doi: 10.17221/513/2017-PSE

Phosphorus effects of recycled products from municipal wastewater on crops in a field experiment

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ABSTRACT

Vogel T., Nelles M., Eichler-Löbermann B. (2017): Phosphorus effects of recycled products from municipal wastewater on crops in a field experiment. Plant Soil Environ., 63: 475–482.

In this study, the phosphorus (P) fertilizing effects of struvite, one thermochemical-treated sewage sludge ash (SSA) based on Ca-P (Ca-SSA) and one full sulfuric acid-digested SSA based on Al-P (Al-SSA) were analysed in comparison to triple superphosphate (TSP) and a control treatment (CON) without P application in a two-year field experiment. In the field experiment, the effects of the recycling products on crop yield, P uptake and labile soil P fractions were analysed. In addition, the effect of nitrogen and magnesium contained in struvite was investigated in the second year of the experiment compared to TSP and CON. In the first year, spring barley was cultivated in the field experiment; and in the second year, it was forage rye followed by sorghum. In the second year, the relative P effectiveness (forage rye, sorghum) of the recycling products compared to TSP increased in the order: Ca-SSA (81%, 91%) ≤ Al-SSA (91%, 96%) = struvite (102%, 110%). In addition, an magnesium fertilizing effect of struvite could be demonstrated. The results show that the recycling products from wastewater treatment are appropriate to substitute rock phosphate-based fertilizers.

Keywords: macronutrient; bioavailability; organic contaminant; plant nutrition; *Hordeum vulgare* L.

Currently, mainly phosphate rock (PR)-based fertilizers are used in agriculture, but the majority of the PR resources are possessed by only a few countries and are enriched with cadmium and uranium (Jasinski 2014). Therefore, phosphorus (P) recycling from waste products for food production is the primary concern to guarantee the security of supply in Europe (Elser et al. 2014). Municipal wastewater is, in addition to manure, one of the most important P sources as it contains 16% of the mined P (Cordell et al. 2009). The direct use of sewage sludge, a residue of wastewater treatment, is declining in some EU member states since sewage sludge entails the risk to contain critical amounts of organic contaminants and heavy metals. For instance, Germany restricted the use of sewage sludge to smaller wastewater treatment plants (capacity in relation to population equivalent < 50 000) and Switzerland banned the direct application of sewage sludge (Kabbe et al. 2015). Therefore, technologies to recycle P from wastewater treatment are in development (Sartorius et al. 2012). Among these recycling products, struvite ($MgNH_4PO_4 \cdot 6\ H_2O$) is of particular significance due to its low concentration in contaminants and its high P availability, which was in the range of highly water soluble P fertilizers (Rahman et al. 2014). However, only a maximum of 40% of P in wastewater inlet can be recovered by struvite precipitation (Kabbe et al. 2015). Higher loads

doi: 10.17221/513/2017-PSE

(up to 90%) of P from wastewater treatment can be recovered by mono-incineration of sewage sludge (sewage sludge ash, SSA) combined with subsequent chemical treatment (Sartorius et al. 2012). The chemical treatment of SSAs is required to increase the P bioavailability and to achieve the legal limit values for heavy metals. The P fractions in treated SSAs vary considerably according to the used precipitating agent during the whole P recycling process and include readily soluble P and P adsorbed and precipitated on Al-, Fe-, Mg- and Ca-compounds (Donatello and Cheesman 2013).

In several pot experiments, a higher P availability of struvite compared to treated SSAs for diverse crops was observed, which was in the range of rock phosphate-based fertilizers (Severin et al. 2013, Vogel et al. 2015). Fewer efforts have been made to evaluate the P availability of struvite and SSAs for crops in the field. Pot experiments are necessary to produce replicable and comparable data, but their results cannot extrapolate to the field since pot conditions are very different from field conditions (e.g. soil temperature, aeration, water content) (Passioura 2006). This highlights the role of field experiments.

In general, the focus of research has been on the P fertilizing effect of struvite, while the research regarding the effect of magnesium (Mg: 10%) and nitrogen (N: 6%) in struvite on crop growth has been less pronounced (Bonvin et al. 2015). Mg is of particular importance, as Mg contents in cereal seeds have declined over the last decades (Cakmak 2013).

