Institute of Crop Science (340)
Department of Biobased Resources in the Bioeconomy (340b)
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Suitability of recycled organic residues from animal husbandry and bioenergy production for use as fertilizers

Dissertation

Submitted to the
Faculty of Agricultural Sciences

In fulfilment of the requirements to obtain the degree
Doctor scientiarum agriculturae (Dr. sc. agr.)

Submitted by
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2021

Date of the acceptance of the dissertation: 19.05.2022
Date of oral exam: 31.05.2022

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List of abbreviations and acronyms

ASN	ammonium sulfate nitrate
BioEcoSIM	Research project: An innovative bio-economy solution to valorise livestock manure into a range of stabilised soil improving materials for environmental sustainability and economic benefit for European agriculture. Grant agreement no. 308637
CAN	calcium ammonium nitrate
CMC	Component Material Category
DAP	diammonium phosphate
DM	dry matter
DMY	dry matter yield
DüV	Düngemittelverordnung, German Fertiliser Ordinance
e.g.	exempli gratia (Latin = for example)
FPR	Fertiliser Product Regulation
GOBi	Research project: General Optimization of Biogas Processes. FKZ 03EK3525A
ha	hectare
i.e.	id est (Latin = that is)
K	potassium
K_2O	potassium oxide
LCA	life cycle assessment
LEX4BIO	Research project: Optimizing Bio-based Fertilisers in Agriculture – Knowledgebase for New Policies. Grant agreement no. 818309
LU	livestock unit
N	nitrogen
N_2O	nitrous oxide
NH_3	ammonia
NH_4^+	ammonium
N_{min}	soil mineral nitrogen
N_{org}	organic nitrogen
P	phosphorus
P_2O_5	phosphate
PA	plant availability
PFC	Product Function Category
SHS	superheated steam

t	tonne (1,000 kg)
TSP	triple superphosphate
VDLUFA	Association of German Agricultural Analytic and Research Institutes

Abstract

In recent years, agriculture has been increasingly faced with the acute need to find a more sustainable practice for dealing with nutrient-rich organic side streams. For ecological and economic reasons, pressure is mounting every day to implement an improved utilisation and take all conceivable measures to close nutrient loops in agriculture to the maximum possible.

Organic residues resulting from pig husbandry and anaerobic digestion are of quantitative significance, both at farm level and for agriculture in general. They are suitable as organic fertilisers because they contain essential plant nutrients. In addition, they provide organic matter that contributes to the maintenance of soil fertility. However, they are increasingly considered as waste - a costly environmental disposal challenge rather than a valuable source of nutrients and bio-based fertilisers. As such, their current use is often insufficient.

Pig manure and biogas digestates can be used as fertilisers either directly or following treatment. Treatment can be as simple as solid-liquid separation. A more advanced approach is the recovery of phosphorus (P) from manure and digestate via precipitation for conversion into phosphate fertilisers. These are referred to as "P-Salts" in this thesis. The remaining solids can be dried, e.g. with warm air or steam, or pyrolysed to biochar. The significant reduction in volume increases the transportability of P-Salts and dried solids compared to the untreated residues. The fertilising effect of P-Salts recycled using innovative technologies needs to be investigated in an agronomic context. The same applies for the integration of separated biogas digestates as organic fertilisers into different biomass production systems.

The primary objective of this thesis is to establish whether recycled fertilisers from organic residues are comparable to mineral fertilisers and can serve as a suitable substitution. For this purpose, five specific objectives were defined: (1) to determine whether separated biogas digestates can complement or substitute mineral fertilisers and whether/how they affect long-term yield performance in different biomass cropping and fertilisation systems; (2) to ascertain which type of separated biogas digestate is suitable for which biomass production system; (3) to test the effect of two recycled P-Salts on yield and quality of different crops and assess their competitiveness with commercial superphosphate; (4) to examine whether the combination of recycled P-Salts with biochar and dried solid digestates results in interaction effects; and (5) to assess whether there are differences in the uptake efficiency of recycled and mineral fertilisers between different crop types.

To explore these objectives, several experiments were carried out. The fertilising effect of separated biogas digestates on three biomass production systems was investigated in multi-year field experiments at two challenging sites in south-west Germany. Three cropping systems were considered: perennial grassland, intercropping of triticale and clover grass, and silage maize. P-Salt and biochar, both obtained from pig manure, were tested in a greenhouse study with spring barley and faba bean. In a second greenhouse study, the P-Salt recycled from manure, a similar P-Salt from biogas digestate, and dried solid digestates were assessed in sunflower, marigold and Chinese cabbage cultivation.

The results revealed that all recycled fertilisers tested resulted in biomass yields for the most part comparable with - and in few cases better than - the conventional treatments.
The long-term yield stability of biomass cropping systems fertilised with separated biogas digestates was clearly demonstrated under field conditions. Separated biogas digestates can substitute mineral fertiliser in perennial and intercropping systems. Solid digestates were most suitable for cropping

systems with soil tillage where their incorporation into soil is possible. For perennial grassland, liquid digestates were better than solids in terms of workload and application. The intercropping of triticale and clover grass was found to be the most stable system, with constantly high biomass yields being maintained using solid or liquid digestates. However for annual crops such as maize, a combined application of digestates and mineral fertiliser proved to be the best option for ensuring sufficient nutrient supply.

The P-Salt recycled from manure was found to have the same or even better effects than triple superphosphate (TSP) on the growth of spring barley and faba bean in two test soils. In the experiment with sunflower, marigold and Chinese cabbage, the two P-Salts recycled from manure and digestate had more or less the same effect as TSP on biomass production. These results suggest that both P-Salts have an equivalent fertilisation effect to TSP and can thus replace it as mineral fertiliser. The good fertilising effect of the P-Salts in cereals, legumes, ornamentals and vegetables confirmed their versatile applicability for a broad range of crop types.

Biochar in combination with P-Salt enhanced the fertilising effect of the latter, especially on poor soils with low organic matter. The combination of P-Salt and air-dried solids resulted in measurable synergistic effects on the biomass production of all test crops. These effects were attributed to the short- and long-term P supply of the two fertilisers and the soil conditioning effect of the solids. The two P-Salts recycled from manure and digestate met the P demand of sunflower, marigold and Chinese cabbage as efficiently as TSP. The P-Salts showed higher suitability for short-term and the steam-dried solids for long-term P supply.

In all three studies included in this thesis, it was possible to achieve competitive yield results with the tested fertilisers and combinations, provided that they are integrated in a suitable fertilising strategy so that the nutrients are plant-available preferably at the time of the demand. The next step is for the recycled fertilisers to be actually used in agricultural practice - a prerequisite for which being that their implementation has agronomic, practical, ecological and economic advantages.

The enhanced use efficiency of N and P already available on farms is a challenging but necessary step to reduce dependency on both N fertilisers synthesised under high energy input and imported P fertilisers derived from phosphate rock. The results of the work described in this thesis present a significant contribution by providing knowledge on the fertilising effect of selected recycled fertilisers necessary for their future implementation in agriculture.

Optimised nutrient management and residue treatment can contribute to the further closing of nutrient cycles with benefits for both the environment and the economy. The highest environmental benefits of nutrient recycling and residue treatment using advanced recovery technologies can be realised on farms with excess residues and limited agricultural land. It is therefore highly recommended that these farms improve their current practice by prioritising the implementation of appropriate residue management measures.

Sound residue management necessitates strategic planning and capital investments from farmers and companies, but is a crucial step towards the sustainable intensification of national and European cropping systems and resilient future agriculture. Consequently, farmers require easy access to targeted funding to implement the necessary changes. In addition, a reliable and clear legal framework is necessary for the production and utilisation of recycled fertilisers, supported by coherent and knowledge-based political decisions.

Zusammenfassung

In den letzten Jahren sah sich die Landwirtschaft zunehmend damit konfrontiert, eine nachhaltigere Lösung für den Umgang mit nährstoffreichen organischen Nebenströmen zu finden. Aus ökologischen und ökonomischen Gründen wird der Druck immer größer, diese besser zu nutzen und alle erdenklichen Maßnahmen zu ergreifen, um Nährstoffkreisläufe in der Landwirtschaft so weit wie möglich zu schließen.

Organische Reststoffe aus der Schweinehaltung und der anaeroben Vergärung sind sowohl auf Betriebsebene als auch allgemein in der Landwirtschaft sehr relevant, weil sie in großen Mengen anfallen. Da sie essentielle Pflanzennährstoffe enthalten, eignen sie sich gut als organische Düngemittel. Zusätzlich trägt die enthaltene organische Substanz zur Erhaltung der Bodenfruchtbarkeit bei. Jedoch werden diese Reststoffe zunehmend als Abfall betrachtet, welcher kostspielig zu entsorgen ist, statt als wertvolle Nährstoffquelle oder biobasierte Düngemittel. Die derzeitige Verwendung ist deshalb häufig unzureichend.

Schweinegülle und Biogasgärreste können als Düngemittel entweder direkt oder nach einer Behandlung verwendet werden. Die Behandlung kann eine einfache Fest-Flüssig-Trennung sein. Ein aufwendigeres Vorgehen ist die Rückgewinnung von Phosphat aus Gülle und Gärresten mittels Fällung und die Herstellung von Phosphatdünger, der in dieser Arbeit als P-Salz bezeichnet wird. Die verbleibenden Feststoffe können beispielsweise mit Warmluft oder Dampf getrocknet oder zu Biokohle pyrolysiert werden. Die deutliche Volumenreduktion erhöht die Transportfähigkeit der P-Salze im Vergleich zu den unbehandelten Reststoffen. Die Düngewirkung von P-Salzen, die mit solch innovativen Technologien zurückgewonnen werden, muss agronomisch untersucht werden. Das Gleiche gilt für die Integration separierter Biogasgärreste als organische Dünger in verschiedene Biomasseanbausysteme.

Das Hauptziel der vorliegenden Arbeit ist es daher, festzustellen, ob aus organischen Reststoffen recycelte Düngemittel mit Mineraldüngern vergleichbar sind und diese möglicherweise ersetzen können. Zu diesem Zweck wurden fünf speziellere Ziele definiert: (1) zu bestimmen, ob separierte Biogasgärreste Mineraldünger ergänzen oder ersetzen können und ob/wie sie die langfristige Ertragsleistung in verschiedenen Biomasseanbausystemen beeinflussen; (2) herauszufinden, welche separierten Biogasgärreste sich für welches Biomasseproduktionssystem eignen; (3) die Wirkung von zwei recycelten P-Salzen auf Ertrag und Qualität verschiedener Kulturpflanzen zu testen und ihre Konkurrenzfähigkeit mit herkömmlichem Superphosphat zu beurteilen; (4) zu untersuchen, ob die Kombination von recycelten P-Salzen mit Biokohle und getrockneten festen Gärresten zu Interaktionseffekten führt; und (5) zu beurteilen, ob es zwischen verschiedenen Pflanzenarten Unterschiede in der Nährstoffaufnahme aus recycelten und herkömmlichen Düngemitteln gibt.

Zur Untersuchung dieser Ziele wurden mehrere Versuche durchgeführt. Die Düngewirkung von separierten Biogasgärresten wurde anhand von drei Biomasseproduktionssystemen in mehrjährigen Feldversuchen an zwei marginalen Standorten in Südwestdeutschland untersucht. Folgende drei Anbausysteme wurden betrachtet: mehrjähriges Grünland, Triticale mit Kleegrasuntersaat (überjährig) sowie einjähriger Silomais. P-Salz und Biokohle, beide aus Schweinegülle gewonnen, wurden in einem Gewächshausversuch mit Sommergerste und Ackerbohnen getestet. Das aus Gülle gewonnene P-Salz, ein ähnliches P-Salz aus Biogasgärresten und getrocknete feste Gärreste wurden in einem zweiten Gewächshausversuch mit Sonnenblumen, Tagetes und Chinakohl untersucht.

Die Ergebnisse zeigten, dass alle getesteten Recyclingdünger zu Biomasseerträgen führten, die überwiegend vergleichbar und in manchen Fällen sogar höher waren als die mit den Mineraldüngern erzielten Erträge.

Die langfristige Ertragsstabilität von Biomasseanbausystemen, die mit separierten Biogasgärresten gedüngt wurden, konnte in Feldversuchen eindeutig nachgewiesen werden. In mehrjährigen und überjährigen Systemen können separierte Biogasgärreste Mineraldünger ersetzen. Feste Gärreste waren am besten für Anbausysteme geeignet, in denen eine Einarbeitung in den Boden möglich ist. Für Dauergrünland waren flüssige Gärreste bezüglich Arbeitsaufwand und Ausbringung besser geeignet als feste Gärreste. Der überjährige Anbau von Triticale mit Kleegrasuntersaat erwies sich als das stabilste System, das konstant hohe Biomasseerträge lieferte und mit festen oder flüssigen Gärresten aufrechterhalten werden kann. Für einjährige Kulturen wie Mais war jedoch die Kombination aus Gärresten und Mineraldünger die beste Option, um eine ausreichende Nährstoffversorgung sicherzustellen.

Das aus Gülle recycelte P-Salz hatte die gleiche oder sogar eine bessere Wirkung als Triple-Superphosphat (TSP) auf das Wachstum von Sommergerste und Ackerbohnen in zwei Bodenarten. In dem Versuch mit Sonnenblumen, Tagetes und Chinakohl hatten die beiden aus Gülle und Gärresten recycelten P-Salze eine vergleichbare Wirkung auf die Biomasseproduktion wie TSP. Diese Ergebnisse deuten darauf hin, dass beide P-Salze eine gleichwertige Düngewirkung wie TSP haben und es somit als Mineraldünger ersetzen können. Die gute Düngewirkung der P-Salze in Getreide, Leguminosen, Zierpflanzen und Gemüse bekräftigt ihre vielseitige Anwendbarkeit für ein breites Spektrum an Kulturarten.

Biokohle in Kombination mit P-Salz verbesserte die Düngewirkung des P-Salzes, insbesondere auf schwachen Böden mit niedrigen Gehalten an organischer Substanz. Die Kombination aus P-Salz und luftgetrockneten Feststoffen führte zu messbaren synergistischen Effekten auf die Biomasseproduktion aller Testpflanzen. Diese Effekte wurden der kurz- und langfristigen P-Versorgung durch die beiden Dünger und der bodenverbessernden Wirkung der Feststoffe zugeschrieben.

Die beiden aus Gülle und Gärresten recycelten P-Salze deckten den P-Bedarf von Sonnenblumen, Tagetes und Chinakohl ebenso wirksam wie TSP. Die P-Salze eigneten sich besser für eine kurzfristige und die dampfgetrockneten Feststoffe besser für eine langfristige P-Versorgung.

In allen drei Studien, die in diese Arbeit einbezogen wurden, konnten mit den getesteten Düngern und Kombinationen gute Ertragsergebnisse erzielt werden, sofern sie in eine geeignete Düngestrategie integriert und die Nährstoffe vorzugsweise zum Zeitpunkt des Bedarfs pflanzenverfügbar werden. Im nächsten Schritt müssen die zurückgewonnenen Dünger in der landwirtschaftlichen Praxis auch tatsächlich eingesetzt werden. Dies setzt voraus, dass der Einsatz agronomische, praktische, ökologische und ökonomische Vorteile hat.

Eine bessere Ausnutzung von bereits auf den landwirtschaftlichen Betrieben vorhandenem Stickstoff und Phosphat ist ein herausfordernder, aber notwendiger Schritt, um die Abhängigkeit von N-Düngern, die mit hohem Energieaufwand synthetisiert werden, und von importierten P-Düngern aus Phosphatgestein zu verringern. Diese Arbeit trägt hierzu bei, indem sie Erkenntnisse zur Düngewirkung ausgewählter Recyclingdünger liefert, die für deren künftige Anwendung in der Landwirtschaft notwendig sind.

Optimiertes Nährstoffmanagement und Reststoffaufbereitung tragen zur besseren Schließung von Nährstoffkreisläufen bei, was sowohl ökologische als auch ökonomische Vorteile hat. Der größte Umweltnutzen von Nährstoffrecycling und Reststoffaufbereitung mit fortschrittlichen Verfahren kann in Betrieben mit Nährstoffüberschüssen und begrenzten landwirtschaftlichen Flächen erzielt werden. Es wird daher dringend empfohlen, dass diese Betriebe ihre derzeitige Praxis verbessern und mit hoher Priorität entsprechende Maßnahmen zum Reststoffmanagement umsetzen.

Ein solides Reststoffmanagement verlangt strategische Planung und Investitionen von Landwirten und Unternehmen, ist aber ein entscheidender Schritt hin zu einer nachhaltigen Intensivierung der nationalen und europäischen Anbausysteme und zu einer resilienten zukunftsfähigen Landwirtschaft. Daher benötigen die Landwirte einen einfachen Zugang zu Fördermitteln, um die notwendigen Veränderungen umzusetzen. Darüber hinaus ist ein verlässlicher und eindeutiger rechtlicher Rahmen für die Herstellung und die Nutzung von Recyclingdüngern erforderlich, der durch einheitliche und wissensbasierte politische Entscheidungen unterstützt wird.

1 General Introduction

In recent years, the agricultural sector has been faced with the acute need to find a more sustainable practice for dealing with nutrient-rich organic sidestreams. For ecological and economic reasons, pressure is increasing every day to implement an improved utilisation and to take all conceivable measures to close nutrient loops in agriculture to the maximum possible. Such a substantial transformation requires a shift away from the sectorial thinking widespread in today's highly specialised agriculture to a more systemic and circular way of thinking.

Agriculture in Germany produces approximately 227 million t of nutrient-rich organic residues every year (Figure 1).
Pig manure and biogas digestates are two types of organic residue of high quantitative importance both at farm level and in agriculture generally. Pig manure consists of liquid and solid manure fractions and slurry. Biogas digestates, also referred to as biogas residues, biogas slurry or biogas effluents, result from the anaerobic digestion of various substrates, including farmyard manure and plant biomass – be it energy crops grown specifically for this purpose or other (residual) crop material that is available "anyway".

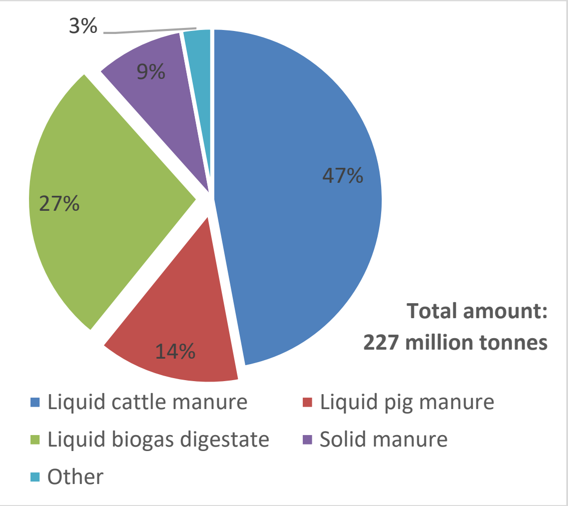

Figure 1: Farmyard manure applied to arable land and grassland in Germany in 2015 (Destatis 2017)

Pig manure and biogas digestates have the following common features:

- They are both organic by-products of practices aimed at the production of major agricultural commodities, i.e. meat from pig husbandry; biogas and electricity from anaerobic digestion of various substrates.
- Both need to be dealt with in one form or another, preferably by putting them to good use. Their application should ideally be performed in a reasonable manner that is as agronomically efficient, environmentally friendly, economically viable and socially acceptable as possible.
- They are both suitable as organic fertilisers, since they contain essential plant nutrients. In addition, they contain organic matter which can contribute to maintaining soil fertility.
- They can be used as fertilisers either directly or following various treatments, ranging from simple to advanced. The use of treated residues is a relatively new topic. As such, many questions still remain unanswered and require research. This is particularly the case for residues treated with novel and more advanced technologies.
- After treatment, there are of course potential alternative uses in other (related) sectors and for other purposes, e.g. in horticulture for the production of vegetables and ornamentals (nutrients, growing media), but also for material utilisation (e.g. carbon for building materials (Essel et al. 2015)), bioenergy generation (e.g. solid fuel for combustion (Kratzeisen et al. 2010)) or a combination of several approaches in holistic biorefinery concepts (Awiszus et al. 2019).

- They are increasingly considered as wastes, representing a costly environmental disposal challenge rather than an underexploited opportunity as a source of nutrients and bio-based fertilisers.

1.1 Significance of residue amounts

A brief glance at the statistics reveals the significance of the accumulating residue quantities:

Pig husbandry and resulting manure:
In November 2018, there were 22,400 farms in Germany keeping approx. 26.4 million pigs, almost 12 million of which were fattening pigs (Destatis 2019a). Assuming 1.2 t manure per animal and year (Foged et al. 2011), this results in an estimated total annual manure production of 31.7 million t. This figure is in agreement with the registered liquid pig manure that was field-applied in Germany in 2015 and amounted to approx. 31 million m³ (Destatis 2017). Field application of pig manure was realised on 33,650 farms, which corresponds to 12% of all farms (Destatis 2017).

Anaerobic digestion and resulting digestates:
In 2019, approximately 9,500 agricultural biogas plants were in operation in Germany (German Biogas Association 2021). The biogas sector has grown exponentially from 2005 onwards as a result of attractive subsidies which were often granted in form of guaranteed feed-in tariffs for the produced electricity (Bahrs and Angenendt 2018). However, a further considerable increase in the number of biogas plants is no longer expected due to changes in the German Renewable Energy Act (EEG 2017), which led to massive cuts in feed-in tariffs. In addition, the proportions of silage maize or cereal grains in biogas substrates were limited to 60% in 2012 and further reduced in two steps to 44% by 2021. Recently, there was only an increase in small-scale biogas plants (max. 75 kW) fed mainly with farmyard manure from the respective farm. A further capacity expansion is expected to slow markedly until 2030 due to economic reasons (Liebetrau et al. 2019).

As the existing biogas plants will still be in operation for several years – provided they receive subsidies or remain profitable - they will continue to produce digestates in the midterm that need to be utilised.

