

# Plant availability of P fertilizers recycled from sewage sludge and meat-and-bone meal in field and pot experiments

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**Abstract** This project is part of the German Research Cluster on P-Recycling. The fertilizer effects of selected recycled P compounds were determined in field and pot experiments in comparison to commercial fertilizers (Triplesuperphosphat (TSP) and rock phosphate). For the field experiments, two magnesium-ammonium-phosphates (MAP) of different sewage treatment plants, a heavy metal depleted sewage sludge ash, and an alkali sinter phosphate made from meat-and-bone meal were used. Additionally in the pot experiments, a meat-and-bone meal ash, a cupola furnace slag made from sewage sludge, and a precipitated apatite of municipal waste water were tested. The field experiments were performed in three loamy soils poor in available P and differing mainly in pH. Phosphate fertilization was for a whole three-year crop rotation with a suboptimal amount of 60 kg of P/ha plus a well fertilized (100 kg P/ha) and unfertilized control. The pot experiment with maize was performed with 5.5 kg of mainly the same soils as in the field in 6L pots. Phosphate was given at a suboptimal rate of 60 mg/kg soil for each fertilizer, plus an unfertilized and well fertilized (200 mg P/kg) control.

In the field, the fertilization with the different materials had no significant effect on the CAL-extractable P or P concentration in soil solution in all three soils. Also grain yield and P uptake was not different. However, the two MAPs tended to perform slightly better than the other compounds. Differences in plant availability became more obvious in the pot experiments. The chemical characterisation of plant availability by CAL-extraction showed that the MAPs, apatite, cupola slag were similar to TSP. These results were partly confirmed by the relative fertilizer efficiency (P uptake from fertilizer in relation to TSP) which was in average highest for MAP, apatite and cupola slag, but not consistent in each soil. Hence, none of the tested compounds reached the plant availability of a water-soluble P fertilizer in the pot experiment in the first year. Long term effects will be studied in the next years.

## INTRODUCTION

Phosphorus (P) is an essential plant nutrient necessary for several metabolic processes. The concentration of plant available P is low in most soils, hence a fertilization is needed. However, phosphate resources are limited. Assuming a constant rate of consumption, the known P resources will be depleted in about 100 years (Berg and Schaum, 2005; Stewart *et al.*, 2005). On the other hand, anthropogenic waste material contains P that can be hazardous for the

environment, e.g. like the discharge of municipal wastewaters can lead to eutrophication of waterbodies (Johnston and Richards, 2003). It is possible by several ways to recover P of different sources such as sewage sludge or animal bones and meal. The soluble P of wastewaters can be precipitated as struvite, i.e. Magnesium-ammonium-phosphate ( $\text{MgNH}_4\text{PO}_4$ , MAP), as well as in other forms such as calcium, aluminium or iron phosphate (Johnston and Richards, 2003). Furthermore, it is possible to recover P with thermo-chemical processes from sewage sludge, as well as from animal residues. It must be taken into account, that the different processes to obtain P from waste material result in fertilizers differing in P solubility and P availability to plants. In previous studies it was demonstrated that finely ground struvites can be as effective as water soluble P fertilizers (Ghosh *et al.*, 1996; Goto, 1998) although struvite is limited in water solubility. This shows that the water solubility of P compounds and their plant availability in the soil are not necessarily related. Hence, the agronomic effectiveness of each recovered-P product must be evaluated before it can be used as commercial fertilizer. In this study, the fertilizer effect of some MAPs and thermally recovered P was investigated in field and pot experiments.

## MATERIALS AND METHODS

Phosphorus sources and soils. Seven different materials were tested with regard to their P fertilizer effect in field and pot experiments on four different soils. Additionally, two commercial fertilizers with different P availability, i.e. triple-superphosphate (TSP) and rock phosphate were taken as control. In the field experiments, two Magnesium-ammonium-phosphates of different sewage treatment plants (MAP-Sb, MAP-Gf), a heavy metal depleted sewage sludge ash (sl-ash), and an alkali sinter phosphate made from meat-and-bone meal (sinter-P) were used. Additionally in the pot experiments, a meat-and-bone meal ash (MB meal ash), a cupola furnace slag (Mephrec procedure, cupola slag) made from sewage sludge, and a precipitated apatite of municipal waste water were tested. These products were not available in sufficient amounts to test them also in the field. The P concentrations of the products are shown in Table 1. All products were finely ground before fertilization.

