are formed. The second step is the puzzolan reaction which is the same reaction that forms cement. Calcium binds with aluminum and silica and forms calcium-alumina hydrates and calcium-silica hydrates. These forms are strong binders and makes aggregates bigger and more stable. The third step is mortar binding which form in the same way as actual mortar, through combining sand, water and  $\text{Ca}(\text{OH})_2$ . Another effect is aggregation due to more clay- polyvalent cation-organic matter bridging.

The only exceptions where the liming may have negative effects on soil structure are soils which are very acidic and Al is an important cation binding element to bring clay particles together. The neutralization of acid Al by  $\text{OH}^-$  and formation  $\text{Al}(\text{OH})_3$  means that the aggregate stability will diminish (Helinä Hartikainen, personal communication, 2013).

Laboratory experiments have shown increased aggregation and aggregate stability after liming of soils. Berglund (1971) added 0.0, 0.25, 1.0, 5.0 and 10.0 % lime in the soil, and the effect on aggregate stability was noticeable with 5 % lime added and thereafter the effect increase further for some soils while other stayed the same. Shanmuganathan and Oades (1983) showed that the addition of cement or gypsum ( $\text{CaSO}_4$ ) to a sandy loam soil with 12% clay also stabilized the aggregates. According to Ledin (1981) improvements in soil structural stability of a clay soil may be sustained 8 years after application of lime in the form of  $\text{CaO}$  or  $\text{Ca}(\text{OH})_2$ .

A field study on liming in Australia has found that the application of  $1.5 \text{ tons ha}^{-1}$  of lime ( $\text{CaCO}_3$ ) after a year promoted a decrease in the structural stability of the soil. According to Chan and Heenan (1998), this was caused by the elevation of the pH, the microbial activity and by the decrease in total organic carbon. However, three years after the application of lime, in the same experimental area, there was an increase in the structural stability of the soil, indicating that the harmful effect of liming was temporary. The effect of applying lime on soil structure is usually seen in the long term.

Helinä Hartikainen *et al.* (2013) carried out an elution test that was performed with structured soil. Their results revealed that liming enhanced the stability of soil aggregates and, thus, diminished the surface area directly in contact with percolating water. It also elevated the ionic strength in the pore water system.

Ulén and Etana (2014) studied the effect of liming on loss of particulate P from two soils with different soil texture (moderate and high topsoil clay content). They found that structural liming significantly affected loss of particulate P through the soil profile on the soil with high clay content, but did not reduce loss of particulate P on the soil with moderate clay content in the topsoil.

## 2.3 Literature available

Table 2.1 presents some available literature pertaining to the role of lime in mitigating P losses by runoff and/or leaching from agricultural fields.

Table 2.1. Knowledge and information in relation to liming effects on P losses and corresponding relevant references.

Knowledge/information on Liming	References
Role of liming in P mobility, P sorption and P solubility in soils.	<ul style="list-style-type: none"> <li>○ Berglund (1971)</li> <li>○ Filipek (2011)</li> <li>○ Hao, et al (2002)</li> <li>○ Mahler (1994)</li> <li>○ Murphy (2007)</li> <li>○ Sanchez, P.A., and Uehara, G. (1980)</li> </ul>
Effects of liming to reduce leaching losses of P.	<ul style="list-style-type: none"> <li>○ Alakukku and Aura, (2006)</li> <li>○ Lee, et al (2011)</li> <li>○ Collin (2010)</li> <li>○ Muukkonen, et al (2009)</li> <li>○ Murphy, P.N.C. and Stevens, R.J. (2010)</li> <li>○ Yang, et al (2007)</li> <li>○ Ulén and Jakobsson (2005)</li> </ul>
Effects of liming soils to maintain aggregate stability	<ul style="list-style-type: none"> <li>○ Berglund, G. (1971).</li> <li>○ Briedis et al (2012)</li> <li>○ Collin (2010)</li> <li>○ Chan and Heenan (1999, 1998)</li> <li>○ Ledin, S. (1981)</li> <li>○ Neufeldt et al (1999)</li> <li>○ McLean (1971)</li> <li>○ Castro (undated)</li> </ul>
Field studies of structure-liming and addition of gypsum	<ul style="list-style-type: none"> <li>○ Alakukku, L. and Aura, E. (2006)</li> <li>○ Ekholm et al (2011)</li> <li>○ Ekholm et al (2012)</li> <li>○ Muukkonen, et al (2007)</li> <li>○ Murphy (2012)</li> <li>○ Ulén, et al (2014)</li> </ul>

## 3. Pilot Study

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A pilot study was conducted to determine the effect of liming on P binding under laboratory conditions.

