

## Effects of farmyard manure and nitrogen fertilizers on mobility of phosphorus and sulphur in wheat and activity of selected hydrolases in soil

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**Abstract.** The paper demonstrates the results of research on the mobility of phosphorus and sulphur in winter wheat fertilized with several rates (0, 20, 40, 60, 80 t ha<sup>-1</sup>) of farmyard manure and nitrogen (0, 40, 80, 120 kg N ha<sup>-1</sup>). The content of these nutrients was related to the activity of acid phosphatase and arylsulphatase in a Haplic Luvisol. The highest content of available phosphorus (91.58 mg P kg<sup>-1</sup>) was reported in the soil amended with farmyard manure at the rate of 60 t ha<sup>-1</sup>. The content of sulphates (VI) in the Haplic Luvisol was high and, on average, equal to 25.22 mg kg<sup>-1</sup>. The activity of acid phosphatase in the soil increased with increasing mineral nitrogen rates. The highest content of sulphates (VI) and the lowest activity of arylsulphatase were identified at the nitrogen rate of 40 kg N ha<sup>-1</sup>. The mobility of phosphorus in winter wheat was the highest when farmyard manure at the rate of 60 t ha<sup>-1</sup> and mineral nitrogen at the rate of 120 kg N ha<sup>-1</sup> were incorporated into the soil. The greatest translocation of sulphur was reported at the high farmyard manure rates (40, 60 and 80 t ha<sup>-1</sup>) and the nitrogen rate of 80 kg N ha<sup>-1</sup>.

**Key words:** acid phosphatase, arylsulphatase, phosphorus, soil, sulphur, winter wheat

### INTRODUCTION

Wheat is considered one of the key cereal crops. About 20% of the cropping area in Poland is covered by the plant (Suwara *et al.*, 2007). Plant production is closely dependent on the soil fertility and hence on the availability of nutrients for plants, which are supplied *eg* with fertilizers (Filipek and Skowrońska, 2009). Creating adequate soil environment conditions, especially supplied with nutrients, is an essential condition of optimal crop yields (Kaczor and Łaszcz-Zakorczenianna, 2009). The application of organic and mineral fertilizers changes the chemical, physical, and biological soil properties. Providing soil with organic fertilizers affects

its physical and chemical properties more than that with nitrogen ones. Farmyard manure (FYM) is the most popular natural fertilizer and is considered as one of the most effective fertilizers in the soil environment (Słowińska-Jurkiewicz *et al.*, 2013). Mineral, organic, and natural fertilization also stimulates the development of microorganisms being a main source of enzymes responsible for nutrients transformations in soils (Bielińska and Mocek-Płóćiniak, 2012; George *et al.*, 2002; Krämer and Green, 2000). Hydrolases represent a third class of enzymes that participate in the substrate decomposition processes with a water molecule. Alkaline (EC 3.1.3.1) and acid (EC 3.1.3.2) phosphatases play an important role in soils since they stimulate the transformations of organic phosphorus compounds into inorganic phosphates (HPO<sub>4</sub><sup>2-</sup> and H<sub>2</sub>PO<sub>4</sub><sup>-</sup>) directly accessible to plants and soil organisms (Bielińska and Mocek-Płóćiniak, 2009; Hopkins *et al.*, 2008). The hydrolysis of sulphate esters occurs due to the O-S bond cleavage by the arylsulphatase enzyme (EC 3.1.6.1) (Tabatabai and Bremner, 1970). A long-term static fertilization experiment in which natural and mineral fertilization overlaps under the same climate conditions and in the same soil represents a credible source in solving the problems connected with the circulation of phosphorus, sulphur, and other macro- and microelements.

The aim of the research was to investigate the effects of various rates of FYM and nitrogen fertilizers in the form of mineral salts on the activity of acid phosphatase, arylsulphatase in soil, and the content of phosphorus and sulphur, and to determine the relationships between the studied parameters in the soil – plant system.

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## MATERIAL AND METHODS

Soil was collected from long-term experimental field located at the Agricultural Experimental Station at Grabów on the Wisła River (Mazowieckie Province, Zwoleń County, and Przyłęk Commune, Poland). The location of the experimental station is determined by the altitude (51°21'8''N), longitude (21°40'8''E), and the lowland climate of moderate altitudes. The experiment was set up in 1980 by the Department of Plant Nutrition and Fertilization, the Institute of Soil Science and Plant Cultivation in Puławy, Poland. According to the Polish Soil Classification (2011), the soils of the Agricultural Experimental Station at Grabów are represented by Haplic Luvisols with class IVa of arable land having a very good rye complex. The studied soils were very similar in granulometric composition with the clay fraction in the range of 2-7%, which corresponds to sandy soils. Soil exchangeable acidity ranged from 5.5 to 5.9, which classifies the soils as slightly acid and acid.