Quantifying the amount of digestates produced depends on several factors. Firstly, it is important to consider the type of biogas plant. This study includes agricultural biogas plants only, meaning they are installed on a farm and fed mainly with farmyard manure and/or crop biomass. Secondly, calculating digestate amounts based on the installed electric capacity is somewhat unreliable, because not every biogas plant is operated at full capacity or at all times. Thirdly, the digestates produced are not really recorded officially, particularly not for smaller biogas plants.
However, field application of biogas digestates has to be monitored in order to fulfil the legal requirements set by the German Fertiliser Ordinance (DüV 2021). Official statistics (Destatis 2017) report that, in 2015, the amounts of digestates applied to arable land and permanent grassland were 62.5 million m³ for liquid digestates (applied by 38,390 farms = 14% of all farms) and 1.7 million t for solid digestates (applied by 3,450 farms = 1% of all farms).
This is consistent with Möller and Müller (2012) who estimated the annual amount of biogas digestates produced at 65.5 million m³ in Germany. More recent studies point to 80 million cubic metres (Wulf and Schultheiß 2017) or 82 million t (Kirsch 2018), in line with the increased number of biogas plants.

These vast amounts of pig manure and biogas digestates represent an enormous potential for nutrient recovery.
Nutrient concentrations in the residues of course vary widely and depend on a number of factors. These include, for example, the production stage of pigs, with differences between mother sows (also

depending on the number of piglets), young animals after weaning, fattening pigs and males for breeding. The feeding regime (standard or NP-reduced) and the housing system (strawless with slatted floors, deep litter, roaming area, water amount needed for cleaning etc.) also influence the manure composition (Chastain et al. 2003). Digestate composition varies according to the biogas substrates used, e.g. nutrient-poor materials naturally lead to low nutrient contents (Zirkler et al. 2014; Möller et al. 2010). Composition of both residue types varies according to storage system (e.g. covered vs. uncovered; uncovered storage results in higher N losses, material may be diluted by rain or other water) and duration (the longer the storage time, the higher the losses are likely to be) (Möller et al. 2010).

Average N concentrations are 2.3 – 6.0 kg N m^{-3} for manure and 4.1 – 4.8 kg N m^{-3} for digestates. They contain 2.7 - 4.0 and 1.8 – 3.5 kg P_2O_5 m^{-3}, respectively (Döhler 2009). Roughly calculated, this gives an annual total of 490,200 t N and 142,500 t P from pig manure and digestates that are available for fertilisation in Germany (Table 1).

Table 1: Annual amounts of pig manure and biogas digestates with corresponding nutrient recovery potential in Germany (typical nutrient concentration ranges are given in brackets)

	Amount	**Average N concentration[1]**	**Resulting N amount**	**Average P_2O_5 concentration[1]**	**Average P concentration**	**Resulting P amount**
	million m^3	kg N m^{-3}	t	kg P_2O_5 m^{-3}	kg P m^{-3}	t
Pig manure	31	4.2 (2.3 – 6.0)	130,200	3.4 (2.7 - 4.0)	1.5	46,500
Biogas digestates	80	4.5 (4.1 – 4.8)	360,000	2.7 (1.8 – 3.5)	1.2	96,000
Total:			490,200			142,500

[1] Döhler (2009)

If we compare this with the annual consumption of synthetic N and mineral P fertiliser in Germany, which contain 1.7 million t N and 126.000 t P (Eurostat 2018), respectively, it becomes evident that almost 30% of the N demand and (more than) the entire P demand could theoretically be met by these two selected residues alone.

This simplified calculation does not take (gaseous) nutrient losses during storage into account. Neither is it realistic to expect a complete recovery of N and P from the treatment of the entire residues available, at least not in the next five to ten years. Nonetheless, the above comparison reveals the considerable fertilising value potential of these residues.

1.2 Problem identification

Today, the predominant use of the two residue types discussed is their application on arable land and grassland. As such, they “remain” within the agricultural sector. Although field application seems an efficient way of closing nutrient cycles, reduces the need for mineral fertiliser inputs, and has been common practice for decades, this strategy must often be deemed insufficient and suboptimal for a number of reasons.

The three main problems involved in the current use are identified as follows:

1) Regional hotspots with nutrient surpluses

Pig production and anaerobic digestion are not equally distributed over Germany, or Europe in general, but are frequently found in regional so-called “hotspots”, where they result in an excess of manure and digestates, and thus an excess of nutrients.

Such large numbers of pig farms with high livestock densities are often coupled with limited land availability. The nutrient loads are too high to be applied to the surrounding fields in a way that is environmentally friendly and in accordance with current legislation (max. 170 kg N ha^{-1}), even under feeding regimes with reduced P and protein input (Oenema et al. 2007).

The problem is exacerbated by imported animal feed, mainly soybean, which further contributes to the accumulation of nutrients. In addition, farm sizes (in terms of livestock numbers) are continuing to grow as a consequence of economic pressure.

Examples of hotspot regions with intensive pig production and resulting manure excess in Germany are the Weser-Ems region around Vechta (3.6 livestock units (LU) per ha agricultural land;) and Cloppenburg (3.1 LU per ha) in Lower Saxony and Borken (2.7 LU per ha) and Kleve (3.3 LU per ha) in North Rhine-Westphalia (Maennel 2018; LWK NRW 2018). Germany is characterised by large regional differences in terms of livestock husbandry and land cultivation types. Bavaria, for example, has the third-highest absolute number of pigs. Nevertheless, the pig density of 45 pigs per km² is not critical in Bavaria due to its relatively large land area. In contrast, the pig density in Schleswig-Holstein is comparatively high at 90 pigs per km² (Destatis 2019a, Destatis 2021). Examples of hotspots at EU level include the Netherlands, Flanders in Belgium, and Normandy and Brittany in France.

Here, manure disposal has become an economic challenge for the farmers. Disposal is performed either by processing it or by transporting it to other regions. Processing is carried out with the main objective of volume reduction in centralised plants using expensive energy-intensive technologies (e.g. biological nitrogen removal, reverse osmosis), recovering little if any of the nutrients. Transporting is also known as so-called “slurry tourism”, where manure is transported over long distances (>200 km) to other regions with nutrient demand or even deficits where arable production and stockless or low-stock farms are predominant. Farmers have to pay up to 30 € for the disposal of one tonne of manure (Bach 2018) and this price is expected to increase in the future. For example, for the first time manure storage tanks were partly not emptied in the Borken region in 2018 due to high disposal costs and, in consequence, pig farmers feared that they would have to discontinue their production (Bach 2018). So, when the manure disposal becomes a limiting factor for pig production, it may involve serious consequences for the continued existence of farms. Apart from the high expenses and the environmental burden, the transporting practice lacks acceptance and is highly - and rightly - criticised.

2) Variability of fertilising effect and nutrient plant availability

The fertilising effect and the nutrient plant availability of organic fertilisers are much more variable and less predictable in comparison to water-soluble inorganic fertiliser (Möller 2009; Hjorth et al. 2010; Odlare et al. 2011).

The highly variable physicochemical characteristics, as explained above, require analysis before field application in order to allow for a targeted nutrient supply. The release of these nutrients in the soil then depends on external factors that cannot be influenced, such as weather conditions, soil properties or soil process dynamics.

The nutrient ratio of N and P in the residues is not ideal to match the crops’ needs. Today, the applied doses are usually calculated based on the N concentration. Thus, more P than necessary may be applied to sites that are already (more than) sufficiently supplied with P (Maltais-Landry et al. 2016). Another challenge is that biogas digestates, for example, combine a short-term fertilisation effect of N (ammonium!) with a long-term fertilisation effect of P (Möller and Müller 2012).

For the user, all of this results in a greater effort and requires higher skills as well as even more anticipatory planning of the fertiliser application in comparison to mineral fertiliser.

3) Negative environmental impacts and resulting consequences

Nitrogen is lost in form of gaseous nitrous oxide (N_2O) and ammonia (NH_3), both climate relevant trace gases with high global warming potential. In 2017, agriculture was responsible for 78% of the anthropogenic nitrous oxide N_2O and for 95% of the anthropogenic NH_3 emissions in Germany (Destatis 2019b). Farmyard manure is hereby considered the most important source for the NH_3 emissions (LWK Niedersachsen 2018).

The emissions not only occur in the stables and during storage but also during and following field application. Application not carried out according to good agricultural practice or not using appropriate techniques leads to avoidable emissions. State-of-the-art techniques include trailing hoses, trailing shoes and slurry injectors, and ensure that manure is applied near the soil surface or directly injected into the soil. However, these are only very slowly becoming the norm. In fact, 56% of all liquid manure applied in Germany in 2015 was spread with some sort of widespreading device (Destatis 2017). Other ways of relevant nutrient losses include run-off (P and N) and leaching (mainly nitrate), resulting in contamination of (ground)water and eutrophication of waterbodies (Guzman-Bustamante et al. 2019). In Germany, for example, the nitrate values in the groundwater exceeded the limit of 50 mg per litre at on average 28% of the monitoring sites from 2012 to 2014 (Keppner et al. 2017). Subsequently, the European Commission European has sued Germany before the European Court of Justice in 2016 for violating the Nitrates Directive (EC 91/676/EEC), resulting in a conviction in 2018 that imposed increased activities against elevated nitrate concentrations in the groundwater (BMU 2020).

In consequence, the German Fertiliser Ordinance (DüV 2021) was amended, i.e. regulations have become stricter and have certainly put increased pressure on the farmers. Examples of measures affecting the practice include that N in digestates needs to be fully taken into account in the max. applicable 170 kg N ha^{-1}; lower surpluses in nutrient budgets for N and P and the gradual introduction of farm-specific material flow balances by 2023 (StoffBilV 2017); changes in the classification of soil P concentration by VDLUFA that now considers soils (over)saturated at a lower P concentration than before (Wiesler et al. 2018); extended storage capacity for liquid manure and digestates to up to nine months; and a larger distance from neighbouring waters to be ensured during N and P fertilisation (DüV 2021).

This outline of main problems shows very clearly that the current practices must not go on as they are. Firstly, losses of valuable nutrients are partly avoidable by using state-of-the-art application techniques. Secondly, the selected way of use does not consider all aspects of sustainability. Thirdly, regulations on field application of organic residues are likely to become more restrictive in the future. Consequently, viable alternatives are urgently needed.

1.3 Potential solutions

One option to address the mentioned challenges is treatment of the residues. Numerous treatment options are conceivable and currently available in different states-of-the-art (lab-scale, pilot-scale, demo-scale, established in practice), ranging from relatively simple to advanced and highly complex approaches. Table 2 gives a brief overview of simple and advanced treatment options that were available during the initial phase of this study in 2012, without claim to comprehensiveness.

A survey on behalf of the German Environmental Agency revealed that at the majority of the responding biogas plants (approx. 80%) there was no treatment at all in place (Scholwin et al. 2019). As expected, there were mainly biogas plants with more than 75 kW_{el} among the small proportion of plants that treated the residues.

Mechanical solid/liquid separation is currently the most widely applied processing method for manure and digestates (ten Hoeve et al. 2014). It represents a relatively simple treatment approach to reduce water content and volume and increase transportability (Hjorth et al. 2010). Screw presses accounted for 68% of digestate separators used on-farm in a study by Guilayn et al. (2019) in the category of low-performance separation. In the category of high-efficiency separation, the great majority of separators were decanting centrifuges (Guilayn et al. 2019).

Centrifuges are characterised by a higher electricity demand and higher susceptibility to malfunctions than screw presses whereas the latter are advantageous because they are affordable, robust and easy to handle (Hjorth et al. 2010). Screw presses are thus even suitable for small farms (Hanserud et al. 2017). The separation changes the nutrient allocation in the fractions compared to the raw digestate (Möller et al. 2009; Hjorth et al. 2010). For this reason and because of the relevance in practice, the integration of the resulting separated digestate fractions into biomass production systems is extensively assessed in field experiments in this study.

Other options for treatment include drying or combinations of separation and drying. Subsequent drying of the separated solid fraction is another relatively easy step. When this is done with waste heat e.g. from the biogas plant, the process can be considered reasonable. Pelletising the dried material further facilitates the handling and field spreading. However, these simple technologies rather focus on volume reduction. This is not sufficient in regions with extreme manure and digestate excesses and land scarcity.

Thus, more advanced treatment technologies have been developed or are currently being developed. They aim ideally at a holistic, possibly complete, low-input process to treat the entire residues in high quantities at reasonable costs, while recovering and reusing nutrients, obtaining secondary raw materials and creating marketable products with added value.

Vacuum evaporation and ammonia stripping ranked among less adopted possibilities according to the above-mentioned survey (Scholwin et al. 2019). Approaches including pyrolysis, hydrothermal carbonisation, micro- and ultrafiltration, ammonia removal, reverse osmosis in order to obtain clean water and several precipitation approaches, usually follow a simple solid/liquid separation and can be implemented separately or in various combinations. Most of them are still mainly far from practical relevance.

Table 2: Overview and simple rating of treatment options available at the initial stage of this thesis (2012)

Technology	Suitable for	Negative impact on environment	Not economically feasible[a]	Tested only at lab-scale[a]
Storage & application	FM, SF, LF	●		
Solid/liquid separation	FM	●[b]		
Composting	FM, SF	●		
Biogas production	FM, SF, LF (pig manure only)	●[b]		
Vacuum evaporation	FM, SF		●	
Drying	SF	●[c]		
Incineration & gasification	SF		●	
Pyrolysis	SF			●
Hydrothermal carbonisation	FM, SF	●[d]		●
Micro- and ultrafiltration	LF		●	
Reverse osmosis	LF		●	
Biological N removal	FM, LF	●		
Stripping and precipitation of ammonium sulfate	LF, SF		●	

Aluminium und iron phosphate precipitation	LF	●	
Struvite and calcium phosphate precipitation	LF		●

FM = untreated fresh material of pig manure or biogas digestate; SF = solid fraction of separated pig manure or biogas digestate; LF = liquid fraction of separated pig manure or biogas digestate

[a] at the beginning of this work (2012), this has partly changed until today (2021)

[b] NH_3 and CH_4 emissions and P and N contamination of groundwater during storage and land application of separated fractions or digested manure

[c] NH_3 emissions unless off-gases are treated

[d] hydrochar has shown negative impacts on soil function compared to pyrolytic biochar (Bargmann et al. 2014)

References: Bonmati and Flotats 2003; Foged 2010; Lehmann and Joseph 2009; Libra et al. 2011; Liao et al. 1995; López-Fernández et al. 2011; Marinari et al. 2000; Masse et al. 2007; O'Shaughnessy et al. 2008.

Even the advanced technologies mainly focus on volume reduction and only partly on nutrient removal instead of true nutrient recovery for further targeted utilisation. None of them was mature enough to simultaneously process residues on a large-scale, recover the nutrients and produce marketable fertilisers while being environmentally benign and economically feasible.

Consequently, the EU-funded BioEcoSIM project (Grant agreement no. 308637) went one step further and aimed at an integrated circular economic concept. During the four years of the project (2012-2016), it succeeded in developing a promising technology to recover P and N separately from pig manure, while pyrolysing the organic matter to biochar and reclaiming the water. During the project duration, the treatment plant was upscaled from lab-scale prototypes of several sizes until it reached the pilot stage, whose function has been demonstrated for more than 15 months. In this way, it was realised for the very first time that raw pig manure was completely utilised and turned into valuable products in a stable continuous process and competitive technological setup, thus, clearly reaching beyond the previous state-of-the-art.

The BioEcoSIM approach starts with a pre-treatment step - acidification of the raw manure to dissolve as much P as possible – followed by a solid-liquid separation (Figure 2). After addition of a base to increase the pH, P is then precipitated from the liquid fraction. It is obtained as a complex of magnesium ammonium phosphate (struvite), calcium phosphate (hydroxyapatite) and magnesium phosphate and is referred to as ‘P-Salt’ in this work. The remaining liquid is microfiltrated, followed by nitrogen recovery in form of crystallised ammonium sulfate. The effluent is clean enough to be used for irrigation purposes or to be discharged into the draining canal. The solid fraction is dried with superheated steam (SHS) and then pyrolysed to produce biochar.

Biochar is reported to act as a soil improver (Laird et al. 2010; Bruun et al. 2014), P source (Bruun et al. 2017), to have beneficial effects on crop yield (Lehmann et al. 2011), and to be an opportunity for carbon sequestration in the soil (Vaccari et al. 2011; Polifka et al. 2018). Potential synergy effects in combination with organic fertiliser are conceivable (Alburquerque et al. 2013). It is thus tested together with the recovered P-Salts in a greenhouse study in the framework of this thesis.

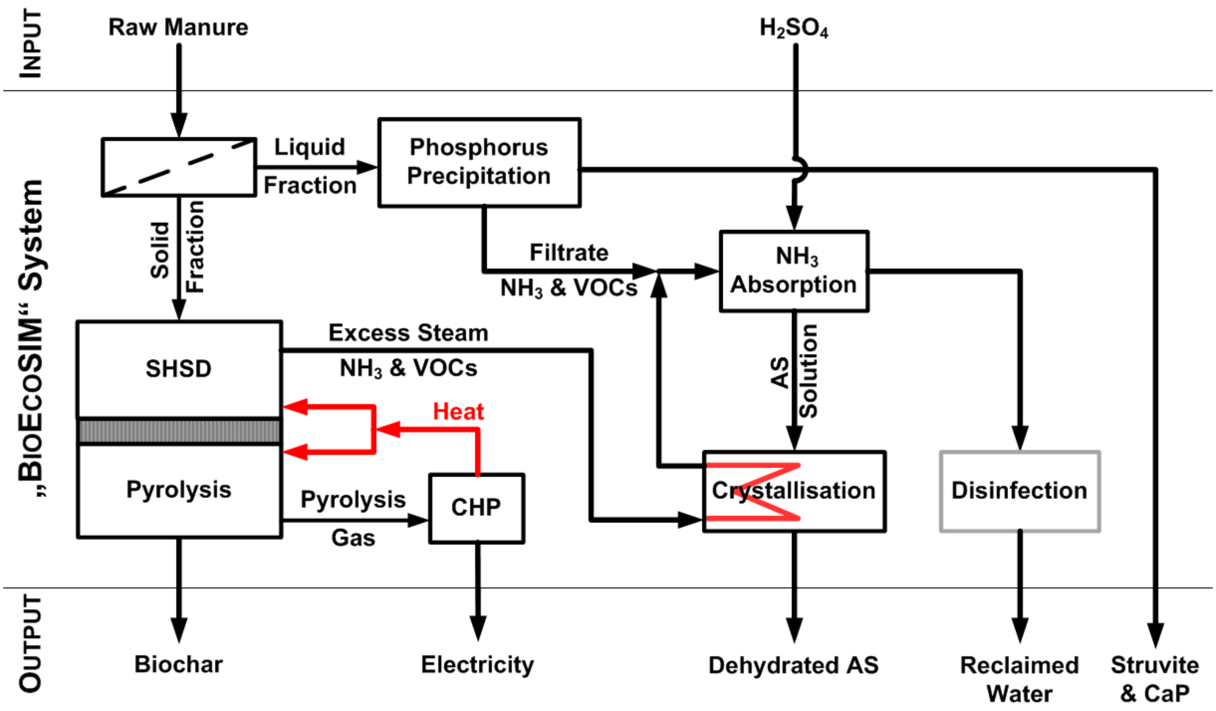

Figure 2: The BioEcoSIM concept for recovery of N and P from pig manure (BioEcoSIM 2016). (SHSD = superheated steam-drying, AS = ammonium sulfate, CaP = calcium phosphate, VOCs = volatile organic carbons, CHP = combined heat and power)

A very similar technology, yet focusing on treating biogas digestates, was subject of the GOBi project funded by the German Federal Ministry of Education and Research (FKZ 03EK3525A). The first steps of acidification and solid-liquid separation, pH increase and P precipitation are basically the same, but adapted to the specific physicochemical properties of digestates. In contrast to BioEcoSIM, the liquid fraction was not further processed after the P recovery step. The solid fraction was simply dried, either using warm air or superheated steam.

The experiments included in this thesis were carried out with products obtained using these two treatment technologies. The anticipated advantages and benefits of both the approaches and the resulting products are numerous:

- The nutrients are recovered separately and can then be formulated to a customised fertiliser, the nutrient ratio of which can be defined to exactly match the crops' specific requirements. Further P addition to soils with already high P stocks is avoided.
- In contrast to the high water content (>90%) of the initial residues (Döhler 2009), the products are dry, easy to handle, storable, and transportable even over long distances.
- The costs for storage, application and potential disposal of treated residues will be lower compared to the untreated material due to volume reduction.
- Mineral fertiliser savings are possible, particularly for N and P, and have benefits from different perspectives. The expenses for fertilisers at farm level are of considerable economic importance and their prices are fluctuating, because they depend on energy prices and market demand.
  - The main negative environmental impact of N fertiliser is its energy-intensive production. NH_3 synthesis via the Haber-Bosch process is responsible for 2% of the global energy consumption (Pfromm 2017). The net energy consumption today is 30 GJ per tonne of NH_3 (Ghavam et al. 2021). This is mainly covered with natural gas, which thus accounts for

    62-84% of the production cost of N fertilisers (ESPP 2018). In addition, Europe highly depends on natural gas imports. N fertiliser supply is basically unlimited as it can be industrially synthesised from atmospheric N. On-farm, N can also be obtained through biological fixation by legumes (organic farming!) or replenished by the plant-soil system.
  - In contrast, P is a finite resource. The EU itself has only an insignificant source in Finland (Römer and Steingrobe 2018). Over 90% of the phosphate for fertilisers used in the EU are imported from external phosphate rock sources. The largest reserves are located in northern Africa, China, the Middle East, and the USA (USGS 2016), some of which are considered politically unstable regions. Thus, the EU is susceptible to market price fluctuations and to economic or political crises. For this reason, phosphate rock has been added to the EC's list of critical raw materials in 2014, followed by phosphorus in 2017 (EC 2018b). Another pressing problem is the diminishing quality of mined phosphate rock. It is increasingly contaminated with pollutants, including uranium and cadmium (particularly in P from Moroccan sources), that end up in the environment with the P fertiliser (Franz 2008). Although removal is technically possible, it renders the process more expensive. P recycling from residues has therefore become a major research focus in recent years, reflected by the fact that it is the subject of numerous research projects at national, European and global level.
- There are no more odour emissions from pig manure spreading that previously displeased residents.
- Both technologies are built as modular systems and are as such adaptable to the respective residue amounts to be treated and to the envisaged degree of recycling (e.g. in certain cases P removal from residues may be sufficient).
- The economic value of manure and digestate can be calculated using the current market prices of nutrients applied as mineral fertilisers. The monetary fertiliser value of manure from fattening pigs was estimated at 4.98 € per m³ (Bastuck 2018) and of biogas digestate at 5.92 € per m³ (Rolink 2013). The benefit of the organic matter is not yet considered in this price. A comparison of this number with the annual manure and digestate production illustrates the huge market potential of innovative nutrient recycling technologies. There is significant scope for increased efficiency of nutrient utilisation itself.
- So, the treatment also enables turning the waste problem into an economic opportunity as the approach opens up considerable potential for products with added value. The concept of nutrient cycling suggests of course that the nutrients preferably stay within agriculture. However, certain excess that comes with imported feedstock suggests the export of e.g. P to related sectors. Such a broader application range beyond direct agriculture may include horticulture (ornamentals, vegetables etc.) or the hobby gardening sector (Herbes et al. 2019).
- The BioEcoSIM approach is unique in that the manure is entirely processed and there is no residue left. Thus, it would fulfil the end-of-waste criteria set by the EU (EC 2018/851), because manure ceases to be waste and obtains the status of a product or a secondary raw material, e.g. P-Salts for fertiliser production.