*Field experiments.* The field experiments were performed on farmland with different crops in three loamy soils low in available P. The soils differed in their pH (Table 2). The phosphate fertilization took place in March 2007 for a whole three-year crop rotation with an amount of  $60 \text{ kg of P ha}^{-1}$  for each fertilizer. This is a suboptimal rate in order to be able to differentiate the P available for the plants among the P fertilizers. Well fertilized ( $100 \text{ kg P ha}^{-1}$ , TSP-100) and unfertilized controls were also established. The crop cultivation was performed

**Table 1.** Description of the P compounds from different P recycled works.

Material	Description	P concentration (%P)
MAP-Sb	Precipitated as $MgNH_4PO_4$	11.0
MAP-Gf	Precipitated as $MgNH_4PO_4$	9.6
sl-ash	Sewage sludge burned over 1000°C	7.8
sinter-P	Meat and Bone meal sintered over 1000°C	11.3
MB meal ash	Ashes from animal meal	16.4
cupola slag	Sewage sludge slagged over 1000°C	2.9
apatite	Calcium phosphate precipitated	11.1
*Triple superphosphate	–	20.1 (19.5 water soluble)
*Rock phosphate	–	11.8

\*Were used as reference.

**Table 2.** Calcium-acetate-lactate extractable P (CAL-P) and pH of the soils used in field and pot experiments.

Soil	CAL-P (mg P kg <sup>-1</sup> )	pH CaCl <sub>2</sub> (1:2.5)	Crops
Sattenhausen	19.0	5.6	Oilseed rape
Lutterbeck	18.0	7.1	Winter barley
Gieboldehausen	21.0	6.8	Winter wheat
Düshorn	24.2	5.5	–

by the farmers as usual. Each treatment was replicated four times. Plot size was 24 m<sup>2</sup>.

*Pot experiments.* The pot experiment was performed with 5.5 kg of soil in 6 L pots. The soils were taken from two sites of the field experiments (Sattenhausen and Gieboldehausen). Unfortunately, the farmer of Lutterbeck did not allow to remove soil from the farm. Additionally, an acid sandy soil (Düshorn) was also used (see Table 2) in the pot experiment, which is not in agricultural use. Phosphate was given at a suboptimal rate of 60 mg of P kg<sup>-1</sup> soil for each fertilizer, plus unfertilized and well fertilized (200 mg P kg<sup>-1</sup>, TSP-200) controls. Furthermore, the pots were fertilized with 1 g K (K<sub>2</sub>SO<sub>4</sub>), 0.2 g Mg (MgSO<sub>4</sub> \* 7H<sub>2</sub>O) and 0.5 g N (NH<sub>4</sub>NO<sub>3</sub>) per pot. Two further applications of N were realized as Ca(NO<sub>3</sub>)<sub>2</sub> in doses of 0.3 g per pot each time. After 3 weeks of incubation without plants, maize (*Zea mais* L., cv. Atletico) was sown (2 plants per pot). Each pot was irrigated daily with demineralized water during the experiment. The plants were harvested at the 80th day after the sowing.

*Measurement.* Soil samples were collected from the upper 30 cm soil depth before fertilizing and at harvest in the field experiments and from the total soil volume in the pot experiments. The available P was determined by extraction with calcium-acetate-lactate (CAL-P; Schueller, 1969) as usual in Germany. Soil solution was obtained according to the displacement method described by Adams (1974). The P concentration in both methods was measured colorimetrically by the method of Murphy and Riley (1962).

In the field, plant samples were taken from 6 m<sup>2</sup> in the middle of the experimental plots. Unfortunately, the farmer of Gieboldehausen harvested the whole field before we could take the samples. Plants were dried at 65°C to constant weight before grain and shoot yield was determined. In the pot experiment, the whole above ground plants were taken as yield. A division in cobs and shoot was not done because the selected maize genotype is usually used as energy plant. Subsamples of plant dry matter were digested in conc. HNO<sub>3</sub> at 180°C and the P concentration herein was measured colorimetrically by the molybdenum-vanadate method (Scheffer and Pajenkamp, 1952). Total P uptake was calculated from shoot dry matter and P concentration.

*Data analysis.* Statistical analysis was performed using SigmaStat 2.0. The experimental design for field experiments was in blocks with 8 treatments and 4 replicates. The pot experiment was completely randomized with 4 replicates for each treatment. The effect of P fertilizers was analyzed with one-way analysis of variance between the treatments as the source of variation, followed with a Tukey test at the 0.05 level of significance to separate the means.