The same conventional soil system management has been applied from the beginning of the field experiment in 1980 to present. It was a two-factor experiment, following the split-plot method, where the first factor was made up by cattle manure fertilization at the rates of 0, 20, 40, 60 and 80 t ha<sup>-1</sup>, while the second factor was nitrogen fertilization in the form of ammonium nitrate (34% N) at the following rates: N0-0, N1-40, N2-80 and N3-120 kg N ha<sup>-1</sup>. The fertilization with phosphorus (granulated triple superphosphate – 46% P<sub>2</sub>O<sub>5</sub>) and potassium (60% potassium salt) in all the experimental treatments was the same and equal to 21 kg P ha<sup>-1</sup> and 60 kg K ha<sup>-1</sup>, respectively. Soil samples for chemical and bio-chemical analyses were collected from the arable layer of 0-20 cm in interrows of winter wheat at the phenological phase of BBCH 87. Winter wheat ('Korweta' cultivar) was grown in a four-year crop rotation: potato, winter wheat, spring barley, maize. All the cultivation measures and care treatments were compliant with the commonly applied principles of good agrotechnical practise for winter wheat. The mean temperature and total precipitation in this region was 8.9°C and 646.6 mm, respectively.

The following properties were determined in the adequately prepared soil samples:

- total organic carbon (TOC) in soil was determined with the TOC analyser Primacs provided by Scalar,
- total nitrogen (TN) in soil with the Kjeldahl method,
- the content of available phosphorus (PE-R) in soil with the Egner-Riehm method – DL (in calcium lactate) (PN-R-04023, 1996),
- sulphate sulphur (S-SO<sub>4</sub><sup>2-</sup>) in soil according to the Bardsley-Lancaster method (Bardsley and Lancaster, 1960),
- the activity of acid phosphatase (AcP) in soil with the Tabatabai and Bremner method (1969),
- the activity of arylsulphatase (Aryl) in soil according to the Tabatabai and Bremner method (1970),

- the content of total phosphorus in the overground parts and in the root of winter wheat according to Mehta *et al.* (1954),
- the content of total sulphur in the overground parts and in the root of winter wheat according to Bardsley-Lancaster (1960).

Based on the total contents of phosphorus and sulphur recorded in winter wheat, values of the index of translocation (IT) for those elements were calculated.

All the assays were made in three replicates. The paper presents mean arithmetic values. The results were subjected to the analysis of variance, and the significance of differences between means was verified with the Tukey test at  $p = 0.05$ . The calculations involved the use of FR-ANALWAR software based on Microsoft Excel. Besides, the results of the analyses of the studied features were exposed to the analysis of simple correlation ( $p < 0.05$ ), which determined the degree of the dependence between the respective features. The analysis of the correlation was made using 'Statistica for Windows Pl' software.

## RESULTS AND DISCUSSION

The highest content of TOC (on average 8.22 g C kg<sup>-1</sup>) was observed in the soil sampled from the treatments with FYM at the rate of 60 t ha<sup>-1</sup>, while the application of the highest FYM rate resulted in a 4% decrease in the TOC content (Table 1). Maćkowiak and Żebrowski (1999) associate this phenomenon with the decrease in the crop yields in the treatments fertilized with FYM at the rate of 80 t ha<sup>-1</sup>, as cereal lodging occurs at high levels of nitrogen fertilization and thus a lower weight of post-harvest residue is observed. In the non-fertilized FYM soil, the TOC content was the lowest (7.20 g C kg<sup>-1</sup>). Long-term agricultural use of soils without proper crop rotation and mineral nitrogen and organic fertilization may lead to a decrease in the soil organic matter content due to the predominance of mineralization processes (Słowińska-Jurkiewicz *et al.*, 2013).

The soil TN content in the soil ranged from 0.84 to 1.02 g N kg<sup>-1</sup> (Table 1). The greatest effect on the soil TN content was attributed to the FYM fertilization. The TN content of soil fertilized with the highest rate of mineral nitrogen was 18% higher than in the soil of the control treatment, where no fertilizers were applied.