  Furthermore, both approaches, BioEcoSIM and GOBi, contribute excellently to the central elements of the EU Circular Economy Package launched in 2015 and the EU Circular Economy Action Plan adopted in 2016. This implicates that waste management should become more efficient by means of an increased resource management, improved nutrient cycles and a sharpened sustainability aspect (EC 2018/851).

1.4 Aim of the study

Given that the two treatment approaches described above are so innovative, they require thorough research activities – on the processes themselves, but of course also on the output materials. It is crucial to investigate how the obtained salts affect crop growth and what ultimately happens to them in the field. This is exactly where this thesis has its starting point, as the fertilising effect of the recycled nutrients needs to be investigated in an agronomic context. The same applies for the integration of separated biogas digestates as organic fertilisers into different biomass production systems.

Thus, the primary objective of this thesis is to establish whether recycled fertilisers from organic residues are comparable to mineral fertilisers and can serve as a suitable substitution.

To help explore this primary objective in more detail, the following specific objectives were defined:

(1) to determine whether separated biogas digestates can complement or substitute mineral fertilisers and whether/how they affect long-term yield performance in different biomass cropping and fertilisation systems;
(2) to ascertain which type of separated biogas digestate is suitable for which biomass production system;
(3) to test the effect of two recycled P-Salts on yield and quality of different crops and assess their competitiveness with commercial superphosphate;
(4) to examine whether the combination of recycled P-Salts with biochar and dried solid digestates results in interaction effects;
(5) to assess whether there are differences in the uptake efficiency of recycled and mineral fertilisers between different crop types.

Several experiments were designed and carried out in order to achieve the aforementioned objectives. An overview is presented in Figure 3. The fertilising effect of separated biogas digestates on three biomass production systems was investigated in two long-term field experiments at the agricultural research station 'Lindenhöfe' of the University of Hohenheim. Recycled P-Salt and biochar, both obtained from pig manure, were tested in a greenhouse study with spring barley and faba bean.

The same P fertiliser was assessed in another greenhouse study with flowering plants, in order to compare its performance with that of the P-Salt recycled from biogas digestate. Dried solid digestates were also included in this comparison, applied alone as well as in combination with the P-Salt from digestate. Hence, this third study represents a neat synthesis of the other two studies, as it combines products obtained from both residue streams that were treated with both simple and advanced techniques.

The experiments presented in Figure 3 resulted in three scientific papers that have already been published in peer-reviewed journals.

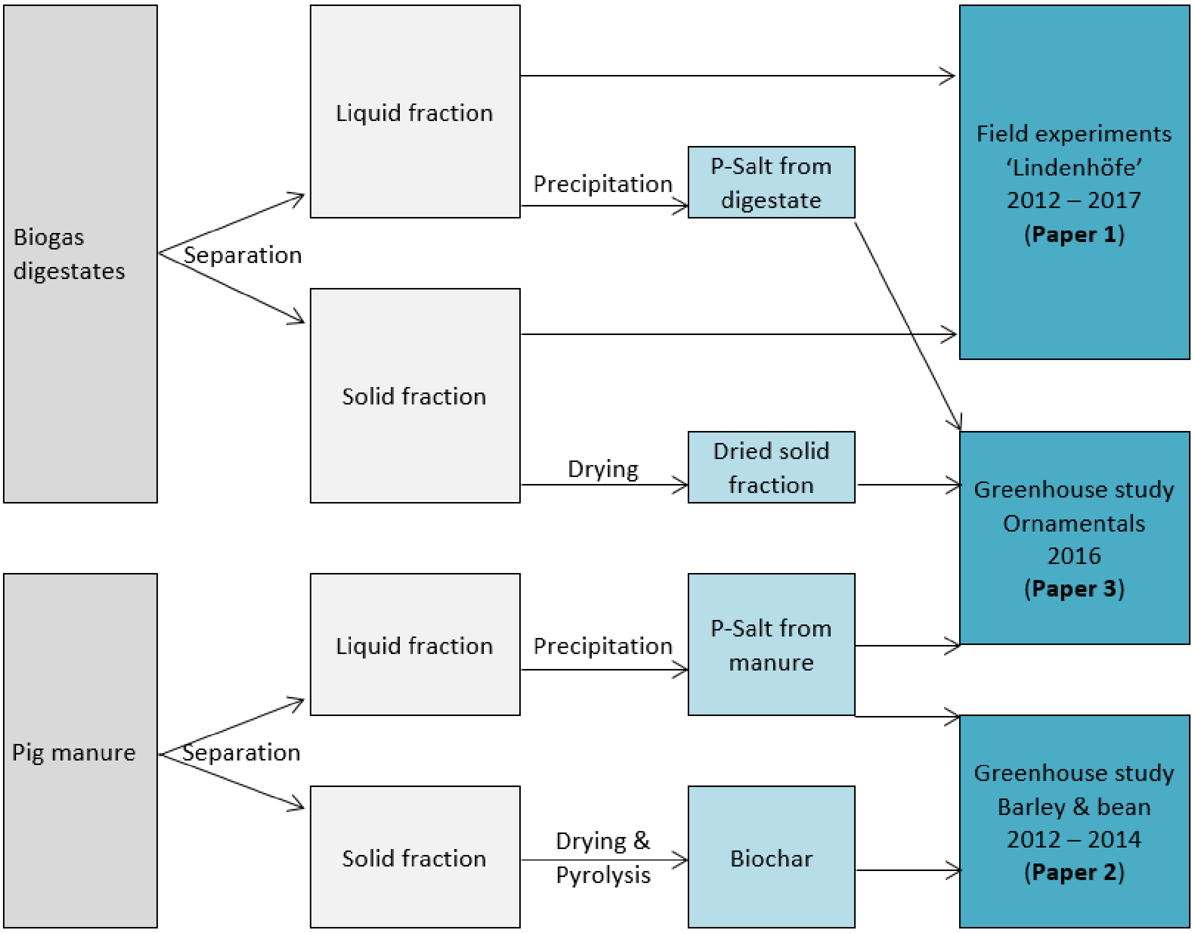

Figure 3: Interrelation between the investigated residue streams, applied treatments, obtained products, experiments and resulting publications

1.5 Publications

This cumulative dissertation comprises three scientific articles on the evaluation of recycled fertilisers from organic residues under field and greenhouse conditions. They all contribute to the objectives stated above. The articles are included in Chapter 2 to 4 of this thesis and have been published in international peer-reviewed journals.

Chapter 2 is entitled *"Fertilising potential of separated biogas digestates in annual and perennial biomass production systems"* and includes the following publication:

> Ehmann, A., Thumm, U. and Lewandowski, I. (2018) Fertilizing potential of separated biogas digestates in annual and perennial biomass production systems. *Front. Sustain. Food Syst.* 2:12. doi: 10.3389/fsufs.2018.00012

Chapter 3 is entitled *"Effect of manure-based phosphate salt on biomass yield of spring barley and faba bean in comparison to conventional fertiliser"* and includes the following publication:

> Ehmann, A., Bach, I.-M., Laopeamthong, S., Bilbao, J. and Lewandowski, I. (2017) Can phosphate salts recovered from manure replace conventional phosphate fertilizer? *Agriculture* 7:1. doi: 10.3390/agriculture7010001

Chapter 4 is entitled *"Suitability of phosphates recycled from semi-liquid manure and digestate as alternative fertilisers for ornamentals"* and includes the following publication:

> Ehmann, A., Bach, I.-M., Bilbao, J., Lewandowski, I. and Müller, T. (2019) Phosphates recycled from semi-liquid manure and digestate are suitable alternative fertilizers for ornamentals. *Scientia Horticulturae* 243, 440–450: doi: 10.1016/j.scienta.2018.08.052

Additional publications and conference contributions are listed in Chapter 6.

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2 Fertilising potential of separated biogas digestates in annual and perennial biomass production systems

In this chapter, the effect of separated biogas digestates applied to three biomass cropping systems – perennial grassland; intercropping of triticale and clover grass; and annual silage maize was assessed. Multi-year field experiments were established at two challenging sites in south-west Germany. A strong focus was on the multi-year aspect in order to evaluate the long-term yield performance of the systems influenced by fertilisation treatment and site. The study mainly addresses the specific objectives (1) and (2) of this thesis that refer to the competitiveness of digestates with mineral fertiliser and their suitability for different biomass production systems. The results from six years are presented here.

This chapter is published in the journal *Frontiers in Sustainable Food Systems*, as:

Ehmann, A., Thumm, U. and Lewandowski, I. (2018) Fertilizing potential of separated biogas digestates in annual and perennial biomass production systems. *Front. Sustain. Food Syst.* 2:12. doi: 10.3389/fsufs.2018.00012

frontiers
in Sustainable Food Systems

ORIGINAL RESEARCH
published: 24 April 2018
doi: 10.3389/fsufs.2018.00012

Fertilizing Potential of Separated Biogas Digestates in Annual and Perennial Biomass Production Systems

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Digestates produced by the increasing number of biogas plants require appropriate treatment or recycling. This study investigates the fertilizing potential of separated biogas digestates. These contain valuable nutrients and can be used in agriculture to close the nutrient cycle. Multi-year field experiments were established at two challenging sites in south-west Germany in 2010; results from 6 years are shown here. The objectives were to determine (1) whether separated digestates can complement or substitute mineral fertilizers and (2) their effect on long-term yield performance in different biomass cropping and fertilization systems. The fertilizing performance was assessed in a split-plot design with four replications using three cropping systems: (1) perennial grassland; (2) intercropping of triticale and clover grass; (3) silage maize. Five N fertilization treatments were applied, each at 150 kg N ha^{-1}:

- mineral fertilizer (calcium ammonium nitrate)
- combined solid digestate fraction and mineral fertilizer
- solid digestate fraction
- combined liquid digestate fraction and mineral fertilizer
- liquid digestate fraction.

The influences of site, cropping system, year and fertilization treatment were highly significant.The mineral fertilizer and combination "liquid digestate fraction + mineral fertilizer" mostly led to the highest quantitative biomass yields in all cropping systems at both sites. Fertilization with solid digestate fraction produced lowest yields in all fertilized plots, with results very often not significantly different from the untreated control. Maize achieved relatively high yields in years with favorable weather conditions; unfavorable conditions led to low yields. The grassland and intercropping systems were less susceptible to weather conditions, producing a more constant biomass supply irrespective of site, treatment and year. The separated biogas digestates were found to have a comparable effect to mineral fertilizer on biomass yield, but this varied with cropping system. In the intercropping system, complete substitution was

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Specialty section:
This article was submitted to
Waste Management in
Agroecosystems,
a section of the journal
Frontiers in Sustainable Food Systems

Received: *31 January 2018*
Accepted: *06 April 2018*
Published: *24 April 2018*

Citation:
Ehmann A, Thumm U and Lewandowski I (2018) Fertilizing Potential of Separated Biogas Digestates in Annual and Perennial Biomass Production Systems. Front. Sustain. Food Syst. 2:12. doi: 10.3389/fsufs.2018.00012

possible. The solid fraction is more likely to contribute positively to soil humus in annual systems. In general, the combined application of digestate and mineral fertilizer is highly recommendable to meet crops' short- and long-term N demand, even on challenging sites. In this study, it allowed a mineral fertilizer input reduction of 66%.

Keywords: biogas digestates, fertilization, cropping systems, bioenergy, alternative biogas substrates, nutrient cycles

INTRODUCTION

Power and heat generated from biogas provide a significant contribution to the increasing amount of bioenergy produced in Europe. Here, more than 17,300 biogas plants were counted in 2015 (EBA, 2016) with a primary production of 654 petajoules (Eurostat, 2015). The biogas sector has experienced a strong impetus in Germany in particular, supported by the German Renewable Energy Sources Act, which was introduced in 2000 and has since been modified several times. In 2017, there were more than 9,300 agricultural biogas plants operating in Germany alone (German Biogas Association, 2017), producing 116 petajoules electric power (BMWi, 2017) and an estimated 65.5 million cubic meters of biogas digestates (Möller and Müller, 2012).

Biogas digestates are the residues left from the anaerobic fermentation of organic matter, such as animal manure and plant biomass specifically grown for this purpose. Through the production of biogas (CH_4 and CO_2) in the fermentation process, the amount of carbon is significantly ($>50\%$) reduced (Tambone et al., 2009). Depending on the operating system (including pH and temperature) of the biogas plant, N can also be lost (as NH_3) to a certain extent (Reinhold et al., 2004). However, most of the N and all other mineral elements contained in the input substrates remain in the biogas digestates (Vaneeckhaute et al., 2017). These include major plant nutrients such as phosphorus, potassium and calcium. Therefore, it is common practice to use biogas digestates as organic fertilizers (Alburquerque et al., 2012a), which at the same time saves costs for both mineral fertilizer and potential disposal of the digestates (up to 25 € t^{-1}, Rolink, 2013). The good fertilizing value of biogas digestates in comparison to mineral fertilizer has been confirmed in several studies (Formowitz and Fritz, 2010; Gunnarsson et al., 2010; Walsh et al., 2012; Barbosa et al., 2014). Also, the remaining carbon bound in the organic matter helps to maintain or even increase soil organic matter (Möller, 2015), which is particularly valuable in marginal soils (Nabel et al., 2014). This effect can be considerable in annual cropping systems.

Although the use of biogas digestates as organic fertilizer seems an efficient way of closing nutrient cycles in agriculture and reducing external inputs of mineral fertilizer, several potential drawbacks need to be considered in order to optimize the efficiency and environmental performance of biomass production systems.

The first is the distribution of digestates. They accumulate at biogas plants and their high water content ($>90\%$) limits their ability to be stored and transported. For this reason, many farms separate the digestates on site in order to reduce the water content and volume and increase transportability (Hjorth et al., 2010). The processing is mostly done using screw press separators, a robust and simple on-farm technology. The separated liquid fraction is characterized by high N (mainly in the form of directly plant-available ammonium) and potassium contents and a total solids content of below 5% (Gutser et al., 2005; Möller et al., 2009; Nkoa, 2014). The solid fraction contains approx. 20% of the total N, a third of the total phosphorus and 15% of the potassium and up to 35% total solids (Rolink, 2013; Vaneeckhaute et al., 2017). Farmers often collaborate with the (more or less) neighboring farms supplying them with biomass in order to optimize operation capacity, particularly in larger biogas plants. The biomass suppliers receive digestates in return, thus helping to manage any oversupply.

Second, the composition of biogas digestates can vary due to variations in substrate supply and, as described above, differs between the solid and liquid fractions. For this reason, farmers are often unsure about the performance of digestates as organic fertilizers and various studies have shown that their fertilizing effect is not always as predictable as that of mineral fertilizer (Möller, 2009; Hjorth et al., 2010; Odlare et al., 2011). Some have reported that such variation in organic fertilizers can lead to fluctuation and/or reduction in biomass yield (e.g. Alburquerque et al., 2012b; Sieling et al., 2013). In order to guarantee biomass yield stability, the yield effect of biogas digestates and their liquid and solid fractions needs to be assessed. One option for overcoming this shortcoming of organic fertilizers may be the use of digestates in combination with mineral fertilizer or gradual supplementation of mineral fertilizers by digestates. To test this, two combinations of digestates and mineral fertilizer were included in this study.

Third, decomposition of the organic matter during the fermentation process leads to an enrichment of NH_4-N in biogas digestates (Reinhold et al., 2004). This increases the probability of gaseous N being lost during storage and application. To avoid such losses, field applications of digestates should be timed to meet the crops' nutrient demand and low-emission application techniques should be used. Nutrient demands and optimal fertilization systems very much depend on the type of cropping system. Application techniques and timing, and also the fertilizing effects of organic fertilizers all differ between annual cropping systems, such as maize, and perennial cropping systems, such as grassland (Svoboda et al., 2013). In grassland for example, the immediate effect of an organic fertilizer is usually not very pronounced due to the high organic matter content of the soil (Conant et al., 2017).

In farming practice, the nutrient-rich biogas digestates are generally applied as fertilizer to crops grown for biomass to be used as biogas feedstock. Three such crops are considered in this study: silage maize, grass and winter triticale. Silage maize has been by far the most important biogas crop in Central Europe (Herrmann, 2013), especially Germany (73%, FNR, 2017), for quite some time now. There are several reasons for this including high biomass and methane yields, relatively simple production system, good availability of the required technical equipment and low demands for plant protection (Herrmann et al., 2017). Another aspect is the availability of a wide range of varieties for various site conditions and applications. In Germany however, the proportion of maize in biogas substrate has been limited to a maximum of currently 50% (Bundesministerium der Justiz und für Verbraucherschutz, 2017). This stems from ecological concerns, for example the fact that maize is often cultivated in large-scale monocultures (and unfortunately often also in combination with poor farming practices), leading to an anticipated increase in pests in the future as well as landscape image issues. In addition, experience has shown that maize cultivation is highly susceptible to N losses (via leaching and gaseous emissions) and soil erosion (Taube and Herrmann, 2009; Svoboda et al., 2013). This has led to a call for alternatives to maize as biogas substrate and for diversification in crop rotations (von Cossel et al., 2017). As a result, alternative and more environmentally benign biomass supply systems are currently being sought, including semi- to fully perennial cropping systems.

Permanent grassland is a fully perennial cropping system and a frequent form of land use, especially in agriculturally disadvantaged regions (Huyghe et al., 2014). Cool temperatures and/or a limited vegetation period render them less productive for maize cultivation. Those with a good water supply are very suitable for forage cropping. On such sites, grassland can achieve top yields, comparable to or sometimes even outperforming those of silage maize (Hartmann and Sticksel, 2010). The biomass from grassland (and also clover grass) can be used as animal feed. Any that is not used for feed, e.g., the second and potential following cuts, can be ensiled and digested in a biogas plant (Hartmann et al., 2011). At 12%, grass silage is the second most used biogas crop substrate in Germany (FNR, 2017).

Whole-crop cereals, notably winter triticale, are the third most frequently used biogas crop substrate (8%, FNR, 2017). Winter triticale has a high biomass yield potential and, as a winter cereal crop, can form a valuable part of the crop rotation (Sticksel, 2010). Its ability to resist unfavorable biotic and abiotic environmental factors allows good yields even at marginal sites (Martinek et al., 2008). In our study, it was harvested as whole green crop in early summer. This harvest time makes it difficult to grow a second crop in the same year (Sticksel, 2010). Thus, when grown in an intercropping system, it is most efficient to establish clover grass by undersowing in spring. In this study, the intercropping of triticale and clover grass is considered a "semi-perennial system." It has positive effects on soil erosion control and N use efficiency due to the year-round soil coverage and the integration of legumes in the crop rotation.

The objectives of this study were to determine whether separated digestates can complement or substitute mineral fertilizers and whether/how they affect the long-term yield performance in different biomass cropping systems.

The research approach was set up to test the following hypotheses:

- The influence of mineral fertilizer and separated biogas digestates on biomass yield is comparable.
- The fertilization effects are stronger in annual cropping systems (with tillage) than in perennial cropping systems.
- The fertilizing effects are influenced by site factors, particularly in the case of organic fertilizers.

These hypotheses were tested by means of a multi-factorial, long-term field experiment allowing a comparison of different fertilization treatments in three cropping systems at two sites. For this purpose, three typical biogas substrate cropping systems (maize; intercropping of winter triticale with clover grass; and grassland) were established on two locations close to a biogas plant. These were chosen to represent an annual, a semi-perennial and a perennial system, respectively. The sites are located at the base and the top of the mountainous region of the Swabian Alb in south-west Germany, both of which display agriculturally challenging conditions (soil quality/growing season, respectively). The fertilizing effects of biogas digestates on these cropping systems were tested using the separated liquid and solid digestate fractions alone and also in combination with mineral fertilizer.

MATERIALS AND METHODS

Site Description

In 2010, two multi-year field experiments were established on marginal sites belonging to the field research station of the University of Hohenheim in south-west Germany: one at the base ("Valley," 48.47° latitude, 9.27° longitude, approximately 480 m above sea level, average annual air temperature 10.0°C, average annual rainfall 779 mm) and the other at the top ("Hill," 48.47° latitude, 9.30° longitude, approximately 700 m above sea level, 7.1°C, 935 mm) of the mountainous region of the Swabian Alb; approximately 35 km south of Stuttgart.

The soil at the "Valley" site is classified as lithoidal clay rendzina with a depth of approximately 0.6 m. The soil at the "Hill" site is a silty clayey loam with a depth of over 1.0 m. The climate data relevant for the field study (2012–2017) are shown in **Figure 1**. Data for the "Valley" site are taken from the nearest weather station at Metzingen, 48.55° latitude, 9.30° longitude, 391 m above sea level.

Experimental Approach

The fertilizing performance of separated biogas digestates was assessed using three cropping systems: (1) perennial grassland; (2) intercropping of winter triticale and clover grass; (3) silage maize.