## RESULTS

*Field experiments.* The fertilization with the different materials had no significant effect on the CAL-extractable P at harvest in all three soils (Table 3). At “Sattenhausen” and “Lutterbeck” the TSP fertilization increased CAL-P in tendency, whereas rock phosphate had not become available at all. The tested P compounds grouped between TSP-60 and rock phosphate. Similar results were obtained for P soil solution concentration (Table 3) on both soils. Only in “Sattenhausen”, the concentration of TSP-100 was significantly higher than that of rock phosphate or sl-ash. The CAL-P of all fertilizers in “Gieboldehausen” did not differ from the unfertilized treatment and also not from the CAL-P before fertilization (not shown). Even the fertilization with water soluble TSP had not increased the CAL-P. This and the very low P soil solution concentration point to the fact that P was fixed after fertilization in non-CAL extractable fractions.

**Table 3.** Influence of recycled P fertilizer on CAL-extractable P and P concentration in soil solution at harvest in the field experiments.

	Sattenhausen (pH 5.6)		Gieboldehausen (pH 6.8)		Lutterbeck (pH 7.1)	
	CAL-P	Solution P	CAL-P	Solution P	CAL-P	Solution P
	mg kg <sup>-1</sup>	μmol L <sup>-1</sup>	mg kg <sup>-1</sup>	μmol L <sup>-1</sup>	mg kg <sup>-1</sup>	μmol L <sup>-1</sup>
Unfertilized	26.0	6.6 b	22.2	0.6	14.3	2.5
TSP-60	30.3	11.7 ab	21.1	2.7	23.8	5.0
TSP-100	34.1	17.8 a	22.3	2.7	20.5	5.1
Rock-P	23.0	6.2 b	21.3	0.7	11.1	1.3
sinter-P	26.2	10.0 ab	23.7	1.2	23.7	2.5
sl-ash	24.3	7.3 b	19.6	0.7	16.0	1.9
MAP-Sb	27.4	12.9 ab	25.1	1.4	14.7	2.3
MAP-Gf	27.3	9.8 ab	24.3	1.2	14.1	2.5

Different letters denote significant differences between treatments (Tukey,  $p < 0.05$ ). Treatments without lettering are not significantly different.

Grain yield on both soils “Sattenhausen” and “Lutterbeck” did not differ between the treatments (Table 4). Unfortunately, there are no yield data for “Gieboldehausen” because the farmer harvested the whole field before we could harvest the experimental plots. On “Sattenhausen” total P uptake was also not different between treatments. However, on “Lutterbeck” a higher P uptake of MAP-Sb indicated a better plant availability of this material compared to the other recycled P fertilizers and rock phosphate. The small differences between the treatments might be due to unusually dry April in 2007. This reduced plant growth and concomitantly P demand. Already the soil-borne P in the unfertilized plots seemed to be sufficient. Furthermore, the fertilizers were not washed into the soil during this period and, hence, were not plant available at this time.

*Pot experiments.* Differences in plant availability between the compounds became more obvious in the pot experiments. The chemical characterizations of plant availability by CAL-extraction after harvest showed that MAP-Sb, apatite, cupola slag and partly MAP-Gf were similar to TSP-60 (Table 5). Rock phosphate and the two ashes of MB meal and sewage sludge were not different to the unfertilized control. Plants take up P only from soil solution, hence, P soil solution concentration may characterize the direct P availability. Only a fertilization with MAP-Sb resulted in a P concentration similar to TSP-60 in all three soils. In “Sattenhausen”, sinter-P and MAP-Gf reached a higher P soil solution concentration than the unfertilized control, whereas all other compounds were not different to the unfertilized control regardless of the soil.

**Table 4.** Yield and P uptake of oilseed rape and winter barley after fertilization with recycled P compounds grown on different soils in the field experiments.

	Sattenhausen (pH 5.6) Oilseed rape		Lutterbeck (pH 6.8) Winter barley	
	Grain yield	P uptake	Grain yield	P uptake
	t ha <sup>-1</sup>	kg ha <sup>-1</sup>	t ha <sup>-1</sup>	kg ha <sup>-1</sup>
unfertilized	2.5	22.9	6.0	24.3 ab
TSP-60	2.6	27.4	6.3	29.6 ab
TSP-100	2.5	28.6	5.6	26.6 ab
Rock-P	2.6	24.6	5.7	23.4 b
sinter-P	2.9	28.6	5.8	24.0 b
sl-ash	2.7	26.7	5.9	23.6 b
MAP-Sb	3.0	33.1	6.6	32.9 a
MAP-Gf	3.3	31.9	6.0	25.3 b

Different letters denote significant differences between treatments (Tukey,  $p < 0.05$ ). Treatments without lettering are not significantly different.

**Table 5.** Influence of recycled P fertilizer on CAL-extractable P and P concentration in soil solution at harvest in the pot experiments.

