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EFFECT OF SULFUR FERTILIZATION ON THE CONCENTRATIONS OF COPPER, ZINC AND MANGANESE IN THE ROOTS, STRAW AND OIL CAKE OF RAPESEED (*BRASSICA NAPUS* L. SSP. *OLEIFERA* METZG)*

Krzysztof Jankowski[1], Łukasz Kijewski[1], Małgorzata Skwierawska[2], Sławomir Krzebietke[2], Ewa Mackiewicz-Walec[2]

[1]Chair of Agrotechnology and Crop Management
[2]Chair of Agricultural Chemistry and Environmental Protection
University of Warmia and Mazury in Olsztyn

Abstract

Sulfur application has a significant effect on the yield of oil bearing plants of the family *Brassicaceae*, especially when the sulfur content of soil is low. Sulfur fertilization also affects the value of plant raw materials, reflected by the concentrations of mineral and biologically active compounds in biomass. The aim of this study was to determine the effect of sulfur application to soil on the concentrations of copper, zinc and manganese in the root residues, straw and oil cake of winter and spring rapeseed. A three-year (2005-2008) field experiment was conducted at the Agricultural Experimentation Station in Bałcyny (NE Poland).

In both spring and winter rapeseed, oil cake contained the highest levels of copper and zinc, followed by root residues and straw. The highest concentrations of manganese per kg dry matter (DM) were found in the root residues of winter rapeseed and in the cake of spring rapeseed. The concentrations of micronutrients (Cu, Zn, Mn) were slightly higher in the roots of winter rapeseed, compared with spring rapeseed. Sulfur fertilization decreased copper levels and increased manganese levels in the root residues of spring and winter rapeseed. Sulfur application to soil increased zinc concentrations in winter rapeseed roots, and it had no significant influence on the zinc content of spring rapeseed roots. Spring rapeseed straw contained considerably higher levels of zinc and manganese than winter rapeseed straw. The copper content of

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dr hab. Krzysztof Jankowski, Chair of Agrotechnology and Crop Management, University of Warmia and Mazury in Olsztyn, 8 Oczapowskiego Street, 10-719 Olsztyn, Poland, e-mail: krzysztof.jankowski@uwm.edu.pl

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straw was comparable in spring and winter rapeseed. Sulfur application to soil increased the concentrations of zinc and manganese in winter rapeseed straw, and it had no significant effect on the levels of those minerals in spring rapeseed straw. Spring rapeseed cake had a significantly higher content of copper and zinc, compared with winter rapeseed cake. Manganese concentrations in the cake of spring and winter rapeseed were similar. Sulfur fertilization contributed to a significant increase in the concentrations of zinc and manganese in winter rapeseed cake. The manganese content of spring rapeseed cake decreased significantly in response to sulfur fertilization, which had no effect on the concentrations of the other micronutrients.

Keywords: winter rapeseed, spring rapeseed, sulfur fertilization, micronutrient content, root residues, straw, cake.

ZAWARTOŚĆ MIEDZI, CYNKU I MANGANU W KORZENIACH, SŁOMIE I WYTŁOKACH RZEPAKU (*BRASSICA NAPUS* L. SSP. *OLEIFERA* METZG) W ZALEŻNOŚCI OD NAWOŻENIA SIARKĄ

Abstrakt

Siarka odgrywa bardzo istotną rolę w plonowaniu roślin oleistych z rodziny *Brassicaceae*, szczególnie w przypadku niskiej jej zawartości w glebie. Wpływa również bardzo silnie na wartość użytkową surowca mierzoną koncentracją w biomasie związków mineralnych oraz biologicznie czynnych. Celem pracy było określenie wpływu doglebowej aplikacji siarki na zawartość miedzi, cynku oraz manganu w resztkach korzeniowych, słomie oraz wytłokach rzepaku ozimego i jarego. Materiał do badań uzyskano w ścisłym doświadczeniu polowym realizowanym w pełnym 3-letnim cyklu badań (2005-2008) na polach doświadczalnych w Bałcynach.