The grassland plots were established in April 2010 using a grassland seed mixture for 3–4 cuts per year (28% *Lolium perenne*, 19% *Festuca pratensis,* 19% *Phleum pretense*, 13% *Poa*

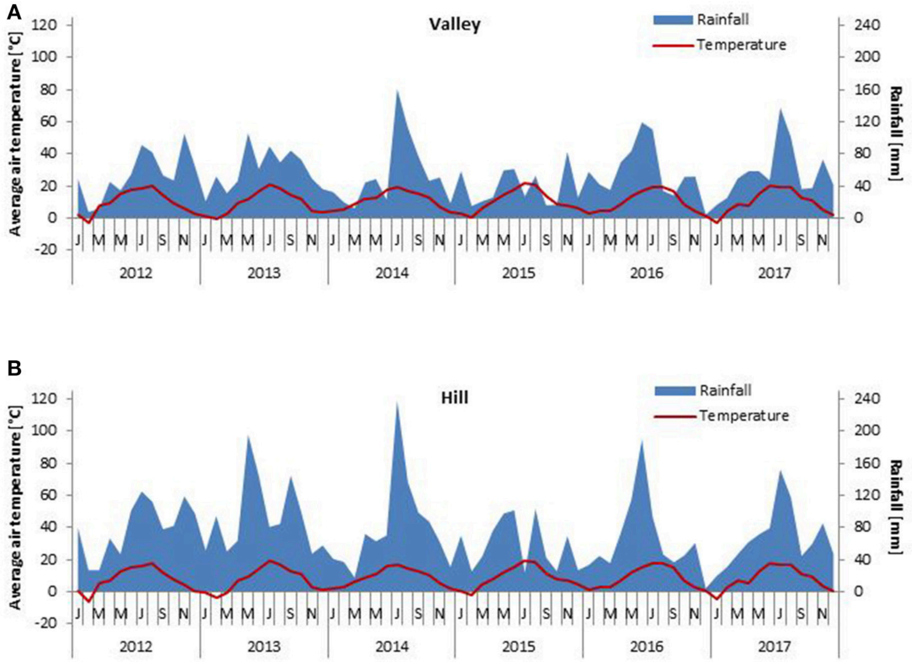

FIGURE 1 | Monthly weather data for field sites "Valley" **(A)** and "Hill" **(B)** for years 2012–2017. Average air temperature was measured 2 m above soil surface (Center for Agricultural Technology Augustenberg 2017).

pratensis, 6% *Festuca rubra,* 6% *Dactylis glomerata*, 9% *Trifolium repens*; LAZBW Aulendorf, Germany) sown at a rate of 32 kg ha^{-1}. Reseeding was carried out in August 2014 at a rate of 23 kg ha^{-1} using a mixture specifically designed for less favorable areas (32% *Lolium perenne*, 20% *Phleum pratense*, 16% *Poa pratensis,* 16% *Dactylis glomerata*, 4% *Alopecurus pratensis*, 12% *Trifolium repens*; LAZBW Aulendorf, Germany) with the aim of maintaining grass cover and counteracting increasing gaps.

The winter triticale (*x Triticosecale* var. "Tarzan") plots were generally sown in the first week of October at a rate of 300 seeds m^{-2}. Clover grass was undersown in the triticale in March/April of the years 2013, 2015 and 2017 at a rate of 30 kg ha^{-1} using a mixture consisting of 83% Italian ryegrass (*Lolium multiflorum* L. var. "Tarandus") and 17% red clover (*Trifolium pratense* L. var. "Titus"). For reasons of clarity, it is referred to as "clover grass" instead of "clover grass mixture." After the last clover grass cut, plots were cultivated with a rotary hoe (8 cm) and chisel plow (16 cm) and then prepared for sowing triticale by rotary harrow (12 cm).

The maize (*Zea mays* L.) plots were sown after seedbed preparation with a rotary harrow (12 cm) at a rate of 13 seeds m^{-2} in rows 0.75 m apart as soon as reasonable, mostly in the first half of May. Varieties were selected according to the vegetation period at each site: "Ronaldinio" (FAO 240) for "Valley" and "Amadeo" (FAO 220) for "Hill." From 2015 onwards, these were switched to newer varieties with the same FAO numbers, respectively ("Frederico" for "Valley" and "Colisee" for "Hill"). Maize seeds were provided by KWS Saat SE, Einbeck, Germany. Soil tillage included stubble cultivation with a chisel plow (16–18 cm) immediately after harvest and plowing (20 cm) later on.

The three crops were fertilized with separated biogas digestates in four different variants (**Table 1**). The digestates were obtained from a 355 $kilowatt_{electric}$ biogas plant at the research station, fed mainly with animal manure and maize silage. Solid/liquid separation was performed with a screw press separator. A mineral fertilizer and an untreated control were included for comparison. All treatments except the control were applied at 150 kg N ha^{-1}; amounts and timing are summarized in **Table 2**. Residual plant-available nitrogen (N_{min}), phosphorus, potassium, calcium, magnesium and the pH in the soil were measured every spring and fall to be used for subsequent research analysis (for methods, see Ehmann et al., 2017). Results from the initial soil sampling (0–30 cm) are summarized in **Table 3**.

Before each application, the NH_4^+ content of the digestates was determined to take account of slight variations over time. Each time, two subsamples were taken; one was analyzed directly using a Quantofix N volumeter (Van Kessel and Reeves, 2000), the other was stored at −18°C and analyzed later in the lab (DIN 38406-E5-2) to validate the first measurement.

Table 4 shows the average NH_4^+ concentrations of the digestates (values for 2012–2017), together with concentrations of other nutrients and pH (values for 2013–2015).

Applications were split into 2-3 portions to suit the crops' requirements as optimally as possible (**Table 2**). In grassland and clover grass, the initial portion was usually applied in spring and the subsequent portions after cutting. Where possible, the digestates were incorporated immediately after

TABLE 1 | Description of fertilization and control treatments.

Variant	Treatment	Mode of application
Control	Unfertilized control	–
Mineral	CAN	Fertilizer spreader
Solid+	Separated solid digestate fraction + CAN (2:1)	Digestate applied manually; CAN with fertilizer spreader
Solid	Separated solid digestate fraction	Manually
Liquid+	Separated liquid digestate fraction + CAN (2:1)	Digestate with slurry trailer; CAN with fertilizer spreader
Liquid	Separated liquid digestate fraction	Slurry trailer

CAN, calcium ammonium nitrate.

application using a harrow (10 cm) to minimize N losses. In the combined treatments, digestates and mineral fertilizer were applied approximately 1 week apart from each other.

The experiments were established in a split-plot design with four replications, resulting in 72 plots (32 m²) at each site. Main plots were the cropping systems and subplots the fertilization treatments. Treatments were randomized for each site separately.

Herbicides and fungicides were only applied when necessary and then according to good agricultural practice.

The grassland plots were cut three (in 2016 two) times per year according to good agricultural practice. The last sparse growth of each year was cut and removed from the plots, but not included in the yield.

For the intercropping plots, the harvesting regime was as follows: in the years 2012, 2014, and 2016, the clover grass was harvested three to four times; in 2013, 2015, and 2017 the winter triticale was harvested wholecrop around the early dough stage (BBCH 83) and the undersown clover grass in September or October.

The maize was harvested wholecrop with a plot-size field chopper around the stage of silage ripeness (BBCH 85) and a dry matter content of 30–35% TS when weather conditions were suitable.

Samples were taken from each cut and the dry matter biomass yield (DMY) determined by drying at 60°C to constant weight.

Statistical Analysis

A mixed model was developed for all traits using the following equation (Piepho et al., 2004):

$$L + C + F + L \bullet C + L \bullet F + C \bullet F + L \bullet C \bullet F : Y + Y \bullet L + Y \bullet L \bullet R + Y \bullet C + Y \bullet F + Y \bullet L \bullet C + Y \bullet L \bullet F + Y \bullet C \bullet F + Y \bullet L \bullet C \bullet F + R \bullet Y \bullet L + C \bullet R \bullet Y \bullet L + \underline{C \bullet F \bullet R \bullet Y \bullet L},$$

where C and F denote effects of the treatments "cropping system" and "fertilization," R, L, and Y denote effects of "replicate," "site," and "year," respectively. Interactions between the treatments "site" and "year" are denoted by a dot between the corresponding main effects. "$R \bullet Y \bullet L + C \bullet R \bullet Y \bullet L + \underline{C \bullet F \bullet R \bullet Y \bullet L}$" denotes replicate effects and effects of main and subplot error in each combination of site and year. Effects from different years are repeated measurements, therefore a first-order autocorrelation was fitted to them. Crop-by-fertilizer-specific variances were assumed but only fitted to sub-plot errors to avoid convergence problems. Fixed effects are given before the colon. To achieve homogeneous residual variances and normality of residuals, data were log-transformed. Both pre-requirements were checked graphically. Where an F-test revealed significant effects, a multiple t-test ($\alpha = 0.05$) was performed. To create the letter display, the %mult macro (Piepho, 2012) was used.

Furthermore, cumulated system-by-site-by-fertilizer treatment estimates across years and their standard errors were calculated as a sum of single-year BLUPs (best linear unbiased prediction), or its standard errors, for each combination of system, site and fertilizer treatment. A single-year BLUP here refers to the sum of the least square estimate for one system-by-site-by-fertilizer treatment mean and the corresponding random year main effect and its interaction effects. Yield data was logarithmically transformed, therefore presented values are interpreted as medians. Thus, cumulated yield estimates were also made from the given model.

The data analysis was carried out with SAS software version 9.3 (SAS Institute Inc., Cary, NC, USA).

RESULTS

Cumulative yields from the 6 years are presented here to compare the long-term yield performance of the cropping systems. **Table 5** shows the results of the statistical analysis of the main effects; these all had a significant influence except the interaction "site*system."

Perennial Grassland

At the "Valley" site, the highest DMY was obtained with mineral fertilizer, followed by the two combination treatments. The liquid digestates only and solid digestates only treatments were not significantly different from the combination treatments or the control (**Figure 2**). At the "Hill" site, the treatments appeared to be more efficient than at the "Valley" site. This is visible from the difference in DMY between control and treatments, both in cumulative as well as annual DMY. All treatments led to a higher DMY than in the control. The highest DMY was obtained with the two treatments containing solids, which were both significantly better than "liquid" only (**Figure 2**). As to be expected, the first of the usual three cuts made up the largest share of the annual yield.

It was noticeable that in 2017 the DMY was considerably lower at the "Hill" than at the "Valley" site. Here, the effect of decreasing DMY over the years becomes especially visible in the control plots (from 114 dt ha^{-1} in 2012 down to 49 dt ha^{-1} in 2017), whereas the "solid+" (average 89 dt ha^{-1}) and "solid" (average 88 dt ha^{-1}) plots were most stable. There were no particular fluctuations visible between the years. At the "Valley" site, the "mineral" plots showed the highest tendency toward decreasing yields.

TABLE 2 | Amounts and timing of fertilization treatments.

Variant	Dose	Grassland		Intercropping				Silage maize	
				Triticale		Clover grass			
		kg N	Time	kg N	Time	kg N	Time	kg N	Time
Mineral	1	80 as CAN	start vegetation period	80 as CAN	start vegetation period	80 as CAN	start vegetation period	90 as CAN	before sowing
	2	40 as CAN	after 1st cut	70 as CAN	start stem elongation	70 as CAN	after 1st cut	60 as CAN	4-leaf-stage
	3	30 as CAN	after 2nd cut	–	–	–	–	–	–
Solid+	1	50 as solids + 50 as CAN	start vegetation period	50 as solids + 50 as CAN	start vegetation period	50 as solids + 50 as CAN	start vegetation period	70 as solids + 25 as CAN	before sowing
	2	50 as solids	after 1st cut	50 as solids	start stem elongation	50 as solids	after 1st cut	30 as solids + 25 as CAN	4-leaf-stage
	3	–	–	–	–	–	–	–	–
Solid	1	70 as solids	start vegetation period	70 as solids	start vegetation period	70 as solids	start vegetation period	90 as solids	before sowing
	2	45 as solids	after 1st cut	45 as solids	start stem elongation	45 as solids	after 1st cut	60 as solids	4-leaf-stage
	3	35 as solids	after 2nd cut	35 as solids	end stem elongation	35 as solids	after 2nd cut	–	–
Liquid+	1	40 as liquids + 50 as CAN	start vegetation period	40 as liquids + 50 as CAN	start vegetation period	40 as liquids + 50 as CAN	start vegetation period	60 as liquids	before sowing
	2	30 as liquids	after 1st cut	30 as liquids	start stem elongation	30 as liquids	after 1st cut	50 as CAN + 40 as liquids	4-leaf-stage
	3	30 as liquids	after 2nd cut	30 as liquids	end stem elongation	30 as liquids	after 2nd cut	–	–
Liquid	1	70 as liquids	start vegetation period	70 as liquids	start vegetation period	70 as liquids	start vegetation period	80 as liquids	before sowing
	2	50 as liquids	after 1st cut	50 as liquids	start stem elongation	50 as liquids	after 1st cut	70 as liquids	4-leaf-stage
	3	30 as liquids	after 2nd cut	30 as liquids	end stem elongation	30 as liquids	after 2nd cut	–	–

CAN, calcium ammonium nitrate.

This general tendency toward declining yields over the years was observed at both sites. It was most likely due to gaps in the grass cover as a consequence of aging plots on the one hand and an infestation with field mice on the other. These gaps increased in frequency and size over time. Although reseeding was performed in August 2014, the plots did not recover satisfactorily as it was too dry during the following weeks. It was observed that the higher-value grass species in the initial seed mix (e.g., including perennial ryegrass *Lolium perenne* L., meadow fescue *Festuca pratensis* L., and timothy *Phleum pratense* L.) disappeared over time and were replaced by species of inferior quality. At the "Valley" site, this was predominantly rough bluegrass (*Poa trivialis* L.). In addition, the occurrence of broad-leaved dock (*Rumex obtusifolius*) reduced the quality of botanical composition at this site. Even the frequent cutting did not displace this persistent weed. The plots at the "Valley" site were also invaded by moss. At the "Hill" site, the initially established perennial ryegrass (*Lolium perenne* L.) was mainly replaced by cocksfoot (*Dactylis glomerata* L.). In general, the patchiness of the grass cover was less pronounced at the "Hill" than at the "Valley" site.

The dry matter content (DMC) of the grass samples was homogeneous and relatively low. At the "Valley" site, the average DMC of all samples was 20% (cut 1) and 23% (cuts 2–3); at the "Hill" site, 21 and 24%, respectively.

Intercropping of Winter Triticale and Clover Grass

The DMY of the intercropping system was fairly homogeneous at both sites. This was particularly the case at the "Valley" site, where all treatments performed equally well and, with the exception of "solid," resulted in significantly higher DMY than the control (**Figure 3**).

At the "Hill" site, all treatments increased the yield compared to the control. The highest DMY was obtained with the "liquid+" treatment. This was significantly higher than with the "solid" treatment (**Figure 3**).

TABLE 3 | Initial soil characteristics (0–30 cm) at the two sites in September 2010.

Site		$P_2O_5^*$	K_2O^*	Mg*	TOC	pH
		mg (100 g soil)$^{-1}$			% DM	
Valley	Mean	44.96 (±15.79)	55.95 (±24.55)	14.30 (±1.86)	4.62 (±0.96)	7.22 (±0.06)
	n	24	24	23	12	5
Hill	Mean	15.42 (±1.84)	19.37 (±1.86)	11.88 (±0.86)	2.68 (±0.27)	5.46 (±0.11)
	n	24	24	24	12	8

Values in brackets indicate standard deviation.
DM, dry matter.
**plant-available concentrations; analyzed with CAL extraction followed by flame photometer (P_2O_5), FIA measurement (K_2O) according to OENORM L 1087:2012-12-01 and $CaCl_2$ extraction followed by AAS measurement (Mg) according to VDLUFA I A 6.2.4.1; soil pH was determined using a glass electrode after $CaCl_2$ extraction (DIN ISO 10390:2005); TOC (total organic carbon) analyzed according to DIN EN 15936:2012-11.*

TABLE 4 | Characteristics of digestates and mineral fertilizer.

		Total solids	C_t	N_t	NH_4^+-N	NO_3^--N	P	K	Ca	pH
		% FM	% DM	% FM	% FM	% FM	% FM	% FM	% FM	–
Solid digestate fraction	Mean	23.58	42.31	0.58	0.26	<0.001	0.22	0.46	0.47	8.51
	STD	4.49	1.36	0.08	0.06	–	0.05	0.10	0.12	0.15
	n	14	14	18	14	14	14	14	14	14
Liquid digestate fraction	Mean	5.06	34.94	0.38	0.24	<0.001	0.07	0.41	0.16	7.79
	STD	1.14	1.18	0.07	0.04	–	0.02	0.07	0.03	0.08
	n	11	11	11	31	11	11	11	11	11
Calcium ammonium nitrate (CAN)		–	–	27.00	13.50	13.50	–	–	10.00	–

FM: fresh matter; DM: dry matter; STD: standard deviation.

TABLE 5 | Results of the statistical analysis of main effects.

Effect	Number of DF	F statistic	*p*-value
site	1	4.59	0.0324
system	2	4.14	0.0162
treatment	5	29.56	<0.0001
system*treatment	10	6.42	<0.0001
site*system	2	0.51	0.6026
site*treatment	5	25.41	<0.0001
site*system*treatment	10	8.74	<0.0001

DF, Degree of freedom; level of significance was $p \leq 0.05$.

The highest yields were obtained at both sites in 2012 and 2013. After this, the yields decreased, but remained at a more or less constant level (120 dt ha^{-1} at the "Valley" site, 112 dt ha^{-1} at the "Hill" site). As expected, the yield difference between control and treatments was larger for triticale than for clover grass, indicating a more prominent fertilizing effect.

In 2015, the triticale DMY was reduced at the "Hill" site due to infestation with yellow rust (*Puccinia striiformis*).

Figure 4 shows that the majority of the triticale plots were harvested too late and the DMC was higher than the optimal value, particularly at the "Valley" site. The DMC varied considerably more at the "Valley" than at the "Hill" site. In 2017, the average DMC at the "Valley" site was 63% with individual maximal values of more than 80%, whereas in the other years values were more within the normal range (46% in 2013, 36% in 2015).

Silage Maize

At both sites, the highest maize DMY was obtained with mineral fertilizer; however, this was only significant at the "Hill" site (**Figure 5**).

At the "Valley" site, both combination treatments performed as well as the mineral fertilizer.

At the "Hill" site, all treatments with digestates except "solid" were comparable to each other and resulted in the second highest DMY after mineral fertilizer.

In general, the DMY standard deviations were higher and fluctuated more at the "Valley" than at the "Hill" site. This is likely due to the relatively high heterogeneity of the field conditions. In addition, problems with regrowth from the preceding crop Jerusalem artichoke (*Helianthus tuberosus*) led to massive yield reductions in certain plots, in one replication in particular, despite frequent manual weeding and the occasional herbicide application. It is interesting that the lowest standard deviation at the "Valley" site was found with mineral fertilizer indicating a reliable fertilizing effect, independent of external influences.

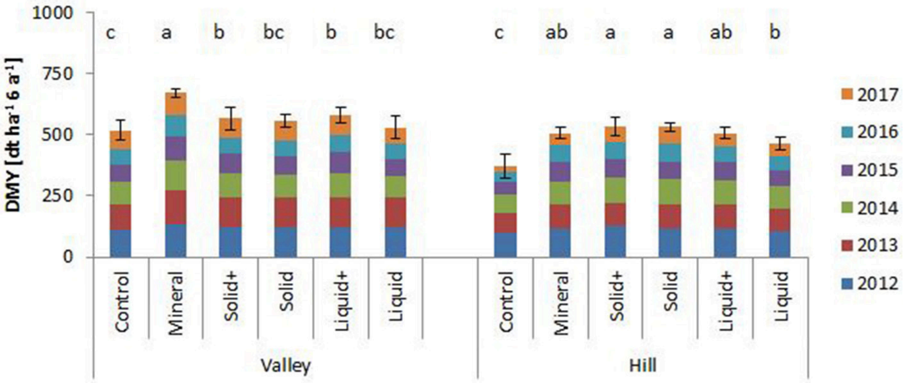

FIGURE 2 | Mean dry matter yield (DMY) of perennial grassland at the sites "Valley" and "Hill" from 2012–2017 fertilized with different treatments of separated biogas digestates and mineral fertilizer in comparison to unfertilized control. Error bars indicate standard deviation of accumulated yields. Means with identical letters are not significantly different from each other ($n = 4$, $p \leq 0.05$). For explanation of treatments, see **Table 1**.

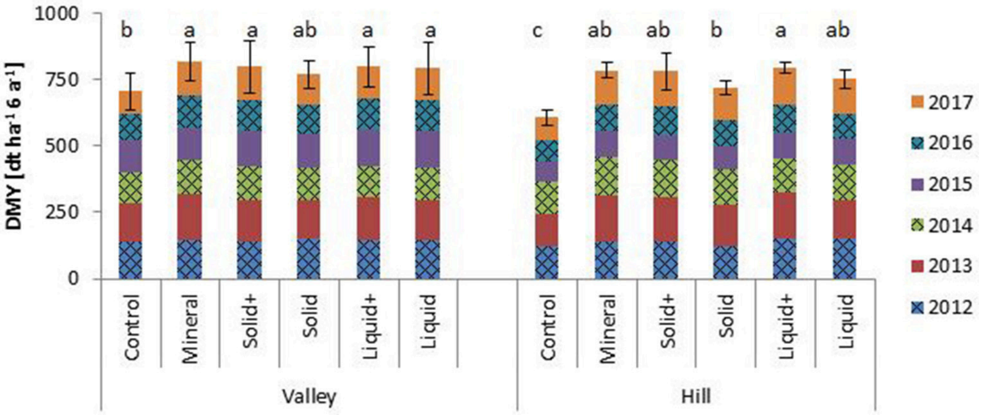

FIGURE 3 | Mean dry matter yield (DMY) of an intercropping system with winter triticale and undersown clover grass grown at the sites "Valley" and "Hill" from 2012–2017 fertilized with different treatments of separated biogas digestates and mineral fertilizer in comparison to unfertilized control. Checked parts of the columns indicate years in which only clover grass was harvested (2012, 2014, 2016). Error bars indicate standard deviation of accumulated yields. Means with identical letters are not significantly different from each other ($n = 4$, $p \leq 0.05$). For explanation of treatment, see **Table 1**.

