The effect of sulphur fertilization on macronutrient concentrations in the post-harvest biomass of mustard

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ABSTRACT

The aim of this study was to determine the effect of sulphur (S) fertilization on macronutrient concentrations in the roots, straw and oil cake of white mustard and Indian mustard. The experiment was conducted in northeastern Poland (2006–2008). The highest content of N, P, K, Mg and S was noted in the oil cake of both mustard species. Sulphur fertilization increased S concentrations and had no effect on Mg concentrations in the roots of white mustard and Indian mustard. Sulphur fertilizers applied to soil significantly increased the content of N, K and Ca in the roots of Indian mustard, whereas they had no significant effect on the content of N, P and Ca, and decreased K concentrations in the roots of white mustard. Sulphur fertilization led to a significant decrease in N content, and an increase in the content of K and Ca in the straw of both mustard species. Sulphur fertilization significantly decreased P concentrations and increased S concentrations in the oil cake of both mustard species. In response to S fertilization, the content of N and K decreased in the oil cake of Indian mustard, but did not change significantly in the oil cake of white mustard. Therefore, S fertilization significantly differentiated the fertilizing value of post-harvest residues and the feeding value of white mustard and Indian mustard seeds.

Keywords: Brassicaceae crops; Sinapis alba; Brassica juncea; mineral composition

In 2009–2013, the global area under mustard cultivation ranged from 634 000–866 000 ha, and annual mustard seed production reached 516 000–705 000 t. Global leaders in mustard seed production are Canada, Nepal and Myanmar. In Europe, mustard seed production in 2009–2013 ranged from 125 000–228 000 t. European leaders in mustard seed production are the Ukraine, Russia and the Czech Republic (FAOSTAT 2014). The economic importance of mustards may increase in the nearest future due to the highly promising results of breeding programs established to develop mustard cultivars with canola quality, i.e. with reduced levels of erucic acid and glucosinolates (GLS). In Europe, hopes are pinned on double low cultivars of white mustard, which are currently being tested as an alternative to spring rapeseed to be grown on lighter and drier soils (Piętka et al. 2010).

On European markets, the demand for mustard seeds is driven mainly by the fact that their oil-free residues accumulate specific biologically active compounds – GLS. Glucosinolate degradation products are used mainly in the production of mustards. Whole mustard seeds and their fat-free residues (mustard powder) are used as spices in the meat processing industry (white, Indian and black mustard), in pickled products (mainly white mustard) and herb pepper (white and black mustard). Mustard seeds, in particular Indian and black mustard, are also applied in the

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pharmaceutical industry to produce heat rubs and mustard plasters (Toboła 2010). In comparison with non-cruciferous crops, plants of the family Brassicaceae have high sulphur (S) requirements, which can be attributed to their ability to biosynthesize GLS (Zukalová and Vašák 2002, Grant et al. 2012, Ray et al. 2015). Sulphur fertilizers applied to soil in order to meet the S requirements of Brassica oilseed crops can significantly modify the chemical properties (including pH) and microbial activity of soil (Chlopecka et al. 1996, Blaylock et al. 1997, Kulhánek et al. 2011), leading to changes in the accumulation of mineral compounds in the tissues of Brassica oilseed crops (Cui et al. 2004, Balík et al. 2006, Markiewicz et al. 2000, Balík et al. 2007, Skwierawska et al. 2014). Changes in the accumulation of micro- and macroelements in the tissues of Brassica crops may affect their feeding value (Smulikowska 2006), the energy content of biomass (Jankowski et al. 2015a) and the fertilizing value of post-harvest residues (Jankowski et al. 2014a,b).

The objective of this study was to determine the N, P, K, Ca, Mg and S content of the root residues, straw and oil cake of white mustard and Indian mustard fertilized with S applied to soil at different rates.

