

Relationship between the 0.01 M CaCl₂- and AL-soluble soil phosphorus contents

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Abstract

The traditional Hungarian method for determining soil phosphorus (P) status is ammonium-lactate acetic acid (AL) extraction. AL is an acidic solution (buffered at pH 3.75), which is also able to dissolve P reserves, so there is a need for extraction methods that also characterize the mobile P pool.

0.01 M CaCl₂-P is considered to directly describe available P forms, because the dilute salt solution has more or less the same ionic strength as the average salt concentration in many soil solutions.

The amount of AL-P may be two orders of magnitude greater than that of CaCl₂-P. Previous studies suggested that the relationship between AL-P and CaCl₂-P was influenced by soil parameters. Regression analysis between AL-P and CaCl₂-P showed medium or strong correlations when using soils with homogeneous soil properties, while there was a weak correlation between them for soils with heterogeneous properties.

The objective of this study was to increase the accuracy of the conversion between AL-P and CaCl₂-P, by constructing universal equations that also take soil properties into consideration.

The AL-P and CaCl₂-P contents were measured in arable soils (n=622) originating from the Hungarian Soil Information and Monitoring System (SIMS). These soils covered a wide range of soil properties.

A weak correlation was found between AL-P and CaCl₂-P in SIMS soils. The amounts and ratio of AL-P and CaCl₂-P depended on soil properties such as CaCO₃ content and texture. The ratio of AL-P to CaCl₂-P changed from 37 in non-calcareous soils to 141 on highly calcareous soils. CaCl₂-P decreased as a function of K_A (plasticity index according to Arany), which is related to the clay content, while the highest AL-P content was found on loam soils, probably due to the fact that a high proportion of them were calcareous.

The relationships between AL-P, CaCl₂-P and soil properties in the SIMS dataset were evaluated using multiple linear regression analysis. In order to select the best model the Akaike Information Criterion (AIC) was used to compare different models. The soil factors included in the models were pH_{KCl}, humus and CaCO₃ content to describe AL-P, and K_A, CaCO₃ content and pH_{KCl} to describe

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CaCl₂-P. AL-P was directly proportional to pH_{KCl}, humus and CaCO₃ content, while CaCl₂-P was inversely proportional to K_A, CaCO₃ content and pH_{KCl}. The explanatory power of the models increased when soil properties were included. The percentage of the explained variance in the AL-P and CaCl₂-P regression models was 56 and 51%, so the accuracy of the conversion between the two extraction methods was still not satisfactory and it does not seem to be possible to prepare a universally applicable equation. Further research is needed to obtain different regression equations for soils with different soil properties, and CaCl₂-P should also be calibrated in long-term P fertilization trials.

Keywords: 0.01 M CaCl₂, ammonium-lactate (AL), extractant, phosphorus, multiple regression

Introduction

The ammonium-lactate acetic acid (AL) extractant is traditionally used in routine soil testing in Hungary. The acidic solution (buffered at pH 3.75) extracts more phosphorus than the readily available pool, because it is also able to dissolve phosphorus (P) reserves (NOVOZAMSKY and HOUBA, 1987). As the accessibility of the P reserves depends on soil parameters such as CaCO₃ content, pH, humus content and soil texture (MENGEL and KIRKBY, 2001; BLUME et al., 2016), an AL-P correction model was elaborated for Hungarian soil conditions, converting AL-P values to standard soil properties (plasticity index according to Arany: 37 /loam/; pH_{KCl}: 6.8; CaCO₃: 0.1%) (THAMM, 1980; SARKADI et al., 1987; CSATHÓ, 2002).

The need to characterize the mobile nutrient pool led to the development and testing of new extraction methods in Hungary, including hot water percolation (FÜLEKY and CZINKOTA, 1998) or extraction with 0.01 M CaCl₂ (HOUBA et al., 1991).

The advantages of the 0.01 M CaCl₂ method were summarized by HOUBA et al. (1991, 2000) and LOCH (2006) as follows: the dilute salt solution has a moderate dissolving and ion exchanging effect; 0.01 M CaCl₂ solution has more or less the same ionic strength as the average salt concentration in many soil solutions; macro- and micronutrients or pollutants are also measurable in 0.01 M CaCl₂ solution, so this method could be an alternative to the many extraction procedures, allowing us to measure relationships and ratios between the amounts of different elements; easily soluble and oxidizable organic N, P and S forms are also measurable in the extraction; the soil:extractant suspension can be easily filtered.