The value of the C:N ratio depended on the fertilization level and ranged from 7.76 to 8.57 (Table 1). According to Gajda and Przewłoka (2012) and Lemanowicz and Bartkowiak (2012), a narrow ratio of C:N may be an indicator of very low participation (10%) of microorganisms in metabolic processes in soil. Triggering nitrogen from organic bonds is to a large extent conditioned by the adequate C:N ratio in soil, which most often corresponds to 10:1. The differences in the values of the C:N ratio point to a varied rate of mineralization of organic carbon and nitrogen compounds. The results suggest that the application of FYM increased the amount of TN (by 13%) slightly more than that of TOC (by 5%).

**Table 1.** Soil properties

Fertilization	Doses	TOC (g kg <sup>-1</sup> )	TN (g kg <sup>-1</sup> )	P <sub>E-R</sub> (mg kg <sup>-1</sup> )	S-SO <sub>4</sub> <sup>2-</sup> (mg kg <sup>-1</sup> )	AcP	Aryl	C:N	P:S
						(mM pNP kg <sup>-1</sup> h <sup>-1</sup> )			
Manure (t ha <sup>-1</sup> )	0	7.20	0.84	61.48	24.53	1.094	0.156	8.57	2.51
	20	6.70	0.80	66.50	24.37	1.135	0.165	8.38	2.73
	40	7.32	0.91	83.08	24.88	1.215	0.124	8.04	3.34
	60	8.22	0.97	91.58	26.00	1.379	0.160	8.47	3.52
	80	7.92	1.02	80.28	26.31	1.221	0.170	7.76	3.05
Nitrogen (kg kg <sup>-1</sup> )	0	7.57	0.91	60.76	25.83	1.091	0.159	8.32	2.35
	40	7.29	0.92	72.42	26.65	1.159	0.136	7.92	2.72
	80	7.22	0.92	90.37	23.54	1.230	0.156	7.85	3.84
	120	7.82	0.94	82.79	24.86	1.355	0.170	8.32	3.33
Mean		7.47	0.92	76.58	25.22	1.209	0.155		
SD		0.69	0.07	16.25	1.939	0.144	0.034		
LSD <sub>0.05</sub>									
Factor I		0.086	0.063	5.311	1.685	0.089	0.022		
Factor II		0.091	n.s.	2.709	1.221	0.036	0.008		

TOC – total organic carbon, TN – total nitrogen, P<sub>E-R</sub> – available phosphorus with the Egner-Riehm method, S-SO<sub>4</sub><sup>2-</sup> – sulphate sulphur, AcP – acid phosphatase, Aryl – arylsulphatase, SD – standard deviation, n.s. – not significant differences.

The P<sub>E-R</sub> content of in the Haplic Luvisol was, on average, 76.58 mg P kg<sup>-1</sup>, which according to PN-R-04023 (1996) classifies it as soils with a high content of phosphorus (class II). Considering the environmental aspect, the optimal state of phosphorus in soil should fall within the average richness class. The higher the rate of natural fertilizers (manure and slurry), the higher the content of P<sub>E-R</sub> in soil. Natural fertilizers are an easily available source of phosphorus to plants since, depending on the degree of mineralization, 50-70% of the total phosphorus contained in those fertilizers occurs in mineral compounds mostly as calcium phosphates. The content of P<sub>E-R</sub> was the highest (91.58 mg P kg<sup>-1</sup>) in the soil sampled from the treatments fertilized with FYM at the rate of 60 t ha<sup>-1</sup>. The lowest content of PE-R (61.48 mg P kg<sup>-1</sup>) was observed in the soil from the treatments without FYM fertilization (Table 1).

The content of sulphates (VI) in the Haplic Luvisol was, on average, 25.22 mg kg<sup>-1</sup> (Table 1), which, given the dependence on the soil richness in S-SO<sub>4</sub><sup>2-</sup> sulphur, classifies it as a soil with a very high content and ensures a good supply of sulphur to cereals (Lipiński *et al.*, 2003). Both FYM and nitrogen fertilization affected the SO<sub>4</sub><sup>2-</sup> content in the Haplic Luvisol. FYM contained from 0.9 to 1.2 kg S ha<sup>-1</sup>. It was estimated that, on average, 20% of total sulphur occur in the form of sulphides available to plants, 40% are made up by organic bonds, and another 40% are organic and inorganic sulphates (Kaczor and Zuzanska 2009). The present research demonstrated that the higher the FYM rates, the higher the content of the fraction of available sulphur to plants. The highest content of sulphate sulphur,

similar to the content of TN, was found in the soil sampled from the treatments fertilized with the highest FYM rate (Table 1). The content of sulphates (VI) in the soil samples from those treatments was 10% higher than in the control samples when no FYM was applied.

