Phosphate fertilizer value of heat treated sewage sludge ash

M. Severin[1], **J. Breuer**[2], **M. Rex**[3], **J. Stemann**[4], **Ch. Adam**[4], **H. Van den Weghe**[1], **M. Kücke**[5]

[1]*Department of Animal Sciences, Faculty of Agricultural Sciences, Georg-August-University of Göttingen, Göttingen, Germany*
[2]*Reterra Service GmbH, Erftstadt, Germany*
[3]*Assosciation Slag, Duisburg, Germany*
[4]*Federal Institute for Materials Research and Testing, Division 4.4. Thermochemical Residues, Treatment and Resource Recovery, Berlin, Germany*
[5]*Institute of Crop and Soil Science, Julius Kühn-Institute, Braunschweig, Germany*

ABSTRACT

This study focuses on the question whether heat treated sewage sludge ashes are more favourable as fertilizers than untreated sewage sludge ashes (USSA) and whether their fertilization effects are comparable with commercial triple superphosphate (TSP). In a pot experiment, maize was fertilized either with one of three heat treated and Na-, Ca- and Si-compounds amended sewage sludge ashes (two glown phosphates, steel mill slag + sewage sludge ash) or USSA or TSP as control. Fertilization with USSA did not increase the biomass yield and the P uptake of maize in comparison to the P0 treatment (7.25 resp. 8.35 g dry matter/pot). Fertilization with heat treated sewage sludge ashes and TSP resulted in significantly higher yields and plant P uptakes which are on average eight times higher than treatment with USSA and P0 control. Biomass yields and P uptake of maize after fertilization with heat treated sewage sludge ashes are not significantly different from those after TSP fertilization. The main P compound in USSA is $Ca_3(PO_4)_2$. By heat treatment and amendment with different sodium, calcium, sulphur and silicon containing additives or steel mill converter slag, $Ca_3(PO_4)_2$ is converted to Ca- and Na-silico-phosphates, which have a higher water solubility than $Ca_3(PO_4)_2$. This increased solubility is responsible for the high plant availability of this phosphates.

Keywords: P recycling; sustainability; P solubility

An alternative to mining geogenic phosphorus (P) is the closure of the natural P cycle by reusing the P in waste materials. The processing of sewage sludge offers a good opportunity to recycle P. However, sewage sludge may contain organic and inorganic pollutants. These include polychlorinated biphenyls, dioxins, persistent pharmaceuticals and heavy metals. Sewage sludge also contains harmful pathogens in high concentrations (Harrison et al. 2006).

On the other hand, sewage sludge ashes have high P contents – between 1.5% and 13.1% (Krüger and Adam 2013, Severin et al. 2013) and so have useful potential for the production of P fertilizers. However, without further processing, sewage sludges or sewage sludge ashes contain P compounds that are not easily available to plants. H_2O solubility of thermally processed sewage sludge ash is very low (0.1–6.4%) in comparison to triple superphosphate (TSP) (> 90%) (Cabeza et al. 2011, Severin et al. 2013). Neutral ammonia citrate solubility of heat treated sewage sludge ashes (Mephrec and Ash Dec process) is between 23–85% (Severin et al. 2013). Cabeza et al. (2011) show a P fertilizer efficiency of 20–30% for thermally treated sewage sludge ashes on an acid sandy soil. Furthermore, the heavy metal contents are quite high in many cases and, of course, concentrations rise as the sludges is dried. Thus field soil heavy metal levels increase annually with repeated applications (Kördel et al. 2007).

A pot trial was done to test the hypotheses that (i) heat treated sewage sludge ashes have a higher P availability and a higher effect on plant growth compared to untreated sewage sludge ash and (ii) at similar levels of application, heat treated sewage sludge ashes behave similarly to triple superphosphate with regard to P plant availability and plant growth promotion after fertilization.

MATERIAL AND METHODS

Production and characterisation of sewage sludge ashes. The test products included three heat treated sewage sludge ashes and two reference materials (untreated sewage sludge ash and TSP).