0.01 M CaCl₂-P is considered to be an intensity parameter (FOTYMA et al., 1999; JÁSZBERÉNYI and LOCH, 2001; KULHÁNEK et al., 2008). According to WUENSCHER et al. (2015), CaCl₂-P is of the same order of magnitude as water-extractable P, and there is a strong, significant correlation between them. In some cases, depending on the soil-water ratio, water extraction may extract higher amounts of P than 0.01 M CaCl₂, as an effect of Ca-phosphate formation (KULHÁNEK et al., 2007).

As to the relationship between traditional Hungarian extraction methods and 0.01 M CaCl₂, it can be concluded that there is a close correlation in the case of nitrate-N and a medium close correlation in the case of K (LOCH and JÁSZBERÉNYI, 1997; HOUBA et al., 1986). It is important to note that the amount of AL-P may be two orders of magnitude greater than that of CaCl₂-P, and that the ratio of AL-P and CaCl₂-P depends on the soil properties (HOUBA et al., 1991.; KÜCKE et al., 1995; BERTA-SZABÓ, 2010). Therefore, the regression analysis between AL-P and CaCl₂-P showed a medium or strong correlation ($r=0.61-0.96$) when using soils from long-term experiments (KÜCKE et al., 1995; CSATHÓ et al., 2011), while the inclusion of a wide variety of soils in the study resulted in a weak correlation ($r=0.04-0.38$) (HOUBA et al., 1991). The relationship between AL-P and CaCl₂-P is influenced by soil parameters, such as pH and CaCO₃ content (HOUBA et al., 1991; BERTÁNE SZABÓ, 2010). Previous research did not include soil parameters in the regression analysis.

The aim of the present research was to increase the accuracy of the conversion between the two extraction methods, so soil parameters were taken into consideration during regression analysis on the extracted CaCl₂-P and AL-P values.

Materials and methods

Experimental soils

To study the relationship between AL-P and CaCl₂-P, the P content was determined in 622 arable soils originating from the Hungarian Soil Information and Monitoring System (SIMS). The soil samples, which were collected by the Hungarian Plant and Soil Conservation Services, originated from various areas of Hungary, covering a wide range of texture, organic matter content and pH (*Table 1*). The soils were sampled in September and October 2007. Samples were taken with an auger at 0-20 cm depth, air-dried and passed through a 2-mm sieve. Large root fragments found after sieving were discarded, along with all soil particles larger than 2 mm.

Chemical and physical analyses

The CaCl₂-P fraction was measured using the CaCl₂ extraction method (HOUBA et al., 2000). P was extracted from dried soil by shaking 5.00 g of soil with 0.01M CaCl₂ for 2 hours at 20 °C, at a soil-solution ratio of 1:10, followed by centrifugation. The concentration of CaCl₂-P in the supernatant was determined spectrophotometrically using a Continuous Flow Analyzer (Skalar, the Netherlands, details in: HOUBA et al., 2000), while AL-P was determined with a UV-VIS spectrophotometer according to EGNÉR et al. (1960). The soil-solution ratio was 1:20 and the shaking time was 2 hours.

The pH, the plasticity index according to Arany (K_A) and the humus (soil organic matter expressed in %) and CaCO₃ contents were determined using standard analytical procedures in the laboratories of the Hungarian Plant and Soil Conservation Services. Soil organic matter was determined by dichromate and sulphuric acid digestion of the organic matter (MSZ-08-0452-80). The pH was

measured in a settling 1:2.5 (w/v) suspension of soil in 1 M KCl solution (MSZ-08-0206/2-78). The plasticity index according to Arany (K_A), which can be used to distinguish between different types of soil texture classes, was determined by the standard test method (MSZ-21470/51-83). The K_A value represents the amount of water taken up by the soil to plasticity capacity ($\text{cm}^3/100\text{g}$). The CaCO_3 content was determined with the Scheibler method (MSZ-08-0206/2-78).

Table 1
Soil properties of the SIMS soils (samples collected in 2004)

Arable soil category*	pH_{KCl}	K_A^{**}	Humus***	CaCO_3	m/m %		
					Clay	Silt	Sand
Chernozem (n=161)	6.94	42	2.68	6.01	23.3	45.6	31
Brown forest soils (n=174)	5.53	39	1.69	0.76	24.2	41.6	34.2
Meadow (n=97)	6.44	52	3.13	5.81	34.8	41.5	23.6
Sandy soils (n=50)	6.35	26	0.96	2.62	6.1	9.3	84.6
Saline soils (n=53)	6.88	49	2.6	4.51	34.8	42.6	22.6
Shallow soils (n=87)	6.16	43	2.21	5.52	23.6	43.8	32.6
Total (n=622)	6.29	42	2.25	3.99	25	40.4	34.6