Therefore, the objectives of the present study were (1) to investigate the P fertilizing effect of struvite and SSAs under natural conditions in a field experiment in relation to their main P-binding forms and (2) to investigate the N and Mg fertilizing effect of struvite. It is hypothesized that the P fertilizing effect of these recycling products is adequate for plant nutrition in the field and that struvite can promote crop growth by its magnesium and nitrogen fertilizing effects.

MATERIAL AND METHODS

Characteristics of fertilizer treatments. The recycling products differed in composition in relation to the recycling process and raw material used (Table 1). The used struvite was produced according to a process developed by the Berliner Wasserbetriebe (BWB), where the digested sludge is aerated in a reactor to raise the pH up to 8 for struvite precipitation by stripping CO_2 and adding $MgCl_2$ (Kern et al. 2008). Two treated sewage sludge ashes were tested (the nomenclature was done according to the main P binding form in the ashes). For Al-SSA, P was precipitated in the wastewater with Al before the sludge was mono-incinerated. The ash was then treated by a H_2SO_4-washing procedure and 300 g H_2SO_4 (96%) per kg SSA was added to increase the P availability. More detailed information of the process is available in Petzet et al. (2011). The Ca-SSA was thermo-chemically treated, whereby heavy metals were evaporated in a rotary furnace at a temperature of 1000°C after the addition of $CaCl_2$ (Ca-SSA) as a chlorine donor at a rate of 100 mg/kg ash. Afterwards, 30% H_2SO_4 was added to increase the P availability. A more detailed description of the process can be found in Adam et al. (2009).

The same recycling products were used in a previous incubation experiment and in a previous pot experiment carried out by Vogel et al. (2015,

Table 1. Application rate, total P (P_t) content and solubility of P (citric acid soluble P, P_{ca}), total nutrient concentration (K, Mg, Ca, N, Al, Fe) as well as the pH of the recycling products and triple superphosphate (TSP) (Vogel et al. 2017)

	Rate (kg/ha)	P_t	P_{ca}	K	Mg	Ca	N	Al	Fe	pH
		(g/kg dry matter)								
Al-SSA	854.7	58.5	45.9	5.1	7.7	42.6	nd	55.8	10.4	1.9
Ca-SSA	1126.1	44.4	29.1	4.4	15.0	96.1	nd	26.2	54.5	3.9
Struvite	410.8	121.7	102.7	0.6	93.4	4.9	41.7	39.6	18.5	7.5
TSP	318.5	157.0	168.9	nd	8.9	106.7	nd	29.4	22.5	2.3

SSA – sewage sludge ashes; nd – not determined

doi: 10.17221/513/2017-PSE

2017). Highly water soluble triple superphosphate (TSP) was used as a reference.

Experimental design and soil characterization. In 2010 and 2011, a field experiment was carried out at the experimental station of the University of Rostock, which is located in Mecklenburg-Vorpommern (MV) in northeast Germany. Soil was low acidic (pH 5.8) loamy sand (Stagnic Cambisol) with total P (P_t) content of about 515 mg/kg. The double lactate soluble P (P_{dl}) and Mg (Mg_{dl}) content in soil averaged at P_{dl}: 55 mg/kg and Mg_{dl}: 114 mg/kg, indicating a suboptimal P supply (category B) and a high Mg supply (category E) (Schweder et al. 2004). In the field experiment, treatments with struvite, Al-SSA, Ca-SSA, TSP and a treatment without P application (CON) were carried out in three repetitions. The crops were cultivated on plots with the size of 4.5 × 6 m or 4.5 × 4.5 m in three repetitions. Edge effects were eliminated by harvesting only the centre of the plots (3 × 1.5 m). In the first year spring barley and in the second year forage rye (catch crop) followed by sorghum (main crop) were cultivated (Table 2). The surface biomass was harvested by cutting the plants within the measurement area.

In the field experiment, 50 kg P_t/ha was inserted into the soil in 2010 for the complete experimental time. N and K were applied in the experiment uniformly by mineral fertilizer. During the first year 110 kg N/ha and 80 kg K/ha and during the second year 140 kg N/ha and 110 kg K/ha were added by commercial fertilizers. Mg was not applied by mineral fertilizer since the Mg concentration in soil was at a level that further yield increase was no longer possible by applying more Mg. In addition, nutrients were supplied in different amounts by the wastewater products and TSP. Soil samples were collected before fertilizer application, after the harvest of spring barley and in the second year after the harvest of the main crop (sorghum) at 0–30 cm depth in order to determine the effect of labile P pools. The effects of N and Mg contained in struvite on crop yield and P availability were investigated in comparison to TSP and CON only in the second year of the field experiment.