Najwięcej miedzi oraz cynku, niezależnie od formy rzepaku, zawierały wytłoki. Średnią koncentrację tego mikropierwiastka stwierdzono w resztkach korzeniowych, a najmniejszą – w słomie. Najwięcej manganu w 1 kg suchej masy zawierały resztki korzeniowe rzepaku ozimego. Rzepak jary zawierał najwięcej manganu w wytłokach. Koncentracja mikropierwiastków (Cu, Zn, Mn) w korzeniach rzepaku ozimego była nieznacznie większa niż u rzepaku jarego. Nawożenie siarką powodowało zmniejszenie zawartości miedzi oraz wzrost zawartości manganu w resztkach korzeniowych obu form rzepaku. Doglebowa aplikacja siarki powodowała wzrost koncentracji cynku w korzeniach rzepaku ozimego, nie różnicując istotnie jego zawartości w resztkach korzeniowych formy jarej rzepaku. Słoma rzepaku jarego zawierała znacznie więcej cynku i manganu w porównaniu z formą ozimą. Zawartość miedzi w słomie badanych gatunków była na porównywalnym poziomie. Doglebowa aplikacja siarki powodowała wzrost zawartości cynku i manganu w słomie rzepaku ozimego, nie różnicując jego koncentracji w słomie formy jarej. Wytłoki rzepaku jarego zawierały istotnie więcej miedzi i cynku w porównaniu z rzepakiem ozimym. Koncentracja manganu w wytłokach obu form rzepaku była podobna. Nawożenie siarką rzepaku ozimego powodowało istotny wzrost koncentracji cynku i manganu w wytłokach. Nawożenie siarką rzepaku jarego powodowało istotne zmniejszenie zawartości manganu w wytłokach, nie różnicując w nich koncentracji pozostałych mikropierwiastków

Słowa kluczowe: rzepak ozimy, rzepak jary, nawożenie siarką, zawartość mikroelementów, resztki korzeniowe, słoma, wytłoki.

INTRODUCTION

Oil bearing plants of the family *Brassicaceae* are characterized by high sulfur requirements, namely they need 15-20 kg of sulfur to produce 1 Mg of biomass (Zhao et al. 1993). The yield-forming effect of sulfur is particularly notable when winter rapeseed is grown in sulfur-deficient soil. Under such conditions, even low rates of sulfur fertilizer can be highly effective (McGrath, Zhao 1996, Krauze, Bowszys 2001, Zukalová et al. 2001*ab*, Podleśna 2004, Jankowski et al. 2008*ab*, Wielebski 2008, 2011). Sulfur applied to plants of the family *Brassicaceae* can considerably affect the quality of raw products such as oil, fat-free seed residues and straw. Sulfur fertilizers exert the strongest effect on the nutritional value of seeds, including concentrations of numerous biologically active compounds, mostly erucic acid in oil and glucosinolates, fiber, tannins, polyphenols and phytic acid in fat-free seed residues (Rotkiewicz et al. 1996). Sulfur fertilization affects the uptake of other nutrients, too. Ahmad et al. (1999), and McGrath and Zhao (1996) demonstrated that sulfur deficiency inhibits the nitrogen uptake by plants, thus suppressing their growth and development, indirectly modifying the chemical composition of the main and side-line crops.

Sulfur fertilization may significantly influence the chemical composition of postharvest residues of *Brassicaceae* species. These plants are grown on a steadily larger acreage partly because of their economic importance as food crops and energy crops and partly owing to their role in agricultural ecosystems. It should be stressed that although the root residues and straw of oil plants are a rich and often the major (in non-livestock farms) source of soil micronutrients, the micronutrient content of postharvest residues of oil crops still awaits more thorough investigations. Roots of winter rapeseed accumulate large amounts of zinc (*ca* 21.6-48.3 mg kg^{-1} d.m.) (Wiśniowska -Kielian 2003). Root residues of spring rapeseed are also a valuable source of zinc (148 mg kg^{-1} d.m.) (Szczebiot, Ojczyk 2002). High levels of manganese and zinc (10.65-14.28 and 6.15-8.68 mg kg^{-1} d.m., respectively) are found in straw of winter rapeseed. The copper content in aerial parts of winter rapeseed does not exceed 2.5 mg kg^{-1} d.m. (Spiak et al. 2007). It should be noted that in winter rapeseed, the copper content is 1.6-fold higher in vegetative organs than in seeds, and the concentrations of zinc and manganese in seeds are four- and three-fold higher, respectively, than in straw. Spring rapeseed straw contains high levels (34.6-37.2 mg kg^{-1} d.m.) of manganese (Krzywy, Iżewska 2007).

The aim of this study was to determine the effect of sulfur application to soil on the concentrations of micronutrients (copper, zinc and manganese) in root residues, straw and cake of winter and spring rapeseed.

MATERIAL AND METHODS

The experiment was conducted at the Agricultural Experimentation Station in Bałcyny (N = 53°35′49″, E = 19°51′20.3″), in 2005-2008. The experimental variables were:

primary variable – crop plant: winter rapeseed, spring rapeseed;

secondary variable – rate of sulfur fertilizer applied to soil: (+S) winter rapeseed – 60 kg ha^{-1}, spring rapeseed – 40 kg ha^{-1}, (-S) control – no sulfur fertilization.

