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INFLUENCE OF NITROGEN DOSES APPLIED IN SEWAGE SLUDGE ON THE CONTENT OF MACROELEMENTS IN THE WILLOW (*Salix viminalis*)

WPŁYW DAWEK AZOTU ZASTOSOWANEGO W OSADZIE ODCIEKOWYM NA ZAWARTOŚĆ MAKROELEMENTÓW W WIERZBIE (*Salix viminalis*)

Abstract: The influence of different doses of nitrogen applied in the sewage sludge as well as NPK in mineral fertilizers in the field experiment carried out during three years on the content of macroelements in the willow biomass *Salix viminalis* was the aim of this work. The doses of nitrogen applied in the form of sewage sludge reached 100, 150 and 200 kg · ha^{-1}. Besides those objects field experiment contained two more objects: NPK with N-150, P-50, and K 150 kg · ha^{-1} in which every year 50 kg of N in urea form was applied with P and K at ratio maintained above and in control. The content of determinated elements P, K, S, Ca, Mg and Na in the branches of willow was not significantly differentiated upon the influence of different nitrogen doses applied in the form of sewage sludge. The content of potassium and sulfur was significantly higher in branches of willow harvested from the object fertilized with doses of nitrogen 200 kg N · kg^{-1} then from others objects. The content of potassium, sulfur, calcium, magnesium and sodium in the branches of willow biomass was not significantly differentiated upon the investigated parameters, but the content of phosphorus was significantly lower in biomass harvested from objects fertilized with sewage sludge than mineral fertilizers. The highest amount of determinated elements was taken up by willow cultivated in the second year and the lowest in the first year of experiment. The uptake of phosphorus increased in each year of experiment from the lowest in the first year up to highest in the third year of experiment.

Keywords: sewage sludge, nitrogen, biomass of willow, macroelements

Introduction

The share of renewable energy in energetic balance of Poland in the 2020 year should reached up to 20 %. Solid biomass comes mainly as the waste from forestry, wood industry, meat and plant processing industry, municipal waste treatment and agriculture. It is predicate that in the future main source of biomass will be the

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plantation of energetic plants. The willow (*Salix viminalis*) is recognized especially in north European (Scandinavian) and North American lands as the very good plant with quick growth for harvesting of biomass for the energetic purpose [1–4]. Yield of the willow (*Salix viminalis*) cultivated and harvested in Poland and in another countries generally reach up to 15 MG (ton) · ha^{-1} dry matter [5–8].

The amount of plant nutrients introduce into the soil should be taken under consideration the fertility of soil as well as the uptake and the yield of those macro- and microelements. For the fertilization of willow can be used organic and mineral fertilizers as well as different waste for example sewage sludge, because this plant shows big possibility for the utilization of plant nutrients from those materials [9, 10] what allow good growth and high yield of this plant.

The aim of this investigation carried out as field experiment during the years 2002–2005 was the estimation of the influence of different nitrogen doses applied into the soil in the form of sewage sludge as well as mineral fertilizers on the content of phosphorus, potassium, calcium, magnesium, sulfur and sodium in the biomass (branches) of willow (*Salix viminalis*). The yield of biomass harvested in this experiment has been published in the other paper [9].

Materials and methods

The field experiment was laid down of spring 2002 year on the soil developed from loamy light sand (according to Polish Soil Science Society) and was carried out during three years. The soil had neutral reaction (pH measured in 1 mol · $CaCl_2$ dm^{-3} reached 6.5 unit) and the high content g · kg^{-1} (of d.m. soil) of organic carbon 42.5 and total nitrogen 2.50, respectively. The total content in g · kg^{-1} of estimated macroelements was as follows, respectively: P – 0.99, K – 1.71, Ca – 9.87, Mg – 1.69 and Na 0.21. The scheme of experiment contained follows objects: different doses of nitrogen applied with sewage sludge (kg · ha^{-1}): 100, 150 and 200; object fertilized with urea applied in 50 kg · ha^{-1} nitrogen in each year (total amount 150 kg · ha^{-1}) and control in which only phosphorus and potassium were applied.