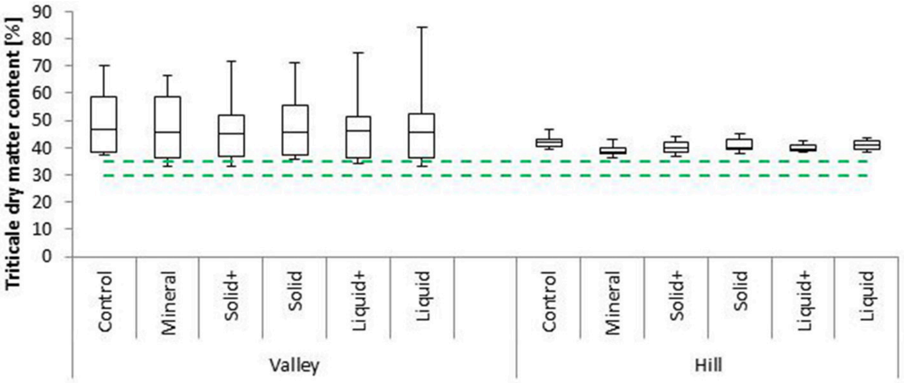

FIGURE 4 | Dry matter contents of triticale grown at the sites "Valley" and "Hill" in 2013, 2015, and 2017 fertilized with different treatments of separated biogas digestates and mineral fertilizer in comparison to unfertilized control ($n = 4$). The green lines indicate the optimal dry matter content range. For explanation of treatments, see **Table 1**.

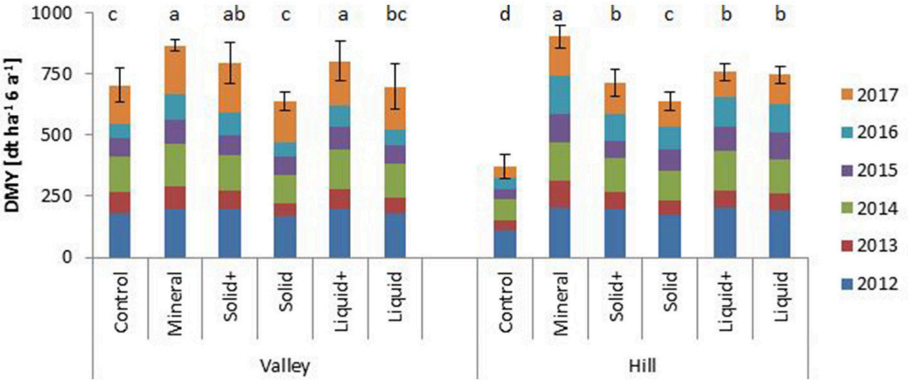

FIGURE 5 | Mean dry matter yield (DMY) of silage maize grown at the sites "Valley" and "Hill" from 2012–2017 fertilized with different treatments of separated biogas digestates and mineral fertilizer in comparison to unfertilized control. Error bars indicate standard deviation of accumulated yields. Means with identical letters are not significantly different from each other ($n = 4$, $p \leq 0.05$). For explanation of treatments, see **Table 1**.

The effect of year was clearly recognizable. 2013 and 2015 were not good years for maize cultivation: wet and cold conditions in spring delayed sowing and/or germination; drought periods with high temperatures in summer months negatively influenced growth (see also **Figure 1** for weather data). In those years, the mineral fertilizer showed the best performance of all treatments. In years with weather conditions favorable for maize cultivation (2014, 2016, 2017) most digestate treatments worked equally well as mineral fertilizer.

The year 2016 was exceptional in that the spring was cold and wet, but there was a short favorable time slot which could be used for sowing. This was followed by a lot of rain in early summer before a dry, hot period set in. At the "Valley" site, this resulted in low DMY (on average 83 dt ha^{-1} for fertilized plots), but a very satisfactory yield at the "Hill" site (118 dt ha^{-1}). Here, the higher altitude and thus lower average temperature, together with the deep soil, were an advantage. Consequently, the water supply lasted longer during the heatwave, ensuring better growth than at the "Valley" site.

The separated solid digestate variant led to the lowest yields of all treatments at both sites (**Figure 5**). This was visible in most years, but also for the accumulated yields. At the "Valley" site, it resulted in yields comparable to the control and to the "liquid" treatment (or even lower in absolute values). At the "Hill" site, it had a DMY higher than the control, but lower than the other treatments.

Figure 6 shows that the majority of the maize plots were harvested with a dry matter content (DMC) within the optimal range of 30 to 35% TS. In general, DMC was lower and fluctuated less at the "Hill" site.

DISCUSSION

Significant differences in yield performance were found between the annual, intercropping and perennial cropping systems subjected to the treatment variants. Interactions with site and year effects were also observed.

The highest and most stable biomass yields were found in the intercropping system with triticale and clover grass, irrespective of the site, treatment and year. This was followed by perennial grassland, which also proved to be relatively stable with regard to treatment and year, but provided lower DMY. Maize only produced high yields in years with favorable climatic conditions. Particularly in years with unfavorable conditions, the best maize yields were achieved with mineral fertilizer, whereas in normal years the DMY difference between treatments was small. Thus the influence of the year effect also varied between cropping systems.

In general, the "Valley" site had higher DMY, but the "Hill" site provided better conditions for growth during hot, dry periods due to the lower average temperature and longer water supply. We also observed that the treatments were more effective in terms of yield at the "Hill" site, as the DMY was significantly higher than the control on all fertilized plots in all three systems here.

Effect of Fertilizer Treatments on Yield Performance of the Three Cropping Systems

The annual system responded most sensitively to influences of treatments, site and year. The yields were significantly influenced by all these factors. As a C4 crop, maize reacts relatively strongly to temperature fluctuations and requires favorable temperatures and sufficient water supply for germination and good establishment, especially if sown in late spring (Maton et al., 2007). In this study, the highest maize yields were achieved with mineral fertilizers, particularly in cooler years and at the cooler "Hill" site. This can be explained by the fact that mineral fertilizer application can be timed to provide plant-available N to coincide with the crop's requirements (Möller, 2009). The N availability of mineral fertilizer is also less dependent on climatic conditions, especially temperature and water supply, than organic fertilizer (Agehara and Warncke, 2005) and the share of mineral N is of course higher than in the digestates (**Table 4**). After sowing, maize first needs to build its root system and is highly dependent on rapidly plant-available N at exactly

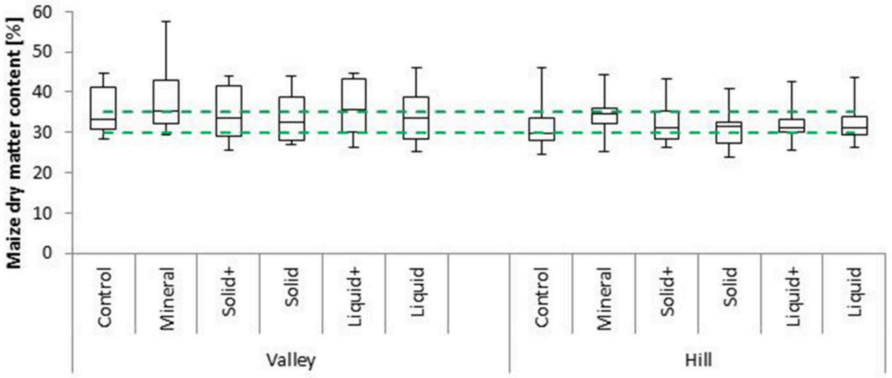

FIGURE 6 | Dry matter contents of silage maize grown at the sites "Valley" and "Hill" from 2012–2017 fertilized with different treatments of separated biogas digestates and mineral fertilizer in comparison to unfertilized control ($n = 4$). The green lines indicate the optimal dry matter content range. For explanation of treatments, see **Table 1**.

the right time (Plénet and Lemaire, 1999). This can best be steered by the application of easily soluble mineral fertilizer.

The effect of more rapid N availability from mineral than from organic fertilizers was particularly evident in the years 2013 and 2015 when temperatures were lower than the long-term average and 2015 also had less than average rainfall during the growing season. In these years, the mineral fertilizer had significantly better results, especially at the "Hill" site, and the solids had a lower performance. In 2013 at the "Valley" site, the solids even resulted in a lower DMY than the unfertilized control. This may have been caused by initial N immobilization, which often occurs after the application of organic matter (Gutser et al., 2005).

Mineral fertilizer had the best effect on maize yield, in terms of both amount and stability, over the years. This was the case for application of mineral fertilizer alone as well as in combination with digestates. The crop's short-term demand for plant-available N was met through the mineral fertilizer and later—once the maize had established—N from the digestate had been mineralized and could provide the maize with a sufficient supply. In addition, the combinations provided at least a certain amount of organic matter (OM). This may be valuable as maize leaves a limited amount of crop residues and its cultivation tends to reduce soil organic matter and humus (Karpenstein-Machan, 2013; Komainda et al., 2018). Several studies have suggested that the combination of organic and mineral fertilizers can improve the regulation of N supply and enhance the effect of the two fertilizer types. As such, it is the most effective way of achieving both high yields and at the same time a build-up of soil organic matter (SOM) (Rauhe, 1987; Körschens et al., 1998; Svensson et al., 2004; Gutser et al., 2005; Möller, 2009). However, as simultaneous application can temporarily immobilize mineral N and increase the risk of N_2O emissions, it is recommended that digestates and mineral fertilizer are applied with a time delay (Möller et al., 2009). We followed this recommendation in our field experiments.

In addition, the effect of combined mineral and organic fertilizers versus the application of mineral fertilization alone depends on site conditions. At the cooler "Hill" site, where the soil only warms up slowly in spring, the mineral treatment worked significantly better than the combinations. By contrast, at the "Valley" site, the mineral fertilizer and the combinations had comparable effects.

As expected, and observed at both sites, the yield effects of the different fertilizer types were less pronounced in permanent grassland than in the annual cropping system. As grassland is characterized by year-round soil cover, it can better exploit the long-term fertilizing effects of the organic treatments than the other two systems. These long-term effects result from the more continuous N release as well as better water retention and other factors improving soil fertility. However, grassland proved to be the system with the lowest total yields over 6 years. In addition, the aging effect of the plots in this system needs to be considered. For this reason, it is difficult to assess the effects of fertilizer treatment and system separately, as the system itself degrades over time (increasing gaps, reduction/loss of valuable grass species) and yields subsequently decrease. Therefore, the aging effect on yields may mask the fertilizer effects.

For permanent grasslands, there were also clear site effects. At the warmer "Valley" site, mineral fertilizer resulted in the significantly highest DMY. In contrast, at the "Hill" site, both treatments with solids led to the highest DMY during the experimental period. This was somewhat unexpected as the solids treatments had lower yields in the annual and intercropping systems at both sites. At the beginning of the experiment, we had assumed that organic fertilizers would be less effective the more marginal the site conditions are. As N mineralization and OM turnover are influenced by temperature (Davidson and Janssens, 2006), it was surprising to find the good performance of the treatments with solids at the site with lower average temperature and limited vegetation period. This result was undoubtedly a consequence of an interaction between system, treatment and site, but cannot be sufficiently explained by the data collected in this study. Repeated application of solid digestates could have increased the soil pH at this site, which was relatively low at the beginning of the experiment (5.5). However, an intermediate soil analysis in fall 2014 showed that the pH had decreased to 5.3 on average on all grassland plots. The smallest decrease was found on plots treated with solids (5.4). Nabel et al. (2017) found that the comparative advantage of digestate fertilization over mineral NPK fertilization on biomass yield became increasingly pronounced over time and explained this through the crucial role of soil carbon content for plant growth. This obviously applies more to perennial systems where the soil is not disturbed and becomes more important the more marginal the soil is. This may serve as an explanation for the surprising performance of the solids at the "Hill" site. However, our hypothesis is that the proportion of nutrient supply provided by OM turnover increases with time and thus renders the grassland system increasingly independent of the direct nutrient effect of the fertilizers.

The intercropping system (here two crops grown in rotation) proved to be a stable and robust system that provided constantly high yields. In this system, the soil was almost always covered (except during early development stages of triticale). Unlike maize, generally all fertilizer treatments worked equally well independent of the site or crop. The yields in the intercropping system appeared to profit from the crop rotation effect, mainly from the biological fixation of atmospheric N_2 by the clover in the mixture (not quantified). This is intended to ensure a more constant N supply independent of fertilizer applications, for example during periods of low N availability due to insufficient amounts of mineralized N. The leguminous component of this system differentiates it from the others. Grassland also contains some clover, but in the intercropping system clover is sown afresh every other year resulting in a higher proportion of legumes in the sward and consequently a higher N fixation rate.

The clover grass and triticale both developed intensive root systems; thus the intercropping system produced a considerable amount of crop residues which additionally contributed to the build-up of SOM and the residual supply of mineralized N (Fouda et al., 2013).

In this study, we focused on the effects of the treatments on biomass yield of the cropping systems and mainly limited the explanation of different fertilizer effects to differences in the timeliness of N availability and the capacity of the various fertilizer types to contribute to SOM production. Another aspect that was considered in explaining differences in yield effects of the various fertilizer types was their interaction with the three cropping systems tested here. All cropping systems have their growth peaks at different times, which clearly affects the nutrient demand and uptake during the vegetation period (Herrmann et al., 2017).

Implications of Different Fertilization Systems

When assessing the suitability of biogas digestates as fertilizers, other aspects in addition to the yield effect need to be considered. Clearly, a farmer who produces biogas needs to dispose of the digestates. In practice, biogas digestates are often separated and used as fertilizer on the farm. However, when other feedstock streams, such as slurry, are co-digested in the biogas plant, the nutrients in the digestates constitute an oversupply at farm level. Therefore, digestates are often transported to other farms. Alternatively, they can be further processed to bio-based mineral fertilizers (Vaneeckhaute et al., 2017). For example, nutrients can be recovered from the liquid fraction by precipitation and filtered off as a mixture of phosphate salts, including struvite (Bilbao et al., 2017; Ehmann et al., 2017). Since this process is costly, the extent to which digestates are directly applied as organic fertilizer or, especially in the case of the liquid fraction, are processed into mineral fertilizer should be carefully considered on a case-by-case basis.

Mineral fertilizer use is always accompanied by the highest costs and environmental impacts, irrespective of whether it is produced chemically (N), from mining (P) or through recovery from biogas digestates (N and P). From a farming practice point of view, mineral fertilizers have the advantage of more predictable N supply on the one hand and easier applicability on the other. The latter is particularly relevant for permanent cropping systems. One major environmental benefit of digestates is that they can help save on mineral N fertilizer, either by complete or partial substitution. In good agricultural practice, gaseous emissions during and after digestate application are kept to a minimum, which was not ensured with the liquid manure spreader used in this study. Application techniques near the soil surface including trailing hoses, trailing shoes and injection would of course reduce gaseous losses (especially in systems and at stages where incorporation is not possible) and at the same time increase the plant-usable N (Möller et al., 2008). The solid fraction should ideally be incorporated into the soil to avoid gaseous N losses (Holly et al., 2017), allow for nutrient release through decomposition and avoid a layer of organic matter remaining on the crop. The application of solids is even more laborious in systems which require multiple cuts over the vegetation period. Although our results showed that solids significantly increased grassland yields, at least at the less favorable "Hill" site, the practicability of solid application remains limited. For this reason, only the liquid fraction is recommended for grassland due to its good infiltration, and also its high N and K but low P contents which correspond well with the nutrient removal by the crops (Messner, 2014).

The application of solid digestates thus appears more appropriate in cropping systems with frequent soil cultivation and on sites where a benefit from OM can be expected. Soil tillage increases the turnover of OM from digestates and crop residues (Blair et al., 2006; Sarker et al., 2018). Although solids were actually not recommendable for maize in terms of their fertilizing effect, their regular application is considered beneficial here for OM replacement (Nkoa, 2014). A study by Nabel et al. (2017) showed that organic fertilization with digestates had a positive influence on soil properties (e.g. increased soil respiration and enhanced water-holding capacity), particularly on marginal sites. The supply of nutrients other than N, including P, K and various microelements, is a further advantage over mineral fertilizer (Risberg, 2015).

In this study, we divided the fertilizer and digestate applications into several doses. In farming practice, this effort may be lowered by reducing the number of fertilizer doses. In grassland, the majority of the N dose would be applied in late winter or early spring, followed by only one more dose later on (Möller et al., 2009). In maize for example, the solids could be applied in one dose before sowing. This may even be possible for the liquids, primarily in the combinations. Lavandier et al. (2011) fertilized silage maize with up to 170 kg N ha^{-1}, applied in form of liquid digestate in one dose and found that this did not lead to increased N_{min} values.

In this study, grassland proved to be the system with lowest yields and highest workload. Nevertheless, permanent grassland is considered the most environmentally friendly way of producing energy crops (Rippel, 2008) and provides a suitable opportunity to maintain ecologically valuable grasslands that are no longer used for fodder production. This is particularly the case when mineral fertilizer is replaced with digestates, because the grassland productivity can be maintained with lower environmental impact (Walsh et al., 2012).

CONCLUSION

The first hypothesis underlying this study, that the influence of mineral fertilizer and separated biogas digestates on biomass yield is comparable, was confirmed. However, the recommendations that can be deduced from this vary depending on cropping system and site. All three systems tested revealed their own specific strengths and weaknesses; the same applies to the treatments. For perennial or intercropping systems, separated digestates can be fully recommended. In the intercropping of triticale and clover grass, separated digestates were able to substitute mineral fertilizer completely. Contrary to our expectations, the solids performed very well in terms of yield in interaction with grassland at the "Hill" site. However, it was seen that the use of solids in permanent grassland does not exploit their full potential. A higher benefit from solids is expected from application in annual systems where they can contribute positively to the build-up of OM. Any short-term N demand of crops is better met by a combination of digestates (liquid preferable to solid, due to high content of plant-available ammonia-N) and mineral fertilizer. The combinations performed equally well as mineral fertilizer alone in most of the systems, sites and years and allowed mineral fertilizer input to be reduced by 66%.

The second hypothesis, that fertilization effects are stronger in annual cropping systems (with tillage) than in perennial cropping systems, could be partly confirmed. If the objective is to maximize yield performance, the preferred option is the use of mineral fertilizer alone or in combination with digestates. Since the application of solid digestates and their incorporation into soil is most difficult in perennial systems, the best balance between the goals of high biomass yield and maintenance/increase of SOM content on the one hand, and the practicability of applying solid digestates on the other, can be achieved in the intercropping system.

The third hypothesis, that fertilizing effects are influenced by site factors, particularly in the case of organic fertilizers, could also be confirmed. The effect of organic fertilizer was found to be unpredictable, especially on cooler sites. To avoid yield fluctuations and N losses on such sites, perennial systems are recommended, as they capture N released at different times in the vegetation period. For these sites, the positive effect of solid biogas digestates on soil fertility and SOM can help improve the long-term stability of biomass production.

In summary, the combined application of organic and mineral fertilizer is the best approach to implement the multiple aims in terms of high yields, low-cost farming and minimal negative environmental impacts. The good performance of the combinations, together with reduced expenses for mineral fertilizer, can help improve farmers' acceptance of organic fertilizers.

AUTHOR CONTRIBUTIONS

AE: carried out the experiments, was involved in sample analysis and analyzed the data. UT and IL: were responsible for the original idea for the research and initial concept of the experiment. All authors contributed to the preparation of the manuscript.

ACKNOWLEDGMENTS

AE was supported by a scholarship from the Faculty of Agricultural Sciences, University of Hohenheim. The authors are grateful to Peter Weckherlin for the excellent maintenance and management of the field experiments and to Sven Schabel and Anja Neuberdt for their assistance with fieldwork. Particular thanks go to Nicole Gaudet for proofreading the manuscript and to Jens Möhring for statistical advice. Weather data were kindly provided by the Center for Agricultural Technology Augustenberg, Karlsruhe, Germany; maize seeds by KWS Saat SE, Einbeck, Germany; and Triticale seeds 'Tarzan' by Pflanzenzucht Oberlimpurg, Schwäbisch Hall, Germany.

Selected yield data from the years 2012 and 2013 were presented at the International Conference Progress in Biogas III, 10 to 11 September 2014, Stuttgart, Germany.

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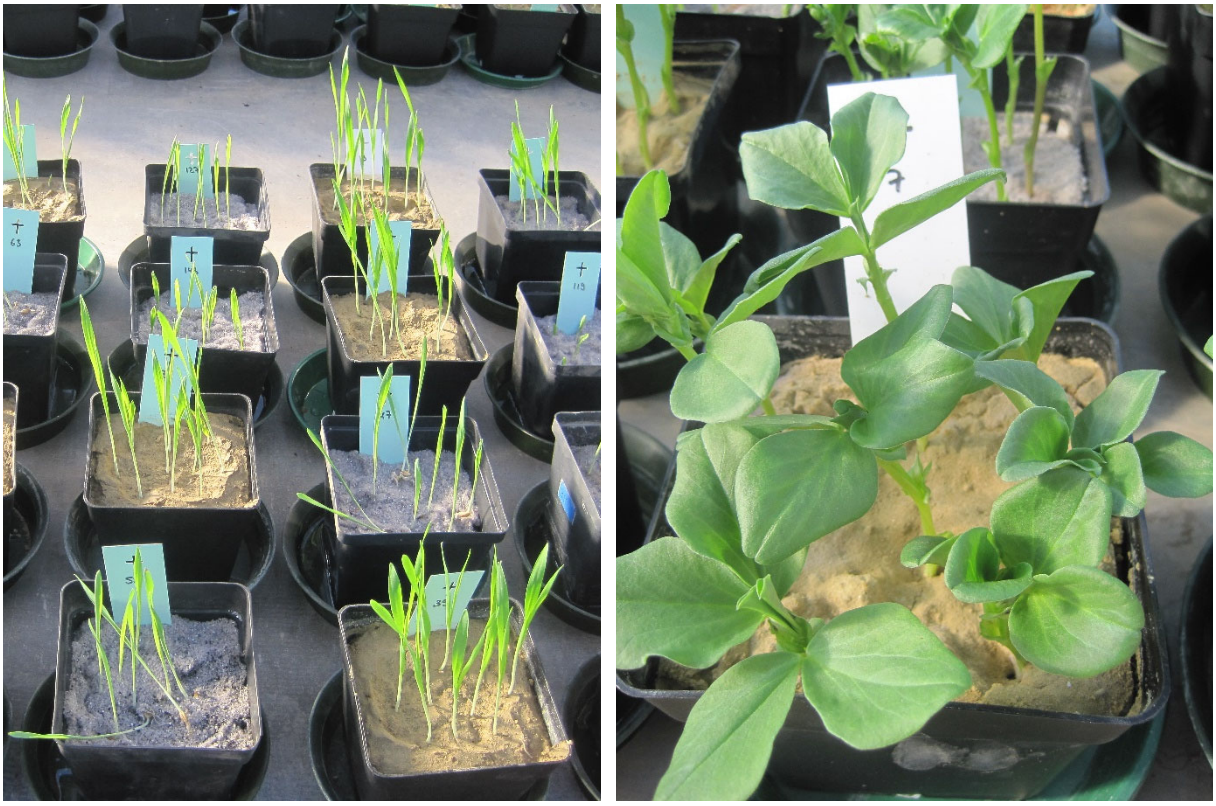
Conflict of Interest Statement: The authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

3 Effect of manure-based phosphate salt on biomass yield of spring barley and faba bean in comparison to conventional fertiliser

The study described in this chapter compared the fertilising performance of manure-based P-Salt and commercial triple superphosphate and determined whether additional biochar application further increased biomass yields. The fertilisers and the biochar were tested in pot experiments with two crop types (spring barley and faba bean) and two nutrient-poor soils (clay and sand) in a greenhouse. P-Salt was applied at three levels and biochar in two concentrations. This experimental setup allowed exploring the specific objectives (3) and (4) of this thesis that deal with the comparison of recycled and commercial phosphate fertilisers and seek to examine potential interactions between P-Salt and biochar. The study also assessed potential differences in the nutrient uptake efficiency from recycled and mineral fertilisers between different crop types which corresponds to specific objective (5).

