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Fertilization efficiency of wood ash pellets amended by gypsum and superphosphate in the ryegrass growth

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ABSTRACT

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Application of biomass ash to soil can save mineral nutrients due to its relatively high contents of Ca, K, and P. The study assessed the effect of powdered ash and pellets made from wood fly ash (WFA), combined moreover with additives rich in S (flue gas desulfurization gypsum – FGDG) and P (single superphosphate – SP) on the yield and uptake of nutrients (Ca, K, P, and S) by ryegrass (*Lolium perenne* L.), the accumulation of nutrients in plant biomass at individual four cuttings, and the available nutrients amount in the acidic loamy soil after the last harvest. Plants grown in pots enriched by wood ash showed significantly higher yield and nutrient uptake than in the unamended treatments. The uptake of nutrients by plants, content of nutrients in plants and in soil was substantially positively influenced by both components added to the wood ash, especially by FGD gypsum. The combination of wood ash with additives proved to be effective. The soil enrichment by WFA + SP + FGDG increased the availability of SP-contained P and available P content in soil even after harvest.

Keywords: plant nutrition; soil amendments; sulfur; phosphorus; plant growth

Currently, there is a growing interest in the agricultural utilization of biomass combustion by-product – ash. Returning biomass ash to agricultural land is beneficial thanks to the fertilizing potential which is determined by the Ca, K, P, and Mg content (Pels et al. 2005) as well as micronutrient contents (Ochecová et al. 2014), and thanks to the highly alkaline pH (Mercl et al. 2016, Ochecová et al. 2016). The application of untreated wood ash creates severe dust problems and stabilization is necessary to make uniform spreading feasible. The use of a compaction technique can help to solve dust problems, decrease heterogeneity of ash application and costs of transport. The compaction process also permits to control particle size and composition of the ash products (Holmberg et al. 2000). Ash contains usually sufficient amounts of Ca and K but P is represented by smaller amounts of approximately 1%, and the most S is lost as gas during combustion process similarly to nitrogen. The biomass ash and materials rich in P or S implemented into ash pellets could be beneficial for plant nutrition. Superphosphate (SP) is a commonly used P fertilizer; flue gas desulfurization gypsum (FGDG) represents a new large source of S. FGDG ($CaSO_4 \cdot 2\,H_2O$) is produced when brown coal is burned (Chen and Dick 2011). Concentration of Ca and S in gypsum usually ranges between 20–24% for Ca and 17–19% for S. FGDG can provide a continual release of S to the soil and it has been

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verified for grass and also for many crops such as corn, soybean, alfalfa, wheat, sorghum, etc. (Chen et al. 2005, 2008, Lee et al. 2008, Chen and Dick 2011, Shi et al. 2011).

The main objective of the present study was to test the efficiency of dust ash application with comparison to the compacted ash pellets and the amendments of P and S in compacted ash on the growth and nutrient uptake by ryegrass (*Lolium perenne* L.) in the pot experiment.

MATERIAL AND METHODS

Soil. The experimental soil was Cambisol (loam – the textural class was classified according to the FAO Soil Classification) originated from the field near the town of Humpolec (49°33'N, 15°21'E) in the Czech Republic. Soil samples were collected from topsoil in the layer at 0–20 cm depth, air-dried at 20°C, ground in a mortar, and passed through a 15 mm sieve before establishment of the pot experiment. Soil characteristics are presented in Table 1.

Pot experiment. The pot experiment was set up in 6 treatments, each in 4 replicates. Air-dried soil (5 kg) was either mixed with powdered or pelleted ash alone or in combination with SP, FGDG or both (SP and FGDG were applied as a constituent of the pellet), and a treatment without ash addition was used as control (Table 2). The amount of the applied ash was 100 g in all treatments, representing 2% w/w per pot. However, since the pellets were partly formed by other additives, 100 g of ash corresponded to different amounts of ash mixture in the different treatments (Table 2). The amounts of nutrients added in each treatments in form of amendment are shown in Table 3.