The value of the P:S ratio ranged from 2.35 to 3.84 (Table 1). Its higher values are reported by Lemanowicz and Siwik-Ziomek (2010) for the soil under maize. The higher the FYM rate, the higher the value of the P:S ratio reaching the highest value (3.52) at the FYM rate of 60 t ha<sup>-1</sup>. The FYM fertilization affected the P<sub>E-R</sub> and S-SO<sub>4</sub><sup>2-</sup> sulphur content, which is reflected by the slow increase in the value of the P:S ratio as well as by the significant positive value of the coefficient of correlation (r=0.73, p.05) between those two parameters (Tables 1 and 3).

The activity of acid phosphatase was the highest (1.379 mM pNP kg<sup>-1</sup> h<sup>-1</sup>) in the soil from the treatments with FYM at the rate of 60 t ha<sup>-1</sup>. The FYM fertilization of winter wheat at the rate of 80 t ha<sup>-1</sup> decreased the activity of the enzyme by 11%. The higher the mineral nitrogen rate, the higher the activity of acid phosphatase in the soil. The activity of the acid phosphatase in the soil from the treatments fertilized with N3 (1.091 mM pNP kg<sup>-1</sup> h<sup>-1</sup>) was 15% higher than the activity of the enzyme in the soil from the treatments without nitrogen fertilization (1.355 mM pNP kg<sup>-1</sup> h<sup>-1</sup>) (Table 1). The activity of acid phosphatase was greater in the soil amended with mineral nitrogen fertilizers, which supports the fact that phosphorus from manure is more mobile than inorganic phosphorus from mineral fertilizers (Koper and Lemanowicz 2008; Gajda and Przewłoka, 2012).

**Table 2.** Content of phosphorus (P) and sulphur (S) in winter wheat at different rates of cattle manure and nitrogen fertilizers

Fertilization	Doses	Yield (t ha <sup>-1</sup> )	P (g kg <sup>-1</sup> )		S (g kg <sup>-1</sup> )		IT	
			above ground part	under ground part	above ground part	under ground part	P	S
Manure (t ha <sup>-1</sup> ) I factor	0	4.614	2.637	0.800	0.258	2.091	3.30	0.124
	20	4.918	2.705	0.846	0.328	2.456	3.19	0.134
	40	5.194	2.879	0.910	0.335	2.147	3.16	0.156
	60	5.615	3.344	1.033	0.351	2.201	3.25	0.160
	80	5.454	2.988	1.008	0.473	2.904	2.96	0.163
Nitrogen (kg kg <sup>-1</sup> ) II factor	0	3.988	1.798	0.639	0.301	2.351	2.81	0.128
	40	5.037	2.658	0.831	0.308	2.230	3.19	0.139
	80	5.747	3.977	1.231	0.387	2.327	3.23	0.167
	120	5.863	3.242	0.978	0.401	2.530	3.32	0.159
Mean		5.159	2.957	0.919	0.349	2.359		
SD		1.098	0.815	0.247	0.094	0.630		
LSD <sub>0.05</sub>								
Factor I		0.014	0.501	0.186	0.046			
Factor II		0.008	0.399	0.115	0.027			

IT – index of translocation.

The activity of arylsulphatase in the soil was, on average, 0.155 mM pNP kg<sup>-1</sup> h<sup>-1</sup> and depended on the degree of fertilization in the experiment (Table 1). Arylsulphatase activity increased after the long-term application of FYM. In the Haplic Luvisol with the highest FYM rate, the activity of arylsulphatase increased by 8.2%, compared with the control treatments (Table 1). Organic matter affects the level of biomass of microorganisms constituting the main source of soil enzymes (Kotkova *et al.*, 2008). Similarly, the highest rate of the nitrogen fertilizer resulted in the highest activity of arylsulphatase. Current research literature provides various views on the effects of mineral fertilization on the soil enzymatic activity. Piotrowska and Wilczewski (2012) claim that mineral fertilization, especially applied for a long-term period and at high rates, can inhibit enzymatic reactions resulting in inactivation of enzymatic proteins by a high concentration of ions (especially anions) and low soil pH values related to it.