	Sattenhausen (pH 5.6)		Gieboldehausen (pH 6.8)		Düshorn (pH 5.5)	
	CAL-P	Solution P	CAL-P	Solution P	CAL-P	Solution P
	mg kg <sup>-1</sup>	μmol L <sup>-1</sup>	mg kg <sup>-1</sup>	μmol L <sup>-1</sup>	mg kg <sup>-1</sup>	μmol L <sup>-1</sup>
unfertilized	25.0 e	0.6 d	10.0 d	0.3 de	19.0 e	0.3 cde
TSP-60	40.2 bcd	2.3 b	22.2 c	0.9 bc	40.4 b	0.6 bc
TSP-200	76.1 a	14.8 a	74.4 a	15.4 a	78.9 a	12.8 a
rock-P	27.4 e	0.5 d	10.9 d	0.2 de	27.5 d	0.3 cde
sinter-P	39.0 cd	1.2 c	17.9 c	0.3 de	33.1 cd	0.4 bcde
sl-slash	29.6 e	0.7 d	12.1 d	0.5 cde	28.9 d	0.1 de
MAP-Sb	44.1 bc	2.0 b	21.9 c	0.9 bc	43.5 b	0.8 b
MAP-Gf	39.5 cd	1.4 c	19.7 c	0.5 cde	38.3 bc	0.4 cde
MB meal ash	29.4 e	0.8 d	12.8 d	0.2 e	26.1 de	0.1 e
apatite	44.5 b	0.6 d	28.0 b	0.4 de	36.6 bc	0.5 bcd
cupola slag	Not tested		28.2 b	1.1 b	40.7 b	0.1 e

Different letters denote significant differences between treatments (Tukey,  $p < 0.05$ ).

The chemical characterization of P availability does not necessarily reflect the 'real' P availability as can be deduced from P uptake and yield. This is due to the fact that plants are able to mobilize P fractions in the soil which are not taken into account by extraction methods. At least 90% of the yield of the TSP-60 treatment was achieved by sinter-P and MAP-Sb in all soils, as well as by apatite

in the acid soils and sl-ash in “Sattenhausen” (Table 6). Phosphate uptake by plants was highest for TSP-200 in all three soils, indicating that a fertilization of  $60 \text{ mg kg}^{-1}$  was suboptimal as intended. The fertilizer effect of the compounds was different for each soil. In “Sattenhausen” only MAP-Sb had a similar uptake to TSP-60. In the second acid soil “Düshorn”, MAP-Sb, apatite and sinter-P were similar to TSP-60, whereas in “Gieboldehausen” uptake of cupola slag and the both MAP’s was even slightly higher than that of TSP-60.

**Table 6.** Yield and P uptake of maize after fertilization with recycled P compounds grown on different soils in pot experiments.

	Sattenhausen (pH 5.6)		Gieboldehausen (pH 6.8)		Düshorn (pH 5.5)	
	yield	P uptake	yield	P uptake	yield	P uptake
	$\text{g pot}^{-1}$	$\text{mg pot}^{-1}$	$\text{g pot}^{-1}$	$\text{mg pot}^{-1}$	$\text{g pot}^{-1}$	$\text{mg pot}^{-1}$
unfertilized	85.6 bcd	92.0 de	55.3 cd	55.6 e	48.2 d	70.0 f
TSP-60	103.3 ab	143.4 b	102.7 a	113.8 b	101.5 ab	120.3 bc
TSP-200	120.5 a	242.2 a	105.5 a	196.2 a	119.4 a	235.9 a
rock-P	71.0 cd	95.0 de	37.8 d	48.0 e	63.7 cd	84.2 def
sinter-P	98.1 abc	117.1 c	92.5 ab	96.0 bc	103.5 ab	108.6 cd
sl-slash	92.4 abcd	110.1 cd	72.6 bc	89.0 bcd	45.3 d	76.3 f
MAP-Sb	103.2 ab	125.0 bc	95.2 ab	116.2 b	104.1 ab	139.2 b
MAP-Gf	86.5 bcd	119.9 c	88.6 ab	116.0 b	87.0 bc	104.0 cde
MB meal ash	69.2 d	88.8 e	67.4 bc	75.8 cde	57.2 d	77.5 ef
apatite	113.5 ab	89.1 e	35.6 d	63.7 de	95.1 ab	111.6 bcd
cupola slag	Not tested		85.0 abc	123.8 b	44.0 d	75.8 f

Different letters denote significant differences between treatments (Tukey,  $p < 0.05$ ).

