https://doi.org/10.34101/actaagrar/150/1739

Study of factors controlling the amount of 0.01 M $CaCl_2$ extractable N_{org} fraction

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Summary

The use of new methods describing the "readily available" nutrient content of the soil is spreading on a global scale. The 0.01 M $CaCl_2$ extractant is a dilute salt solution in which the easily soluble inorganic (nitrate-N and ammonium-N) and organic N fractions, P, K and micronutrients are also measurable. The 0.01 M $CaCl_2$ has been tested in the University of Debrecen, Institute of Agricultural Chemistry and Soil Sciences since the 90's. The results of the researches related to organic N fraction, performed in the last decades, and the results of the present study (originating from the long-term experiment of Karcag, 2007–2009) can be concluded as follows:

The measurement of easily soluble and oxidizable organic nitrogen (N_{org}), besides inorganic fractions, could improve the nutrient management.

The amount of the N_{org} fraction is determined by the soil conditions, therefore it is considered to be a site-specific parameter.

Management practices and cropyear affect the amount of N_{org} as well. The present research confirmed that, the effect of fertilization on the amount of N_{org} can be explained by the changing of the yield (related to total biomass production), while the effect of cropyear is related to the differences in mineralization circumstances and yield as well.

The measurement of the N_{org} fraction is increases the accuracy of N-supply, therefore it could prevent the environmentally harmful excess N application as well.

Keywords: 0.01 M $CaCl_2$, long-term fertilization, liming, cropyear

Introduction

The need for the measurement of "readily available" nutrient content rather than nutrient reserves of the soil is increasing worldwide since the 50's. Therefore the use of water (Sissingh, 1971) and dilute salt solutions (Schachtschabel, 1954; Schofield, 1955; Houba et al., 1990) as an extractant is spreading. Among dilute salt solutions, the 0.01 M $CaCl_2$ extraction method has been investigated in the last decades, and the nutrient contents, extracted by 0.01 M $CaCl_2$, are considered to be related to nutrient intensity (Diest et al., 1993; Fotyma et al., 1996; Baier és Baierova, 1997; Fotyma et al., 1999; Jászberényi and Loch, 2001; Kulhánek et al., 2008).

Inorganic and organic N fractions, P, K and micronutrients are also measurable in 0.01 M $CaCl_2$, therefore ratios between the amounts of different elements and fractions could be also studied. Other advantages of the method are: the soil:extractant suspension can be easily filtered; the 0.01 M $CaCl_2$ solution has the same ionic strength as the average salt concentration in soil solution (Houba et al., 1991, 2000; Loch, 2006).

The applicability of 0.01 M $CaCl_2$ as an extractant has been studied in the University of Debrecen, Institute of Agricultural Chemistry and Soil Sciences since the 90's (Jászberényi et al., 1994; Jászberényi and Loch, 1995; Lazányi and Loch, 2006; Loch, 2006). The most important results are concluded by Loch (2006) as follows:

In the case of pH, nitrate-N, Mg and K contents the amounts determined by $CaCl_2$ and conventional methods are closely related, therefore $CaCl_2$ and the conventional extractants are replaceable.

P excess or deficiency could be detectable by $CaCl_2$ method more accurately than by AL method (ammonium-lactate acetic acid, standard method of Hungary).

The organic (N_{org}) and inorganic (ammonium-N, nitrate-N) N fractions extracted by $CaCl_2$ are related to EUF N fractions.

The amount of N_{org} was on average 34% of $CaCl_2$-N_{total} fraction in Hungarian soils.

The exact determination of the soil N supply is essential from an economical and an environmental point of view as well, therefore the measure of available N forms, beside reserves, is also important.