Table 1. Physico-chemical characteristics of the experimental soil

Soil property		Cambisol
Altitude (m a.s.l.)		527
Mean annual temperature (°C)		7.1
Mean annual precipitation (mm)		603
pH_{CaCl_2}		5.0
Cation exchange capacity ($mmol_+$/kg)		68
Total organic carbon (%)		1.3
Pseudototal Ca (%)		0.2
Pseudototal K (%)		0.04
Pseudototal P (%)		0.09
Pseudototal S (%)		0.03
Soil texture		loam
Soil particle size (%)	sand	30.2
	silt	48.4
	clay	21.4

All values represent means (n = 3)

Wood fly ash (WFA) originated from a combustion plant with production of 27 000 tons of ash per year. The producer uses a specific combustion technology – fluid burning (820°C).

Soil-ash mixtures were mixed thoroughly, placed into 5 L plastic pots, and moistened by deionized water to keep 60% of water holding capacity (determined for each treatment separately).

As the experimental plant, ryegrass (*Lolium perenne* L.) was chosen in the seed rate 20 g/m^2. Sowing took place on 9 May 2013. During the vegetation, weeds were removed to avoid interplant competition. The pots received N fertilization (0.5 g N in the form NH_4NO_3) before sowing, and after 1st and 2nd harvest.

Table 2. Experimental design

Treatment	Ash form	Ash portion in amendment (%)	Amount of applied amendment (g/pot)	Pellet enrichment (%)
I	no ash	0	0	–
II	powdered	100	100	–
III	pelleted	90	111.1	–
IV	pelleted	88	113.6	SP (2)
V	pelleted	78	128.2	SP (2) + FGDG (10)
VI	pelleted	70	142.9	FGDG (20)

SP – single superphosphate; FGDG – flue gas desulfurization gypsum. Pelleted amendments contained 10% (w/w) of inert binder

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Table 3. Total amount of nutrients applied as an amendment (g/pot)

Treatment	Ca	K	P	S
I	–	–	–	–
II	10.2	5.10	0.79	1.20
III	10.2	5.10	0.79	1.20
IV	10.8	5.11	0.95	1.58
V	13.6	5.13	0.95	4.02
VI	16.4	5.15	0.79	6.61

The experiment was conducted in the rain-controlled vegetation hall (50°13'N, 14°37'E). The pots were randomized side by side on the bench exposed to daylight and outdoor climate. Plants were irrigated regularly by deionized water to maintain optimal growth conditions.

Biomass was harvested manually with scissors at a height of 2 cm from soil surface on 27 June, 23 July, 26 August, and 8 October (four cuts) in order to distinguish possible differences in nutrient release dynamics among treatments. For each pot, plant material was dried at 40°C, dry matter was weighed, ground through 0.75 mm sieve and homogenized. The soil samples were taken from 25 cm deep profile using the soil sampler (five samples from different places of each pot with total weight of approximately 100 g) at the end of the experiment. Then, the soil samples were air-dried at ambient temperature, ground in a mortar, passed through a 2-mm plastic sieve, and homogenized.

Analytical procedures. Non-destructive X-ray fluorescence (XRF) spectrometry (Spectro IQ, Kleve, Germany) was used for the determination of total nutrient contents in the ash and amendments.

Harvested plant material was pressure-digested according to Száková et al. (2013). The Ca and K concentrations were determined by flame atomic absorption spectrometry (F-AAS, Varian 280FS, Varian, Mulgrave, Australia), while P and S were determined by inductively coupled plasma optical emission spectrometry (ICP-OES, Varian, VistaPro, Mulgrave, Australia) (Száková et al. 2013). RM IAEA V-10 Hay Powder (Analytika, Prague, Czech Republic) was used as the certified reference material. The soil pH was determined after extraction with 0.01 mol/L $CaCl_2$ (w/v =

Table 4. Nutrient concentration in dry matter biomass of plants (%) at individual harvests