The activity of arylsulphatase in the soil depended on the content of sulphate sulphur. The highest concentration of sulphates (VI) and the lowest activity of arylsulphatase were determined at the N1 nitrogen rate (Table 1). Low SO<sub>4</sub><sup>2-</sup> levels stimulated soil microorganisms to produce sulphatases or activate enzymes (Saviozzi *et al.*, 2006).

A good supply of phosphorus to winter wheat plants results in an increase in content of protein, gluten, essential amino acids, vitamin B1. The content of total phosphorus in winter wheat depended on the FYM and nitrogen fertilization and ranged from 2.437 g kg<sup>-1</sup> in the samples taken from

the no-nitrogen treatments to 5.174 g kg<sup>-1</sup> in the dry weight of the species sampled from the treatments fertilized with N2 nitrogen (Table 2). At the highest level of nitrogen (N3) applied, a decrease in the content of total phosphorus in wheat was observed. Sądej (2000) confirmed that plants fertilized with manure showed, in general, a higher content of phosphorus than those treated with mineral fertilization only. The most effective treatment was fertilization with manure combined with NPK, which increased the content of phosphorus in plants (potato, barley, wheat, clover with grasses, rye, maize, and rape) by, on average, 4%, compared with the fertilization with FYM alone (by 9%) and with mineral fertilization. The content of total phosphorus was lower in the roots of winter wheat by 69%, compared with the phosphorus content in the overground parts of plants, and that content varied significantly depending on the experimental factors (Table 2).

Sulphur is an indispensable nutrient and its deficit in soil makes it impossible for plants to develop an adequate biomass yield and, at the same time, deteriorates the biological value of the yield (Scherer, 2009). The content of sulphur in wheat biomass ranged from 2.347 to 3.996 mg S kg<sup>-1</sup> d.m. (Table 2). The content of this bioelement in the wheat biomass was affected by the FYM and nitrogen fertilization. The application of the highest FYM rate for this cereal increased the sulphur content by 38%, compared with the no-fertilization treatment. Increasing rates of natural fertilizers for the Haplic Luvisol ensured a successive release of available forms of sulphur to the soil solution. Gradually

**Table 3.** Correlation coefficients (r) of the contents of phosphorus and sulphur with soil and plant characteristics (n = 20)

Parameters	TOC	TN	P			S		
			straw	roots	soil	straw	roots	soil
TOC	n.s.	0.54	0.35	0.34	0.61	0.44	n.s.	0.61
TN	0.54	n.s.	n.s.	0.45	0.54	0.67	n.s.	0.75
pH in KCl	0.39	0.57	n.s.	n.s.	n.s.	0.51	n.s.	0.54
AcP	0.50	0.53	0.35	0.34	0.81	0.51	n.s.	0.69
Aryl	0.38	0.43	n.s.	n.s.	0.34	n.s.	-0.31	0.60
Yield of grain	0.39	0.44	0.80	0.74	0.74	0.60	n.s.	0.55
Yield of straw	0.47	0.58	0.75	0.71	0.79	0.72	n.s.	0.64

Explanations as in Table 1. Significant at  $p < 0.05$ .

progressing processes of biochemical and biological mineralization (Scherer, 2009) of the organic material applied resulted in an increase in the content of the fraction of sulphur available to plants in the soil and in the plants, both in their overground parts and roots (Tables 1, 3). The present research also demonstrated a high correlation ( $R = 0.70$ ,  $p < 0.05$ ) between the content of sulphates (VI) in the soil and their content in the plants, mostly in its overground parts (Table 3). On the other hand, Gondek and Filipek-Mazur (2008) did not find any significant effect of manure fertilization and mixtures of sewage sludge with peat on the average content of this bioelement in maize biomass. The content of available forms of sulphur depended on the processes of mineralization and immobilization that occurred in soils and on the soil type, soil moisture content, aeration, temperature, pH as well as on rates of natural fertilizers and methods of agricultural use (Scherer, 2009).

To determine the degree of mobility of phosphorus and sulphur in winter wheat, the value of the translocation index (IT) was calculated for evaluation of the direction of nutrient mobility in a given species (Siwik-Ziomek and Lemaniowicz, 2011). The value of the parameter was calculated as the ratio of the P and S contents in the overground parts to their contents in the winter wheat roots. The IT value for phosphorus ranged from 2.81 to 3.32 (Table 2) depending on the rate of the fertilizers applied. Phosphorus moved mostly from the roots to the overground parts of a given crop species at the FYM rate of  $60 \text{ t ha}^{-1}$  ( $IT_P 3.25$ ) (Table 2), while FYM fertilization slightly differentiated the IT value. The higher the amount of mineral nitrogen applied, the higher the mobility of phosphorus from the roots to the overground part of plants. At the N3 rate, the value of the translocation index was the highest ( $IT_P 3.32$ ) (Table 2).