Total N content of the topsoil is between 0.02–0.4% (Mengel and Kirkby, 2001). More than 90% of total N is fixed in an organic form (Blume et al., 2016), thus the amount of total N is closely related to organic matter content (OM). OM content could be divided to passive/stabile and active/labile pools (Brady and Weil, 2004). A part of the labile OM pool consists of dissolved organic compounds (dissolved organc matter – DOM). DOM considered to be an important fraction regarding the movement of nutrients in the soil profile (Murphy et al., 2000), and also influences microbial activity (Chantigny, 2003). However the amount of N fixed in DOM is relatively small – according to Haynes (2005) the maximum concentration of extractable organic nitrogen (EON) is typically only 5% of the total N in agricultural soils – it is often considered to be the most dynamic and bioavailable organic fraction of the soil N cycle. The main constituents of this fraction are free and combined amino compounds such as peptides and proteins (Matsumoto and Ae, 2004, Paul and Williams, 2005). Because of its dynamic nature, EON has been used as an estimate of the N supplying capacity of soils (Ros et al., 2011) and as an indicator of the effects of changes in soil and fertilizer management (Wander, 2004; Haynes, 2005). Among numerous soil extraction methods, which are used to measure EON, the organic N fraction (N_{org}) extractable by 0.01 M $CaCl_2$ has been investigated intensively (Houba et al., 1986; Appel and Mengel, 1993, 1992, 1990, 1998; Groot and Houba, 1995; Appel, 1998; Kulcsár and Jászberényi, 1999; Nunan et al., 2001; Lazányi et al., 2002; Bregliani et al., 2010; Ros et al., 2011).

Mengel et al. (1999) found that $CaCl_2$-N_{org} fraction is related to a dynamic N pool originated from the yearly produced plant materials and organic fertilizers. Similarly, Ros et al. (2010) observed a very dynamic behavior of N_{org} after crop residue addition. Incorporation of residues immediately increased the amount of N_{org}, than it stabilized at a concentration that was slightly higher than the concentration in the control soil within 10–30 days. Researchers found significant relation between $CaCl_2$-N_{org} and microbial biomass (Olfs and Werner, 1989; Madsen et al., 1994; Černý et al., 2003), but it does not means that $CaCl_2$ method extract N from microbial biomass (Ros et al., 2010), rather there is an indirect relation between these.

The N_{org} fraction is controlled by abiotic and biotic processes such as mineralization and immobilization, adsorption and desorption, precipitation and solubilization. Variation in environmental factors and management practices are therefore likely to affect the size of the N_{org} fraction. Among environmental factors, the effect of soil properties on the amount of N_{org} fraction is significant (Ros et al., 2009; Bertáné, 2016). The most important soil properties affecting N_{org} are the humus content, what might be explained by its strong correlation with total N, and soil texture, which could be related to mineralization conditions.

According to our previous results N_{org} is a site-specific parameter (Bertáné, 2016). The average N_{org} content of chernozem (N=184), meadow (N=97) and sandy (N=50) soils, originating from the Hungarian Soil Information and Monitoring System, is 9, 12 and 5.9 mg kg^{-1}, respectively, and differ significantly.

Ros et al. (2009) also found that management factors as organic and inorganic fertilization had a significant influence on N_{org} levels in soil. However, this effect of fertilization seems to be short-term since Chantigny (2003) suggested that N_{org} levels on the long term are determined by vegetation type. Cropyear is another factor controlling many soil processes and hence it affects soil N_{org} content.

Understanding the impact of management practices and cropyear on the dynamics of N_{org} could lead to an increase in the sustainability of agriculture and more accurate soil tests to predict N mineralization.

Therefore the aim of this study was to measure the effect of cropyear and fertilizer management on N_{org} fraction. Under field conditions, the effect of management practices might be hard to study, because many factors influence N_{org} at the same time. Therefore, the present study is based on three years results derived from a long-term experiment (management practices differed for more than 40 years), where the effects of treatments could be measured, even in the case of the same soil type and climatic conditions.

Materials and methods

Experimental soils

The effects of management factors and cropyear were studied in the experiment of Karcag (Karcag Research Institute of the University of Debrecen) which is located in the Great Hungarian Plain, and was established in 1967.

The arrangement of the small-plot field experiment is split-plot with four replications. The sequence in the crop rotation is: winter wheat – maize – maize – winter wheat. The fertilization treatments are summarized in *Table 1*.