Note: *Each category includes soil types with similar fertility and agronomic properties (according to the Hungarian fertilization advisory system; BUZÁS & FEKETE, 1979); ** plasticity index according to Arany: water quantity taken up by soil to plasticity capacity ($\text{cm}^3/100\text{g}$); ***soil organic matter content

Statistical analysis

All data were checked for normality using descriptive statistics (skewness and kurtosis) and graphical methods. If the value of skewness for the data was more than twice the standard error, the data were transformed using a natural log transformation (Table 2). Pearson correlation tests were used to evaluate the relationships between individual variables. The relationships between AL-P, CaCl_2 -P and soil properties in the SIMS dataset were evaluated using multiple linear regression analysis. The variance inflation factor (VIF) was used for filtering the multicollinearity of the explanatory variables (FOX and MONETTE, 1992). If VIF was > 4 for an explanatory variable, it was omitted from the analysis. Ordinary least square (OLS) regression was used to determine the regression coefficients. All the possible regression models (2^n , where n means the number of possible explanatory variables) were determined using the all subset regression method. The Akaike Information Criterion (AIC) was used to select the best model (AKAIKE, 1973, 1974). AIC was calculated as follows:

$$\text{AIC} = (-2)\log(\text{maximum likelihood}) + 2(\text{number of independently adjusted parameters within the model})$$

Based on this equation, AIC considers both the fit of the model and the number of explanatory variables. The aim was to choose the model with the best

explanatory power and the fewest parameters. A lower AIC number means a better model.

Table 2
Changes in the skewness and kurtosis of the distribution as the effect of logarithmic transformation

Soil parameters	Skewness	Kurtosis
CaCl ₂ -P	2.24	5.44
ln (CaCl ₂ -P)	-0.47	0.45
AL-P	1.63	2.90
ln (AL-P)	-0.52	0.34
Humus	1.13	2.73
ln (humus)	-1.01	2.14

Results and discussion

The relationship between AL-P and CaCl₂-P in SIMS soils is presented in *Figure 1*. When the CaCl₂-P values were plotted against AL-P, the correlation was weak, so the P values determined with the two methods could not be converted into each other. This can be explained by the heterogeneous soil conditions of the soils used for this study.

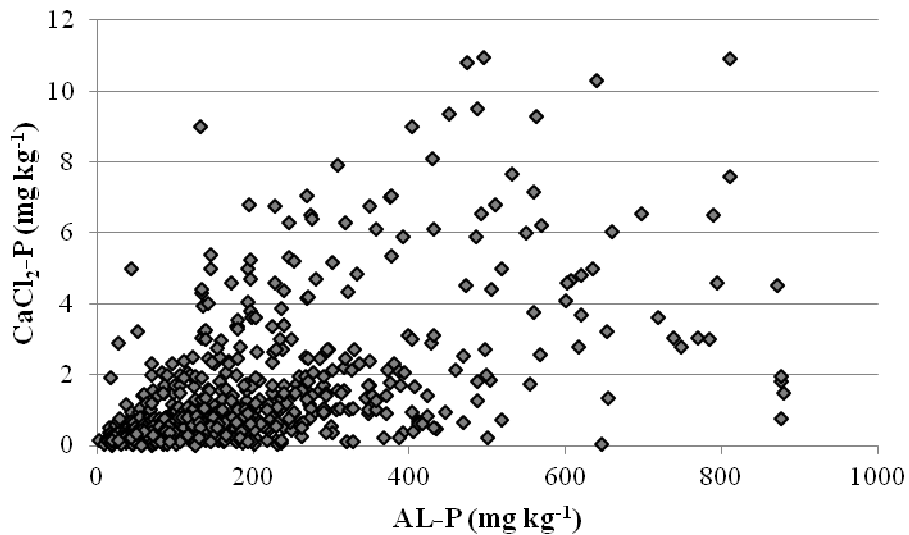


Figure 1
Relationship between AL-P and CaCl₂-P in SIMS (Soil Information and Monitoring System) soils covering a wide range of soil properties (n=622)

As the soil CaCO_3 content influences the relationship between AL-P and $\text{CaCl}_2\text{-P}$, the soil samples were classified based on their CaCO_3 content (*Table 3*).