Harvest/Treatment		I	II	III	IV	V	VI
Ca	1st	0.67^{aB}	0.69^{aB}	0.67^{aB}	0.65^{aB}	0.61^{aBC}	0.69^{aB}
	2nd	0.53^{aA}	0.52^{aA}	0.49^{aA}	0.51^{aA}	0.46^{aA}	0.50^{aA}
	3rd	0.62^{cAB}	0.59^{bcAB}	0.52^{abcA}	0.51^{abcA}	0.49^{abAB}	0.47^{aA}
	4th	0.67^{aB}	0.59^{aAB}	0.66^{aB}	0.66^{aB}	0.63^{aC}	0.71^{aB}
K	1st	3.61^{aBC}	4.20^{abC}	4.37^{abC}	4.21^{abB}	4.67^{bC}	4.69^{bC}
	2nd	3.21^{abB}	3.39^{abB}	3.64^{bB}	3.03^{aA}	3.50^{abA}	3.12^{abA}
	3rd	3.82^{aC}	4.65^{cD}	4.67^{cC}	4.40^{bcB}	4.04^{abB}	4.00^{abB}
	4th	2.47^{aA}	2.80^{abcA}	2.94^{bcA}	2.65^{abA}	3.08^{cA}	2.85^{abcA}
P	1st	0.32^{aB}	0.31^{aB}	0.31^{aB}	0.24^{aA}	0.28^{aA}	0.29^{aC}
	2nd	0.24^{aA}	0.25^{aA}	0.22^{aA}	0.24^{aA}	0.25^{aA}	0.18^{aA}
	3rd	0.25^{abcA}	0.29^{cAB}	0.24^{abA}	0.28^{bcA}	0.28^{bcA}	0.22^{aAB}
	4th	0.23^{aA}	0.26^{aAB}	0.26^{aA}	0.27^{aA}	0.25^{aA}	0.25^{aBC}
S	1st	0.22^{aA}	0.48^{bA}	0.68^{bBC}	0.48^{bA}	0.57^{bA}	0.93^{cC}
	2nd	0.31^{aAB}	0.44^{bA}	0.54^{cA}	0.48^{bcA}	0.50^{bcA}	0.53^{bcA}
	3rd	0.37^{aB}	0.80^{bB}	0.78^{bC}	0.77^{bB}	0.80^{bB}	0.79^{bB}
	4th	0.32^{aAB}	0.49^{bA}	0.62^{cAB}	0.60^{bcAB}	0.69^{cAB}	0.80^{dB}

All values represent means (*n* = 4). Different lower case letters indicate significant differences ($P < 0.05$) among the treatments in individual harvests. Different capital letters indicate significant differences ($P < 0.05$) among harvest times for each nutrient and treatment individually

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1/2.5) and measured by WTW pH 340i meter with ion-selective electrode (WTW, Weilheim, Germany). Cation exchange capacity (CEC) was calculated according to the ISO 11260 (1994). Total organic carbon (TOC) was determined by the method described by Sims and Haby (1971). For determination of potentially available elements in soils after harvest, Mehlich 3 extraction procedure was applied (Mehlich 1984).

Statistical analysis. The effects of WFA application were examined by one-way analysis of variance including all treatments and harvests. When this test revealed significant differences, the mean values were compared by Tukey's means test ($P < 0.05$). The software Statistica 12.0 (Statsoft, Tulsa, USA) was used.

RESULTS AND DISCUSSION

The highest Ca concentration in ryegrass was found out at the first and last cuttings (Table 4), which correspond with the lowest biomass yield, but no significant differences were observed among the treatments even in the treatments with FGDG. Bailey (1995) observed no response to gypsum only at first cut of ryegrass.

The highest K concentration in ryegrass was recorded at the first and third cuttings. Treatments V and VI were significantly different from the control treatment I in the first cut although almost no K was present in SP or FGDG. The explanation was provided in the experiment of Sárdi et al. (2012) who found out that the better levels of P supply had a beneficial influence on K uptake and K concentrations in plants. During the uptake of K^+, root may exchange another cation, such as H^+ for K^+, or it may absorb an anion as NO_3^- or $H_2PO_4^-$ in order to maintain the electrical balance in cells (Marschner 1995). If the external pH declines, cation-anion imbalance may occur in the root tissues and therefore, anion absorption may be preferred. In our case, treatment VI had 70% of wood ash and 20% of FGDG. The acidic reaction of FGDG could therefore increase solubility of ash-contained nutrients, like P or K. On the other hand, synergism between K and S could also take place.