MATERIAL AND METHODS

Field experiments with white mustard (Sinapis alba L.) and Indian mustard (Brassica juncea (L.) Czern. et Coss.) were conducted in 2006–2008 at the Agricultural Experiment Station in Balczyna (53°35’46.4”N, 19°51’19.5”E, 137 m a.s.l.), owned by the University of Warmia and Mazury in Olsztyn (northeastern Poland). The experimental variable was the presence or absence of S fertilization: +S and –S treatments, respectively. The applied S rates were considered optimal based on the forecast yield and S uptake, and they were within the ranges recommended for soils with moderate levels of S for the analyzed oilseed crops of the family Brassicaceae (Toboła 2010, Barczak et al. 2011 (white mustard), Malhi et al. 2007 and Toboła 2010 (Indian mustard)). In +S treatments, sulphur was applied to soil at 25 kg/ha (Indian mustard) or 40 kg/ha (white mustard). Sulfur and N were applied to soil as ammonium sulfate and ammonium nitrate, respectively, in +S treatments, whereas –S treatments received only ammonium nitrate. Phosphorus was applied to soil as triple superphosphate and K as 60% potash salt (+S and –S).

The experiment had a randomized complete block design with three replications. Plot size was 18 m². Each year, the experiment was established on Haplic Luvisol developed from boulder clay (IUSS 2006). The soil had a slightly acidic pH ranging from 5.75–6.39 in 1 mol/L KCl. Soil nutrient levels were as follows: 1.47–1.75% Corg (Kurmies method), 85–143 mg P/kg (Egner-Riehm method), 104–133 mg K/kg (Egner-Riehm method), 51–103 mg Mg/kg (atomic absorption spectrometry – AAS, Carl Zeiss Jena, Germany), 3.3–8.3 mg S/kg (Bardsley and Lancaster method), 2.8–4.4 mg Cu/kg (AAS), 11–23 mg Zn/kg (AAS) and 180–235 mg Mn/kg (AAS).

The applied farming operations (including dates) are presented in Table 1. The seeding rate, seed-

<table>
<thead>
<tr>
<th>Farming operation</th>
<th>Month of operation and agricultural inputs</th>
</tr>
</thead>
<tbody>
<tr>
<td>Skimming (5–8 cm)</td>
<td>July</td>
</tr>
<tr>
<td>Fall ploughing (18–22 cm)</td>
<td>July</td>
</tr>
<tr>
<td>Sowing</td>
<td>October</td>
</tr>
<tr>
<td>Mineral fertilization</td>
<td>October</td>
</tr>
<tr>
<td>pre-sowing N/P/K</td>
<td>cv. Borowska (130a) April</td>
</tr>
<tr>
<td>top-dressing N</td>
<td>70/17/100; April</td>
</tr>
<tr>
<td>Chemical crop protection</td>
<td>70/13/66; April</td>
</tr>
<tr>
<td>herbicides</td>
<td>30; May</td>
</tr>
<tr>
<td>insecticides</td>
<td>105; May</td>
</tr>
<tr>
<td>Seed and straw harvest</td>
<td>10–83; April, May, June</td>
</tr>
<tr>
<td></td>
<td>58–82; April, May, June</td>
</tr>
<tr>
<td></td>
<td>August</td>
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<td></td>
<td>August</td>
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</tbody>
</table>

a germinating seeds per 1 m²; b kg/ha; c g/ha active ingredient; d variations resulting from differences in pest intensity in each year of the study
ing depth, NPK fertilization rates, and chemical protection against weeds, pests and diseases were determined for each mustard species at locally optimal levels for the analyzed region. The only exception was the rate of S fertilization which was selected based on the second experimental factor (locally optimal rate and control – no S fertilization). The biomass yield per 1 ha of white mustard and Indian mustard has been given by Jankowski et al. (2014b).

Macronutrient content was determined in the roots and stubble, straw and oil cake of white mustard and Indian mustard on a dry matter basis. Samples for chemical analyses were collected upon harvest. Roots with stubble and soil were sampled to a depth of 30 cm. Seed samples were cold pressed in a laboratory press with the estimated output of 50 kg/ha. Samples of dried roots with stubble, straw and oil cake were ground in a laboratory mill. The samples were wet mineralized in H₂SO₄, and Norg content was determined by the Kjeldahl method, P content – by the vanadium-molybdenum method, K and Ca content – by atomic emission spectrometry (ESA), and Mg content – by AAS. Total S was determined turbidimetrically in plant material that had been incinerated with nitric acid and magnesium nitrate to sulfate form. The results were checked against certified reference materials, Virginia Tobacco Leaves (CTA VTL-2), with the error of: P – 4.5%, K – 2%, Ca – 2.8%, Mg – 1.5% (Dybczyński et al. 1998).