Table 1. *Fertilizer rates applied in the experiment of Karcag (kg ha^{-1}) (1971–2007)*

Treatments	1971–1987			1987–2007		
	N	P	K	N	P	K*
$N_0P_0K_0$	-	–	–	-	–	–
$N_1P_0K_0$	50	–	–	100	–	–
$N_1P_1K_0$	50	22	–	100	26	–
$N_1P_2K_0$	50	44	–	100	52	–
$N_2P_0K_0$	100	–	–	150	–	–
$N_2P_1K_0$	100	22	–	150	26	–
$N_2P_2K_0$	100	44	–	150	52	–
$N_3P_0K_0$	150	–	–	200	–	–
$N_3P_1K_0$	150	22	–	200	26	–
$N_3P_2K_0$	150	44	–	200	52	–
$N_1P_0K_1$	50	–	83	100	–	83/166
$N_1P_1K_1$	50	22	83	100	26	83/166
$N_1P_2K_1$	50	44	83	100	52	83/166
$N_2P_0K_1$	100	–	83	150	–	83/166
$N_2P_1K_1$	100	22	83	150	26	83/166
$N_2P_2K_1$	100	44	83	150	52	83/166
$N_3P_0K_1$	150	–	83	200	–	83/166
$N_3P_1K_1$	150	22	83	200	26	83/166
$N_3P_2K_1$	150	44	83	200	52	83/166
$N_4P_3K_2$	200	65	83	250	79	83/207

Note: * 83 kg K ha^{-1} for winter wheat 166 and 207 kg K ha^{-1} for maize.

Ammonium-nitrate was used as N-fertilizer in split-application (50% in autumn, 50% in spring), the full PK-doses were distributed in autumn. On the 1^{st} and 3^{rd} replications in 1991 and 2003, 14.5 and 11.05 t ha^{-1} of lime was applied respectively. The tillage system was based on autumn ploughing. The soil of the experiment is Luvic Phaeosem/Calciustolls (FAO/USDA soil type). The main properties of the experimental soil: 5–5.5 pH_{KCl}; clay loam texture; 2.5–2.9% humus content; 90 cm deep humus layer; P-supply is very weak; K-supply is good (Buzás and Fekete, 1979). The climate is warm temperate continental, and the mean annual precipitation is 527 mm. Representative soil samples (0–20 cm depth) were taken from each plot after the harvest in 2007, 2008 and 2009. All crops were harvested at the ground level and removed from the plots.

Chemical analyses

Soil samples were air dried and sieved (<2 mm) for further analysis. We measured the N_{org} contents of the soil samples in 0.01 M $CaCl_2$ solution with 1:10 soil:solution ratio (Houba et al., 1990). During the extraction, samples were shaken for two hours, centrifuged in a bench centrifuge (MLW T54 centrifuge, Democratic Republic of Germany) at 2500 rpm for 10 minutes and the nitrate-, ammonium- and total N contents of the supernatant were measured with an autoanalyzer (Continuous Flow Analyzer, Scalar SANPLUSSYSTEM, Scalar, Breda, the Netherlands) (Houba et al., 2000). The N_{org} was calculated by subtracting nitrate- and ammonium-N from total N.

Statistical analyses

All statistical analyses were performed with SPSS for Windows, version 13 (SPSS Inc., Chicago, Illinois). The evaluation of the effects of management factors on N_{org} was performed by General Linear Model Univariate Analyses. The treatments were four N, three P and two K fertilizer rates, and liming. The model includes N, P, K fertilizer rates, liming and the interactions of these (GLM table is not shown). The effect of cropyear was studied by univariate ANOVA. Significant differences were examined by Duncan post hoc test. The model fits were expressed as coefficients of determination (R^2).

The relationship between N_{org} and yield was measured by linear regression analysis.

Results and discussion

The N_{org} contents of the treatments in 2007, 2008 and 2009 are presented in *Table 2*. The difference between the average N_{org} content of the treatments could reach 72.3% in 2008. Significant differences were observable between the average N_{org} contents of each studied year. The largest average N_{org} content was found in 2008, it was 70% higher than that of in 2007. It means that there is a high variability in the amount of N_{org} as an effect of cropyear.