Only minor significant differences were found for P concentrations in ryegrass biomass. Generally, no amendment treatment was different from the control. This was probably due to soil's ability to supply plant-available P and therefore the possible P fertilization effects of amendment treatments were masked. However, from the results of Sárdi et al. (2012) it was evident that P accumulation by ryegrass and amounts of P taken up by plants both responded to the level of applied P.

The highest S concentration in ryegrass was reported mostly in the third and the last cut. Among the treatments, the most significant differences were observed from all tested nutrients. In the treatment VI with FGDG, on average almost 60% higher S concentration was noted compared to

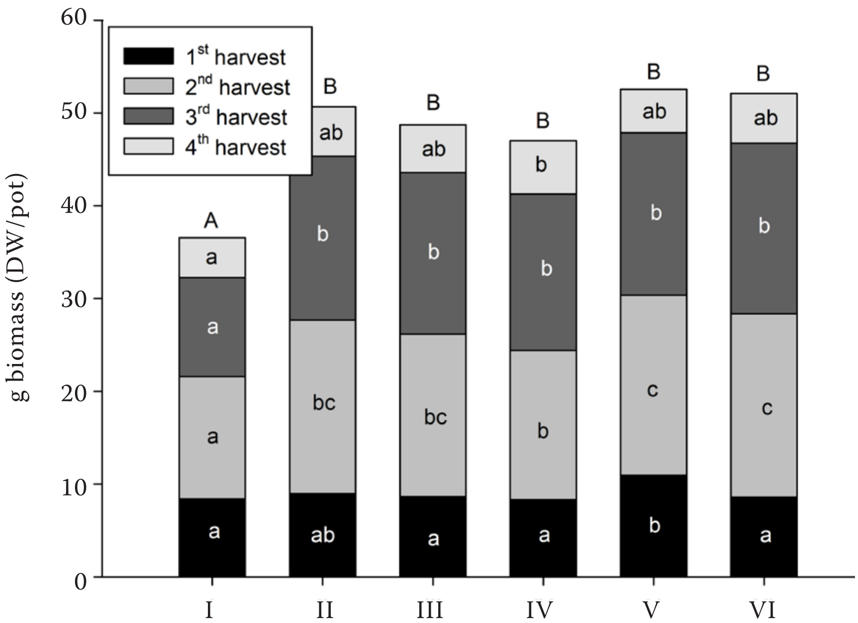

Figure 1. Total dry aboveground biomass yield of ryegrass (g biomass/pot). Different letters indicate significant differences ($P < 0.05$) among treatments. Differences among the individual harvests are represented by small letters, differences among the total biomass yield are represented by capital letters

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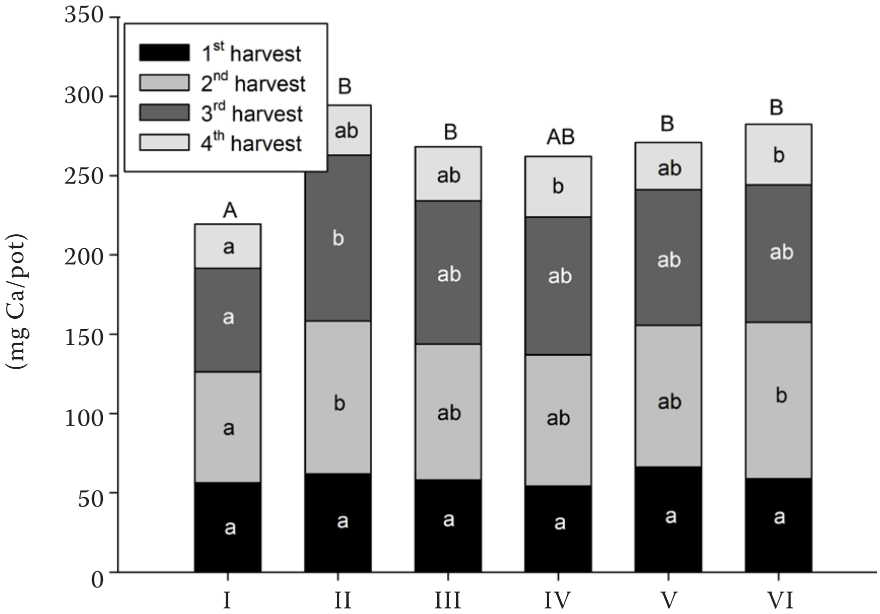