Data were analyzed by ANOVA and treatment means were compared with the Duncan’s test (P ≤ 0.05) using Statistica 10.1 PL software (StatSoft, Inc. 2011).

### RESULTS AND DISCUSSION

**Root residues.** In the group of oilseed crops examined by Szczebiot and Ojczyk (2002), the root residues of spring rapeseed had the lowest N content (9.8 g/kg dry matter (DM)), and the root residues of Indian mustard and white mustard accumulated the highest N concentrations (12.0 and 12.7 g/kg DM, respectively). The root residues of white mustard, compared with the root residues of Indian mustard, had a higher content of K (by 1.9 g/kg DM), Ca (by 1.3 g/kg DM), Mg (by 0.8 g/kg DM) and S (by 0.7 g/kg DM) (Szczebiot and Ojczyk 2002). Different results were noted in our study, where the root residues of Indian mustard had a significantly higher content of K, Ca and S than the root residues of white mustard. The content of N, P and Mg in the roots of both mustard species was similar (Table 2). The difference could be attributed to varied agricultural conditions during the field experiment. In our study, precipitation levels during the spring/summer growing season were 15–20% higher on average than in the study by Szczebiot and Ojczyk (2002). White mustard develops a much stronger root system than Indian mustard (Toboła 2010). In soils characterized by a lower moisture content (Szczebiot and Ojczyk 2002), plants with more extensive root systems (white mustard) accumulate more nutrients (K, Ca and S) than plants with weaker roots (Indian mustard) (Grzebisz 2008). At higher levels of precipitation (this study), the concentrations of those macronutrients in root residues are less influenced by the size of the root system and are more likely to be determined by their involvement in the

<table>
<thead>
<tr>
<th>Oilseed crop</th>
<th>Content (g/kg dry matter)</th>
<th>N</th>
<th>P</th>
<th>K</th>
<th>Ca</th>
<th>Mg</th>
<th>S</th>
</tr>
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<tbody>
<tr>
<td>Root residues</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>(roots + stubble)</td>
<td>White mustard</td>
<td>6.3</td>
<td>1.5</td>
<td>9.9b</td>
<td>4.5b</td>
<td>1.0</td>
<td>1.2b</td>
</tr>
<tr>
<td></td>
<td>Indian mustard</td>
<td>6.3</td>
<td>1.6</td>
<td>11.7a</td>
<td>4.9a</td>
<td>1.1</td>
<td>1.8a</td>
</tr>
<tr>
<td>Straw</td>
<td>White mustard</td>
<td>6.7a</td>
<td>1.2b</td>
<td>7.2b</td>
<td>10.8a</td>
<td>1.2</td>
<td>1.4</td>
</tr>
<tr>
<td></td>
<td>Indian mustard</td>
<td>6.5b</td>
<td>1.7a</td>
<td>7.9a</td>
<td>9.6b</td>
<td>1.3</td>
<td>1.5</td>
</tr>
<tr>
<td>Oil cake</td>
<td>White mustard</td>
<td>56.2a</td>
<td>11.8</td>
<td>11.4</td>
<td>4.8a</td>
<td>4.0</td>
<td>10.0a</td>
</tr>
<tr>
<td></td>
<td>Indian mustard</td>
<td>50.8b</td>
<td>12.1</td>
<td>11.0</td>
<td>4.1b</td>
<td>4.2</td>
<td>8.7b</td>
</tr>
</tbody>
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Values marked with the same letter are not significantly different at P ≤ 0.05
biosynthesis of compounds specific for *Brassica* oilseed crops: GLS and fat. The synthesis of those compounds is directly influenced by the levels of plant-available S (GLS) and K (fat) (Grzebiś 2008, Toboła 2010, Jankowski et al. 2015b). Indian mustard accumulates 3- to 10-times more GLS in roots and 8–9% more fat in seeds than white mustard (Jankowski et al. 2015b). Therefore, under supportive agricultural conditions, Indian mustard can accumulate more K and S than white mustard.