Table 3
CaCl₂-P and AL-P contents of soils with different CaCO₃ content

CaCO ₃ content**	N	CaCl ₂ -P (mg kg ⁻¹)			AL-P (mg kg ⁻¹)			AL/CaCl ₂
		average	min.	max.	average	min.	max.	
Non-calcareous	302	1.86 ^{a*}	0.01	18.5	68 ^a	1.3	510	37
Slightly calcareous	148	1.69 ^a	0.05	11.0	110 ^{bc}	8.7	470	65
Moderately calcareous	142	1.31 ^a	0.01	6.8	130 ^c	7.4	420	99
Highly calcareous	30	0.69 ^b	0.05	6.8	96 ^b	19.2	227	141
Total	622	1.64			94			57

* Means designated by the same letter did not differ significantly at the P = 5% level; ** CaCO₃ content: 0 – non-calcareous, 0.1-4.9 – slightly calcareous, 5-19.9 – moderately calcareous, >20 – highly calcareous

The $\text{CaCl}_2\text{-P}$ content of highly calcareous soils was significantly lower than that of other soils, due to the low solubility of Ca-phosphate compounds. An opposite trend was detectable in the case of AL-P. The AL-P content of moderately calcareous soils was nearly two times higher than that of non-calcareous soils. This can be explained by the acidic nature of the extraction solution, which caused the dissolution of Ca-phosphate reserves. The ratio of AL-P to $\text{CaCl}_2\text{-P}$ changed from 37 in non-calcareous soils to 141 on highly calcareous soils.

As the soil texture also affects the availability of phosphate compounds, the average $\text{CaCl}_2\text{-P}$ and AL-P contents of soils with different texture were studied (*Table 4*). There were significant differences between the average $\text{CaCl}_2\text{-P}$ and AL-P contents of the soil texture categories. $\text{CaCl}_2\text{-P}$ decreased from coarse-textured to heavy-textured soils, while the highest AL-P content was found on loam soils. The ratio of AL-P to $\text{CaCl}_2\text{-P}$ was higher in heavier soils.

As the ratio of AL-P to $\text{CaCl}_2\text{-P}$ was found to be influenced by the CaCO_3 content and soil texture, the quantitative relationship between AL-P and $\text{CaCl}_2\text{-P}$ was measured by multivariate regression analysis taking into account the CaCO_3 content and K_A of the soils. Additionally, pH_{KCl} and humus content were used as independent variables, due to the importance of these soil parameters for P accessibility.

Table 4
CaCl₂-P and AL-P contents of soils with different texture

Soil texture**	n	CaCl ₂ -P (mg kg ⁻¹)			AL-P (mg kg ⁻¹)			AL-P/CaCl ₂ -P
		average	min.	max.	average	min.	max.	
Coarse sand	48	3.15 ^a *	0.16	10.95	80 ^a	7.4	347	25
Sand	51	2.57 ^a	0.05	10.80	86 ^a	12.2	354	34
Sandy loam	104	1.76 ^b	0.10	9.50	91 ^{ab}	8.3	470	52
Loam	143	1.82 ^b	0.04	18.50	117 ^b	1.3	510	64
Clay loam	145	1.21 ^{bc}	0.01	10.30	96 ^{ab}	5.2	428	80
Clay	86	0.97 ^c	0.01	9.00	79 ^a	7.0	380	81
Heavy clay	45	0.80 ^c	0.02	6.20	70 ^a	11.4	285	87
Total	622	1.64	-	-	94	-	-	57

* Means designated by the same letter did not differ significantly at the P = 5% level; ** K_A (plasticity index according to Arany) was used to distinguish between soil texture categories

As soil parameters may be related to each other, correlations between them were investigated (Table 5), and correlations were found between CaCO₃ and pH_{KCl} and between K_A and humus. A close correlation between the explanatory variables may cause problems due to the multicollinearity of the model. In the present case the correlation matrix indicated only moderate collinearity, so these parameters were not excluded from the regression analysis.

Table 5
Correlations between soil parameters

	CaCl ₂ -P	AL-P	pH _{KCl}	K _A	Humus	CaCO ₃
CaCl ₂ -P	1					
AL-P	0.547 ^{**}	1				
pH _{KCl}	-0.020	0.377 ^{**}	1			
K _A	-0.293 ^{**}	-0.043	0.038	1		
Humus	-0.113 ^{**}	0.163 ^{**}	0.104 [*]	0.644 ^{**}	1	
CaCO ₃	-0.127 ^{**}	0.284 ^{**}	0.590 ^{**}	0.033	0.143 ^{**}	1

Note: * Significant at the 0.05 level; ** Significant at the 0.01 level

The relationship between AL-P and CaCl₂-P is controlled by several parameters, some of which are not known, so different equations are needed for calculating AL-P and CaCl₂-P. The number of possible models including 0, 1, 2, 3 or all of the above-mentioned soil parameters (CaCO₃, K_A, pH_{KCl} and humus) is 16 for both AL-P and CaCl₂-P. The Akaike Information Criterion (AIC) was utilized to compare the models and to decide which variables should be included.