Figure 2. Calcium (Ca) uptake by ryegrass during four harvests. Different letters indicate significant differences ($P < 0.05$) among the treatments. Differences among the individual harvests are represented by small letters, differences among the total Ca uptake by plants are represented by capital letters

unamended treatment I. Warman and Sampson (1994) confirmed that gypsum was effective for supplying plant available S.

Effects of fertilizer treatments on biomass production. Application of wood ash in different forms and with different amendments resulted in significantly ($\alpha = 0.05$) higher total biomass yield production compared to the control treatment (Figure 1). At the first harvest, yields were small, on average about 9 g of dry matter. The ash pelletized together with FGDG and SP resulted in the significantly ($\alpha = 0.05$) largest yield. During the second harvest, plants grown in treatments enriched by FGDG resulted also in larger biomass. At third harvest, ryegrass that had been fertilized with ash developed well and resulted in the significantly ($\alpha = 0.05$) larger yields compared to unamended treatment. At the last, fourth harvest, yields were small in general, probably because of lower temperature and light in autumn, and also due to the fact that the plants were not fertilized by nitrogen after the third harvest.

Overall, treatments with FGDG resulted generally in larger total yield than with SP alone, but

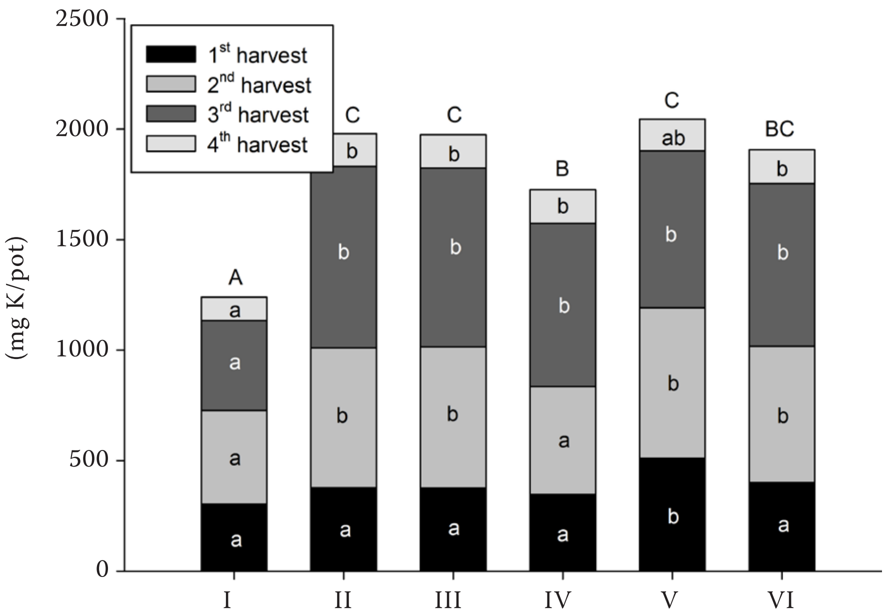

Figure 3. Potassium (K) uptake by ryegrass during four harvests. Different letters indicate significant differences ($P < 0.05$) among the treatments. Differences among the individual harvests are represented by small letters, differences among the total K uptake by plants are represented by capital letters