Sulphur fertilization contributes to a significant increase in the S content of *Brassica* oilseed crops (Filipek-Mazur and Gondek 2005, Balk et al. 2009). Sulphur fertilization also led to an over two-fold increase in S concentrations in the roots of white mustard (from 2.2–5.9 g/kg DM) (Filipek-Mazur and Gondek 2005). In our experiment, S fertilizers applied to soil significantly increased S concentrations in the root residues of white mustard and Indian mustard (by 0.3–0.4 g/kg DM), but they had no effect on Mg content (Figure 1). Sulphur fertilization had varied effects on the concentrations of the remaining macronutrients in the root residues of both mustard species. Sulphur fertilizers applied to soil significantly increased the content of N and Ca in the root residues of Indian mustard (by 0.7 and 1.4 g/kg DM), whereas they had no significant influence on the content of those macronutrients in the root residues of white mustard. The application of S led to a significant decrease (by 0.5 g/kg DM) in the K content of roots and stubble of white mustard, and to a significant increase (by 0.8 g/kg DM) in the K content of root residues of Indian mustard. Sulphur fertilization caused a significant decrease in the P content of root residues of Indian mustard (by 0.2 g/kg DM), whereas it had no influence on the P content of roots and stubble of white mustard (Figure 1).

**Straw.** The straw of *Brassica* oilseed crops is a rich source of Ca (17–20 g/kg DM) (Jakubus 2006, Spiak et al. 2007). The straw of spring rapeseed is also an abundant source of Ca, but it contains less of this element than winter rapeseed (by 8–9 g/kg DM). High Ca concentrations in the straw of white mustard (10.8 g/kg DM) and Indian mustard (9.6 g/kg DM) were also noted in our study (Table 2). In comparison with Ca, the content of K and N in mustard straw was 25% and 35% lower, respectively, and the content of P, Mg and S was 7- to 8-fold lower. The straw of white mustard accumulated more N and Ca than the straw of Indian mustard, by 0.2 and 1.2 g/kg DM, respectively. The content of P and K was higher (by 0.5 and 0.7 g/kg DM) in the straw of Indian mustard, compared with white mustard. The content of Mg and S was similar in the straw of both mustard species (Table 2).

In a pot experiment conducted by Ryant and Hlušek (2007), S fertilization of white mustard grown on slightly acidic soil decreased the content of N, K and Ca in straw (by 3.0, 0.8 and 2.2 g/kg DM), but it did not affect the content of P and S. Sulphur fertilizers contributed to an increase in the concentrations of macronutrients (N, P, K and S) in the straw of Indian mustard (Tripathi et al. 2011). In our study, S applied to soil had a weak influence on the macronutrient content of straw of white mustard and Indian mustard (Figure 2). Sulphur fertilization significantly decreased the

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**Figure 1.** The effect of sulphur fertilization on the macronutrient content of root residues (roots + stubble) of white mustard (a) and Indian mustard (b) in 2006–2008. Values marked with the same letter are not significantly different at $P \leq 0.05$ according to the Duncan’s test; DM – dry matter
N content of straw of white mustard and Indian mustard (by 1.3 and 0.4 g/kg DM, respectively) and increased the concentrations of K (by 0.8 g/kg DM) and Ca (by 0.4 and 0.2 g/kg DM, respectively) (Figure 2).

**Oil cake.** The potential fertilizing value of oil cake of *Brassica* oilseed crops results mostly from its high N content. In the work of Jankowski et al. (2015b), the oil-free seed residues of white mustard and Indian mustard contained 49.9 g N/kg DM and 45.6 g N/kg DM, respectively. In the present study, the oil cake of both mustard species accumulated higher concentrations of N, in comparison with the remaining macronutrients, from 4- to 6-fold (P, K and S) up to 12- to 13-fold (Ca and Mg) (Table 2). The oil cake of white mustard had a significantly higher content of N, Ca and S than the oil cake of Indian mustard, by 5.4, 0.7 and 1.3 g/kg DM, respectively. The oil cake of both mustard species accumulated similar amounts of the remaining macroelements (Table 2).