### 3.1 Materials and Methods

Composite soil samples from the topsoil depth (0-20 cm) were collected from the cereal field nearby The Norwegian University of Life Sciences (NMBU) campus in Ås, Norway. The field has uniform soil characteristics and a relatively flat topography. According to Njos (1978), the texture of the topsoils in the field is silty clay loam with a particle size distribution of 27% clay, 45% silt and 28% sand that has marine origin. And the average total P content of the soils in top layer was 800 mg/kg Dry Matter (DM) rated as moderately high, the soil P status was 8-9 mg P-AL/100g, and the pH was 6, which has a neutral reaction. The soils were sampled after crop harvest in the beginning of October 2012. The soils were then air-dried and sieved through 2 mm mesh.

In the Norwegian Institute for Agricultural and Environmental Research (Bioforsk) laboratory, three buckets with a capacity of 10 liters (~10 kg) were prepared for the lime treatments. Three treatments were used. No lime (T1) used as a control, and two liming materials namely calcium oxide, CaO (T2) and calcium hydroxide, Ca (OH)<sub>2</sub> (T3) which were supplied from Franzefoss kalk AS (Oslo, Norway). About 10 kg of sieved soils (2 mm) were mixed with 25 g of CaO and 33 g of Ca (OH)<sub>2</sub> taking into account the recommended applications rate of 5 tons per ha by Ulén *et al.* (2007, 2010). In addition, about 1.25 g of P fertilizer was also applied to the 10 kg of soil following the recommendation rate of 20 kg P per ha (Anne Falk Øgaard, pers.comm, 2013). The P fertilizer (with 750 g weight) contained ingredients of 8% P, 12% S, 20% Ca). The soils were moistened by periodically adding water to maintain 35-40% of field water capacity of the soil using HH2 moisture meter (Delta-T devices Ltd. UK). In total, the soils were incubated for 15 weeks at room temperature. Composite soil samples were taken from upper, middle and lower part of the bucket using soil probes after 3, 6, 15 weeks of incubation.

The sampled soils were analysed for pH, Electrical Conductivity (EC), water soluble P in soils (P-CaCl<sub>2</sub>) and plant available P in soils (P-AL) in the NMBU-IPM soils laboratory. The pH-value was determined by mixing 5 mL air-dried soil with 25 mL water and shaking for 5 min., standing for 16 h, brief shaking again and leaving to settle so that the particles could sediment out. The pH and EC were determined according to Norwegian standard (pH: NS-EN ISO 10523:2012 and EC: NS-EN 13038:2011). The plant available P (P-AL) was determined by the ammonium lactate colorimetric method according to (Egner *et al.*, 1960). The water soluble P (P-CaCl<sub>2</sub>) was determined by an extraction method using 0.0025 M calcium chloride (CaCl<sub>2</sub>).

All analytical results are averages of three soil samples except for the soils analysed after 3 weeks of incubation where only one sample was analysed from each treatment. A one way ANOVA and a comparison using Tukey`s family errors rate was analysed using Minitab 16 software (Minitab Inc., State College, PA, USA) to compare the means of the different treatments.

## 3.2 Results

### 3.2.1 Soil pH and EC

The soil pH in the limed soils (CaO and Ca(OH)<sub>2</sub>) was higher than the un-limed soils in the first 3 weeks of incubation as liming raises soil pH (Brady and Weil, 2002). After 6 weeks, the pH in both liming materials had increased by 0.6 units (i.e. from 6.3 to 6.9) compared to un-limed soils. However, after 15 weeks the pH of both the soils with lime and without lime had decreased slightly. Other factors (temperature, moisture) may have caused the decrease in soil pH.

The electrical conductivity of soils with lime was higher than un-limed soils. The increase in electrical conductivity was expected because of the high solubility of the liming material. However, there was no statistically significant difference in the EC between the two liming materials. The reason could be that CaO and Ca(OH)<sub>2</sub> were added in different amounts to adjust for differences in neutralizing capacity.

Table 3.1 Average pH and EC after liming (CaO, Ca(OH)<sub>2</sub>) and incubation (3, 6 and 15 weeks).