Sewage sludge ash samples originated from a mono-incineration facility in Bonn, Germany. The untreated sewage sludge ash (USSA) was not further processed. For glown phosphate 1 (GP1), 875 g of sewage sludge ash (dry matter > 99%), 375 g of Na_2CO_3 and 1250 g of $CaCO_3$ were thoroughly mixed calculated on their dry matter and subsequently granulated with deionized water in an Eirich mixer (Hardheim, Germany). The granules were thereafter spread on an aluminium tray and partially dried at 105°C for about 1 h. For glown phosphate 2 (GP2), 875 g of sewage sludge ash (dry matter > 99%), 502 g of Na_2SO_4 and 1550 g of black lime (residual lime from nitrogen fertilizer processing: 38% CaO and 1% N) were mixed calculated on their dry matter, granulated and dried in the same manner. Additional phosphates were not added during the processing of the sewage sludge products.

Then the granulated sewage sludge products were heat treated at 950°C for about 20 min. The heat treatments were performed in a pilot scale rotary kiln (TL 1100, Thermal Technology, Bayreuth, Germany) with indirect electrical heating. GP1 was produced in a nitrogen atmosphere without addition of reducing agents whereas GP2 was produced under slightly reducing conditions due to the addition of carbon containing lime. Calcination converts the phosphates into compounds which are more available to plants (Gunawardane and Glasser 1979). Heavy metals from sewage sludge ash are partly volatilised at these high temperatures and can be separated via the exhaust gas (Struis et al. 2009).

In contrast to the two heat treated sewage sludge ashes (GP1 and GP2), steel mill slag + sewage sludge ash (S + S) was produced from basic oxygen furnace slag (converter slag) from a steel mill (prepared by the Linz-Donau method) which was enriched in the melt with sewage sludge ash (dry matter > 99%). Mixing ratio of converter slag:sewage sludge ash is 12.2:0.8. The sewage sludge ash was added to the still liquid slag at 1500°C.

For determining the acid neutralization capacity the fertilizer is solved in a known amount of hydrochloric acid. The excess acid is back titrated with soda. The basic active ingredients are given in % CaO (Quaggio et al. 1995, VDLUFA 2008c).

P content, neutral ammonium citrate solubility (NAC) and acid neutralisation capacity (% CaO content, basic effective at pH 4.8) of all tested products can be seen in Table 1.

Experimental setup. A two factorial pot experiment with maize (*Zea mays* L., cv. Bonfire) was carried out in Braunschweig, Germany, outdoors, at average daily air temperatures between 20°C and 30°C. A sandy soil was selected (82% sand; 12% silt; 6% clay; pH 3.8; 0.7% C_{org}; 1.14 mg P 100/g soil as P CAL). Phosphorus is extracted with a buffered solution (pH 4.1) of calcium acetate, calcium lactate, and acetic acid of air dried soil. The phosphate concentration is detected photometrically after extraction (VDLUFA 2012). The soil pH is very low and the soil shows P deficiency (VDLUFA 1997). The soil was sieved (10 mm) before mixing with the products. Of this, 6000 g were mixed with processed sewage sludge ash (grind before mixing with soil) or with one of the reference materials and placed in pots.

Table 1. P-content, neutral ammonium citrate solubility (NAC) and neutralizing value of four different sewage sludge products and triple superphosphate (TSP)

Treatment	P-content*	P-NAC**	Acid neutralisation capacity***
		(%)	
S + S	1.6	106	47
GP1	5.4	94	39
GP2	4.8	86	38
TSP	19.5	103	0
USSA	9.6	54	13

USSA – untreated sewage sludge ash; GP1, GP2 and S + S – heat treated sewage sludge ashes. *Total P after *aqua regia* digestion (VDLUFA 2008a); **relative neutral ammonium citrate solubility; ***% CaO basic effective at pH 4.8

The effectiveness of the products as P fertilizers was examined at three levels of P per pot: level 1 (0.18 g P); level 2 (0.35 g P) and level 3 (0.52 g P) (total P after *aqua regia* digestion) (Table 2). The products differed in their base contents (acid neutralization capacity, Table 1). In order to avoid differences in the soil pH, finely ground calcium carbonate was added to the pots according to the differences in the base content (Table 2, compensation liming). The rate of liming was based on the predetermined levels of base active materials in each of the products. Compensation liming was adjusted to match the levels in S + S.