Chemical analysis. To determine the dry matter (DM) yield of the crops, the harvested biomass was dried in an oven at 60°C for seven days and then weighed. The P and Mg concentration of the ground plant samples was measured after dry ashing using the vanadate-molybdate method (Page et al. 1982) and the N content was determined after wet digestion of the ground plant material using the Kjeldahl apparatus. The relative P effectiveness (RPE) (%) of the recycling products was calculated by comparing their P uptake to the P uptake of TSP.

$$\text{RPE} = \frac{(\text{P uptake of the recycling product})}{(\text{P uptake of the TSP})} \times 100$$

Air-dried and sieved (2 mm) soil samples were analysed for double lactate extractable P using the method described by Blume et al. (2000). The water-extractable P (P_w) was determined by extracting the soil samples with distilled water with a soil:water ratio of 1:25 (Van der Paauw 1971).

Statistics. Statistical analysis was performed with the software package PASW Statistics 18 (SPSS, Hong Kong, Ltd.). Analysis of variance (ANOVA, general linear model) was followed by a Duncan's multiple range test at the 0.05 level of significance to compare the means of soil and plant parameters.

RESULTS AND DISCUSSION

First year of the field experiment. In the first year of the field experiment, the rain was much lower in April (44%) as well as in June (46%) and July (81%) compared to the average values of the years 1991 to 2010, while at harvest time (August)

Table 2. Cultivated crops, cultivar of crops and seed rate in the field experiments

Crop	Cultivar	Seed rate	Sowing time	Time of harvest
Spring barley (*Hordeum vulgare* L.)	Marthe	300 gs/m²	08. 04. 2010	10. 08. 2010
Forage rye (*Secale cereal* L.)	Varda	140 kg/ha	01. 10. 2010	11. 05. 2011
Sorghum (*Sorghum bicolor* Moench)	Zerberus	25 gs/m²	20. 05. 2011	30. 09. 2011

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doi: 10.17221/513/2017-PSE

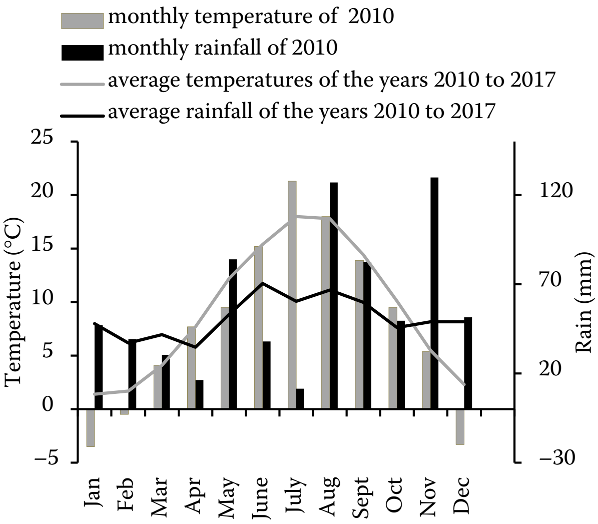

Figure 1. Monthly temperature and monthly rainfall in 2010 for Rostock; the values were compared to the average values of the years 1971–2010 (mean) for Rostock (Deutscher Wetterdienst)

the rain was twice as high as the average values of the years 1991 to 2010 (Figure 1). The temperature in 2010 was in the range of the average values of the years 1971 to 2010. As a consequence, the dry matter yield of spring barley in the field experiment was rather low due to the dry July and the wet August in 2010.

In the field experiment, no increases of P uptake and yield of spring barley by the wastewater products and TSP compared to CON were observed (Table 3). Concerning the low yield, this low effect on yield is most likely due to the soil P concentration in the field experiments, which was adequate for spring barley.