The experiment had a split-plot design with three replications. The plot size was 18 m^2. Each year, the experiment was established on grey-brown podsolic soil developed from light loam, of good wheat complex. The soil had a slightly acid pH of 5.75-6.39 in 1 M KCl, medium copper and manganese content, and medium to high zinc content (Table 1). The preceding crop was spring barley grown after spring wheat (first and second growing season) or after winter wheat (third growing season).

Table 1

Soil conditions (2005-2008)

Specification	Growing season		
	2005/2006	2006/2007	2007/2008
Soil type	grey-brown podsolic soil		
Soil texture group	light loam		
Soil quality class	IIIa	IIIa	IIIb
Soil suitability for agriculture	good wheat complex		
Organic carbon content of soil (%)	1.47	1.75	1.57
Soil pH (1 M KCl)	6.39	6.08	5.75
Concentrations of available nutrients (mg kg^{-1} soil)			
– P	107	85	143
– K	104	133	104
– Mg	103	85	51
– S	163	140	144
– $S\text{-}S0_4$	25	10	10
– Cu	4.4	2.7	2.8
– Zn	23.1	11.1	10.9
– Mn	230	180	235

Winter rapeseed was fertilized pre-sowing with 30 kg N ha^{-1}, 22 kg ha^{-1} P and 166 kg ha^{-1} K. In spring, nitrogen was applied in three split doses: 120 (52.50* + 67.50) kg ha^{-1} before lateral branch development (BBCH 20), 80 kg ha^{-1} at inflorescence emergence (BBCH 50). Spring rapeseed was fertilized pre-sowing with 70 (35*+35) kg N ha^{-1}, 17 kg ha^{-1} P and 100 kg ha^{-1} K. At inflorescence emergence (BBCH 50), supplemental nitrogen was applied at 30 kg ha^{-1}. Boron was sprayed over leaves of winter rapeseed plants (BBCH 53) at 43.75 g ha^{-1}, as aqueous solution of Solubor DF. Phosphorus was applied to soil as triple superphosphate, potassium – as 60% potash salt, sulfur – as ammonium sulfate (*), nitrogen – as ammonium nitrate (treatments without sulfur fertilization) or ammonium sulfate (*) and ammonium nitrate (treatments with sulfur fertilization).

Winter rapeseed cv. Californium was sown in the first week or in the middle of August, at the density of 90 germinating seeds per 1 m^2 of plot area. Spring rapeseed cv. Hunter was sown in the first week or in the middle of April, at the density of 140 germinating seeds per 1 m^2 of plot area. Both winter and spring rapeseeds were dressed with thiamethoxam, metalaxyl-M and fludioxonil. The inter-row spacing was approximately 19 cm.

Dicotyledonous weeds were controlled with metazachlor and quinmerac, at 1 166 g ha^{-1} and 290.5 g ha^{-1} in winter rapeseed, and at 999 g ha^{-1} and 249 g ha^{-1} in spring rapeseed. In the winter rapeseed plantation, haloxyfop-R was applied at 52 g ha^{-1} at the stage of 4-6 leaves unfolded (BBCH 14-16).

In the first growing season, pest control required four and six insecticide treatments in winter and spring rapeseed fields, respectively. In the second and third growing season, high pest infestation coincided with a period of high pest susceptibility of spring rapeseed. Therefore, only one insecticide treatment was carried out in winter rapeseed, and four to six in spring rapeseed plots. Chemical control of plant pathogens was necessary only in winter rapeseed, and it involved the use of 100 g ha^{-1} dimoxystrobin and 100 g ha^{-1} boscalid at the stage of flower fading: most petals fallen (BBCH 66-67).

Harvest was carried out in two stages, at processing maturity. Winter rapeseed was swathed (cutting height of 8 cm) at the end of June or in mid-July. Spring rapeseed reached maturity in the first half of August.

The organic carbon content of soil was determined according to the research protocol of the Chemical and Agricultural Research Laboratory, SCHR PB 24, second edition of 21 June 2004. The available nutrient content and soil pH were determined in the plough layer, in accordance with the Polish Standards: PN ISO 10390:1997 (pH), PN-R-04023:1996 (phosphorus), PN-R-04022/Az1:2002 (potassium), PN-R-04020:1994 (magnesium), PN-92/R-04017 (copper), PN-92/R-04016 (zinc), and PN-93/R-04019 (manganese). The content of total sulfur and sulfate sulfur was calculated according to the research protocol of the Chemical and Agricultural Research Laboratory, SCHR PB 27 second edition of 21 June 2004 and SCHR PB 26, second edition of 21 June 2004.