Fresh sewage sludge produced on the municipal sewage purification plants at Siedlce was applied at spring before plantation of willow plant. The used sewage sludge contained 20.2 % of dry matter in which the following amount of investigated elements were determinated in g · kg^{-1} of d.m.: N – 50.6, P – 25.8, K – 3.25, Ca – 41.8, Mg – 8.90, S – 6.51, Na – 1.68 and in mg · kg^{-1} of d.m.: Fe – 13499, Mn – 253, Zn – 1573, Cu – 139, Mo – 5.92, Co – 5.10. The content of heavy metals in the sewage sludge was in the amount accepted for fertilizer use in agriculture [11].

The urea was applied in each year three times during whole duration of experiment in dose 50 kg N · ha^{-1} before the starting of vegetation. In the first year of experiment on all objects fertilized with sewage sludge, urea and control (without nitrogen) additional fertilization with potassium (potassium salt) in dose to reach the ratio N : K equal 1 : 1 was applied. On the object fertilized with urea additional fertilization with phosphorus and potassium were applied in the form of mineral fertilizer (triple superphosfate and potassium salt) in dose to reach the ratio of N : P : K equal 1 : 0.35 : 1. The amount of P and K applied in the control object equal to the amount of those elements introduced into the soil with the middle dose of sewage sludge which contained 150 kg · ha^{-1} of nitrogen.

The experiment was laid down in four replicates in which willow (*Salix viminalis*) clone 1056 was cultivated in the area plot on 8 m^2 and at the density 8 pieces of plant per 1 m^2. The biomass of willow was harvested in each year of experiment. Whole biomass of the willow branches were harvested in February. These branches were shreaded, representative samples were separated, dried and finely grounded. So prepared samples were digested by the "method of dry combustion" in the oven at the temperature 450 ^{o}C. Ash was treated by 6 mol HCl · dm^{-3} to decompose carbonate. The excess of HCl from crucible was evaporated to dryness. The chlorides of elements were transferred by 10 % HCl to volumetric flask. This was the base solution in which investigated elements were determined by ICP-AES method on the spectrometer Optima 3200 manufactured by Perkin-Elmer.

The results were statistically calculated using the analyses of variance for the experiment with two investigated parameters in completely randomized scheme. The values of $LSD_{0.05}$ were calculated by Tukey's test.

Results and discussion

The cultivation of willow in the every year harvesting system allow the production of biomass in small and big areas of energetic plants for it using in own farms or for sale to energetic plants in small towns. The cultivation of willow in this system makes easy its fertilization and harvesting [12]. Independently from the cycle of biomass harvesting system(every year or once per 3 years) to obtained high yield of biomass, it is necessary to applied high doses of plant nutrients, which can be supplied in organic and mineral fertilizers by mixing their with soil. Utilization of sewage sludge in fertilization this plant is quite possibility but needs to their cover (mixing) with soil. The sewage sludge used in this experiment for fertilization of willow, were the main source of macro- and microelements, beside some quantity of those elements which can be taken up by plants from soil. Sewage sludge applied in this experiment contained very low amount of potassium and therefore additional potassium dose was applied in mineral fertilizer.

The content of calcium and sodium in willow branches was not significantly differentiated by the doses of sewage sludge (Table 1). The content of Mg, K, P and S was significantly higher in the branches of willow harvested from the plots fertilized with the highest doses of sewage sludge (200 kg · ha^{-1} of nitrogen) and the lowest at medium doses of nitrogen (150 kg N · ha^{-1}). The willow biomass harvested from plants fertilized with sewage sludge in dose contained 150 kg · ha^{-1} of nitrogen contained lower content of phosphorus, potassium and sulfur than it biomass harvested from control object (fertilized only with PK). Content of calcium, magnesium and sodium in willow branches harvested from above described objects were similar. The content of all determinated elements in willow biomass harvested from the objects fertilized with urea were not significantly differentiated from the content of these element in biomass