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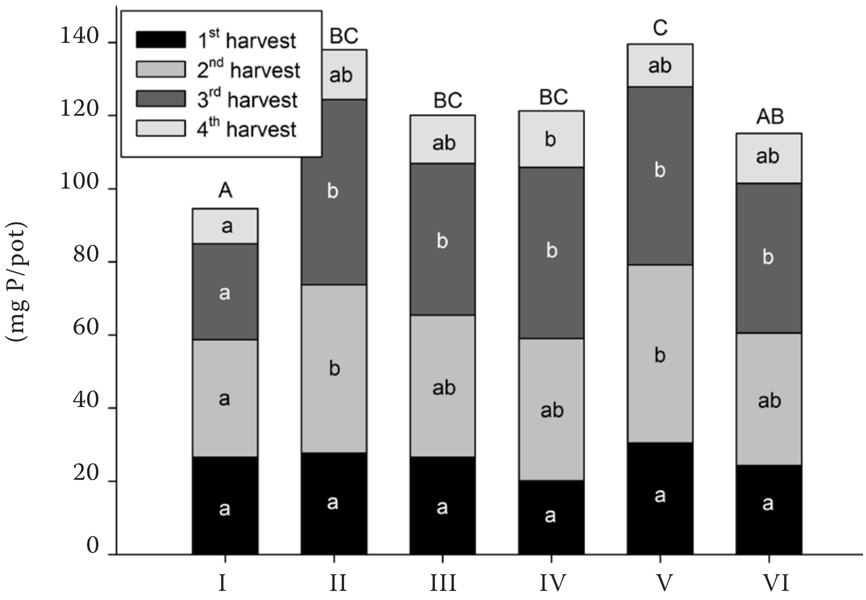

Figure 4. Phosphorus (P) uptake by ryegrass during four harvests. Different letters indicate significant differences ($P < 0.05$) among the treatments. Differences among the individual harvests are represented by small letters, differences among the total P uptake by plants are represented by capital letters

differences between these effects were not significant (α = 0.05), although the relative increase at the treatment V were 30% compared to untreated soil. The yield increase after FGDG application was observed also by Chen et al. (2005) in the experiment with alfalfa growing on silt loam soil.

Effects of fertilizer treatments on Ca uptake by plant biomass. Although plants utilized less than 1% of Ca applied with the powdered ash, Ca uptake in the treatment II was by 25% higher compared to the treatment I. The treatment II was the most successful in the case of Ca uptake by ryegrass but not significantly compared to the other enriched treatments (Figure 2). During the vegetation period, plants in the amended treatments, except the treatment IV, took up significantly more Ca than in the control treatment. It corresponds to the results of Mercl et al. (2016) that the addition of biomass ash increased Ca concentrations in soil. The higher Ca content in the FGDG treatment did not affect the Ca uptake by plants. It is contrary to the statements of Álvarez-Ayuso et al. (2011) that Ca in gypsum is well soluble but in agreement with the experiment of Buckley and

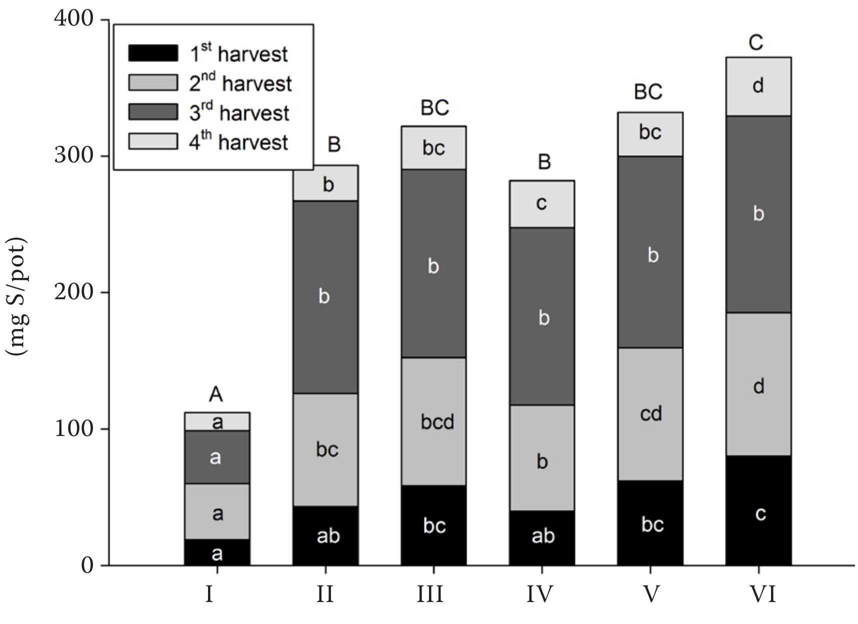

Figure 5. Sulfur (S) uptake by ryegrass during four harvests. Different letters indicate significant differences ($P < 0.05$) among the treatments. Differences among the individual harvests are represented by small letters, differences among the total S uptake by plants are represented by capital letters