The concentrations of copper, zinc and manganese were estimated in the dry matter of root residues (roots + stubble), straw and cake of winter and spring rapeseed. Samples for chemical analysis (roots + stubble, straw, and seeds) were collected at harvest. Root and stubble samples were collected with soil, by drilling to the depth of 30 cm with a steel cylinder (diameter – 22.57 cm, area – *ca* 400 cm^2). The samples were washed under running water in a 1 mm mesh sieve. Seeds were cold-pressed in a laboratory press with a pressing capacity of *ca* 50 kg h^{-1}. The oil content of cake ranged from 127 g kg^{-1} d.m. (winter rapeseed) to 129 g kg^{-1} d.m. (spring rapeseed). Dried samples of roots + stubble, straw and cake were ground in a laboratory mill. The concentrations of copper, zinc and manganese in biomass were determined by atomic absorption spectometry (AAS). The chemical properties of soil and the micronutrient content of plant material were analyzed in the Chemical and Agricultural Research Laboratory in Olsztyn.

The results of chemical analyses were processed by Anova in accordance with the experimental method. Mean values from every treatment were compared by the Tukey's test. LSD values were calculated at a 5% error rate.

RESULTS AND DISCUSSION

The application of sulfur fertilizers (ammonium sulfate) to winter and spring rapeseed had no significant effect on changes in the pH of the plough layer, which in turn were largely dependent on the form of rapeseed (Table 2). On average (means from three years), the pH of soil tended to increase under winter rapeseed and to decrease under spring rapeseed (Tables 1 and 2).

Table 2

Soil pH (1 M KCl) after rapeseed harvest

Crop plant	Growing season						Mean	
	2005/2006		2006/2007		2007/2008			
	+S	-S	+S	-S	+S	-S	+S	-S
Winter rapeseed	6.44	6.54	6.16	6.05	5.76	5.76	6.12±0.21	6.11±0.21
Spring rapeseed	6.06	6.23	5.87	5.89	5.44	5.38	5.79±0.22	5.83±0.22

The root dry weight of spring rapeseed in the plough layer (0-30 cm) was higher (by *ca* 12% on average) than that of winter rapeseed. The yields of straw and oil cake per ha were considerably higher (by 67% and over four-fold, respectively) by winter rapeseed than spring rapeseed. Sulfur fertilization had no significant effect on the yields of root residues and straw from winter and spring rapeseed. Sulfur application to soil resulted in a significant decrease in the yield of winter rapeseed cake per ha, and it had no significant effect on the yield of spring rapeseed cake (Figure 1).

The postharvest biomass of winter and spring rapeseed (root residues, straw and cake) differed considerably with respect to the concentrations of micronutrients (Cu, Zn, Mn) – Figure 2. In the biomass of winter and spring rapeseed, the cake was the richest source of copper and zinc, followed by root residues and straw. Manganese concentrations in the postharvest residues of winter and spring rapeseed were different. Winter rapeseed contained the largest amounts of manganese in root residues (46.4 mg kg^{-1} d.m.).

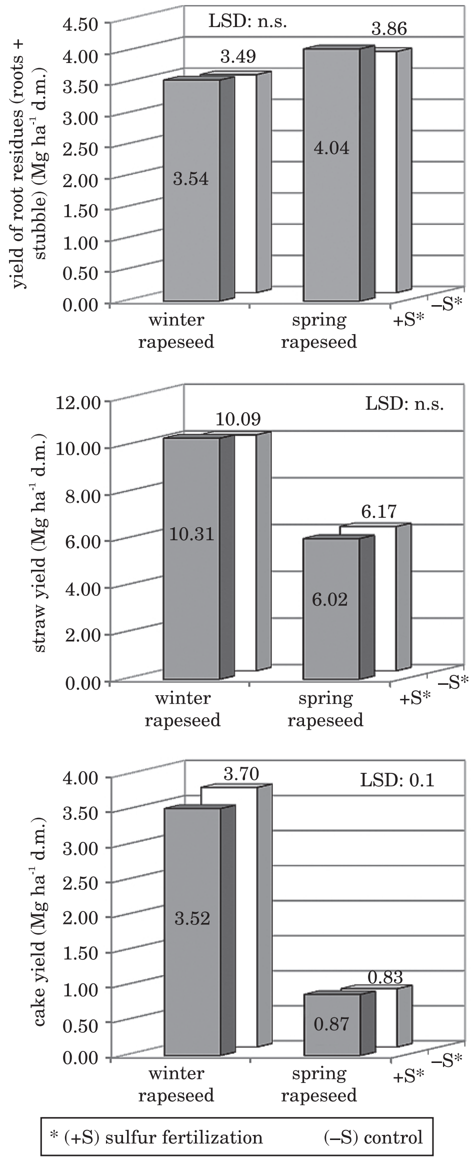

Fig. 1. Effect of sulfur fertilization on the biomass yield of winter and spring rapeseed in 2005-2008