Table 1

The content of determinated macroelements [g · kg^{-1} d.m.] in willow branches

Doses of nitrogen	Calcium				Magnesium				Potassium			
	Years of cultivation			Mean	Years of cultivation			Mean	Years of cultivation			Mean
	I	II	III		I	II	III		I	II	III	
Control object	4.86	7.11	4.81	5.59	0.656	0.734	0.585	0.658	14.19	3.30	3.62	7.04
100kg N (sludge)	4.28	6.00	4.81	5.03	0.563	0.719	0.670	0.651	10.59	4.01	4.05	6.22
150kg N (sludge)	4.20	3.82	6.04	4.69	0.525	0.543	0.700	0.589	9.75	1.88	3.78	5.14
200kg N (sludge)	4.10	7.51	4.53	5.38	0.649	0.710	0.589	0.649	12.04	4.03	3.99	6.69
150kg N (urea)	5.20	6.03	5.59	5.61	0.718	0.918	0.710	0.782	12.08	4.23	3.93	6.75
Mean	4.53	6.10	5.16	5.26	0.622	0.725	0.651	0.666	11.73	3.49	3.87	6.37
$LSD_{0.05}$	0.75*			n.i.***	0.090*			0.137**	0.95*			1.45**
	Sodium				Phosphorus				Sulfur			
Control object	0.137	0.589	0.011	0.246	2.25	1.05	1.31	1.54	0.866	0.600	0.346	0.604
100kg N (sludge)	0.106	0.664	0.024	0.265	1.62	1.23	1.33	1.39	0.623	0.594	0.361	0.526
150kg N (sludge)	0.131	0.633	0.020	0.261	1.66	0.58	1.44	1.23	0.573	0.447	0.375	0.465
200kg N (sludge)	0.129	0.625	0.014	0.256	1.89	1.18	1.41	1.49	0.777	0.586	0.364	0.576
150kg N (urea)	0.128	0.660	0.038	0.275	2.26	1.34	1.37	1.66	0.933	0.679	0.380	0.664
Mean	0.126	0.634	0.065	0.261	1.94	1.08	1.37	1.46	0.754	0.581	0.365	0.567
$LSD_{0.05}$	0.053*			n.i.***	0.15*			0.23**	0.070*			0.102**

* $LSD_{0.05}$ for years of cultivation, ** $LSD_{0.05}$ for fertilizations objects, *** n.i. – differences among average unimportant.

harvested from control object (only PK). Also the content of all investigated elements in biomass harvested from the objects fertilized with urea (object with mineral fertilizer) was slightly higher (most often not significantly differentiated) than in biomass harvested from objects fertilized with all doses of sewage sludge. Only the content of P, K, Mg and S was higher in willow biomass harvested from plants fertilized with urea (mineral fertilizers) than after sewage sludge applied with dose contained 150 kg · ha^{-1} of nitrogen.

The content of all determinated elements in willow biomass was significantly differentiated under the influence of the harvesting year. The highest concentration of phosphorus, potassium and sulfur were determinated in the willow biomass harvested in the first year of cultivation whereas the content of calcium, magnesium and sodium were the highest in the biomass harvested in the second year of cultivation. The lowest content of calcium and magnesium in the willow branches were determinated in first year of cultivation, but phosphorus and potassium in second year, however sodium and sulfur in third year.

The uptake of determinated elements (kg · ha^{-1}) by harvested part of willow (branches) was depended up their content in biomass (Table 1) and yield [9]. The amount of macroelements taken up by harvested biomass significantly depended upon investigated parameters; it is year of cultivation and different kind and dose of fertilizers (Table 2). The highest amount of P, K were taken up by willow biomass harvested in the third and the lowest in the first year of cultivation, whereas Ca, Mg, S and Na the highest in the second and the lowest in the first year of cultivation. It is also very interesting that the amounts of P and K taken up by willow biomass increased in each year of cultivation from the lowest in the first up to the highest in the third year of cultivation. The highest amount of investigated elements were harvested most often with biomass from plots fertilized with the highest dose of sewage sludge in which 200 kg · ha^{-1} of and the lowest with the dose 150 kg · ha^{-1} nitrogen introduces into the soil. The contents of P, K, Mg and S in biomass harvested from plants fertilized with urea (mineral fertilization) were not significantly differentiated from the amount of determinated elements harvested from control plots. The amount of determinated elements harvested with willow biomass from plots fertilized with sewage sludge in dose 100 kg · ha^{-1} and 200 kg · ha^{-1} of nitrogen generally was higher than from control plot.