The value of the coefficient of sulphur translocation ranged from 0.124 to 0.167 (Table 2), which indicated greater accumulation of this element in the wheat roots than in the overground parts of the wheat plants. Sulphur was translocated to the overground parts from the roots mostly at

the nitrogen rate of N2 (Table 2). The FYM fertilization at the rates of 40, 60 and  $80 \text{ t ha}^{-1}$  also affected the mobility of sulphur more efficiently than the control treatment and the FYM rate of  $20 \text{ t ha}^{-1}$  (Table 2).

The activity of acid phosphatase and arylsulphatase was closely connected with the content of phosphorus and sulphur forms available to plants (Table 3). The value of the coefficient of correlation between the activity of AcP and the content of  $P_{E-R}$  in the soil was equal to 0.81 at  $p < 0.05$ . A linear relationship between the activity of acid phosphatases and the release of inorganic phosphorus forms to the soil solution was commonly observed (Dodor and Tabatabai, 2003; Yadav and Tarafdar, 2001; Krzyżaniak and Lemaniowicz, 2013). The interpretation of this phenomenon is complex due to the possibility of long-term occurrence of extracellular enzymes in the bonds with soil colloids. As is known for phosphomonoesterases, the phosphoro-organic compounds in soils constitute a substrate. To a large extent, the knowledge of the values of these two parameters should make it possible to evaluate the content of available phosphorus, which can be determined by the Egner-Riehm method.

There was also a significant correlation between the content of available fractions of phosphorus in the soil and that of total phosphorus both in the overground parts ( $R = 0.83$ ,  $p < 0.05$ ) and in the roots ( $R = 0.82$ ,  $p < 0.05$ ) of winter wheat. Kucharski *et al.* (2006) identified a significant correlation between the AcP activity in soil contaminated with copper and the content of phosphorus ( $R = 0.73$ ) in spring barley. Significant positive coefficients of correlation were observed between the TOC content in the soil and the activity of acid phosphatase ( $R = 0.50$ ,  $p < 0.05$ ) as well as between the TN content and the activity of acid phosphatase ( $R = 0.53$ ,  $p < 0.05$ ). Aon and Colaneri (2001) identified a significant correlation between the TOC and TN contents in soil and the activity of both alkaline and acid phosphatase. The authors claimed that the activity of the enzymes depended directly on the content of organic substances in soil, and an inadequate management system with a low amount of

applied organic matter deteriorated the biological and biochemical soil fertility. Similarly, Deng and Tabatabai (1997) reported a significant correlation between the content of TOC and acid phosphatase ( $R = 0.71$ ) in 40 studied soils. A positive correlation ( $R = 0.38$  at  $p = 0.05$ ) was also recorded between the activity of arylsulphatase and the TOC content (Table 3). As reported by Shai *et al.* (2007), the value of the coefficients of the above-mentioned correlation was equal to 0.68, which supported the fact that the organic substance protected soil enzymes against unfavourable factors and thus prolonged the period of their activity. Arylsulphatase is not directly related to the transformations of nitrogen in soil and, therefore there were no significant correlations between its activity and the content of this bioelement incorporated into the soil in the form of mineral salts. The correlation between the TN content and the activity of arylsulphatase is a result of permanent bonds of enzymes with the organic matter of soil. The activity of arylsulphatase showed a correlation with various fractions of sulphur in the soil (Table 3). The soil samples revealed a negative correlation between the content of sulphur in the roots of wheat and the activity of arylsulphatase as well as a positive correlation between the content of sulphates in the soil and the activity of the enzyme (Table 3).

Significant coefficients of correlation were recorded between the activity of acid phosphatase in the soil and the yields of grain ( $R = 0.74$ ,  $p < 0.05$ ) and straw ( $R = 0.82$ ,  $p < 0.05$ ) of winter wheat. Similarly, Gajda and Przewłoka (2012) reported a significant statistical correlation between microbiological and biochemical parameters of soil *ie* TOC and the yield of winter wheat.

#### CONCLUSIONS

1. The cattle manure fertilization and the application of different rates of nitrogen fertilizers affected the content of available phosphorus in soil.