The manganese content of cake and straw was lower by 8.6 and 36.2 mg kg^{-1} d.m., respectively. In spring rapeseed, manganese concentrations were the highest in cake (40.8 mg kg^{-1} d.m.), and lower in root residues (36.1 mg kg^{-1} d.m.) and straw (20.9 mg kg^{-1} d.m.) – Figure 2.

In the study by Gondek and Filipek-Mazur (2003), roots of winter rapeseed accumulated considerable amounts of zinc (*ca* 21.6-48.3 mg kg^{-1} d.m.).

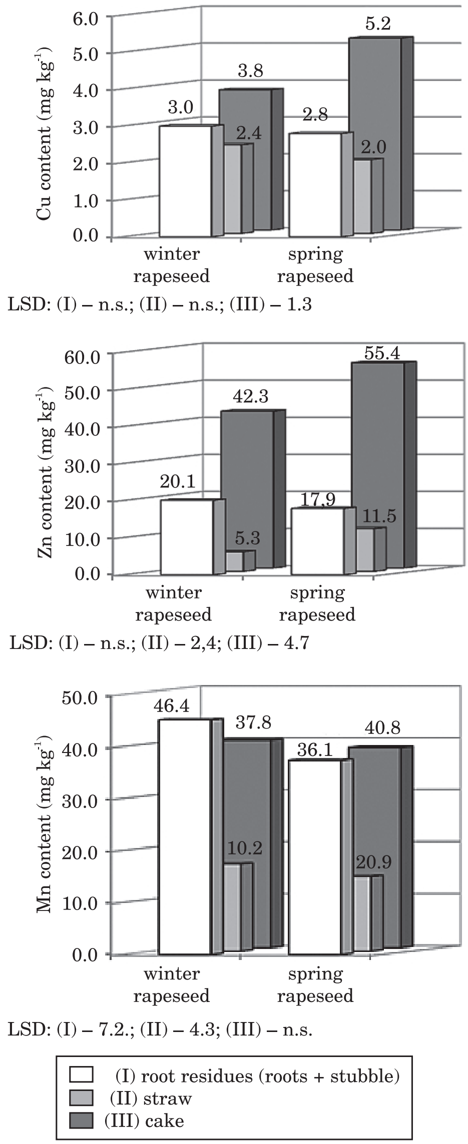

Fig. 2. Concentrations of copper, zinc and manganese in the root residues (roots + stubble), straw and cake of winter and spring rapeseed (means for crops), 2005-2008

SZCZEBIOT and OJCZYK (2002) also demonstrated that roots of spring oil plants contained mostly zinc; the highest zinc concentrations (148 mg kg^{-1} d.m.) were determined in spring rapeseed roots, followed by roots of Indian mustard (123 mg kg^{-1} d.m.) and white mustard (110 mg kg^{-1} d.m.). The post-harvest residues (roots + stubble) of spring oil crops are also a valuable source of manganese. In the study by SZCZEBIOT and OJCZYK (2002), root residues of white mustard were characterized by the highest manganese concentrations (124 mg kg^{-1} d.m.). The manganese content in roots of Indian mustard and spring rapeseed were by around 27% and 44% lower, respectively. The copper content of the root residues of winter rapeseed determined by GONDEK and FILIPEK-MAZUR (2003) was 2.6-3.1 mg kg^{-1} d.m., whereas SZCZEBIOT and OJCZYK (2002) reported that the copper content of spring rapeseed roots reached 3.0-3.5 mg kg^{-1} d.m.