Table 2. *N_{org} content of the treatments in the long-term fertilization experiment of Karcag (mg kg^{-1})*

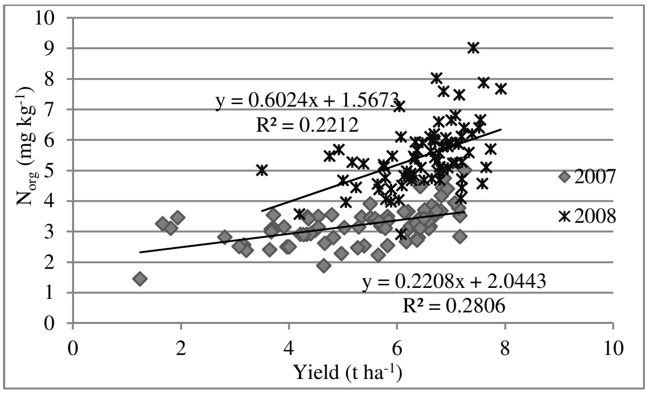
Treatments	2007		2008		2009	
	Mean	SD*	Mean	SD	Mean	SD
$N_0P_0K_0$	2.82	0.92	4.62	0.73	4.48	2.01
$N_1P_0K_0$	2.84	0.37	4.22	0.89	2.87	1.39
$N_1P_1K_0$	3.13	0.42	5.23	0.75	3.42	0.95
$N_1P_2K_0$	3.53	0.23	5.68	0.74	3.73	0.28
$N_2P_0K_0$	2.90	0.54	4.61	0.76	4.30	2.11
$N_2P_1K_0$	3.33	0.26	5.43	1.01	3.86	0.67
$N_2P_2K_0$	3.66	0.43	5.25	1.18	4.47	1.58
$N_3P_0K_0$	2.97	0.38	5.51	0.65	3.23	1.10
$N_3P_1K_0$	3.37	0.19	6.03	1.15	3.84	2.57
$N_3P_2K_0$	3.92	0.34	5.69	0.87	4.30	1.85
$N_1P_0K_1$	3.22	0.18	4.59	0.54	3.58	1.33
$N_1P_1K_1$	2.83	0.54	5.85	0.70	3.70	1.97
$N_1P_2K_1$	2.91	0.43	5.98	1.34	4.24	1.82
$N_2P_0K_1$	2.58	0.51	5.03	0.73	2.50	1.61
$N_2P_1K_1$	3.14	0.35	5.36	0.60	4.10	1.14
$N_2P_2K_1$	2.78	0.32	5.64	0.25	4.64	1.70
$N_3P_0K_1$	2.85	0.41	5.49	0.68	3.30	0.36
$N_3P_1K_1$	3.51	0.27	5.52	0.63	4.59	1.62
$N_3P_2K_1$	3.70	0.59	6.46	1.37	4.74	1.04
$N_4P_3K_2$	4.47	0.58	7.27	1.74	4.67	1.10
Total	3.22^{a**}	0.60	5.47^{b}	1.06	3.93^{c}	1.47

Note: * standard deviation, ** means designated by the same letter were not significantly different at P=0.05 level. Remark: treatments are 0 (N_0), 100 (N_1), 150 (N_2), 200 (N_3), 250 (N_4) kg N ha^{-1}; 0 (P_0), 26 (P_1), 52 (P_2), 79 (P_3) kg P ha^{-1}; 83 (for wheat)/166 (for corn) (K_1), 83 (for wheat)/207 (for corn) (K_2) kg K ha^{-1} rates in four replications.

In the present study the effect of yield – which is in close relation with total plant biomass production and also with root density and activity in the soil – on N_{org} was observable (*Figure 1*). Significant correlation was found between the winter wheat yield and N_{org} content in 2007 and 2008.