## DISCUSSION

In the field experiment, the fertilizer effect was very low regardless of the P compound. Even TSP increased CAL extractable P or solution P only in tendency. This indicates a fixation of P in fractions that are not CAL soluble. Hence, it can not clearly be deduced whether the recycled P compounds became soluble and were immediately fixed as soil-P or not. However, P uptake and grain yield points to a slightly better plant availability of TSP, MAP-Gf, MAP-Sb and sinter-P compared to the other compounds, although the yield effects were not significant. The similar P uptake of the TSP-100 treatment and the unfertilized control indicates that plants were able to use mainly soil-born P despite the low P supply level of the soils as indicated by the CAL-P.

The pot experiments confirmed the field results with a somewhat better differentiation between the treatments. The CAL extractable P and solution P of

both MAPs, sinter-P and partly the apatite was comparable to the TSP-60 treatment whereas the other compounds differed not much from the unfertilized control. The plant availability can be assessed by deriving the relative fertilizer effect (RFE). The difference between P uptake of a specific fertilizer treatment and the unfertilized control reveals the amount of P that came from the fertilizer. This is compared to the fertilizer effect of a readily available fertilizer, i.e. the TSP-60 treatment, which is set to 100% (Table 7). Depending on soil and fertilizer this RFE ranged from 12% (cupola slag, “Düshorn”) up to 138% (MAP-Sb, “Düshorn”). A RFE close to TSP, i.e. more than 70%, was only achieved by MAP-Sb, MAP-Gf and cupola slag in “Gieboldehausen” soil and MAP-Sb, apatite and sinter-P in “Düshorn” soil. In “Sattenhausen” none of the tested compounds reached a RFE higher than 70% (Closest was MAP-Sb with 64%).

**Table 7.** Relative fertilizer efficiency for P recycled products utilized in pot experiments.

	Sattenhausen (pH 5.6)	Gieboldehausen (6.8) %	Düshorn (5.5)
TSP-60	100.0	100.0	100.0
TSP-200	292.2	241.6	329.8
rock-P	5.8	-13.1	28.2
sinter-P	48.8	69.4	76.7
sl-slash	35.2	57.4	12.5
MAP-Sb	64.2	104.1	137.6
MAP-Gf	54.3	103.8	67.6
MB meal ash	-6.2	34.7	14.9
apatite	-5.6	13.9	82.7
cupola slag	-	117.2	11.5

At all, the MAPs performed best of all tested compounds. These results are consistent with other studies. Plaza *et al.* (2007) tested the effectiveness of MAP in comparison with sewage sludge and conventional P fertilizer (TSP) by calculating the relative agronomic effectiveness (RAE) in relation to TSP and found also that MAP reached 94% of effectiveness. The P uptake of ryegrass was equal in all levels of P, indicating that MAP under certain conditions can be totally available. Johnston and Richards (2003) noted that struvite was as effective as monocalcium phosphate (MCP). A comparison with rock phosphate on acid soils indicates that struvite had the same fertilizer potential as rock phosphate (Gonzalez Ponce and De Sa, 2007). Ghosh *et al.* (1996) found that struvite was equally efficient as diammonium phosphate which is also a water soluble fertilizer. These evidences that struvite can be as effective as water

soluble P fertilizers are somewhat astonishing because the P in struvite is not water soluble (Johnston and Richards, 2003). There is also no clear indication that a low soil pH would be necessary to bring MAP in solution. The RFE of both MAPs on the acid soil “Sattenhausen” was comparably low (Table 7) and much higher on the neutral soil “Gieboldehausen”, whereas on the second acid soil “Düshorn” they react differently. Gonzalez Ponce and De Sa (2007) attributed the good effectiveness of struvite to the fine particle size of the material. It is well documented that the particle size, i.e. the surface area, affects the solubility of fertilizers. In our experiment all P compounds were finely ground, which might explain the relatively good availability of the MAPs. Looking closer at the soil and yield data revealed that both used MAPs differ in their solubility and plant availability, i.e. MAP-Sb performs better than MAP-Gf. This is most probably due to the high Fe concentration of MAP-Gf (26.6 g Fe/kg) compared to MAP-Sb (5.9 g Fe/kg). The phosphate in the Gf sewage works is precipitated by Fe leading to these high concentrations. Römer and Samie (2002) and Römer *et al.* (2004) could show that P availability of sewage sludge is strongly reduced by a high Fe concentration in the sludge.

## CONCLUSION

The results showed that none of the tested compounds reached in all soils the plant availability of a water-soluble P fertilizer in the pot experiment. Closest to this was the MAP-Sb in most soils. The ashes had a low availability, whereas the availability of sinter-P and cupola slag depended much on soil properties. However, these results so far are preliminary. Both, the field and pot experiments, were designed for a three-year period. They will be continued without a further P fertilization to examine the long-term fertilization effect of the tested compounds.

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