Sulphur fertilization increases the content of N and S in the seeds of Indian mustard, by 0.7–11.4 and 0.3–2.7 g/kg DM, respectively (Markiewicz et al. 2000, Tripathi et al. 2011). It should be noted that S fertilizers contribute to a greater increase in S concentrations in the seeds of traditional cultivars of Indian mustard, compared with double low (canola) cultivars, by 5.1 and 1.0 g/kg DM, respectively (Malhi et al. 2007). Markiewicz et al. (2000) demonstrated that S concentrations in the seeds of Indian mustard fertilized with S increased with increasing rates of top-dressed N. Sulphur fertilization increases S content also

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**Figure 2.** The effect of sulphur fertilization on the macronutrient content of straw of white mustard (a) and Indian mustard (b) in 2006–2008. Values marked with the same letter are not significantly different at $P \leq 0.05$ according to the Duncan's test; DM – dry matter

**Figure 3.** The effect of sulphur fertilization on the macronutrient content of oil cake of white mustard (a) and Indian mustard (b) in 2006–2008. Values marked with the same letter are not significantly different at $P \leq 0.05$ according to the Duncan's test; DM – dry matter
in white mustard seeds. In a study by Ryant and Hlušek (2007), S content increased (by 1.0 g/kg DM) and P content decreased (by 0.5 g/kg DM) in the seeds of white mustard grown on slightly acidic soil and fertilized with S. In the cited study, the content of the remaining macronutrients in white mustard seeds did not change significantly in response to S fertilization. Markiewicz et al. (2000) also reported that S fertilizers applied to soil had a weak influence on the content of macroelements (except for S) in white mustard seeds. In our experiment, S fertilization contributed to a significant decrease in P concentrations (by 1.0 g/kg DM) and a significant increase in S concentrations (by 1.7–2.2 g/kg DM) in the oil cake of white mustard and Indian mustard grown on slightly acidic soil (Figure 3). In response to S fertilization, the content of N and K decreased in the oil cake of Indian mustard (by 2.1 and 1.0 g/kg DM, respectively), but remained unchanged in the oil cake of white mustard. Sulphur fertilizers had no significant effect on the content of Ca and Mg in the oil cake of both mustard species (Figure 3).

In conclusion, our findings suggest that S fertilization significantly differentiated the fertilizing value of root residues and straw, and the feeding value of white mustard and Indian mustard oil cake. Sulphur fertilization induced more pronounced changes in the macronutrient content of root residues and oil cake in Indian mustard than in white mustard. Sulphur fertilization had a similar effect on the macronutrient content of straw in both mustard species. Sulphur fertilization clearly improved the fertilizing value (determined based on macronutrient levels) of root residues in Indian mustard (N, K, Ca and S concentrations increased by 0.7, 0.8, 1.4 and 0.3 g/kg DM, respectively). In white mustard, S fertilization significantly increased (by 0.4 g/kg DM) only the S content of root residues and contributed to a significant decrease in K levels (by 0.5 g/kg). Sulphur fertilization significantly lowered the N content of straw in white mustard and Indian mustard (by 1.3 and 0.4 g/kg DM, respectively) and increased the concentrations of K (by 0.8 g/kg DM) and Ca (by 0.4 and 0.2 g/kg DM, respectively). In Indian mustard, S fertilization reduced the feeding value of oil cake by decreasing N (by 2.1 g/kg DM), P (by 1.0 g/kg DM) and K (by 1.0 g/kg DM) levels. In white mustard, only P concentrations decreased (by approximately 1.0 g/kg DM) in response to S fertilization. Sulphur was the only macronutrient whose content increased (by 2.2 and 1.7 g/kg DM, respectively) in the oil cake of white mustard and Indian mustard fertilized with this element.

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