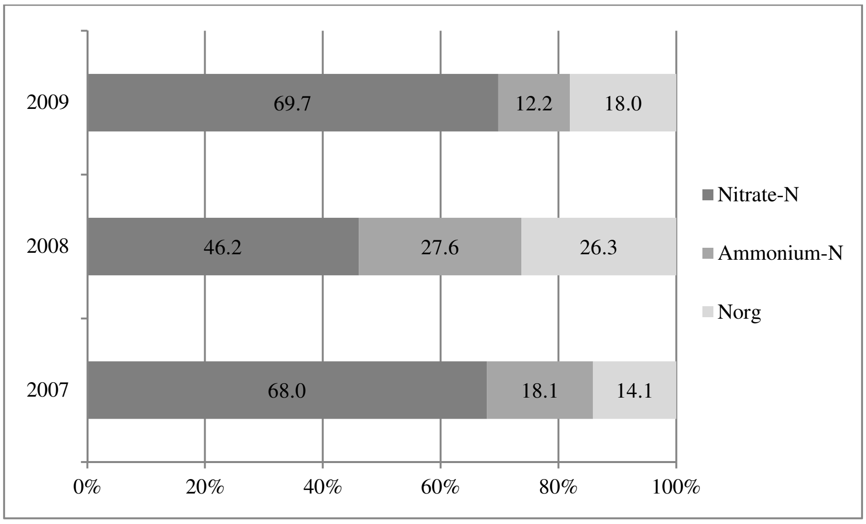
The coefficient of correlation was 0.47 in 2007 and 0.53 in 2008. By the increasing winter wheat yield, the amount of N_{org} fraction increased in 2007 and in 2008 as well, however in 2009, the relation between corn yield and N_{org} was not observable. These results can be explained by that the higher the yield is (e.g. as an effect of fertilization), the higher the amount of plant residues is, which constitute the main part of easily degradable organic matter. In addition, fertilization may increase soil microbial biomass due to an increase in root biomass and exudates.

Figure 1. *Relationship between $CaCl_2$-extractable N_{org} and winter wheat yield in the long-term fertilization experiment of Karcag*



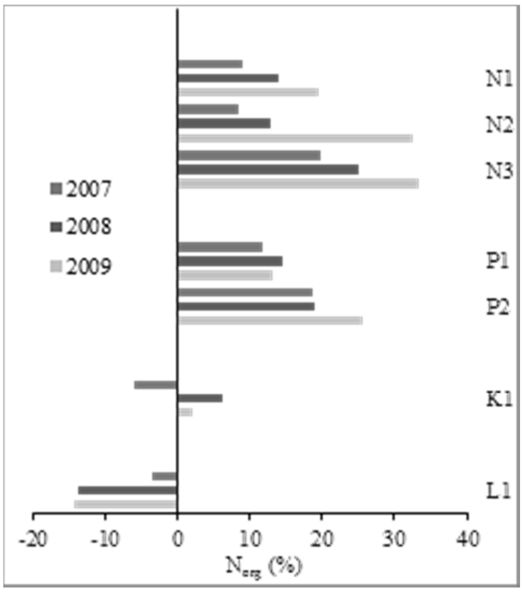
As the amount of N_{org} was influenced by cropyear, the importance of this fraction compared to inorganic N fractions also changed (*Figure 2*). In 2008, 26.3% of $CaCl_2$-extractable total N was in organic form, while in 2007 the ratio of N_{org} was only 14.1%.

Figure 2. *The ratio of $CaCl_2$-extractable N fractions in the long-term fertilization experiment of Karcag*



We studied the effect of long-term inorganic fertilization and liming on N_{org} fraction in the 2007, 2008 and 2009 cropyears (*Figure 3*). Significant and positive effect of P fertilization was observable in each studied year (2007: P=0.001; 2008: P=0.001; 2009: P=0.05) which is related to the fact that the P-supply of the experimental soil is very weak, therefore P fertilization increased the yield. N fertilization significantly increased (P=0.05) N_{org} in 2007 and in 2008, while K fertilization decreased N_{org} in 2007 (P=0.05). The N_{org} content of plots, where lime was applied in 1991 and in 2003, was significantly lower in 2008 comparing them to plots without liming. It can be related to the large difference between the pH_{KCl} of not limed (pH_{KCl}=4.4) and limed plots (pH_{KCl}=6.1), which can cause problems regarding the availability of some nutrients (e.g. most of the micronutrients). In 2007, 2008 and 2009, 70, 75 and 54% of the variation in N_{org} could be explained by management factors (long-term fertilization and liming), respectively. The effect of N and P fertilization could reach a 25% significant increase in the amount of N_{org} (*Figure 3)*.