	No lime	CaO	Ca(OH) <sub>2</sub>
3 weeks*	6.1	6.8	6.9
6 weeks	6.3c	6.9a	6.9a
15 weeks	5.9d	6.7b	6.6ab
(EC) 15 weeks	103.7 ± 5.4a	178.6 ± 12b	180.9 ± 28b

\*only one sample

### 3.2.2 Lime effects on P-CaCl<sub>2</sub> and P-AL

Figures 3.1 and 3.2 show the concentrations of P-CaCl<sub>2</sub> and P-AL after 3, 6 and 15 weeks of incubation treated with no lime, CaO and Ca(OH)<sub>2</sub>. From 3 to 6 weeks incubation period, there was a slight decrease in P-CaCl<sub>2</sub> for all three treatments. And, from 6 to 15 weeks there was an increase of P-CaCl<sub>2</sub> concentration in the un-lime treatment (1.3 to 2.7 mg P/kg) which is statistically significant ( $p < 0.05$ ). The change in the two limed soils was not statistically significant ( $p < 0.05$ ). After 15 weeks, there was a significant lower concentration of P-CaCl<sub>2</sub> in the limed samples compared to un-limed soils.

The P-AL concentrations increased in all treatments throughout the incubation period but did not change significantly (Figure 3.2). The limed treatments had higher concentrations of P-AL throughout the experiment. After 15 weeks the P-AL concentration in Ca(OH)<sub>2</sub> treated soils (10.5 mg P/100g) and CaO treatments (10.3 mg P/100g) was higher than the un-limed (9.1 mg P/100g). The Ca(OH)<sub>2</sub> treated soils were significantly higher than the un-limed soils at  $p < 0.05$  level.

No clear effects were found on P-CaCl<sub>2</sub> or P-AL after liming in this experiment. A challenge was that the control sample showed greater/similar changes as the limed treatments.

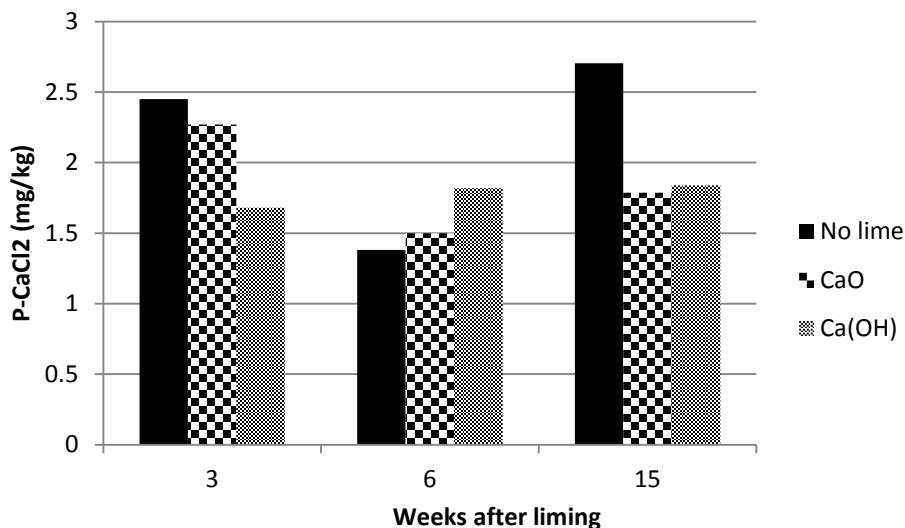


Figure 3.1 Concentrations of P-CaCl<sub>2</sub> (mg/kg) after liming (CaO, Ca(OH)<sub>2</sub>) and incubation (3, 6 and 15 weeks).

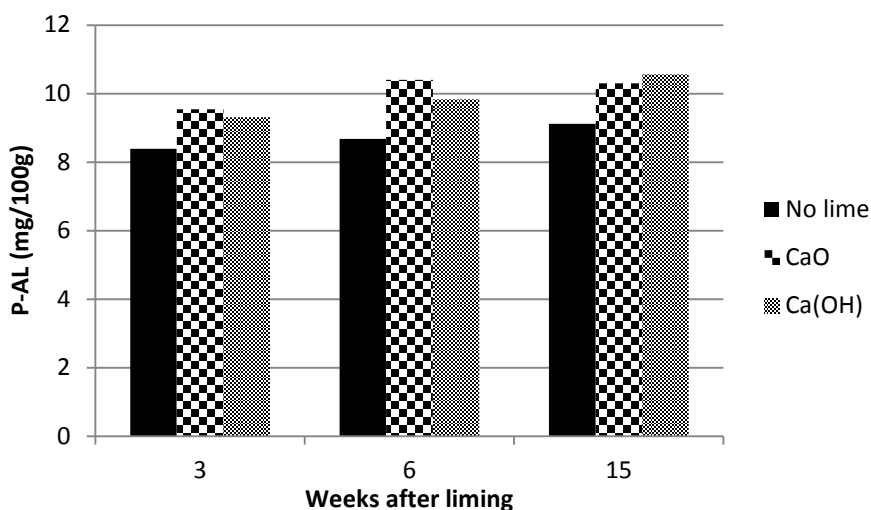


Figure 3.2 Concentrations of P-AL (mg/kg) after liming (CaO, Ca(OH)<sub>2</sub>) and incubation (3, 6 and 15 weeks).