This chapter is published in the journal *Agriculture*, as:

Ehmann, A., Bach, I.-M., Laopeamthong, S., Bilbao, J. and Lewandowski, I. (2017) Can phosphate salts recovered from manure replace conventional phosphate fertilizer? *Agriculture* 7:1. doi: 10.3390/agriculture7010001



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Article

Can Phosphate Salts Recovered from Manure Replace Conventional Phosphate Fertilizer?

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Academic Editor: Guodong Liu
Received: 16 November 2016; Accepted: 16 December 2016; Published: 7 January 2017

Abstract: Pig farming produces more manure than can reasonably be spread onto surrounding fields, particularly in regions with high livestock densities and limited land availability. Nutrient recycling offers an attractive solution for dealing with manure excesses and is one main objective of the European commission-funded project "BioEcoSIM". Phosphate salts ("P-Salt") were recovered from the separated liquid manure fraction. The solid fraction was dried and carbonized to biochar. This study compared the fertilizing performance of P-Salt and conventional phosphate fertilizer and determined whether additional biochar application further increased biomass yields. The fertilizers and biochar were tested in pot experiments with spring barley and faba beans using two nutrient-poor soils. The crops were fertilized with P-Salt at three levels and biochar in two concentrations. Biomass yield was determined after six weeks. Plant and soil samples were analysed for nitrogen, phosphorus and potassium contents. The P-Salt had similar or even better effects than mineral fertilizer on growth in both crops and soils. Slow release of nutrients can prevent leaching, rendering P-Salt a particularly suitable fertilizer for light sandy soils. Biochar can enhance its fertilizing effect, but the underlying mechanisms need further investigation. These novel products are concluded to be promising candidates for efficient fertilization strategies.

Keywords: manure; phosphorus recovery; struvite; biochar; spring barley; faba bean

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1. Introduction

European agriculture is currently facing the problem of the accumulation of large amounts of slurry and manure, particularly in regions with high livestock densities, for example northwest Germany, Flanders and the Netherlands. Slurry and manure contain considerable amounts of important plant nutrients, including phosphorus (P) and nitrogen (N). It has been estimated that if the Netherlands applied its manure up to the allowed amount of phosphate on all its agricultural land, in 2015 there would have still been excess manure containing 40–60 million kg of phosphate [1]. Dealing with these manure and nutrient excesses is becoming an increasingly urgent challenge, and is heightened by the trend towards larger farm sizes as a consequence of increasing economic pressure. Manure storage is not only cost-intensive but is also associated with nutrient losses [2], leading to environmental problems such as air pollution (gaseous N emissions in the form of ammonia and nitrous oxide) and groundwater contamination (nitrate leaching).

Today, large livestock producers often buy a substantial proportion of their animal feed instead of growing it on their own farm. Most protein feed used in Europe, for example, is soybean meal, which has to be imported from South America. Farmers are no longer limited by regional feed supply and availability of arable land. Nutrients are imported along with the feed and remain in surplus on the farm within the manure. The livestock farms have too small a land area for the environmentally friendly field application of the accumulating nutrient load without exceeding the legal limits set by the European Union (EU) Nitrates Directive [3] and the EU Water Framework Directive [4]. Consequently, manure is considered a waste rather than a valuable resource. The situation is aggravated by the lack of regionally available, environmentally sound manure treatment solutions and the high costs of storage and disposal. As an example, Dutch farmers pay between €5 and €20 per tonne for the transport of surplus manure to other locations within the Netherlands [5] or even abroad.

By contrast, in other regions nutrients are needed—for example, at sites where arable farming is predominant and animal feed is produced for export. However, the high water content (>90%, [6]) makes long-distance transportation of manure neither profitable nor ecological. As a consequence, soil organic matter contents are depleted at these sites and nutrient deficits replaced through synthetic (N) or mineral (P, and potassium, K) fertilizers [7], which considerably interferes with the global P cycle [8].

Synthetic N fertilizers are mainly produced through the Haber–Bosch process. This process uses N from the air (thus unlimited in availability), but also consumes high amounts of natural gas and energy [9]. In contrast, mineral fertilizers are mainly derived from fossil resources and are, as such, limited. This is especially true for fossil P sources.

As a vital component of DNA and ATP, P is essential for all living organisms. Thus, it is one of the main nutrients needed for crop nutrition. The goal of achieving food security for a growing world population, the increasing use of biomass for biofuel production and the progressive degradation of arable land have all led to P fertilizer becoming more important for agricultural production than ever before.

In 2013/14, annual phosphate fertilizer consumption in Germany was 284,000 t [10]. In 2011, total EU phosphate consumption (fertilizer and industrial use) stood at approx. 4.6 million t per year. This represents 10% of global phosphate demand [11].

Phosphate fertilizer used in agriculture is mainly produced from rock phosphate (RP). However, RP is a finite resource, as with all mined resources. For this reason, in 2014, the EC added it to the list of critical raw materials [11]. Contrary to assertions in previous studies, there are still sufficient supplies of RP, but its extraction is very complex and not (yet) economically viable [12]. In addition, mined RP is increasingly contaminated by uranium and cadmium [13]. As 82% of the phosphorus extracted is used for fertilizers, these pollutants end up in the environment [11].

For this reason, prudent management of available P resources is of paramount importance. Exploiting "fresh" RP resources is one option. Another is the recycling of already "exploited" P, for example from livestock manure.

Livestock manure contains highly plant-available forms of P (inorganic) and N (ammonium) [14]. As such, it is a valuable organic fertilizer and a promising resource for P and N recovery. The manure excreted in EU-27 every year contains 1.8 million t of P, which corresponds to 150% of the amount of P used annually in fertilizers in Europe [2]. Thus, P recovery from manure could theoretically more than meet the entire demand for P fertilizer in Europe—providing the fertilizing effect of the recovered product is comparable.

The EC-funded research project "BioEcoSIM" ("An innovative bio-economy solution to valorise livestock manure into a range of stabilised soil improving materials for environmental sustainability and economic benefit for European agriculture"; grant No. 308637) has succeeded in developing an innovative technology at pilot-scale to recover P and N from pig manure. In a first step, manure is pretreated, so that the P completely dissolves. Subsequently, the manure is separated into a solid and a liquid fraction. The solid fraction is dried and then pyrolyzed to biochar. The P is recovered from the liquid fraction by precipitation and filtered off as a mixture of calcium phosphate (hydroxyapatite), magnesium phosphate and magnesium ammonium phosphate (MAP, struvite). The raw manure contains sufficient magnesium (1.7% dry matter) to allow struvite formation; no additional magnesium source is necessary. In this study, the obtained product is referred to as phosphate salts or "P-Salt".

This innovative technology has several advantages. It contributes to an environmentally friendly solution to the problem of manure disposal. It addresses the unfavourable nutrient ratio of manure, which often leads to an oversupply of P, as the amount of manure used in fertilization is usually calculated based solely on its N content. This also avoids the accompanying negative environmental consequences, such as P accumulation in soil, surface runoff and eutrophication of waterbodies. As the nutrients P and N are recovered separately, they can be used to create customized fertilizers as transportable and marketable products. This allows the fertilization of crops according to their respective requirements and the balancing of disrupted nutrient cycles. The technology could also reduce the EU's dependency on P imports. The improvement in P-use efficiency could help to conserve fossil P resources and reduce energy consumption in mining.

Struvite has been shown to be a highly effective, slow-releasing P fertilizer [15,16]. Several studies have found that struvite recovered from different materials can improve the yields of various crops compared to untreated controls [17–19]. Struvite recovered from swine wastewater has been shown to increase the biomass yield of maize more than commercial P fertilizer [20].

However, the plant availability of P in recovered products is often low, or at least unpredictable [21]. The assessment of fertilizers based on analytical results alone is not sufficient, because the predicted and actual availability and uptake of P by plants can differ substantially. Johnston and Richards [22] as well as Römer [23] confirmed that some P fertilizers ensure relatively good P availability and supply despite the small amounts contained in water-soluble form. Cabeza et al. [17] concluded that the dissolution of P in soil is a much more accurate indicator of the fertilizing effectiveness of recycled P products than their solubility in water or citric acid. Thus, plant experiments are crucial to evaluate the actual efficacy of the P-Salt in terms of P-fertilizing performance.

Biochar is produced from the solid manure fraction in the BioEcoSIM process and can serve as a potential soil improver. Biochar made from different substrates was reported to have beneficial effects on crop yield, soil quality and soil biological activity [24]. It can be used as an amendment to increase the water and nutrient retention capacity of light soils [25,26], thus aiding the sustainable production of food, feed and energy crops on progressively degrading soils—one measure to help meet the demand of an increasing world population. It also functioned as a means of carbon sequestration in soil [27,28] and has been shown to contribute to the mitigation of greenhouse gas emissions [29,30]. However, the use of biochar as a soil-improving substance is controversial and some studies have found biochar application to have no effect or even adverse effects on crop yield [31,32]. A meta-analysis review concluded that biochar application had a small, but statistically significant influence on crop productivity [33]. In this study, the biochar produced is used together with the recovered P-Salt, underlining the integrated concept of the project.

The combined application of P-Salt and biochar recovered from the same material has not been tested before. Based on results from the use of biochar in combination with conventional fertilizer [34–36], we assume that biochar prevents the leaching of nutrients contained in the P-Salt and increases crop yield. Biochar application may promote root development [37] through improved soil structure, resulting in more efficient nutrient uptake from the P-Salt and thus better crop development [38].

There are only a few studies [15,39,40] on the use of P fertilizer recovered from pig manure that used a comparable technique and none of these tested and compared its fertilizing effect on different crop types.

For that reason, this study aimed to test the fertilizing effect of the manure-based P-Salt on two crop types and assess its competitiveness with conventional superphosphate. A further objective was to determine whether the combined application of P-Salt and biochar improves the fertilizing effect through synergy effects. A third objective was to assess whether there are differences in the uptake efficiency of recovered and synthetic nutrients between different crop types.

Based on these objectives, the following hypotheses were set up for the study:

- P-Salts recovered as struvite from pig manure work equally well as or better than mineral P fertilizer.
- There is a synergetic effect/an interaction between P-Salt and biochar application with regard to improved soil productivity and biomass yield.
- Different crop types (cereals/legumes) react differently to P-Salt treatment, and this is also influenced by soil.

These hypotheses were tested by means of pot experiments with spring barley and faba beans. However, an important prerequisite for the use of novel products (in this case P-Salt and biochar) as fertilizers is that they do not have any undesirable effects on plants or soil biota. For this reason, a comprehensive chemical analysis and two bioassays were carried out on the products prior to the pot experiments.

2. Materials and Methods

The experimental part of this study included (1) the comprehensive determination of the chemical composition of P-Salt and biochar; (2) two bioassays to detect any eco-toxic effects on seed germination and crop development; and (3) two pot experiments to assess the fertilizing and soil-improving performance of the products.

This three-stage approach enabled detection of both desired and undesired impacts of the products on plants and soil biota at an early stage of the research project and, if necessary, the adaptation of the production process towards ecologically sound fertilizer products. Manure does not usually contain excessive amounts of problematic substances, such as heavy metals or organic pollutants. The bioassays were performed to determine whether these contaminants are concentrated in the products during the recovery process and to ensure that they do not affect crops.

2.1. Chemical Characterization

The P-Salt used in this study is a complex of struvite, magnesium phosphate and calcium phosphate obtained via the BioEcoSIM process. Pig manure was collected at a farm in Kupferzell (Germany). It was acidified with sulfuric acid to pH 5 and subsequently separated by coarse filtration into a solid and a liquid fraction. The solid fraction was dried and pyrolyzed in a superheated steam atmosphere (45 min at 450 °C). The P-Salt was recovered from the liquid manure fraction by precipitation and then filtered off. It serves as a potential source of P, but also contains N (Table 1). Contents of additional macro- and micronutrients as well as heavy metals are provided in Table A1.

Table 1. Characteristics of phosphate salts (P-Salt) and biochar.

Parameter	Unit	Method	P-Salt	Biochar
Total volatile solid content	% DM	DIN EN 15935:2012-11	17.3	-
P total	% DM	DIN EN ISO 11885	5.0	6.0
of which				
P water soluble	% DM	VDLUFA II, 4.1.4	1.2	0.4
P citric acid soluble	% DM	VDLUFA II, 4.1.3	9.5	13.5
P neutral ammonium citrate soluble	% DM	VDLUFA II, 4.1.4	9.5	13.2
N total	% DM	DIN ISO 13878	8.1	3.0
Ammonium N (NH_4-N)	% DM	DIN 38406-E5	2.4	<0.05
Nitrate N (NO_3-N)	% DM	$CaCl_2$-extraction	-	<0.00051
K	% DM	DIN EN ISO 11885	2.0	2.1
S	% DM	DIN EN ISO 11885	4.7	0.3
pH	-	DIN EN 12176	7.0	8.8

DM, dry matter; N, nitrogen; P, phosphorus; K, potassium; S, sulfur; VDLUFA, Association of German Agricultural Analytic and Research Institutes.

2.2. Toxicity Studies

Preliminary testing in petri dishes showed the germination capacity of barley to be 98% and that of faba beans to be 100%.

Two bioassays were then carried out on the P-Salt and biochar to detect any inhibiting effects on seed germination and early crop growth (Tables 2 and 3). Both tests employed a direct exposure approach. The P-Salt and biochar were applied to cress and barley at five different levels. The P-Salt applications ranged from 50% to 200% of the optimal P supply (=100%) of 150 mg P per kg substrate. The biochar application rates were calculated based on mass percentage of the cultivation substrate, not nutrient content. Both products were mixed with the substrate and filled into pots. The cress seeds were sown on top of the substrate and lightly covered. The barley seeds were sown at a depth of approximately 1 cm. The pots for the germination test were placed in a climate chamber and taken out regularly to count the number of germinated seeds. The pots for the growth test were placed on tables in a greenhouse. At the end of the test, the crops were cut 0.5 cm above the soil surface, weighed and dried at 60 °C for 48 h. Dry weight was determined and dry matter content calculated.

Table 2. Experimental set-up of seed germination test.

Crop	Cress (*Lepidium sativum*) 10 seeds per pot	Spring barley (*Hordeum vulgare* var. 'Grace') 2 seeds per pot
Substrate + pots	30 g (biochar)/50 g (P-Salt) cultivation substrate (TKS 1, Floragard) per pot (polypropylene, 7 × 7 × 8 cm^3, Goettinger)	
Treatments + replications	P-Salt: 0, 0.125, 0.25, 0.313, 0.375 and 0.5 g P-Salt per pot (control, 50%, 100%, 125%, 150% and 200% of optimal P supply); 10 replications Biochar: 0, 0.03, 0.06, 0.15, 0.3 and 0.6 g biochar per pot (control, 0.1%, 0.2%, 0.5%, 1.0% and 2.0%); 8 replications	
Duration	14 days	19 days
Conditions	20 °C, 16 h light, 8 h dark; climate chamber KBK/LS 4600 (Ehret GmbH & Co. KG, Emmendingen, Germany) Initial watering with 100 mL deionized water per pot; additional spraying when required	

TKS, the product name of the substrate.

Table 3. Experimental set-up of crop growth test.

Crop	Cress (*Lepidium sativum*) 20 seeds per pot	Spring barley (*Hordeum vulgare* var. 'Grace') 10 seeds per pot; after germination reduction to 3 seedlings per pot
Substrate + pots	250 g cultivation substrate (TKS 2, Floragard) per pot (polypropylene, 11 × 11 × 12 cm^3, Goettinger)	
Treatments + replications	P-Salt: 0, 0.375, 0.75, 0.938, 1.125 and 1.5 g per pot; 4 replications Biochar: 0, 0.25, 0.5, 1.25, 2.5 and 5 g per pot; 4 replications	
Duration	2 weeks	6 weeks
Conditions	Greenhouse; initial watering with 250 mL deionized water per pot to soak substrate; additional watering when required	

2.3. Pot Experiments

The pot experiments were carried out using two soil substrates. Clay and sand were chosen due to their low concentration and plant availability of P. The P content measured by calcium-acetate-lactate extraction (P(CAL)) in both soils is classified as very low according to Association of German Agricultural Analytic and Research Institutes (VDLUFA, Table 4). Additionally, the clay soil had a high phosphate immobilization potential due to a high concentration of carbonates. The N mineralization potential was low in both soils. Both soils were of low fertility and thus not representative of agricultural soils. The clay soil had good water retention properties, but became very hard when dry and warmed only slowly. The sand soil had zero water retention capacity; water immediately flowed to the bottom of the pots.

Table 4. Characteristics of soil substrates.

Soil	N_{min}	P(CAL)	K(CAL)	pH
	$mg \cdot (kg \cdot soil)^{-1}$	$mg \cdot (100 \cdot g \cdot soil)^{-1}$		
Clay	1.7	0.7	2.9	8.1
Sand	0.8	0.01	0.17	8.0

N_{min}, mineralized nitrogen, CAL, calcium-acetate-lactate method.

The two soils were mixed with varying amounts of P-Salt, P-Salt in combination with biochar or conventional fertilizer (Table 5). The application rates of the P-Salt were calculated based on its total P content. Optimal P supply was defined as 150 mg total P per kg·soil [41], i.e., 0.225 g P or 4.5 g P-Salt pot^{-1}, and is referred to as 100%. A reduced dose (50%) to simulate nutrient shortage and an elevated dose (200%) were included. Levels higher than 200% were not considered reasonable and thus not tested.

The performance of the P-Salt was compared to conventional mineral fertilization with ammonium nitrate NH_4NO_3 (35% N) and calcium dihydrogen phosphate $Ca(H_2PO_4)_2$, (24.6% P). Mineral N and P were applied in the same amount as in the P-Salt (Table 5). Other main plant nutrients (K, Mg, Ca) and trace elements were not considered in this experiment.

Biochar (BC) was applied in two concentrations (0.1% and 0.2%, equivalent to 1.5 and 3.0 $g \cdot pot^{-1}$) in combination with the 100% level of P-Salt (Table 5). The experiment also included control pots that remained completely unfertilized. The pot experiments were carried out first with barley, then with faba beans, and with both soils for each test crop.

Table 5. Overview of all treatments and corresponding N and P application rates.

Treatment	N Applied	P Applied	Biochar
	$g \cdot pot^{-1}$		
Control	-	-	-
P-Salt 50%	0.180	0.113	-
P-Salt 100%	0.360	0.225	-
P-Salt 200%	0.720	0.450	-
Mineral 100%	0.360	0.225	-
P-Salt 100% + BC 0.1%	0.360	0.225	1.5
P-Salt 100% + BC 0.2%	0.360	0.225	3.0

BC: biochar.

The required amounts of P-Salt and biochar were mixed thoroughly with 1.5 kg·soil and filled into polypropylene pots (13 × 13 × 13 cm^3, Goettinger). The conventional fertilizers (analytical grade NH_4NO_3 and $Ca(H_2PO_4)_2$) were dissolved in water to ensure exact dosage of the small amounts and then added to the soil. Pots were initially watered with 300 mL deionized water each.

The prepared pots were sown with either ten seeds of spring barley (*Hordeum vulgare* L. var. 'Grace') or eight seeds of faba bean (*Vicia faba* L. var. *minor* var. 'Isabell'). All pots were set up on a table in a greenhouse with no additional lighting in a randomized complete block design with four replications. After germination, plants were reduced to five per pot. The pots were watered from above with deionized water when necessary to keep the moisture near field capacity. Any leachates were collected and returned to the pots. Air temperature in the greenhouse was approx. 20 °C during the day and 16 °C at night.

The barley plants were treated once against powdery mildew with a combination of propiconazol, tebuconazol and fenpropidin. The bean plants were sprayed once against black bean aphids with Lambda-Cyhalothrin. Both treatments were carried out according to the manufacturer's (Syngenta Agro GmbH, Maintal, Germany) instructions for the respective crop.

After six weeks (barley BBCH 29/31, faba beans BBCH 39/51), the shoots were cut 0.5 cm above the soil surface, weighed and then dried at 60 °C for 48 h. Dry weight was determined and dry matter content calculated. Soil samples were taken from each individual pot. Roots were washed and dried at 60 °C for 48 h to determine the root dry weight.

2.4. Sample Analyses

The dried shoots were ground in a mixer mill (duration 40 s, frequency 30 min^{-1}; Retsch GmbH, Haan, Germany). Total N concentration in the biomass was determined according to DUMAS (DIN EN 13654-2). Concentrations of P, K, Ca and Mg were determined using microwave digestion followed by ICP-OES measurement (DIN EN ISO 11885). All samples were analysed in duplicate. Plant P uptake was calculated from dry matter yield (DMY) and P concentration.

The soil samples were used to determine plant-available N (NO_3 and NH_4; referred to as N_{min}) in fresh soil using $CaCl_2$ extraction followed by FIA (Flow injection analysis) measurement (DIN ISO 14255:1998-11). Plant-available P and K were then determined in air-dried soil using CAL extraction followed by flame photometer or FIA measurement, respectively (OENORM L 1087:2012-12-01). Soil pH was measured using a glass electrode after $CaCl_2$ extraction (DIN ISO 10390:2005).