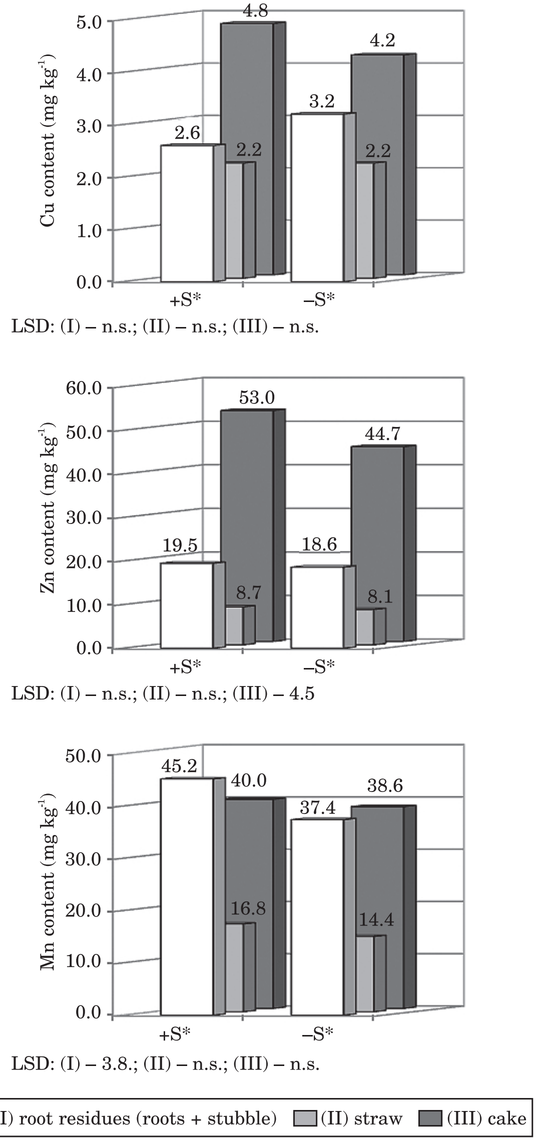

Fig. 3. Concentrations of copper, zinc and manganese in the root residues (roots + stubble), straw and cake of winter and spring rapeseed (means for sulfur fertilization), 2005-2008

The analysis of the data presented in Figure 3 shows that sulfur fertilization had no significant effect on copper concentrations in roots of winter and spring rapeseed. Sulfur application to soil caused a significant (14%) increase in zinc concentrations in roots of winter rapeseed, having no significant influence on the zinc content of spring rapeseed roots (Figure 4). Manganese content per kg root d.m. increased (by ca. 21% on average) in response to sulfur fertilization in both winter and spring rapeseed (Figures 3).

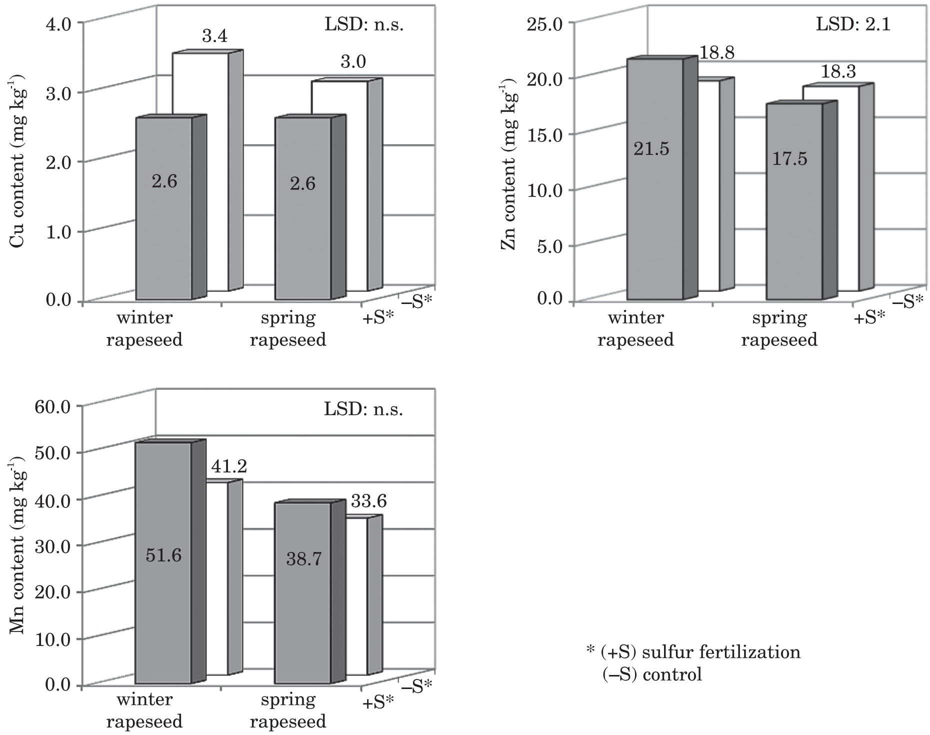

Fig. 4. Effect of sulfur fertilization on the concentrations of copper, zinc and manganese in the root residues (roots + stubble) of winter and spring rapeseed (interaction: crop · sulfur fertilization), 2005-2008