Two models for predicting AL-P, one involving only CaCl₂-P and one also including the variables selected based on AIC, are presented in *Table 6*. Besides CaCl₂-P the most important soil parameters were pH_{KCl}, humus and CaCO₃ content.

The explanatory power of the models is also presented. The coefficient of correlation increased from 0.57 to 0.75 when soil parameters were included. The proportion of the variation in the dependent variable explained by the regression model also increased from 32 to 56%. The standard error of the estimate decreased, suggesting that the accuracy of the prediction increased.

Table 6
Explanatory power of the models explaining AL-P

Independent variables included in the model	r*	R square**	Adjusted R square	SE***
ln (CaCl ₂ -P)	0.569	0.324	0.323	0.730
ln (CaCl ₂ -P), pH _{KCl} , ln humus, CaCO ₃	0.749	0.562	0.559	0.589

Note: *Coefficient of correlation; **Coefficient of determination; ***Standard error of the estimate

The multiple regression equations were the following:

$$\ln \text{AL-P} = 0.404 \ln \text{CaCl}_2\text{-P} + 4.23$$

$$\ln(\text{AL-P}) = 0.434 \ln(\text{CaCl}_2\text{-P}) + 0.238 \text{pH}_{\text{KCl}} + 0.29 \ln(\text{humus}) + 0.024 \text{CaCO}_3 + 3.303$$

Models for predicting CaCl₂-P, either involving only AL-P or also including variables selected using AIC are presented in *Table 7*. Besides AL-P the most important soil parameters were K_A, CaCO₃ content and pH_{KCl}. These soil properties were also used in the AL-P correction model as modifying factors (THAMM, 1980; SARKADI et al., 1987). As indicated by the correlation matrix, CaCl₂-P was inversely proportional to these parameters, so the reciprocals of K_A and pH_{KCl} were included in the model. In the case of CaCO₃ content the reciprocal transformation is not applicable, because its value may be zero.

Table 7
Explanatory power of models explaining CaCl₂-P

Independent variables included in the model	r*	R square**	Adjusted R square	SE***
ln (AL-P)	0.569	0.324	0.323	1.029
ln (AL-P), 1/K _A , CaCO ₃ , 1/pH _{KCl}	0.715	0.511	0.508	0.877

Note: *Coefficient of correlation; **Coefficient of determination; ***Standard error of the estimate

The coefficient of correlation increased from 0.57 to 0.72 when soil parameters were included. The explained variance also increased from 32 to 51%, while the standard error of the estimate decreased.

The multiple regression equations for predicting CaCl₂-P were the following:

$$\ln(\text{CaCl}_2\text{-P}) = 0.802 \ln(\text{AL-P}) - 4.187$$

$$\ln(\text{CaCl}_2\text{-P}) = 0.96 \ln(\text{AL-P}) + 50.577 (1/K_A) - 0.046 \text{CaCO}_3 + 5.017$$

$$(1/\text{pH}_{\text{KCl}}) - 6.946$$

Conclusions

In accordance with the results of previous studies, a medium correlation was found between AL-P and CaCl₂-P, which can be explained by the wide range of soil properties. The effect of CaCO₃ content differed for the two methods. AL-P increased as a function of CaCO₃ content, due to the fact that it is able to dissolve calcium-phosphate reserves, while CaCl₂-P decreased as the CaCO₃ content increased. The effect of soil texture also differed for AL-P and CaCl₂-P. The results suggest that the regression equations obtained for soils with similar soil properties (e.g. in long-term experiments) are not universally applicable for conversion between the two extraction methods in practice, even in the case of a strong correlation.

Regression models that included soil properties such as pH_{KCl}, K_A, CaCO₃ and humus content explained 56 and 51% of the variation in AL-P and CaCl₂-P (n=622). However, the accuracy of the conversion between the two extraction methods showed that the regression equations obtained were still not appropriate for universal application.

Therefore, further research is needed to obtain different regression equations for soils with different soil properties, which will allow conversion between the two extraction methods. Before using CaCl₂-P in the fertilizer recommendation system, calibration in long-term P fertilization trials will also be required.

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