Averaged over all three application levels, the pH values were: 7.25 (P0 control), 7.35 (USSA), 7.61 (GP1), 7.63 (GP2), 7.66 (S + S) and 7.46 (TSP).

Eighteen maize seeds were sown in each pot and nine were left at 14 day after germination. The pot trial was carried out with four replicates. The pots were placed randomly in a block design and watered regularly with tap water. The soil used in the pot trial is rather nutrient poor. The plants were fertilized twice (15 and 25 days) with N (1.96 g/pot), K (5.03 g/pot), S (1.04 g/pot) and Mg (0.78 g/pot). Micronutrients were sprayed on the foliar (15, 25 and 35 days) using a commercial micronutrient fertilizer (Fetrilon: 0.5% B, 1.5% Cu, 4.0% Fe, 4.0% Mn, 0.1% Mo, 1.5% Zn, 2% Mg). This was done due to the different processing of the sewage sludge ash products (e.g. different additives, heating procedures, relations between additives and sewage sludge ash, processing steps). Due to this it is most likely that the ash products do not differ only in their calcinations capacity (Table 1) but also in their plant nutrients contents and also in the plant availability of these nutrients. On the other hand, no additional nutrients are applied in the TSP and the P0 treatment. Therefore, plant nutrients except of P were applied in amounts which ensured a sufficient nutrient supply and prevent additional nutrient effects besides those from phosphate. To prevent micronutrient deficiency a micronutrient solution containing Mg was applied by spraying.

The above ground biomass was harvested at 42 day. After harvest, the biomass was dried in a ventilated oven for 48 h at 60°C to determine the dry matter content. Dried plant material was milled for laboratory analysis. From each pot, a soil sample was taken for analysing P and pH.

Analyses. The mineral composition of the sewage sludge products and TSP were determined with an X-ray refractometer (Panalytical, Almelo, Netherland).

To determine the total contents of phosphate, the plant samples as well as the products were digested in a mixture of hydrochloric acid and nitric acid (*aqua regia*) and were extracted of the acid under reflux (VDLUFA 2008a). To determine the plant availability of P, the sewage sludge ash products were extracted in neutral ammonium citrate (NAC) (Anonymous 2012). To analyze the contents of phosphate in the plant samples, the milled plant material was placed in a polytetrafluorethylen tube with nitric acid and hydrogen peroxide. The tubes are placed in a microwave heated pressure digestion system. In this system, the elements are extracted from the matrix at a defined temperature. The mass concentration of the elements in the extracted sample of fertilizer and plant material can be determined by optical emission spectrometry with inductively coupled plasma (ICP OES, Varian, Palo Alto, USA). The quantification of the element concentration is

Table 2. Setup of the pot fertilization experiment with different products made of sewage sludge

Treatment	P (g/pot)	Product/pot	Acid neutralisation value	Ca	Compensation liming
		(g)		(g/pot)	
Control	–	–	0.0	10.7	27.3
S + S	0.18	10.7	5.0	10.7	18.2
	0.35	21.4	10.0	10.7	9.1
	0.52	32.1	15.0	10.7	0.0
GP1	0.18	3.2	1.3	10.7	25.0
	0.35	6.5	2.5	10.7	22.7
	0.52	9.7	3.8	10.7	20.4
GP2	0.18	3.6	1.4	10.7	24.8
	0.35	7.3	2.8	10.7	22.3
	0.52	10.9	4.1	10.7	19.8
TSP	0.18	0.9	0.0	10.7	27.3
	0.35	1.8	0.0	10.7	27.3
	0.52	2.7	0.0	10.7	27.3
USSA	0.18	1.8	0.2	10.7	26.8
	0.35	3.7	0.5	10.7	26.4
	0.52	5.5	0.7	10.7	26.0

USSA – untreated sewage sludge ash; GP1, GP2 and S + S – heat treated sewage sludge ashes; TPS – triple superphosphate based on linear calibration functions (VDLUFA 2008b, 2011).