A stronger effect of the P treatments on labile P fractions was found. P_{dl} in soil was increased by 34% to 46% after application of struvite and TSP compared to CON, whereas the P_{dl} value in soil was not increased after application of the Al-SSA and the Ca-SSA compared to CON. In addition, the P_w concentration in soil was increased by struvite (by 22%) and by TSP (by 21%) compared to CON, while no effect of the Ca-SSA and the Al-SSA on P_w compared to CON was observed.

The lower effect of the Ca-SSA on labile soil P pools (P_{dl}, P_w) compared to struvite can be explained by different reaction mechanisms of Mg-P and Ca-P in soil. Over time, Mg-P reacts in soil to the plant available trimagnesium phosphate, while Ca-P converts to the plant unavailable hydroxyapatite (Vogel et al. 2013). In addition, the chlorine donor ($CaCl_2$) used during thermochemical treatment of the Ca-SSA resulted in the formation of poor soluble P compounds, which were not completely decomposed by the partial H_2SO_4 treatment (Nanzer et al. 2014). This was visible in the low concentration of citric acid soluble P (plant-available proportions of P_t) of the Ca-SSA (Table 1). The lower P effect on labile soil P pools of the Al-SSA compared to struvite can be related to its high concentration of amorphous Al (54.2 g/kg) since Al-rich P recycling products reduced the bioavailability of P in the first months after application to acidic soils due to the increase of P sorption capacity (Bøen et al. 2013). This effect of the Al-SSA on the P sorption capacity in acidic soil was demonstrated in a previous incubation experiment (Vogel et al. 2017). In addition, it is assumed that the nitrification of NH_4 contained in

Table 3. Dry matter yield, phosphorus (P) uptake of spring barley as well as double lactate soluble P (P_{dl}) and water soluble P (P_w) concentration in soil after addition of wastewater products, triple super phosphate (TSP) and control treatment without P addition (CON) in the first year after the cultivation of spring barley

	Dry matter yield (t/ha)	P uptake (kg/ha)	P_{dl}	P_w
			(g/kg soil)	
CON	3.7 ± 0.27[a]	10.8 ± 3.4[a]	51.9 ± 6.5[a]	16.6 ± 2.1[a]
Struvite	4.0 ± 0.30[a]	13.9 ± 1.6[a]	69.7 ± 3.2[b]	20.2 ± 0.4[b]
TSP	4.1 ± 0.14[a]	14.8 ± 4.1[a]	76.0 ± 1.0[b]	20.0 ± 1.4[b]
Al-SSA	4.3 ± 0.10[a]	11.6 ± 0.8[a]	60.7 ± 1.8[a]	15.4 ± 1.8[a]
Ca-SSA	4.4 ± 0.24[a]	11.8 ± 0.7[a]	58.0 ± 0.6[a]	16.4 ± 1.2[a]

SSA – sewage sludge ashes. Small letters indicate significantly different means between fertilizer treatments. Duncan's test a > 0.05; ± the standard error of the means

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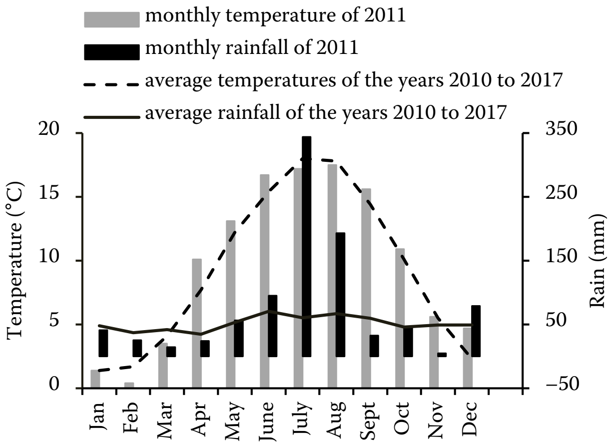

Figure 2. Monthly temperature and monthly rainfall in 2011 for Rostock; the values were compared to the average values of the years 1971–2010 (mean) for Rostock (Deutscher Wetterdienst)

struvite further enhanced its P solubility compared to the SSAs in soil by decreasing the soil pH near the struvite crystals since P dissolution of struvite is thought to increase under acidic conditions (Achat et al. 2014). This is confirmed by Daumer et al. (2007), who demonstrated that the dissolution of struvite increased during the treatment of piggery wastewater due to the fall in pH near the nitrifying bacterial floc by the release of H^+ ions.