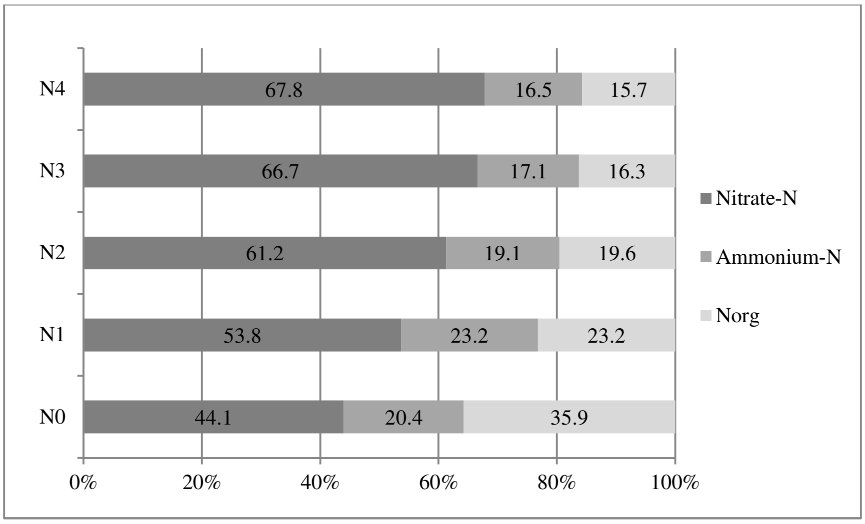
Figure 3. *The effect of long-term NPK fertilization and liming on the amount of N_{org} fraction (long-term experiment of Karcag)*



Note: N_{org} (%) means the N_{org} content of treated plots [fertilized with 100 (N_1), 150 (N_2), 200 (N_3) kg N ha^{-1}; 26 (P_1), 52 (P_2) kg P ha^{-1}; 83/166 (K_1) kg K ha^{-1} rates; limed (L1)] compared to control groups (0 kg N ha^{-1}; 0 kg P ha^{-1}; 0 kg K ha^{-1}; without liming).

Mineral fertilization affected each N fraction, especially inorganic N forms, therefore the importance of N fractions also changed as an effect of treatments (*Figure 4).* The ratio of N_{org} in the studied period (2007–2009) was 35.9% of the $CaCl_2$-extractable total N in the control, and only 15.7% in the $N_4P_3K_2$ treatment. It is in the same order of magnitude as inorganic N, which is in accordance with previous studies (Murphy et al., 2000; Chen and Xu, 2005).

Figure 4. *The ratio of $CaCl_2$-extractable N fractions in the long-term fertilization experiment of Karcag*



Conclusions

On the studied chernozem soil, N_{org} content varied between 14–36% of the total soluble N, depending on the cropyear and management practices, therefore the amount of this fraction was in the same order of magnitude as inorganic fractions.

Among management factors, fertilization had a significant effect on N_{org}. The effect of long-term N or P fertilization could reach 25%, but the difference between the average N_{org} content of treatments could reach 72.3%. From 54 to 70% of the total variation of N_{org} could be explained by long-term fertilization and liming. These results confirm that N_{org} is a dynamic N pool, and its amount reflects the effect of management practices.

Cropyear had a major effect on N_{org}, which can be related to different mineralization conditions, microbial activity and biomass production. Even 70% difference was observable between cropyears. However N_{org} is a site-specific parameter, our results suggest that cropyear and management factors also affect the amount of it. Therefore the measurement of this fraction could be important in nutrient management.

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