Plant available soil P contents after harvest were determined in CAL extracts as described in materials and methods (VDLUFA 2012). Soil pH was determined in $CaCl_2$.

Statistics. Statistical analyses were performed as a two factorial analysis of variance using SAS 9.2 (SAS Institute, Cary, USA). The data were examined using the Tukey's test (level of significance $P < 0.05$).

RESULTS AND DISCUSSION

Dry matter yields of maize were significantly affected by both the type of the sewage sludge product and the application level.

The yields of all treatments fertilized with heat treated sewage sludge ash (GP1, GP2 and S + S) and with TSP significantly exceeded those from the USSA and the P0 treatment. The highest yield was achieved with GP2 fertilization at level 3 with 55.3 g dry matter (DM)/pot. All treatments with heat treated sewage sludge ashes had similar yields than the TSP treatment. Plant development was slow in two of the four S + S replicates of level 2 and the reason is unknown (Figure 1a).

The plants fertilized with heat treated sewage sludge ashes and the TSP show in all three fertilization levels significantly ($P < 0.05$) higher P contents in maize biomass than those of the USSA and the P0 treatment (Table 3).

After fertilization with GP1, GP2, S + S and TSP, P uptake by maize increased with increasing application level (exception S + S).

Phosphorus uptake in the P0 treatment and USSA were similar, but both were significantly ($P < 0.05$) below those of the heat treated sewage sludge ashes and the TSP treatments (Figure 1b).

The finding that fertilization with processed sewage sludge ashes results in higher P uptakes might be influenced by different effects of different sewage sludge ash products on the soil pH (experimental setup). Due to the fact that we minimized these alterations of soil pH (7.3–7.7) by compensation liming (Table 2) we are confident that these small differences in soil pH had no obvious effect on P uptake and our conclusions with regard to the fertilization effect of the tested products.

At the end of the experiment, the soil in each pot was analysed for plant available P concentrations (CAL P content, Figure 1c). The soil of the treatments with heat treated sewage sludge ashes and TSP showed significantly higher plant available P concentrations than the soils from the P0 and USSA treatments. The maximum P content reached the treatment S + S at application level 3 (5.6 mg 100/g soil). The phosphate forms of heat treated sewage sludge ashes are recognized as equally available to plants (P CAL), such as the phosphate forms of TSP. The USSA did not increase the P CAL content in the soil. After fertilization with USSA soil shows P deficiency as well as before the trial started (VDLUFA 1997).

The X-ray refractometry study showed that the dominant P compound in USSA is whitlockite $Ca_3(PO_4)_2$, an anhydrous, sparingly soluble mineral phosphate, with low soluble P fractions. The heat treated sewage sludge ashes contain water soluble Ca-Na-silico-phosphates (Table 4).

Correlation between NAC solubility and P uptake is $r = 0.6357$. This shows that high NAC solubility is responsible for high P plant uptake.

In this study, the effect of heat treatment of sewage sludge ashes in combination with different additives on plant availability on processed sewage sludge ashes, were tested the first time.

Previous studies (Biermann and Rosen 1994, Cabeza et al. 2011) have shown that unprocessed sewage sludge ashes have no or only little P fertilization effects. On the other hand, Cabeza et al. (2011) found that sewage sludge that is heat treated (sewage sludge ash melted with coke by 2000°C in a cupola oven) but without Na, Si or S additives resulted in a very limited improvement of the fertilization value (10–30% P fertilizer effect compared to TSP), which was comparable to Thomas phosphate. Römer (2013) also tested fertilization effects of sewage sludge ashes which were heat treated and processed with Mg and Ca additives. He found no significant fertilization effects of these ashes. Drissen (2012) could show that a steel mill slag enriched with a sewage sludge ash shows very high P availability (> 90%) compared to other heat treated sewage sludge ashes (30–91% P availability for plants), this finding is comparable to our results. This high P availability is due to the conversion of apatite and whitlockite P minerals in the heat treatment (1500°C) in which the poorly available forms of phosphate are converted into calcium-silico-phosphate (Rex et al. 2013). After World War I in Germany, processing technologies similar to the process technologies of GP1 and GP2 were used to produce rhenania phosphate. Rhenania