2. The activity of arylsulphatase in the soil depended on the content of sulphate sulphur. At the nitrogen rate of 40 kg N ha<sup>-1</sup>, the highest content of sulphates (VI) and the lowest activity of arylsulphatase were identified. The higher nitrogen rate, the higher the activity of acid phosphatase in the soil.

3. The long-term overlapping fertilization resulted in diversification of the content of total phosphorus in winter wheat. The use of the highest rate of cattle manure (80 t ha<sup>-1</sup>) and nitrogen fertilizers (120 kg N ha<sup>-1</sup>) resulted in an unfavourable significant decrease in the phosphorus content.

4. Increasing rates of natural fertilizers incorporated into the Haplic Luvisol ensured successive release of the available sulphur forms to the soil solution and resulted in a greater content of this nutrient in the wheat plants.

5. The translocation index of the nutrients studied was the highest after the application 60 t ha<sup>-1</sup> of cattle manure without mineral nitrogen fertilization.

6. Long-term mineral nitrogen fertilization significantly affects soil properties and may lead to changes in soil enzymatic activity and nutrient contents in the soil and plant tissues. The results of the study show that it is necessary to continue research of soil chemical and enzymological properties during application of fertilizers in order to select of a proper management system.

#### REFERENCES

- Aon M.A. and Colaneri A.C., 2001.** Temporal and spatial evolution of enzymatic activities and physico-chemical properties in an agricultural soil. *App. Soil Ecol.*, 18(3), 255-270.
- Bardsley C.E. and Lancaster J.D., 1960.** Determination of reserve sulfur and soluble sulfates in soil. *Soil Sci. Soc. Am. Proc.*, 24, 265-268.
- Bielińska E.J. and Mocek-Plóćiniak A., 2009.** Phosphatases in soil environment (in Polish). Univ. Life Sci. Press, Poznań, Poland.
- Bielińska E.J. and Mocek-Plóćiniak A., 2012.** Impact of the tillage system on the soil enzymatic activity. *Archiv. Environ. Protec.*, 38(1), 75-82.
- Deng S.P. and Tabatabai M.A., 1997.** Effect of tillage residue management on enzyme activities in soils: III. Phosphatases and arylsulphatase. *Biol. Fert. Soils*, 24, 141-146.
- Dodor D.E. and Tabatabai M.A., 2003.** Effect of crop rotation systems on phosphatases in soil. *J. Plant Nutr. Soil Sci.*, 166, 7-13.
- Filipek T. and Skowrońska M., 2009.** Optimizations of soil reaction and nutrient management in Polish agriculture (in Polish). *Adv. Agric. Sci.*, 1, 25-37.
- Gajda A.M. and Przewłoka B., 2012.** Soil biological activity as affected by tillage intensity. *Int. Agrophys.*, 26, 15-23.
- George T.S., Gregory P.J., Wood M., Read M., and Buresh R.J., 2002.** Phosphatase activity and organic acids in the rhizosphere of potential agroforestry species and maize. *Soil Biol. Biochem.*, 34, 1487-1494.
- Gondek K. and Filipek-Mazur B., 2008.** Changes of sulphur content in maize fertilized with organic materials (in Polish). *Acta Agrophysica*, 158, 633-646.
- Hopkins D.W., Sparrow A.D., Shillam L.L., English L.C., Dennis P.G., Novis P., Elberling B., Gregorich E.G., and Greenfield L.G., 2008.** Enzymatic activities and microbial communities in an Antarctic dry valley: Responses to C and N supplement. *Soil Biol. Biochem.*, 40, 2130-2136.
- Kaczor A. and Łaszcz-Zakorczemna J., 2009.** The effect of sulphur and potassium fertilization of barley and rape on the content of available phosphorus, potassium and magnesium in soil (in Polish). *Adv. Agricul. Sci. Probl.*, 538, 103-110.
- Kaczor A. and Zuzajska J., 2009.** Importance of sulphur in agriculture (in Polish). *Chemistry-Didactics-Ecology-Metrology*, 14(1-2), 69-78.
- Koper J. and Lemanowicz J., 2008.** Effect of varied mineral nitrogen fertilization on changes in the content of phosphorus in soil and in plant and the activity of soil phosphatases. *Ecol. Chem. Eng.*, 15(4), 465-471.
- Kotkova B., Balik J., Cerny J., Kulkanek M., and Bazalova M., 2008.** Crop influence on mobile sulphur content and arylsulphatase activity in the plant rhizosphere. *Plant Soil Environ.*, 54, 100-107.