Second year of the field experiment. In 2011, there was too dry spring with lower rain levels in February (31%), March (61%) and April (30%) compared to the average values of 1971 to 2010, while the level of rain in June (44%), July (81%) and August (46%) was much higher compared to the average values (Figure 2). The temperature was again in the same range compared to the average values of 1971 to 2010.

In 2011, the yield of forage rye was in the lower range of values that can be achieved in Mecklenburg-Vorpommern and can be related to the drought from February to April (Figure 2). The dry matter yield of forage rye was increased by struvite (by 36%), the Al-SSA (by 21%) and TSP (by 33%) compared to CON, while the Ca-SSA had no effect on the dry matter yield of forage rye compared to CON (Table 4). The P effectiveness of struvite (102%) and the Al-SSA (91%) after cultivation of forage rye was in the same range compared to TSP, while the P effectiveness of the Ca-SSA (81%) was lower compared to TSP. This can be explained by the dissolution behavior of the Ca-SSA in soil and its concentration of low

Table 4. Dry matter yield, phosphorus (P) uptake of cultivated crops as well as double lactate soluble P (P_{dl}) and water soluble P (P_w) concentration in soil after addition of wastewater products, triple superphosphate (TSP) and control treatment without P addition (CON) in the second year after cultivation of forage rye and sorghum

	Dry matter yield (t/ha)	P uptake (kg/ha)	P_{dl} (g/kg soil)	P_w (g/kg soil)
Forage rye (catch crop)				
CON	3.3 ± 0.17^a	7.3 ± 1.0^a	nd	nd
Struvite	4.1 ± 0.30^b	9.9 ± 0.8^b	nd	nd
TSP	4.2 ± 0.30^b	9.7 ± 1.1^b	nd	nd
Al-SSA	3.8 ± 0.35^{ab}	8.8 ± 0.9^b	nd	nd
Ca-SSA	3.3 ± 0.38^a	7.9 ± 1.3^a	nd	nd
Sorghum (main crop)				
CON	8.1 ± 0.94^a	17.1 ± 0.7^a	47.2 ± 8.8^a	12.7 ± 1.4^a
Struvite	11.1 ± 1.40^b	25.6 ± 2.9^c	66.4 ± 11.2^a	17.3 ± 1.8^b
TSP	11.3 ± 1.22^b	23.3 ± 1.2^{bc}	63.3 ± 4.8^a	16.6 ± 1.0^b
Al-SSA	10.8 ± 0.84^b	22.4 ± 2.8^{bc}	57.5 ± 2.8^a	11.3 ± 1.8^a
Ca-SSA	11.0 ± 0.58^b	21.2 ± 0.7^b	56.8 ± 4.8^a	13.0 ± 1.0^a

SSA – sewage sludge ashes. Small letters indicate significantly different means between fertilizer treatments. Duncan's test a > 0.05; ± the standard error of the means

doi: 10.17221/513/2017-PSE

soluble P compounds, as described above. The yield of sorghum in the field experiments was rather low since in north Germany the cultivation of sorghum imposes generally late sowing due to the relatively low temperatures in spring (Ercoli et al. 2004). In this field experiment the yield (by 32% to 44%) and P uptake (by 33% to 50%) of sorghum were increased by the wastewater products and TSP compared to CON (Table 4).

The yield and P uptake after application of the SSAs and struvite were in the same range compared to TSP. This can be illustrated by the P effectiveness of the recycling products compared to TSP after cultivation of sorghum, which increased in the same order as after the cultivation of forage rye: Ca-SSA (91%) ≤ Al-SSA (96%) ≤ struvite (110%). After the cultivation of forage rye followed by sorghum no differences between the P_{dl} concentration in soil of the P fertilizer treatments and the CON treatment were observed, while P_w in soil was higher in the TSP treatment (by 39%) and in the struvite treatment (by 32%) compared to CON. This indicates again that struvite has higher P bioavailability than the Al-SSA and the Ca-SSA.

However, the high P effect of the Ca-SSA on the yield of sorghum is in contrast to their effect on the yield of forage rye, which was lower compared to TSP. It cannot be fully clarified with our study, why sorghum and forage rye showed different yield responses to the fertilizer treatments, but the rapidly growing root system of sorghum may have increased soil exploitation and consequently the P supply in the Ca-SSA-treatment (Khalili et al. 2008).