Spiak et al. (2007) reported that 1 kg d.m. of winter rapeseed straw contained 2.4-2.7 mg Cu, 6.2-8.7 mg Zn and 10.7-14.3 mg Mn. The straw of spring rapeseed is a rich source of manganese, whose content ranges from 34.6 to 37.2 mg kg^{-1} d.m. Krzywy and Iżewska (2007) reported that the zinc and copper content of spring rapeseed straw reached 4.0-4.4 mg kg^{-1} d.m. and 27.9-41.9 mg kg^{-1} d.m., respectively. In our study, the straw of winter and spring rapeseed contained primarily manganese (8.3-21.4 mg kg^{-1} d.m.) and zinc (4.7-11.6 mg kg^{-1} d.m.) – Figure 5. Spring rapeseed straw was a richer source of zinc and manganese than winter rapeseed straw. The concentrations of these minerals were over twofold higher in the straw of spring rapeseed than winter rapeseed. The copper content of straw was comparable in winter and spring rapeseed (2.0-2.4 mg kg^{-1} d.m.), and the noted differences were within the margin of error (Figure 2). Sulfur fertilization had no significant effect on copper concentrations in the straw of winter and spring rapeseed (Figure 3). Sulfur application to soil contributed to a substantial increase in the zinc and manganese levels in straw of winter rapeseed (by 23% and 46%, respectively), but it had no significant effect on the concentrations of those micronutrients in straw of spring rapeseed (Figure 5).

The fat-free seed residues of oil plants are a rich source of iron. The cake of spring oil-bearing crops (spring rapeseed, Indian mustard) contains higher levels of iron (138-266 and 133-162 mg kg^{-1} d.m.), compared with winter rapeseed (111-175 mg mg kg^{-1} d.m.). The concentrations of zinc and manganese in cake of winter rapeseed are similar (37-64 mg Zn and 41-63 Mn mg kg^{-1} d.m.) (Banaszkiewicz 1998, Kalembasa, Adamiak 2010). Fat -free seed residues of spring rapeseed are a richer source of manganese (52-61 mg kg^{-1} d.m.) than of zinc (37-43 mg kg^{-1} d.m.) (Bell et al. 1999). The copper content of fat-free seed residues was comparable in winter and spring rapeseed (*ca* 4.3-6.1 mg kg^{-1} d.m.). The cake of mustards contains higher concentrations of copper (by *ca* 35-57% on average) than the cake of rapeseed (Banaszkiewicz 1998, Bell et al. 1999, Kalembasa, Adamiak 2010).

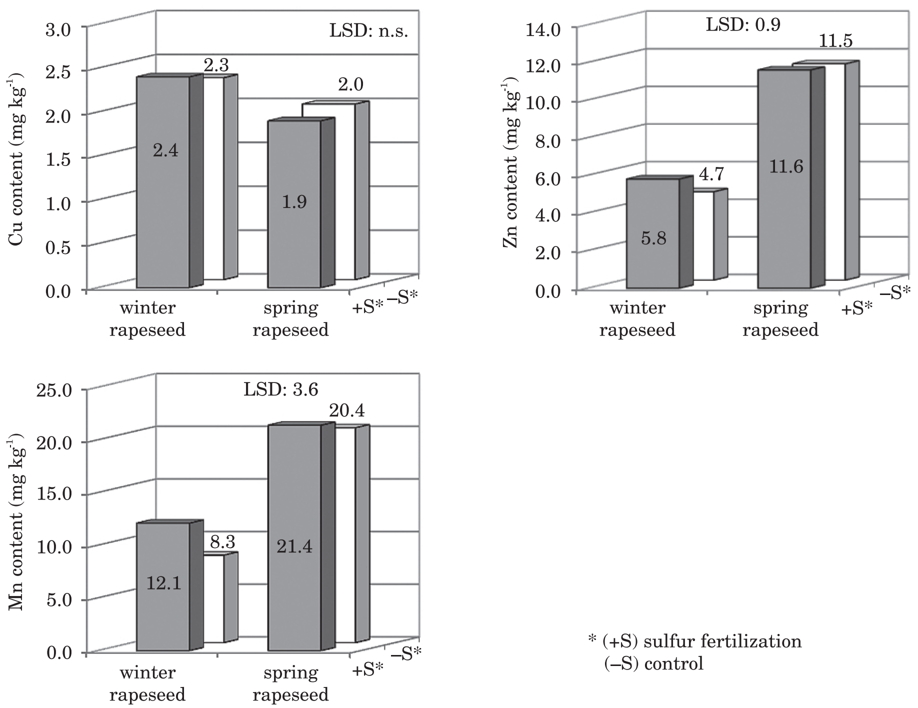

Fig. 5. Effect of sulfur fertilization on the concentrations of copper, zinc and manganese in the straw of winter and spring rapeseed (interaction: crop · sulfur fertilization), 2005-2008