2.5. Statistical Analysis

Data analysis was performed using SAS software version 9.3 PROC MIXED (SAS Institute Inc., Cary, NC, USA). Soil and treatment as well as their interaction were handled as fixed effects with DMY and nutrients in plant and soil samples as dependent variables. Data were log transformed where necessary. The graphs shown here were plotted with untransformed data. As large differences in biomass development were expected for the two soils, the treatments were compared separately for each soil. The level of significance was $\alpha = 0.05$. Standard errors (SE) given in tables were calculated as pooled standard error of the mean.

3. Results

3.1. Toxicity Studies

The growth and germination tests with biochar gave somewhat contradictory results (Tables 6 and 7). In summary, neither P-Salt nor biochar exposed any major risks to soil, crops or environment in terms of their chemical composition and resulting characteristics, as long as the amounts applied are in line with common fertilizing practice.

Table 6. Results of germination test.

	Cress	Barley
P-Salt	Seed germination up to 27% lower following application in the tested ranges.	Seed germination enhanced by up to 30% by doses up to and including the 100% dose; no further increases at higher doses.
Biochar	No effect in any of the tested concentrations.	Moderate concentrations of up to 1% did not have any negative effect.

Table 7. Results of crop growth test.

	Cress	Barley
P-Salt	Dry matter yield (DMY) was not significantly influenced by doses up to and including the 150% dose. The 200% dose resulted in 19% lower DMY compared to the control.	Tendency for decreasing DMY with increasing P-Salt dosage; however, the growth-retarding effect was only statistically significant for the two highest levels (31% and 18% lower DMY).

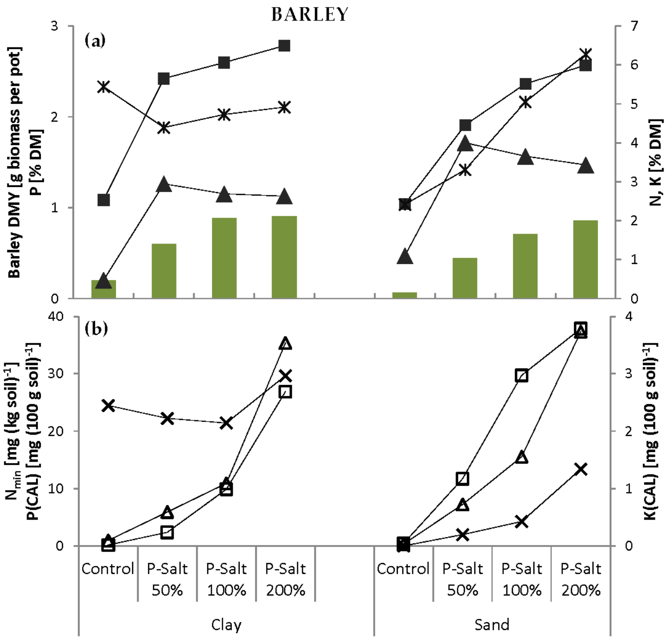
Table 7. *Cont.*

	Cress	Barley
Biochar	DMY appeared to decrease with increasing concentration. However, the adverse effect was only significant for the two highest concentrations with 19% and 20% lower DMY than in the control.	DMY not influenced by any concentration tested.

3.2. Pot Experiments

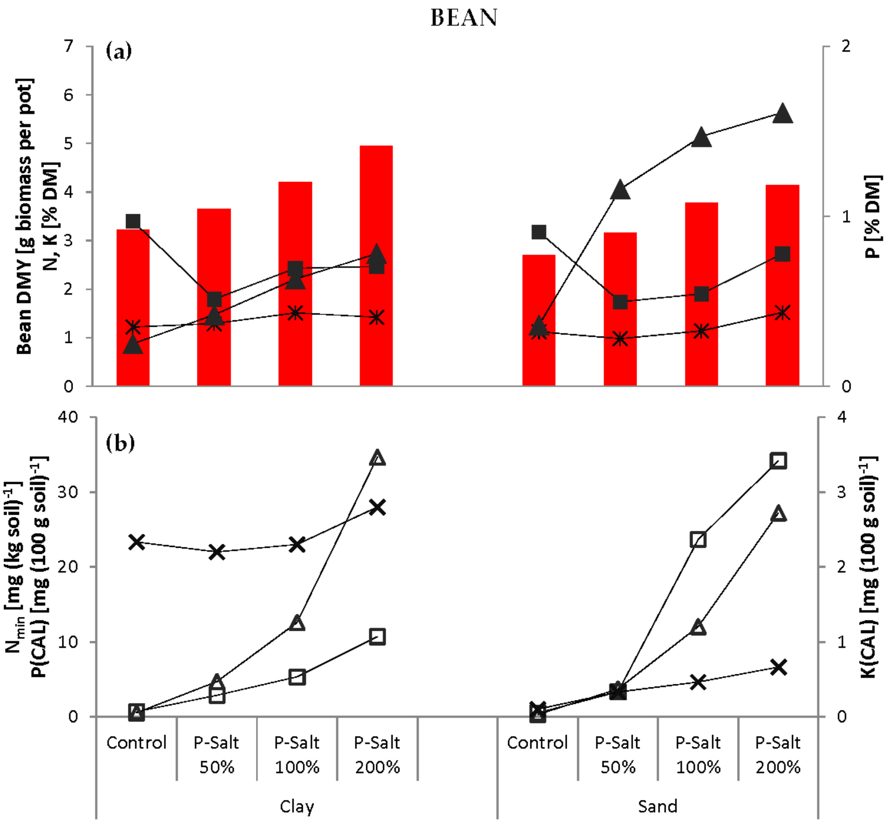
3.2.1. Effect of Increasing P-Salt Doses on Biomass Yield and Nutrient Concentrations

All P-Salt treatments led to an increase in DMY in both crops (Figures 1 and 2). In barley, this increase was significant even from the moderate 50% dose upwards, but in beans only from 100% upwards. High concentrations (200%) further increased the DMY. However, for barley this was significant only in sand, but not in clay, and for beans vice versa. The DMY of both crops was generally higher in clay than in sand. The effects of the factors 'treatment', 'soil' and their interaction 'soil*treatment' were highly significant ($p < 0.0001$) in both crops. For reasons of clarity, error bars have not been included in the figures. Instead, variances are expressed as standard errors in the corresponding tables.



		Control	P-Salt 50%	P-Salt 100%	P-Salt 200%	Control	P-Salt 50%	P-Salt 100%	P-Salt 200%	SE
		Clay				Sand				
Biomass	DMY ■	a	b	c	c	a	b	c	d	0.036
	N ■	a	b	bc	c	a	b	c	d	0.044
	P ▲	a	b	b	b	a	b	bc	c	0.038
	K ∗	a	b	bc	c	a	b	c	d	0.049
Soil	N$_{min}$ □	a	b	c	d	a	b	c	d	0.241
	P(CAL) △	a	b	c	d	a	b	c	d	0.088
	K(CAL) ×	a	a	a	b	a	b	c	d	0.075

Figure 1. Dry matter yield (DMY) and nutrient concentration in biomass, graph upper panel, (**a**), and soil, graph lower panel, (**b**), of barley treated with increasing P-Salt levels compared to untreated control. Different letters in the table indicate statistically significant differences between treatments ($\alpha = 0.05$, $n = 4$). SE: pooled standard error of the mean.



		Control	P-Salt 50%	P-Salt 100%	P-Salt 200%	Control	P-Salt 50%	P-Salt 100%	P-Salt 200%	SE
		Clay				Sand				
Biomass	DMY 🟥	a	a	b	c	a	a	b	b	0.069
	N ■	a	b	c	c	a	b	b	c	0.057
	P ▲	a	b	c	d	a	b	c	d	0.026
	K ∗	a	ab	c	bc	a	a	a	b	0.041
Soil	N_{min} □	a	b	c	d	a	b	c	d	0.135
	P(CAL) △	a	b	c	d	a	b	c	d	0.076
	K(CAL) ×	a	a	a	b	a	b	c	d	0.054

Figure 2. Dry matter yield (DMY) and nutrient concentration in biomass, graph upper panel, (**a**), and soil, graph lower panel, (**b**), of faba beans treated with increasing P-Salt levels compared to untreated control. Different letters in the table indicate statistically significant differences between treatments ($\alpha = 0.05$, $n = 4$). SE: pooled standard error of the mean.

The plant N concentration showed different patterns in the two crops, although both crops showed higher values in clay than in sand. In barley, it increased with the P-Salt dosage and was highest (6.5% DM) with the 200% dose in clay. In beans, by contrast, it was relatively high in the controls (3.4% in clay, 3.2% in sand), and only between 1.7% and 2.7% DM in the treated plants. In clay, the N uptake calculated per pot puts this into perspective, where it was similar in all variants (except the 50% dose).

The plant concentration and uptake of P were higher in sand in both crops. In barley, the plant P concentration did not vary between the treatments in clay, but decreased with increasing P-Salt dose in sand. In beans, it increased steadily with P-Salt dose in both soils. The P and K concentrations were lower in beans than in barley; however, beans took up substantially higher amounts of P and K due to their higher DMY. The plant K concentration of barley grown in clay increased with P-Salt dosage. Although levels in treated plants remained below those of the control (5.4% DM), this was relativized by the higher DMY. In contrast, K increased significantly with every P-Salt level in sand. The K concentration of beans only rose with the 100% (clay) and 200% (sand) doses.

Plant-available soil nutrients measured at the end of the experiment showed the same pattern for both crops (Figures 1 and 2). The N_{min} and P(CAL) values increased significantly with P-Salt dosage in both soils, and K(CAL) only in sand. The N_{min} and P(CAL) contents were mostly higher in sand than in clay. The P(CAL) contents increased sharply from the 100% to the 200% doses. In contrast, K(CAL) contents in clay were similar for all variants except the 200% dose. Higher K(CAL) contents were found in clay than in sand.

3.2.2. Effect of Biochar Addition and Comparison of P-Salt and Mineral Fertilizer

All fertilizer treatments increased DMY in both crops compared to the control, with the one exception of the mineral fertilizer treatment of beans grown in sand (Figures 3 and 4). Biochar addition alone did not have any significant effect on DMY (Appendix A). The application of 0.1% and 0.2% biochar in addition to P-Salt enhanced barley DMY compared to fertilization with P-Salt only; however, this effect was only statistically significant in sand (Figure 3). All P-Salt treatments—with or without biochar—outperformed the mineral fertilizer in terms of DMY, except for beans grown in clay. In sand, it was not possible to harvest any barley biomass from pots treated with mineral fertilizer. The highest DMY overall was obtained with the P-Salt + 0.1% BC treatment (4.5 $g \cdot pot^{-1}$ for bean grown in clay; 1.0 $g \cdot pot^{-1}$ for barley grown in sand).

The highest plant N concentration was found in the minerally fertilized plants (7.0% DM in barley, 6.9% DM in beans), followed by the P-Salt treatment in barley and the biochar variants in beans. However, the N uptake in barley was lower with mineral fertilizer than with the P-Salt treatments (Table 8). The N concentration seemed remarkably high in minerally fertilized beans grown in sand, but this was partly an effect of the lower DMY.

The plant P concentration was higher in sand than in clay. There was no difference between the P-Salt alone and the combined treatments with biochar in barley in either soil (on average 1.2% DM in clay, 1.5% DM in sand). Mineral fertilizer considerably increased plant P (to 2.8% DM) in barley grown in clay, whereas it decreased plant P in beans in both soils.

In both crops and soils, the highest plant K concentration was found in plants treated with the combination of P-Salt and 0.2% biochar. Biochar addition almost always significantly increased plant K relative to P-Salt alone and mineral fertilizer. Application of P-Salt with and without biochar resulted in a higher uptake of K than with mineral fertilizer.

By far the highest N_{min} contents were found in minerally fertilized pots in both crops and soils. These were followed by the P-Salt variants, but with much lower values. Again, higher values were found in sand. Biochar addition, particularly the 0.2% concentration, seemed to lower N_{min} compared to P-Salt alone.

Soil P(CAL) was close to zero in all controls and continuously increased following P-Salt and particularly biochar treatments. The P(CAL) of pots treated with mineral fertilizer was between the control and P-Salt variants, yet unexpectedly low.

The K(CAL) values closely followed the pattern of plant K: highest values were found in pots treated with P-Salt and 0.2% biochar and lowest values in minerally fertilized pots. Application of P-Salt alone and each of the combinations significantly increased K(CAL). Levels were generally higher in clay than in sand. Practically no K(CAL) was measured in sand in the control (0.0 $mg \cdot (100 \cdot g \cdot soil)^{-1}$) and the minerally fertilized pots (0.1 and 0.2 $mg \cdot (100 \cdot g \cdot soil)^{-1}$ for beans and barley, respectively).

3.2.3. Influence of Fertilizer Form on Nutrient Uptake

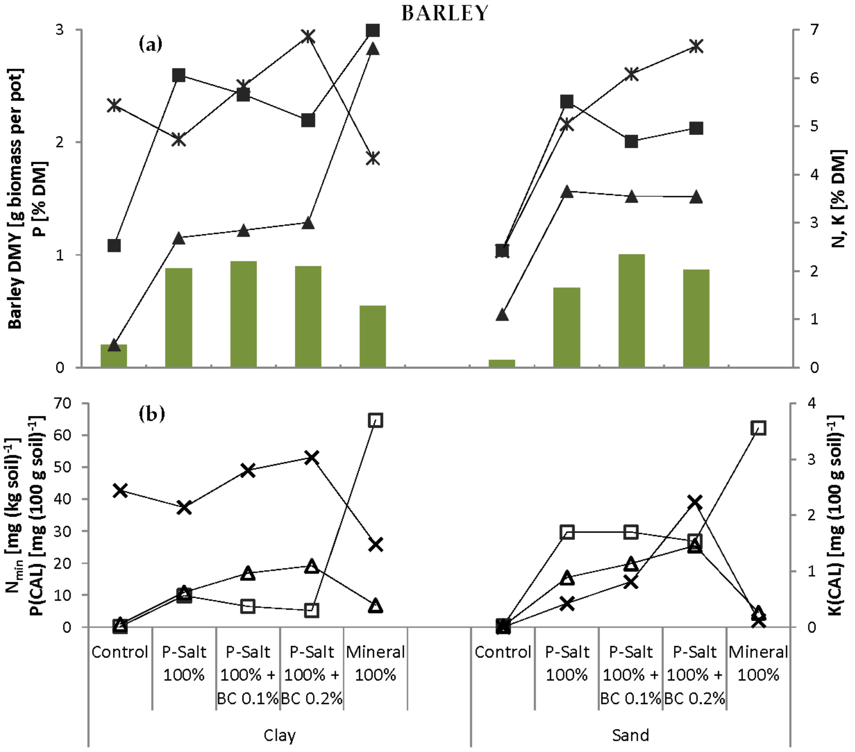
The N uptake of barley was higher from the P-Salt treatments than from mineral fertilizer. For P uptake, it was the other way around. This was observed in both soils (Table 8).

The nutrient uptake of beans was the reverse for both N and P. The nutrient uptake was of course closely related to the DMY obtained and the concentration of N and P in the crops (Figures 3 and 4).

3.2.4. Influence of Treatment and Soil on Root Dry Matter and Shoot:Root Ratio

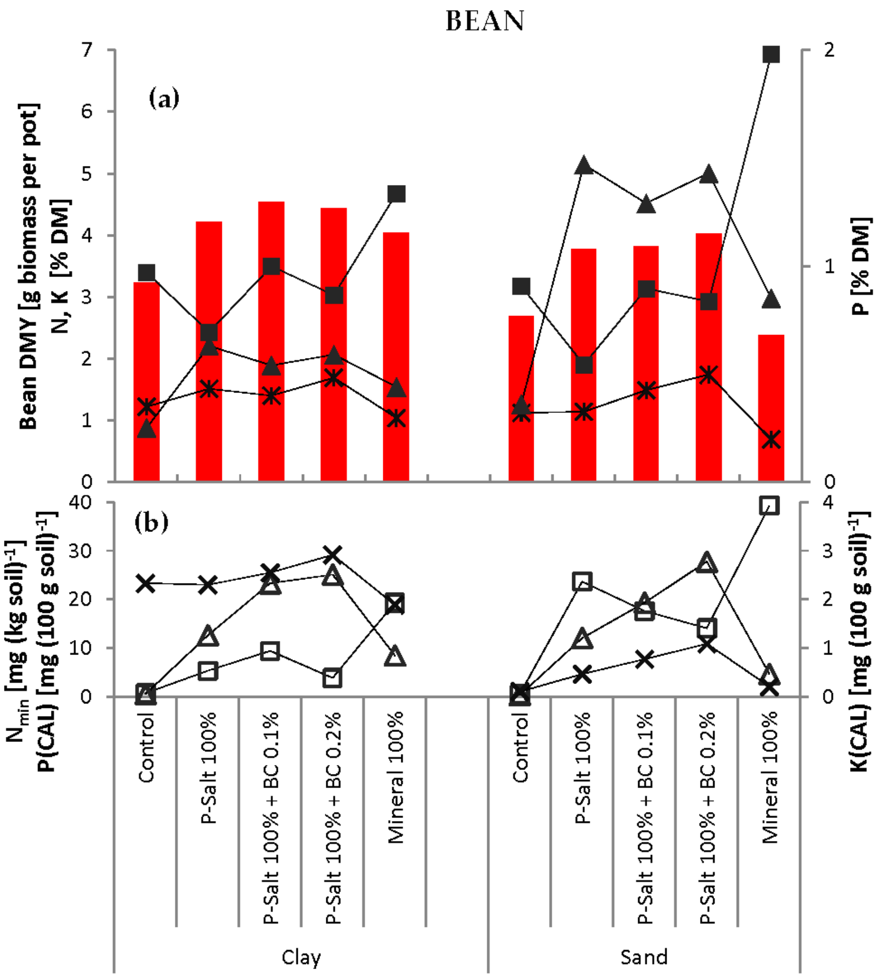
As expected, the root development of barley was much more pronounced in sand soil than in clay (Table 9). In sand, even the smallest plants had developed a relatively extensive root system. This is reflected by the low shoot:root ratio. In clay, the two biochar treatments led to a particularly high shoot:root ratio (>9).

In contrast, beans formed more root biomass in clay than in sand and in general considerably more than barley. The shoot:root ratio of the beans followed the same pattern for all treatments in both soils; however, values reached a slightly higher level in sand.



		Control	P-Salt 100%	P-Salt 100% + BC 0.1%	P-Salt 100% + BC 0.2%	Mineral 100%	Control	P-Salt 100%	P-Salt 100% + BC 0.1%	P-Salt 100% + BC 0.2%	Mineral 100%	SE
				Clay					Sand			
Biomass	DMY ■	a	b	b	b	c	a	b	c	c	-	0.036
	N ■	a	b	b	c	d	a	b	c	c	-	0.044
	P ▲	a	b	b	b	c	a	b	b	b	-	0.038
	K ∗	a	b	a	c	b	a	b	c	c	-	0.049
Soil	N_{min} □	a	b	c	c	d	a	b	b	b	c	0.241
	P(CAL) △	a	b	c	c	d	a	b	c	d	e	0.088
	K(CAL) ×	ab	a	bc	c	d	a	b	c	d	e	0.075

Figure 3. Dry matter yield (DMY) and nutrient concentration in biomass, graph upper panel, (**a**), and soil, graph lower panel, (**b**), of barley treated with P-Salt only ("P-Salt 100%"), P-Salt and biochar ("P-Salt + BC 0.1%", "P-Salt + BC 0.2%") and mineral fertilizer ("Mineral 100%") compared to untreated control. Different letters in the table indicate statistically significant differences between treatments ($\alpha = 0.05$, $n = 4$). SE: pooled standard error of the mean.



		Control	P-Salt 100%	P-Salt 100% + BC 0.1%	P-Salt 100% + BC 0.2%	Mineral 100%	Control	P-Salt 100%	P-Salt 100% + BC 0.1%	P-Salt 100% + BC 0.2%	Mineral 100%	SE
		Clay					Sand					
Biomass	DMY 🟥	a	b	b	b	b	a	b	b	b	a	0.069
	N ■	ab	c	a	b	d	a	b	a	a	c	0.057
	P ▲	a	b	c	bc	d	a	b	c	b	d	0.026
	K ∗	a	bc	ac	b	d	a	a	b	c	d	0.041
Soil	N_{min} □	a	b	c	d	e	a	b	c	c	d	0.136
	P(CAL) △	a	b	c	c	d	a	b	c	d	e	0.076
	K(CAL) ×	a	a	a	b	c	a	b	c	d	e	0.054

Figure 4. Dry matter yield (DMY) and nutrient concentration in biomass, graph upper panel, (**a**), and soil, graph lower panel, (**b**), of faba beans treated with P-Salt only (“P-Salt 100%”), P-Salt and biochar (“P-Salt + BC 0.1%”, “P-Salt + BC 0.2%”) and mineral fertilizer (“Mineral 100%”) compared to untreated control. Different letters in the table indicate statistically significant differences between treatments (α = 0.05, *n* = 4). SE: pooled standard error of the mean.

Table 8. Mean nutrient uptake into shoots for fertilizer forms with/without biochar (BC) addition tested. Different letters in the table indicate statistically significant differences between treatments ($\alpha = 0.05$, $n = 4$).

			Clay					Sand					SE
			Control	P-Salt 100%	P-Salt 100% + BC 0.1%	P-Salt 100% + BC 0.2%	Mineral 100%	Control	P-Salt 100%	P-Salt 100% + BC 0.1%	P-Salt 100% + BC 0.2%	Mineral 100%	
Barley	N	mg·pot^{-1}	5.13a	53.48b	53.37b	41.69bc	38.11c	1.64a	38.88b	46.88b	43.20b	no crop	0.138
	P		0.41a	10.17b	11.48b	11.59b	15.46c	0.32a	11.04b	13.74c	11.69b	no crop	0.145
	K		11.02a	41.71b	54.96bc	61.78c	23.64d	1.63a	35.60b	60.83c	57.96c	no crop	0.166
Bean	N	mg·pot^{-1}	109.69a	102.24a	158.47b	117.83a	189.14c	85.56a	71.89b	119.48c	117.86c	165.11d	0.077
	P		8.08a	26.52b	24.48b	26.21b	17.80c	9.70a	55.57b	49.18b	57.63b	20.25c	0.085
	K		39.41a	63.57b	63.46bc	69.71c	42.07a	30.18a	43.09b	56.81c	70.12d	16.44e	0.091

SE: pooled standard error of the mean.