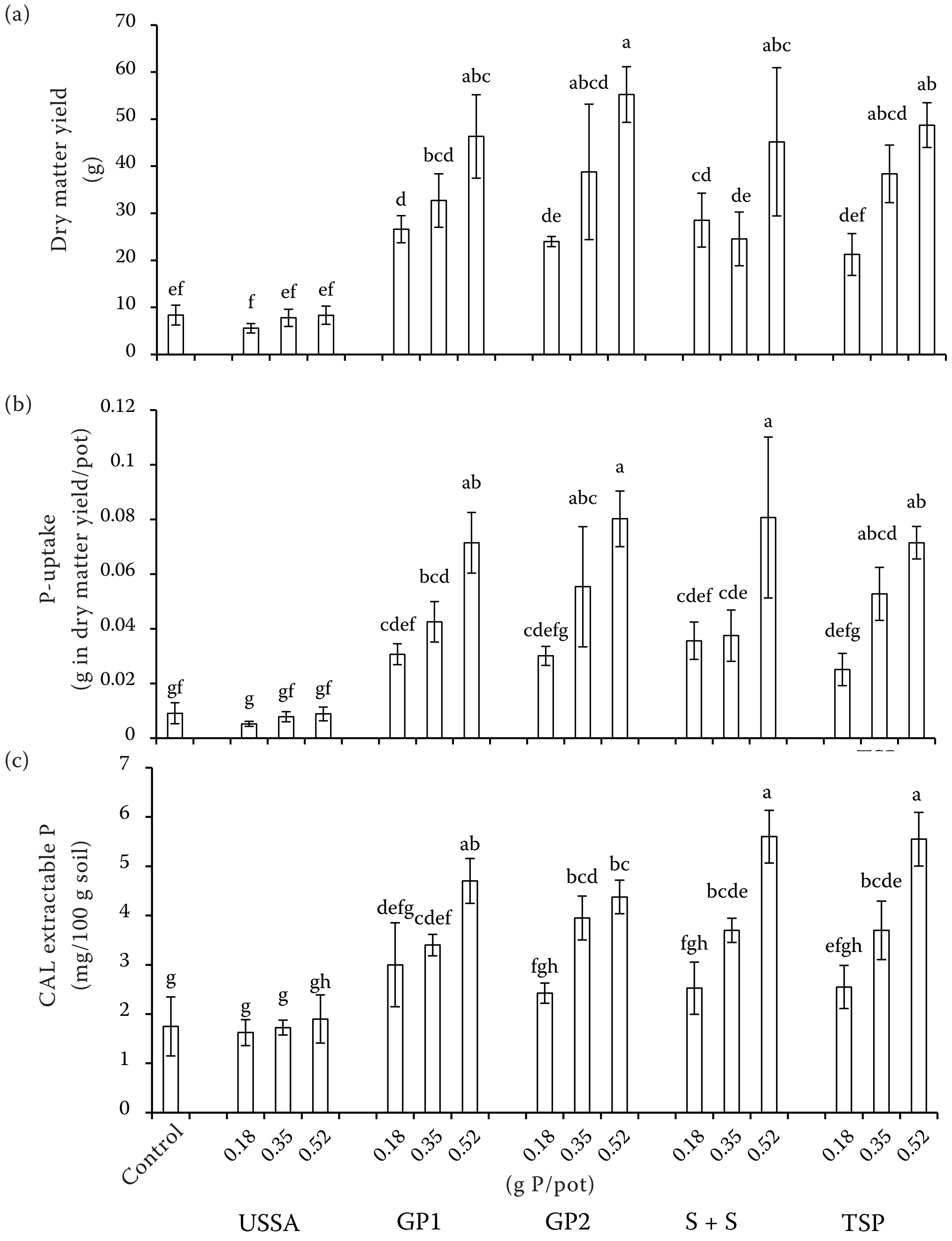

Figure 1. (a) Dry matter yield responses of maize; (b) P-uptake of maize plants, and (c) concentration of CAL extractable P in the soil after fertilization with four different sewage sludge products (USSA – untreated sewage sludge ash; GP1, GP2 and S + S – heat treated sewage sludge ashes; TPS – triple superphosphate; P fertilization levels (level 1: 0.18; level 2: 0.35, level 3: 0.52). Values marked by different letters are significantly different (Tukey's test, $P < 0.05$). Error bars are standard deviations