The relation of chemical elements one- to bivalent ([K + Na]/[Ca + Mg] counted as their the gram-equivalents) in first year of the willow cultivation ranged from 0.99 in object fertilized with urea to 1.24 on control once (Table 3). The willow sprouts fertilized with sewage sludge had in first year the lowest relation of chemical elements one- to bivalent on object with dose 150 kg N · ha^{-1} (1.01), and the highest after applied dose 200 kg N · h^{-1} (1.22). Willow sprouts harvested in second and third year had the similar relation of chemical elements one- to bivalent (average value for all studied objects had value 0.32 and 0.33, respectively). It means that in the first year of cultivation willow plant have taken mainly potassium and sodium and much less calcium and magnesium. It is explained by physiology of this plant connected with the amount of woody materials which in the first year of cultivation is very low in whole biomass of willow branches.

Table 2

Uptake of determinated macroelements [kg · ha^{-1}] by willow

Doses of nitrogen	Calcium					Magnesium					Potassium				
	Years of cultivation			Mean	Sum	Years of cultivation			Mean	Sum	Years of cultivation			Mean	Sum
	I	II	III			I	II	III			I	II	III		
Control object	24.4	219.6	120.8	121.6	364.8	3.3	22.7	14.7	13.6	40.7	71.4	101.9	90.9	88.1	264.2
100 kg N (sludge)	26.2	203.2	133.6	121.0	363.0	3.4	24.4	18.6	15.5	46.4	64.7	135.8	112.5	104.3	313.0
150 kg N (sludge)	17.6	121.6	184.9	108.0	324.1	2.2	17.3	21.4	13.6	40.9	41.0	59.8	115.7	72.2	216.5
200 kg N (sludge)	26.7	262.3	166.8	151.9	455.8	4.2	24.8	21.7	16.9	50.7	78.4	140.8	146.9	122.0	366.1
150 kg N (urea)	16.7	163.5	143.4	107.9	323.6	2.3	24.9	18.2	15.1	45.4	38.8	114.7	100.8	84.8	254.3
Mean	22.3	194.0	149.9	122.1	366.2	3.1	22.8	18.9	14.9	44.8	58.9	110.6	113.4	94.3	282.9
$LSD_{0.05}$	8.5*			12.9**	—	1.9*			2.9**	—	6.8*			10.4**	—
	Sodium					Phosphorus					Sulfur				
Control object	0.7	18.2	0.3	6.4	19.2	11.3	32.4	32.9	25.5	76.6	4.4	18.5	8.7	10.5	31.6
100 kg N (sludge)	0.6	22.5	0.7	7.9	23.8	9.9	41.7	36.9	29.5	88.5	3.8	20.1	10.0	11.3	33.9
150 kg N (sludge)	0.6	20.1	0.6	7.1	21.3	7.0	18.5	44.1	23.2	69.6	2.4	14.2	11.5	9.4	28.1
200 kg N (sludge)	0.8	21.8	0.5	7.7	23.1	12.3	41.2	51.9	35.1	105.4	5.1	20.5	13.4	13.0	39.0
150 kg N (urea)	0.4	17.9	1.0	6.4	19.3	9.6	34.0	40.2	27.9	83.8	3.0	18.4	9.8	10.4	31.2
Mean	0.6	20.1	0.6	7.1	21.3	10.0	33.6	41.2	28.3	84.8	3.7	18.3	10.7	10.9	32.8
$LSD_{0.05}$	1.0*			1.5**	—	3.0*			4.5**	—	1.4*			2.1**	—

* $LSD_{0.05}$ for years of cultivation, ** $LSD_{0.05}$ for fertilizations objects.