### 3.3 Discussion

The chemical effect of liming on P binding is highly dependent on soil type and its initial pH level (Murphy 2007). In structure liming, the large amounts of Ca<sup>2+</sup> ions added increases the ionic strength by suppressing the diffuse double layer of the soil particles (Murphy, 2007) thereby increasing P adsorption in the soil particles. The same effect can be achieved with gypsum. Liming is also expected to increase pH and influence phosphate availability in several ways, according to Murphy (2007) «Lime has been reported to increase, decrease, or have no effect on P solubility, plant availability or sorption». Several lab experiments have shown a reduced leaching of P after addition of lime or gypsum (Lee et al., 2011; Yang et al., 2007; Muukkonen et al., 2009). In this pilot study, no clear effects of liming soils were found on P-CaCl<sub>2</sub> or P-AL after 15 weeks incubation. The un-limed soils showed greater/similar changes as the limed treatments. Possible explanations could be 1) Changes in temperature or/and soil moisture content during the 15 weeks incubation. To investigate further the effect of structural liming on P binding, a better control over temperature and moisture regimes is needed. 2) A too short time period or too low clay content. However, CaO

and  $\text{Ca}(\text{OH})_2$  are much more soluble than  $\text{CaCO}_3$  and there are reports by Berglund (1971) that only a few weeks are needed to bring changes in soils fixing ability to P by liming. 3) The soil P status (P-AL) in this experiment was somewhat lower than the soil P status in the Swedish experiment by Ulén and Etana (2014). Murphy (2012) found that initial soil P status was more important than liming in determining P loss, and found a higher effect with liming on high-P soil. This could be possible explanations for the lower effect in this experiment compared to literature. And it could indicate that the effect of structure liming on solubility of P only has an effect in soil with high soil P status.

The structural effects of liming are shown in several experiments (Berglund 1971; Shanmuganathan and Oades 1983; Ledin 1981) and are caused by several reactions described in section 2.2. The effect seems to be primarily caused by the addition of large amounts of  $\text{Ca}^{2+}$  ions that among others increases the ionic strength of the soil solution and provides mutual attraction between organic matter and soil clay particles. Investigations of the effect of structure lime and gypsum on aggregate stability and leaching of P through soil columns are suggested in the future. There are several reports from the field showing reduced loss of P after both structure liming and addition of gypsum (Alakukku and Aura 2006; Ekholm et al., 2012; Murphy, 2012, Ulén et al., 2014). Research in Sweden seems to be focused on structured lime, while Finland is focused on gypsum amendments. Both could be promising mitigation measures in Norway in the future. Further research should include both materials.

## 4. Conclusion

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In this pilot study, no clear effects of liming soils were found on P-CaCl<sub>2</sub> or P-AL after 15 weeks of incubation. The un-limed soils showed greater/similar changes as the limed treatments. Possible explanations could be

- 1) Changes in temperature or/and soil moisture content during the 15 weeks incubation.
- 2) A too short time period or too low clay content.
- 3) A low soil P status (P-AL) in the soil in this experiment.

The experiment and literature review indicate that the effect of structure liming on solubility of P only has an effect in soil with high soil P status.

The structural effects of liming are shown in experiments with heavy clay soil. Investigations of the effect of structure lime and gypsum on aggregate stability and leaching of P through soil columns in soil with lower clay content are suggested in the future. Both lime and gypsum could be promising mitigation measures in Norway in the future. Further research should include both materials.



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## 6. Annex

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Table 6.1. Concentrations of P-CaCl<sub>2</sub> (mg/kg) after liming (CaO, Ca(OH)<sub>2</sub>) and incubation (3, 6 and 15 weeks).

	Control	CaO	Ca(OH) <sub>2</sub>
3 weeks*	2.5	2.3	1.7
6 weeks	1.4 ± 0.3a	1.5 ± 0.2a	1.8 ± 0.1a
15 weeks	2.7 ± 0,2b	1.8 ± 0,3a	1.8 ± 0,1a

Table 6.2. Concentrations of P-AL (mg/100g) after liming (CaO, Ca(OH)<sub>2</sub>) and incubation (3, 6 and 15 weeks).

	Control	CaO	Ca(OH) <sub>2</sub>
3 weeks*	8.4	9.5	9.3
6 weeks	8.7 ± 0.6c	10.4 ± 0.8ab	9.8 ± 0.2abc
15 weeks	9.1 ± 0,1bc	10.3 ± 0,2ab	10.6 ± 0,6a

\*only one sample