phosphate is a mixture of tricalciumphosphate and soda heat-treated by 900–1200°C. By soda addition the solubility of the glown phosphates is improved. Bad soluble apatite structures were destroyed in this process. Solubility in citric acid is > 95% (Werner 1967). Huck und Munk (1979) made a pot trial and showed that sewage sludge ash treated with soda show a fertilization effect comparable to Thomas phosphate.

In this study we could show that heat treatment in the presence of Na-, Ca-, Si-containing additives is an effective way to convert the P compounds in sewage sludge ash to P minerals with high NAC solubility (Table 1) and thus a high plant availability

Table 3. Phosphorus concentrations of maize biomass (% P in dry mass, means of four replicates) after fertilization with four different sewage sludge products

Level	Control	USSA	GP1	GP2	S + S	TSP	$\bar{x}$
1	–	0.09^{a}	0.12^{ad}	0.13^{bdf}	0.13^{bdf}	0.12^{ade}	0.11^{a}
2	–	0.10^{ab}	0.13^{cdg}	0.14^{efg}	0.15^{gh}	0.14^{dg}	0.13^{b}
3	–	0.11^{abc}	0.16^{gh}	0.15^{fg}	0.18^{h}	0.15^{fg}	0.15^{c}
$\bar{x}$	–	0.10^{a}	0.13^{bc}	0.14^{bc}	0.15^{be}	0.13^{bc}	0.13
Control	0.11^{fgh}	–	–	–	–	–	–

USSA – untreated sewage sludge ash; GP1, GP2 and S + S – heat treated sewage sludge ashes; TPS – triple superphosphate; P fertilization levels (g P/pot: level 1: 0.18; level 2: 0.35, level 3: 0.52). Values marked by different letters are significantly different (Tukey test, $P < 0.05$)

(Figures 1a,b, Table 3). The heat treated sewage sludge ashes contain Ca-Na-silico-phosphates. The mineral P constituents are responsible for high P availability. Untreated sewage sludge ash shows no P fertilizer effects. Obviously, the three processing techniques used in this study can produce products with similar plant availability of their phosphates. This shows that the processing technique (Mephrec and Ash Dec) in previous studies were far less successful to improve the plant availability of the P in the sewage sludge ashes.

This study confirms that phosphates in sewage sludge ash cannot be converted without chemical additives into a phosphate form that is an alternative to conventional fully digested phosphates obtained from rock phosphate.

It was shown in this paper that heat treated sewage sludge ash can achieve P fertilizer effect similar to that of TSP whereas untreated sewage sludge ash shows no P fertilizer effects. The technologies for fertilizer production from sewage sludge ashes were already successfully proven in demonstration scale.

Table 4. The main P constituents of the sewage sludge products and triple superphosphate (TSP) measured by X-ray refractometry

Fertilizer	Mineral phosphate
TSP	$Ca(H_2PO_4)_2 \times H_2O$
USSA	$Ca_3(PO_4)_2$
S + S	$Ca_2SiO_4 \times 0.05\ Ca_3(PO_4)_2$
GP1	$Na_2Ca_4(PO_4)_2SiO_4$ and $Ca(OH)_2$
GP2	$Na_2Ca_4(PO_4)_2SiO_4$ and CaS

USSA – untreated sewage sludge ash; GP1, GP2 and S + S – heat-treated sewage sludge ashes

Further trials are required to confirm the efficacy of the heat treated sewage sludge ash fertilizer under semi-commercial conditions in the field.

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Corresponding author:

Msc. agr. Maximilian Severin, Georg-August-University of Göttingen, Faculty of Agricultural Sciences,
Department of Animal Sciences, Universitätsstraße 7, 493 77 Vechta, Germany
e-mail: mseveri@gwdg.de