Table 3

The ratio of chemical elements one- to bivalent (K + Na)/(Ca + Mg) calculated as gramequivalent

Doses of nitrogen	(K + Na) / (Ca + Mg)			
	Years of cultivation			Mean
	I	II	III	
Control object	1.24	0.26	0.32	0.61
100kg N (sludge)	1.06	0.37	0.35	0.59
150kg N (sludge)	1.01	0.32	0.27	0.53
200kg N (sludge)	1.22	0.30	0.37	0.63
150kg N (urea)	0.99	0.36	0.30	0.55
Mean	1.10	0.32	0.33	0.58

The content of investigated elements determinated in willow biomass in own research was contained in a range of papers published by others authors [13, 14]. Results presented in this paper related to phosphorus indicated on the lower degree of phosphorus availability for willow from sewage sludge that from mineral fertilizer mainly in the second and third year of cultivation. The lower content of sulfur determinated in willow biomass harvested from plots fertilized with lower doses of sewage sludge than that from object with mineral fertilizer as well as in comparison with the sulfur content in others energetic materials [12] indicates that the pollution of environment should be lower when biomass of willow harvested from plots fertilized with sewage sludge will be used as energetic materials.

Conclusions

In the field experiment carried out during three years with the fertilization of willow (*Salix viminalis*) in which biomass was harvested every year by different doses of sewage sludge on the loamy light sandy soil the following conclusions were drawn out:

1. The content of all determinated P, K, S, Ca, Mg and Na elements were significantly differentiated upon the year of harvesting. The highest content of P, K and S were determinated in willow biomass harvested in the first, whereas Ca, Mg and Na in the second year of cultivation.
2. The highest content of all determinated elements was stated in biomass harvested from plots fertilized with mineral fertilizers (urea).
3. The highest content of all determinated elements contained the biomass harvested from plots fertilized with the highest dose from plots fertilized with the highest dose of sewage sludge (200 kg $\cdot$ ha^{-1} of N) and the lowest with plots fertilized with sewage sludge in the middle dose (150 kg $\cdot$ ha^{-1} of N).
4. Uptake by willow biomass of Ca, Mg, S and Na was the highest in the second year, whereas the P and K increase in the following years of cultivation.
5. The value of relation of chemical elements one- to bivalent (K + Na)/(Ca + Mg) in small degree were diverse in dependence from applied fertilization, however they were clearly larger in first year of the willow cultivation than in second and third.

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WPŁYW DAWEK AZOTU ZASTOSOWANEGO W OSADZIE ODCIEKOWYM NA ZAWARTOŚĆ MAKROELEMENTÓW W WIERZBIE (*Salix viminalis*)

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Abstrakt: Określono wpływ zróżnicowanych dawek azotu wprowadzonych do gleby w osadach ściekowych oraz nawożenia mineralnego na zawartość i pobranie fosforu, potasu, siarki, wapnia, magnezu i sodu przez wierzbę krzewiastą (*Salix viminalis*). W doświadczeniu polowym uprawiano wierzbę na obiektach nawożonych osadami ściekowymi w dawkach odpowiadających wprowadzeniu do gleby 100, 150, i 200 kg N · ha^{-1}. Ponadto wydzielono obiekt nawożony corocznie przez 3 lata mocznikiem w dawce 50 kg N · ha^{-1}, co w sumie stanowiło 150 kg N. Zawartość badanych pierwiastków w pędach wierzby na ogół nie była istotnie uzależniona od dawki osadu ściekowego. Tylko zawartość potasu i siarki w uzyskanej biomasie była istotnie największa po zastosowaniu największej dawki osadu. Zawartość potasu, siarki, wapnia, magnezu i sodu w pędach wierzby nawożonej osadami ściekowymi i NPK w postaci mineralnej najczęściej nie różniła się istotnie, natomiast zawartość fosforu była istotnie mniejsza po zastosowaniu osadów niż nawożenia mineralnego. Najwięcej badanych pierwiastków (potasu, siarki, wapnia i magnezu) rośliny wierzby pobrały w II roku uprawy, a najmniej w I roku po założeniu plantacji. Pobranie fosforu zwiększało się w kolejnych latach uprawy wierzby.

Słowa kluczowe: osady ściekowe, azot, wierzba, makroelementy