The high effectiveness of P contained in the Al-SSA found in the field experiment for forage rye and sorghum is in contrast to the results of a previous pot experiment (over eight weeks), where a lower P uptake in the Al-SSA treatment compared to TSP after cultivation of forage rye and sorghum was found in acidic soil (pH 5.2) (Vogel et al. 2015). As described above, the low P availability for crops found in the pot experiment can be related to its effect on P sorption capacity in the first month after addition to acidic soils. However, Al-P contained in soil after the first growing season is thought to be available to plants in the coming growth season (e.g. Krogstad et al. 2005), which was confirmed by this field experiment.

In addition, it must be considered that the SSAs have to be applied in higher amounts to the soil compared to struvite due to their lower P concentrations. This might increase the risk of heavy metal accumulation in soil still further, in particular since the SSAs contain higher amounts of heavy metals compared to struvite (Vogel et al. 2017). Furthermore, struvite precipitating is already implemented and operating in full scale, while the processes for recovering P from mono-incinerated SSA are currently tested in a pilot phase and the whole P recovery process including the mono-incineration and the post-treatment of the SSA is a cost and energy-extensive process (Kabbe et al. 2015).

The fertilizing effect of struvite and TSP on N and Mg uptake was only analysed during the second year after cultivation of forage rye and sorghum. The N uptake was increased by struvite and TSP compared to CON after cultivation of sorghum (Table 5). This can be attributed to the P-fertilizing effect of struvite and TSP (Achat et al. 2014), which increased the yield and consequently the N uptake of sorghum as described above. Concerning the low initial N dose applied by struvite (17.1 kg/ha) and the N supply by commercial fertilizer, no N fertilizing effect of struvite could be demonstrated. The Mg uptake of forage rye and sorghum was increased by struvite compared to CON and TSP (Table 5), which can be related to the Mg dose applied by struvite (38.4 kg/ha).

Table 5. Nitrogen (N) and magnesium (Mg) uptake of (kg/ha) cultivated crops after addition of struvite and triple superphosphate (TSP) and control treatment without phosphorus (P) addition (CON) in the second year after cultivation of forage rye and sorghum

	N uptake	Mg uptake
Forage rye (catch crop)		
CON	38.8 ± 6.3^a	3.14 ± 0.2^a
Struvite	43.1 ± 3.9^a	3.82 ± 0.3^b
TSP	44.5 ± 5.4^a	3.34 ± 0.2^a
Sorghum (main crop)		
CON	72.3 ± 7.3^a	17.4 ± 4.0^a
Struvite	103.0 ± 5.4^b	22.6 ± 3.8^b
TSP	102.3 ± 3.0^b	18.7 ± 1.2^a

SSA – sewage sludge ashes. Small letters indicate significantly different means between fertilizer treatments. Duncan's test a > 0.05; ± the standard error of the means

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This is confirmed by González-Ponce et al. (2009), who observed a higher Mg uptake by lettuce after struvite application compared to single superphosphate.

In conclusion, P contained in the wastewater products studied here was readily available for the cultivated crops in the acidic soil under field conditions, and consequently these recycling products can reduce the dependence on rock phosphate-based fertilizer. The results in the first year of the field experiment indicate that the bioavailability of struvite is higher compared to the Ca-SSA and the Al-SSA. In addition, the Mg fertilizing effect of struvite found in this field experiment showed that struvite can contribute to an increase of the Mg-concentration in food crops. In case of the Al-SSA, its P availability is likely too low for crops in the first growing season, and therefore needs to be applied in combination with highly water soluble P fertilizer. However, possible negative effects of these recycling products on crop growth need to be excluded in further pot experiments and in long-term field experiments (e.g. Al toxic effects on crops) and under diverse soil conditions.

Acknowledgements

The authors thank the Faculty of Interdisciplinary Research of the University of Rostock for the financial support to this work. Further support was given by Leibniz Science Campus Phosphorus Research Rostock. We also thank Outotec, the Berliner Wasserbetriebe, IST Anlagenbau GmbH and the University of Darmstadt for providing the recycling products.

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Received on August 14, 2017
Accepted on October 19, 2017
Published online on November 1, 2017