- Krämer S. and Green D.M., 2000.** Acid and alkaline phosphatase dynamics and their relationship to soil microclimate in a semi-arid woodland. *Soil Biol. Biochem.*, 32, 179-188.
- Krzyżaniak M. and Lemanowicz J., 2013.** Enzymatic activity of the Kuyavia Mollic Gleysols (Poland) against their chemical properties. *Plant Soil Environ.*, 59(8), 359-365.
- Kucharski J., Jastrzębska E., and Wyszowska J., 2006.** Contamination of soil with hard coal ash as modifier of physico-chemical and biological properties of soil. *EJPAU, Agronomy*, <http://www.ejpau.media.pl/volume9/issue1/art-35.html>
- Lemanowicz J. and Bartkowiak A., 2012.** Activity of phosphatases in humus horizons of Unisław basin arable soils. *Polish J. Soil Sci.*, 45(1), 1-7.
- Lemanowicz J. and Siwik-Ziomek A., 2010.** Concentrations of available phosphorus and sulphur and activities of some hydrolytic enzymes in a luvisol fertilized with farmyard manure and nitrogen. *Polish J. Soil Sci.*, 43(1), 37-47.
- Lipiński W., Terelak H., and Motowicka-Terelak T., 2003.** Suggestion for limiting values of sulphate sulphur content in mineral soils for fertilization advisory needs (in Polish). *Soil Sci. Ann.*, 54(3), 79-84.
- Maćkowiak C. and Żebrowski J., 1999.** Effect of farmyard application and selection of crops in rotation on organic carbon and total nitrogen content in the soil (in Polish). *Adv. Agri. Sci. Probl.*, 465, 341-351.
- Mehta N.C., Legg J.O., Goring C.A., and Black C.A., 1954.** Determination of organic phosphorus in soils. *Soil Sci. Soc. Am. Proc.*, 44, 443-449.
- Piotrowska A. and Wilczewski E., 2012.** Effects of catch crops cultivated for green manure and mineral nitrogen fertilization on soil enzyme activities and chemical properties. *Geoderma*, 189-190, 72-80.
- PN-R-04023, 1996.** Chemical and Agricultural Analysis (in Polish). PWN Press, Warsaw, Poland.
- Polish Soil Classification, 2011.** *Soil Sci. Ann.* (in Polish), 62(3), 5-142.
- Saviozzi A., Cardeli R., Cipolli S., Levi-Minzi R., and Riffaldi R., 2006.** Arylsulphatase activity during the S mineralization in soils amended with cattle manure and green waste. *Agrochimica*, 50, 231-237.
- Sądej W., 2000.** Studies on phosphorus transformation in the soil and its utilization by cultivated plants under conditions of differentiated fertilization (in Polish). *Dissertations and Monographs, ART Olsztyn*, 33, 1-78.
- Scherer H.W., 2009.** Sulfur in soils. *J. Plant Nutr. Sci.*, 172, 326-335.
- Shai W., Hanoch L., and Pariente S., 2007.** Temporal dynamics in arylsulfatase enzyme activity in various microenvironments along a climatic transect in Israel. *Geoderma*, 140, 30-41.
- Siwik-Ziomek A. and Lemanowicz J., 2011.** Phosphorus and sulphur accumulation and mobility in maize cultivated for silage as dependent on farmyard manure and nitrogen rate (in Polish). *J. Res. Appl. Agri. Eng.*, 56(1), 124-129.
- Słowińska-Jurkiewicz A., Bryk M., and Medvedev V.V., 2013.** Long-term organic fertilization effect on chernozem structure. *Int. Agrophys.*, 27, 81-87.
- Suwara I., Lenart S., and Gawrońska-Kulesza A., 2007.** Growth and yield of winter wheat after 50 years of different fertilization and crop rotation (in Polish). *Acta Agrophysica*, 153, 695-704.
- Tabatabai M.A. and Bremner J.M., 1969.** Use of p-nitrophenol phosphate for assay of soil phosphatase activity. *Soil Biol. Biochem.*, 1, 301-307.
- Tabatabai M.A. and Bremner J.M., 1970.** Factors affecting soil arylsulfatase activity. *Soil Sci. Soc. Am. Proc.*, 34, 427-429.
- Yadav R.S. and Tarafdar J.C., 2001.** Influence of organic and inorganic phosphorus supply on the maximum secretion of acid phosphatase by plants. *Biol. Fertil. Soils*, 34, 140-143.