In the present study, the cake of winter rapeseed contained the following amounts of copper, zinc and manganese: 3.8, 42.3 and 37.8 mg kg^{-1} d.m., respectively. The cake of spring rapeseed contained significantly higher concentrations of copper (by 1.4 mg kg^{-1} d.m.) and zinc (by 13.1 mg kg^{-1} d.m.) than the cake of winter rapeseed. Manganese levels in the cake of spring and winter rapeseed were similar (37.8-40.8 mg kg^{-1} d.m.) – Figure 2. Sulfur fertilization contributed to a significant increase in the concentrations of zinc and manganese in winter rapeseed cake. Sulfur application to soil considerably decreased the manganese content of spring rapeseed cake (by 24%), having no effect on the concentrations of copper and zinc (Figure 6).

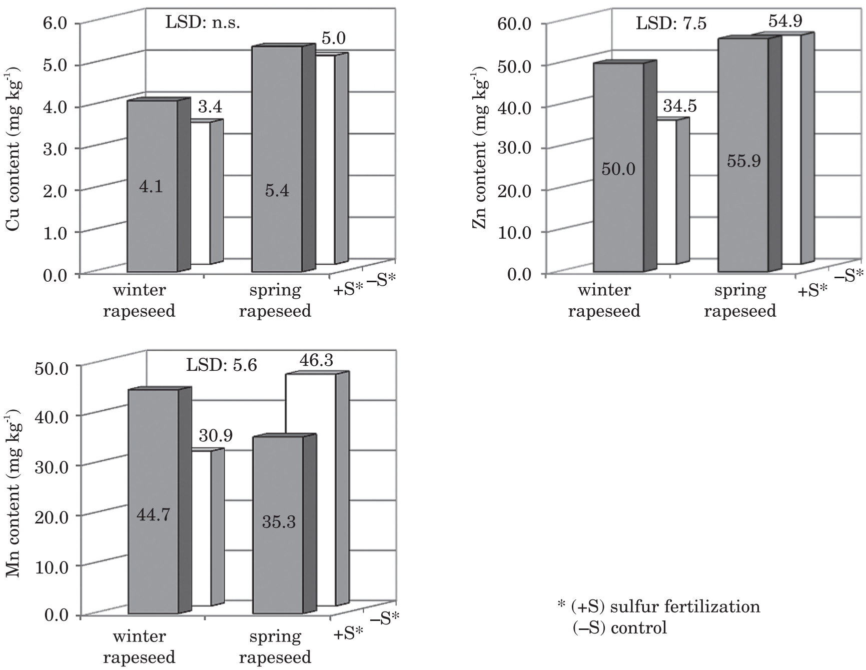

Fig. 6. Effect of sulfur fertilization on the concentrations of copper, zinc and manganese in the cake of winter and spring rapeseed (interaction: crop · sulfur fertilization), 2005-2008

CONCLUSIONS

1. In both spring and winter rapeseed, oil cake contained the highest levels of copper and zinc, followed by root residues and straw. The highest concentrations of manganese were found in the root residues of winter rapeseed and in the cake of spring rapeseed.

2. The concentrations of micronutrients were higher in roots of winter rapeseed than in roots of spring rapeseed. Sulfur fertilization decreased copper levels and increased manganese levels in the root residues of spring

and winter rapeseed, whereas zinc concentrations increased only in winter rapeseed roots.

3. Spring rapeseed straw contained considerably higher levels of zinc and manganese than winter rapeseed straw. The copper content of straw was comparable in spring and winter rapeseed. Sulfur application to soil increased the concentrations of zinc and manganese in winter rapeseed straw, but it had no significant effect on the levels of these minerals in spring rapeseed straw.

4. Spring rapeseed cake had a significantly higher content of copper and zinc than winter rapeseed cake. Manganese concentrations in the cake of spring and winter rapeseed were similar. Sulfur fertilization contributed to a significant increase in the concentrations of zinc and manganese in winter rapeseed cake. The manganese content of spring rapeseed cake decreased significantly in response to sulfur fertilization.

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