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Table 5. Plant available nutrients in soil after the fourth harvest (mg/kg DW)

Treatment	I	II	III	IV	V	VI
Ca	1084^{a}	1759^{b}	1504^{ab}	1398^{ab}	1794^{b}	1710^{b}
K	55^{a}	131^{b}	130^{b}	126^{b}	111^{ab}	105^{ab}
P	36^{a}	61^{a}	58^{a}	73^{a}	111^{b}	46^{a}
S	12^{a}	30^{a}	27^{a}	65^{a}	251^{ab}	420^{b}

All values represent means (n = 4). Different letters indicate significant differences (P < 0.05) among the treatments in individual harvests. DW – dry weight

Wolkowski (2012) where the concentration of Ca in corn and soybean were largely unaffected by application of FGDG.

Effects of fertilizer treatments on K uptake by plant biomass. There were significant differences in K uptake after WFA fertilization (Figure 3). Overall, utilization of K supplied in WFA significantly increased the K uptake on average more than 40% compared to the treatment I. The highest total K uptake by ryegrass was in the treatment V although only 2% of K contained in these pellets was utilized by plants. K content in SP and FGDG was negligible (Table 3) but presence of these additives could improve the K uptake as also observed Blum et al. (2014).

The application of the WFA pellets with SP or FGDG alone was not as successful as their combination.

Effects of fertilizer treatments on P uptake by plant biomass. The total P amount taken up by ryegrass was significantly higher by 26% on average in the treatments II–VI compared to the treatment I (Figure 4). Although a significant increase of P uptake by plants after SP addition alone was expected, surprisingly, these pellets were not such efficient as the combination of SP with FGDG. It is in agreement with Philips et al. (2000) who also found out that applications of P and gypsum increased wheat grain and forage yields compared to P banded without gypsum. The latter authors suggested that P fertilizer bands with respect to Ca^{2+} could induce precipitation of applied P as dicalcium phosphate or dicalcium phosphate dihydrate which would slowly become plant available with time. Contrary to the findings stated above, results from the experiment of Silva et al. (2013) with strawberries or the studies of Murphy and Stevens (2010), and Clark et al. (2001) showed that the combination of P and gypsum were not beneficial for plants, and P solubility decreased with increased Ca concentration.

Effects of fertilizer treatments on S uptake by plant biomass. Among the analysed nutrients, S was found to be the most sensitive element to application of different additives to soil. In all harvests of amended treatments were found significantly higher uptakes of S compared to the treatment I (Figure 5). As expected, the highest uptake was observed at treatment VI – by 70% higher than in the treatment I. Our results are in agreement with Baligar et al. (2011) who stated that FGDG is an excellent source of S to plants.

As seen from Table 5, the amount of plant available Ca, P, and S in the soil was significantly influenced by the amendments added to the WFA. The most noticeable changes were observed in the case of S where the amount of plant available S in the treatment VI was 35 times higher than in the control treatment. Even though, WFA increased the uptake of Ca, K, P and S in our experiment, a significant increase in plant-available portions in soil after harvest was found for K only. This indicates a possible effect of long-term K fertilization with WFA. Ohno (1992) observed that the increase of soil available K levels resulted from the release of wood ash K as well as from the replacement of K on soil exchange sites by Ca and other exchangeable cations released directly from wood ash into the soil suspension.

The application of WFA in various forms to the soil resulted in the increase of yield, nutrient contents and nutrient uptake by ryegrass biomass and plant available portion of nutrients in soil after harvest. Compaction of ash did not significantly affect nutrient release and biomass growth.

The addition of FGDG into WFA pellets did not influence total biomass yield but resulted in a significant increase of S concentrations in ryegrass

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biomass. This may represent strong potential for further waste-recycling in agriculture. However, it was shown that nutritional effects are difficult to predict and due to complex character of waste materials may not necessarily lead to higher yields. More effort is therefore needed to optimize final composition of waste-based fertilizers.

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