Table 9. Mean root dry matter and shoot:root ratio of barley and faba beans for fertilizer forms with/without biochar (BC) addition tested. Different letters in the table indicate statistically significant differences between treatments ($\alpha = 0.05$, $n = 4$).

		Unit	Clay					Sand					SE
			Control	P-Salt 100%	P-Salt 100% + BC 0.1%	P-Salt 100% + BC 0.2%	Mineral 100%	Control	P-Salt 100%	P-Salt 100% + BC 0.1%	P-Salt 100% + BC 0.2%	Mineral 100%	
Barley	Root dry matter	g·pot^{-1}	0.22ns	0.19ns	0.10ns	0.11ns	0.14ns	0.42a	0.44a	0.66b	0.40a	no crop	0.067
	Shoot:root ratio	-	1.01a	4.42b	9.83c	9.64c	4.20b	0.16a	1.79b	1.65b	2.88b	no crop	0.180
Bean	Root dry matter	g·pot^{-1}	4.65b	4.37b	4.20b	5.05b	3.11a	2.00a	2.66b	2.78b	3.02b	1.60a	0.087
	Shoot:root ratio	-	0.71a	1.00bc	1.13cd	0.96ab	1.37d	1.35ab	1.45ab	1.49ab	1.33a	1.50b	0.114

SE: pooled standard error of the mean, ns: not significant.

4. Discussion

The findings of this study confirmed the hypotheses that (1) the fertilizing performance of P-Salt recovered from manure is equivalent to that of mineral P fertilizer; (2) there are positive synergies between biochar and P-Salt; and (3) there are differences in reaction to fertilization between crops. These are discussed in the following sections.

4.1. The Fertilizing Performance of P-Salt Is Equivalent to that of Mineral P Fertilizer

The fertilizing performance of P-Salt was evaluated on the basis of DMY and nutrient concentration. In terms of DMY, P-Salt performed better than mineral P fertilizer in both barley and bean crops and in the two soils sand and clay. This is particularly remarkable, as the fertilizers were compared based on total rather than water-soluble P content. The latter differed considerably, with commercial triple superphosphate supplying 43.5% and P-Salt only 1.2% of P in water-soluble form. Analysis by Mazeika et al. [42] of the molecular and morphological structure of manure-derived fertilizer (poultry manure) showed a colocalization of K, S, and P within the derived organo-mineral fertilizers (OMF). This, and the specific structure of the OMF at the molecular and crystalline levels may affect their performance, which can thus be different than that of minerally-derived P fertilizer.

Although barley had a higher DMY with P-Salt fertilization, its P uptake was higher with mineral fertilizer. We concluded that this is an effect of the large water-soluble P-fraction in mineral fertilizer. We hypothesize that in general both fertilizer types have similar yield effects, but that they are based on different dynamics of P-availability over time.

Contrary to expectations, both P concentration and uptake were higher in beans from the P-Salt treatment than from the conventional fertilizer treatment. As a legume, the bean was able to stimulate P mobilization by releasing root exudates, which very likely increased P availability [16] from the P-Salt.

Previous studies comparing P fertilizers/struvites recovered from various materials to commercial P fertilizer have reported that the recycled products increased DMY in maize [20], led to comparable DMY in perennial ryegrass [22], or at least improved DMY compared to untreated controls in several crops [17–19,43].

Our findings support the hypothesis that P-Salt is able to compete with commercial products in terms of yield effect and nutrient supply under the conditions tested.

However, we observed a few potential disadvantages of P-Salts compared to mineral fertilizer. The increase in both P and N concentration in barley biomass was considerably higher with mineral fertilizer. This can most likely be attributed to the higher plant-availability of P and N from mineral fertilizer immediately from the beginning of the experiment. These plants probably took up all their required nutrients within the first weeks. In contrast, the crops receiving P-Salt—whose main component struvite is known for its gradual P release [16] and low solubility—were not able to catch up within the remaining time. However, they compensated for the lower nutrient concentration through higher DMY, resulting in a type of nutrient dilution effect. A test duration longer than six weeks may have produced slightly different results, particularly because the amount of plant-available P from both fertilizer types may then have equalized.

In general, the fertilizing effect of mineral fertilizer was more uniform than that of P-Salt. This was apparent from the lower standard deviation of the DMY between replications. The reason for this remains unclear. To ensure a sufficiently uniform distribution, the P-Salt was ground very finely before mixing it with the soil. Fine particle size can positively influence the nutrient availability and thus the fertilizing effect [44]. For future experiments, granulation of the P-Salt should be considered to prevent possible demixing.

4.2. Biochar Improves P-Salt Fertilization Effects

The results of this study confirmed the findings of Schulz and Glaser [36] that biochar enhanced the effects of fertilizer and led to an increase in yield. In addition, we found that the biochar effect differed depending on soil and its positive effect appeared to increase with decreasing soil organic matter and an increasing sand content. Therefore, it was concluded that biochar has huge potential as a soil improver, particularly for more unproductive soils with low organic matter content, such as sand.

Light soils are more often subject to nutrient leaching due to lack of organic matter. Biochar addition may prevent these losses by improving the physical properties of the soil, namely the nutrient and water retention capacity of the soil [45], both valuable in sand. Biochar can absorb considerable amounts of water due to its large specific surface area. This water then remains available for the crops, along with the nutrients dissolved in it. However, the subsequent increased root growth reported by Bruun et al. [45] was only seen to a small extent in this study. The shoot:root ratio of the biochar variants significantly increased in barley grown in clay. This could be an indication of P accumulation in the soil. An increase in soil pH following biochar application [46] can have the indirect effect of higher P availability. This, in combination with the direct effect of a small amount of P from the biochar itself, results in improved P uptake and increased growth [47]. There are certainly interactions between the physical and the biological effects, but it was not possible to draw a conclusion here.

Towards the end of the study, significantly increased contents of P(CAL) and K(CAL) were recorded following biochar application in both crops and soils, which for P is consistent with previous studies [48,49]. The same was observed for plant K concentration and uptake. Hence, the biochar served as a source of P and K for the crops, despite the fact that the analysis found the P contained in the biochar to have very low water solubility. Biochars made from solid manures [1], poultry litter and swine manure [50] or beech-wood [36] are often reported to act as a nutrient source.

Biochar's normally positive property of retaining nutrients, thus preventing them from leaching can of course also have the negative effect of immobilization and therefore reduced plant-availability of certain nutrients. The treatments with biochar had lower soil N_{min}. Although these pots received the same amount of N as those in the "P-Salt only" treatments, it was not entirely plant-available. This suggests the—at least temporary—immobilization of nitrogen by biochar, as also observed in other studies (e.g., [34,35,37,48]). Beans showed a higher plant N concentration in the combined treatments than with P-Salt alone, whereas barley was unable to maintain the N concentration level of the P-Salt treatment. Although the bean seeds were not inoculated with rhizobia, by harvest, N fixation nodules had developed in the majority of pots. Thus, beans were able to meet their N demand by taking up additional N from biological fixation and possibly also mobilizing the N bound to biochar.

It is possible that biochar applied in combination with fertilizer binds nutrients released by the fertilizer. The nutrient release from P-Salt is slow. Therefore, it is assumed that biochar binds fewer nutrients from P-Salt than from mineral fertilizer, which provides the entire nutrient amount applied in readily plant-available forms. Enhanced DMY following the combined application of biochar and P-Salt may be explained by reduced nutrient leaching [48]. Furthermore, this result must stem from a synergistic effect, as the combined application led to higher DMY than with application of either P-Salt or biochar alone (Table A2, [36]). Therefore, it can be concluded that the fertilizing effect of P-salt can be enhanced by combined application with the biochar—a by-product of the BioEcoSIM process. The two biochar concentrations applied in this study did not significantly differ in terms of DMY. However, the 0.2% concentration showed a trend to decreasing DMY in barley in both soils and beans grown in clay. As biochar concentrations ten times as high (1% and 2%) did not show any adverse effect in the preliminary bioassays, a toxic effect of the low concentrations in the main experiment can be discounted. Bruun et al. [45] concluded that rates of 1%–2% by mass improve soil quality. The slight, but statistically insignificant decreases in yield following the 0.2% concentration may be in some way related to limited plant-availability of nutrients as discussed above.

In summary, the positive yield effect of biochar in sand was probably a consequence of factors such as improved soil structure (including water retention and increased soil organic matter), retention of fertilizer nutrients and limited nutrient supply. In combination, this promoted crop growth and yield.

4.3. Crop Types (Cereals/Legumes) React Differently to the P-Salt Treatment

The essential difference between the crop types was the significantly higher positive effect of P-Salt on cereal than legumes. This is revealed by a comparison of the controls with the P-Salt variants. Barley showed a highly positive reaction to N and P supplied by the P-Salt in terms of DMY and both plant concentration and uptake of N and P. Beans, in contrast, produced the same DMY in the control and the 50% treatment. The extremely low soil N_{min} and P(CAL) in the controls recorded at the beginning of the experiment suggests that beans were able to meet their nutrient demands using other sources, for example atmospheric N.

The main explanation here is of course that the bean as a legume has the ability to (1) take up additional N from biological fixation; and (2) mobilize P with low plant availability by releasing organic acids. The latter, for instance, has been reported for the uptake of native soil P by white lupin *Lupinus albus* L. [51].

In addition, the bean has a higher thousand grain weight than barley, providing more nutrients and thus making it less dependent on external nutrient supply during germination and early growth stages. Cereals, in contrast, develop an extensive root system to ensure access to nutrients provided both by the soil and by fertilizer [52].

The moderate DMY response to the P-Salt treatments as well as the lower plant N concentration in beans might be explained by inhibited biological N fixation as a consequence of applied N. This can also cause yield losses [53], yet this was not observed. The benefit of N fertilization of legumes is controversial, although minor N fertilization is sometimes recommended for faba bean production under unfavourable growing conditions, poor seedbed environment or low soil pH.

In sum, the different reactions are ascribable more to the crop type than to the P-Salt. For beans, it would be recommendable to modify the precipitation process in order to obtain a P-Salt with lower N content. We conclude that P-Salt worked well for both crop types tested, supporting the hypothesis that P-Salt could replace conventional P fertilizer.

5. Conclusions and Recommendations

This study explored the potential use of a P-Salt recovered from pig manure as a replacement for conventional mineral P fertilizer.

The P-Salt was found to have the same or even better effects than mineral fertilizer on growth in both crops in both soils. Thus, firstly, the recovered product can replace conventional mineral P in terms of the fertilizing effect for the two crop types tested here. Secondly, and perhaps more importantly, the demand for P fertilizer in European agriculture could theoretically be met by P recycling from manure alone. Ideally, this would render the extraction of "new" P from rock phosphate for fertilizer production superfluous in the medium to long term.

This study did not consider the potential fertilizer replacement value of the P-Salt. Organic products are usually applied in higher amounts in order to compensate for the slower release and lower plant availability of nutrients than with conventional products. If the amounts applied had been adjusted accordingly, the P-Salt would have certainly led to considerably better results than those obtained in this study. In addition, the P-Salt can supply plants and soil with additional microelements and a small amount of organic matter. These aspects render P-Salt recovered from manure by the BioEcoSIM process even more advantageous than conventional fertilizers.

However, the acceptance of such recycled fertilizers by agriculture and horticulture is currently fairly low. One constraint is certainly the reliability of the novel product. The combination of P-Salt and conventional products could serve as a convincing solution for users/farmers: conventional fertilizer provides readily available, water-soluble P in the early growth stages, whereas the slow-releasing P-Salt ensures a continuous supply during the entire growth period. This would allow the entire P fertilizer amount to be administered in one application without the risk of P deficiency in heavy soils with high P immobilization potential (e.g., clay) of water-soluble P. P-Salt also has a strong advantage in light soils with low buffer capacity (e.g., sand) where the slow release of P prevents its leaching or surface runoff. The fertilizing effect of P-Salt can be enhanced by combined application with biochar, which is also a product of the manure recycling process in which P-Salts are extracted.

The results indicate that biochar improves the soil status of sand, suggesting that biochar can be a valuable addition to sandy or degraded soils. However, no significant benefit was seen in the clay soil.

Granulation or pelletizing of finely ground P-Salt and biochar can considerably simplify their handling and turn them into marketable products. A reduction in N content of the P-Salt would avoid the accompanying N application, thus increasing flexibility. The next steps will be a detailed assessment of how the properties of the raw manure influence the emerging products and validation of the presented findings in field-scale experiments.

Acknowledgments: This work was performed within the project "BioEcoSIM", which received funding from the European Commission under grant agreement No. 308637. We would also like to thank Dagmar Mezger and Astrid Stadtaus for their support with the laboratory analyses, Sven Schabel and Anja Neuberdt for assistance with the pot experiments, Jens Möhring for statistical advice and Nicole Gaudet for revising the manuscript.

Author Contributions: A.E. and I.-M.B. conceived and designed the experiments; A.E. performed the experiments and analysed the data; S.L. and J.B. developed the manure processing technology and contributed materials to be tested; A.E. and I.L. are the lead authors and all authors contributed to paper writing.

Conflicts of Interest: The authors declare no conflict of interest. The founding sponsors had no role in the design of the study; in the collection, analyses, or interpretation of data; in the writing of the manuscript, and in the decision to publish the results.

Appendix A

Table A1. List of additionally analysed parameters measured in phosphate salts (P-Salt) and biochar and methods used.

Parameter	Unit	Method	P-Salt	Biochar
Calcium (Ca)	% DM	DIN EN ISO 11885	3.3	8.3
Magnesium (Mg)	% DM		2.7	3.9
Sodium (Na)	mg/kg		17,600	5,310
Boron (B)	mg/kg		39.1	98.2
Cobalt (Co)	mg/kg		<5.00	5.52
Manganese (Mn)	mg/kg		588	1070
Molybdenum (Mo)	mg/kg		15.3	10.9
Selenium (Se)	mg/kg	DIN EN ISO 17294-2 (E29)	5.8	<2.0
Iron (Fe)	mg/kg	DIN EN ISO 11885	2200	2300
Aluminium (Al)	mg/kg		280	870
Lead (Pb)	mg/kg		<5.0	<5.0
Cadmium (Cd)	mg/kg		0.6	<0.5
Chrome (Cr)	mg/kg		5.9	11.0
Copper (Cu)	mg/kg		226	158
Nickel (Ni)	mg/kg		8.2	7.9
Zinc (Zn)	mg/kg		2390	1500
Arsenic (As)	mg/kg		<4.0	<4.0
Thallium (Tl)	mg/kg	DIN EN ISO 17294-2 (E29)	0.3	<0.2
Mercury (Hg)	mg/kg	DIN EN 1483-E12-4	0.07	<0.05

DM, dry matter; DIN, German Organization for Standardization; EN, European Standard; ISO, International Standards Organization.

Table A2. Mean dry matter yield (DMY) of barley and bean treated with increasing biochar (BC) concentrations (n = 4).

		Clay				**Sand**			
		Control	**0.1% BC**	**0.2% BC**	**0.5% BC**	**Control**	**0.1% BC**	**0.2% BC**	**0.5% BC**
Barley	g·pot^{-1}	0.20	0.28	0.29	0.24	0.07	0.11	0.15	0.13
Bean	g·pot^{-1}	3.23	3.23	3.87	3.80	2.70	2.83	2.95	3.53

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4 Suitability of phosphates recycled from semi-liquid manure and digestate as alternative fertilisers for ornamentals

In this chapter, the suitability of recycled fertilisers produced from pig manure and biogas digestate was assessed. A pot experiment was carried out to test the effect of these fertilisers on shoot and flower development and P concentration in biomass. Two recycled phosphate salts (from manure and digestate), two dried solid digestates (air-dried and steam-dried), a combination of salt and solid, and commercial triple superphosphate as reference, were applied to sunflower, marigold and Chinese cabbage. Hence, this chapter also addresses this thesis' specific objectives regarding the comparison of recycled and commercial phosphate fertilisers (3), potential interaction effects (4) and differences in their uptake efficiency (5), yet with a focus on ornamentals.

This chapter is published in the journal *Scientia Horticulturae*, as:

Ehmann, A., Bach, I.-M., Bilbao, J., Lewandowski, I. and Müller, T. (2019) Phosphates recycled from semi-liquid manure and digestate are suitable alternative fertilizers for ornamentals. *Scientia Horticulturae* 243, 440–450: doi: 10.1016/j.scienta.2018.08.052

Scientia Horticulturae 243 (2019) 440–450

Contents lists available at ScienceDirect

Scientia Horticulturae

journal homepage: www.elsevier.com/locate/scihorti

Phosphates recycled from semi-liquid manure and digestate are suitable alternative fertilizers for ornamentals

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ARTICLE INFO

Keywords:
Recycled phosphorus fertilizer
Digestates
Semi-liquid pig manure
Sunflower
Marigold
Chinese cabbage

ABSTRACT

In several regions in Europe, the amounts of both manure produced by pig husbandry and biogas digestates from anaerobic digestion are too high to be sustainably applied to the surrounding fields. In these regions, nutrient surpluses are therefore often a problem. The research projects GOBi and BioEcoSIM succeeded in developing innovative recycling technologies for the recovery of phosphorus (P) from biogas digestates and manure, converting them into valuable fertilizers. This study tested the suitability of recovered phosphate salts ("P-Salts") and dried solids as P fertilizers for sunflower, marigold and Chinese cabbage in a greenhouse experiment. Treatments included two recovered P-Salts (from manure and digestate), two dried solids (air-dried and steam-dried), a combination of salt and solid, and triple superphosphate (TSP) as reference, each at two fertilization levels. Measurements included biomass production (ornamentals separated into shoots and flowers), P concentration in the biomass and plant-available P in the growing medium. Both P-Salts had more or less the same effect as TSP on biomass production. The combination of P-Salt and air-dried solids resulted in a synergistic effect on sunflower in terms of biomass yield, P concentration and number of flowers. The P concentration was mostly higher in plants treated at the higher P fertilizer level.

A fast P uptake into plants and thus high plant availability is particularly important in the horticultural sector due to the short production periods of potted plants. In general, all the tested recycled products except the air-dried solids could be adapted to the requirements of different ornamentals, met their P demand as efficiently as TSP and thus have high potential as P fertilizers. The P-Salts are more suitable for short-term and the steam-dried solids more for long-term P supply. The combination of both may ensure optimal P supply and guarantee long-term product quality.

1. Introduction

Phosphorus (P) is required for good flowering quality and quantity of ornamental plants. Nowadays, it is mainly applied in the form of fertilizer manufactured from phosphate-rich rocks. It is well-known that fossil P resources are limited. Assuming future consumption continues to increase at a constant rate, the economically exploitable reserves will be exhausted in about 350 years (USGS, 2016). Total resources are estimated to last up to 1300 years (USGS, 2016). However, there is a high degree of uncertainty in this prediction as it includes all naturally occurring material for which an economic extraction is currently or potentially feasible. Today, the entire P requirements for chemical fertilizers and feed are derived from phosphate-rich rocks. About 75% of the identified global reserves are located in Morocco (Western Sahara), which is also the main exporter of phosphate ore (Schoumans et al., 2015). Koppelaar and Weikard (2013) reported that 17.6 Mt of P were utilized in fertilizer production in 2009, representing more than 80% of the total mined P. The manufacturing process of chemical P fertilizers produces waste that contaminates soil and water resources and the use of these fertilizers contributes to heavy metal contamination of soils, resulting in increased expenses for soil remediation (Moura Filhio and Dantas Alencar, 2008).

The high demand for phosphate fertilizers in food and flower production makes finding affordable alternative products crucial. Such alternatives should be available in relatively high quantities, have consistent quality and equivalent fertilization effects and plant nutrient availability to conventional fertilizers.

One possibility is the recycling of P from manure and biogas digestates produced in agriculture. The accumulation of large amounts of

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https://doi.org/10.1016/j.scienta.2018.08.052
Received 9 August 2017; Received in revised form 9 May 2018; Accepted 26 August 2018

semi-liquid manure is a particular problem in regions with intensive livestock production. Moreover, livestock husbandry is often found in combination with anaerobic digestion as an efficient method of converting animal manures into biogas and heat by co-fermentation with energy crops (Nkoa, 2014), resulting in biogas digestates. In Germany, the amount of biogas digestates is estimated to be around 65.5 million m^3 per year (Möller and Müller, 2012). The quantities of manure and digestate produced are likely to increase in future due to ongoing intensification in livestock breeding, worldwide trends in energy consumption and the predicted need for a 30% increase in energy production in the next 25 years, especially from renewable energy (IEA, 2015).

Both manure and digestates are particularly nutrient-rich and their positive effect on crop growth has frequently been demonstrated. These positive effects are mainly attributed to the supply of nitrogen and phosphate (Alburquerque et al., 2012), and the return of organic matter (Möller and Müller, 2012).

However, farmers are often reluctant to use organic fertilizers as the release of nutrients is slower and more weather-dependent compared to soluble chemical fertilizers (Figueiredo et al., 2008). In addition, the high water content (around 90%) of manure and digestates (Risberg et al., 2017) makes the application of large quantities per hectare necessary and renders the handling and transport in horticulture challenging. Moreover, untreated anaerobic digestate may not always be a perfect organic fertilizer due to its unbalanced nutrient ratio (Westerman and Bicudo, 2005) and specific requirements for soil application techniques as a consequence of the increasing necessity to avoid ammonia losses (Kreith and Tchobanoglous, 2002; BMEL, 2017).

In order to produce fertilizers with reduced volume that can be easily stored, transported and applied, efficient solutions are required to increase the nutrient content and decrease the water content of residues such as digestates and manure. The plant availability of P in recovered products is often low, or at least unpredictable (Kahiluoto et al., 2015). Bilbao et al. (2017) have developed a recovering process for manure and digestates in which 95% of the insoluble P is first converted into a dissolved form. The pretreated manure or digestate is then subjected to a solid-liquid separation. Phosphate salts (P-Salts) are obtained from the liquid fraction by precipitation. The resulting P-Salts are in the form of a powder that can be dried and granulated and, as such, are easy to dose and mix with horticultural growing media. The separated solid fractions of digestate and manure are also dried and used as organic P fertilizer with a texture comparable to wood shavings.

All of these recycled P-fertilizers are expected to have a high potential as alternative P-fertilizers (Bilbao et al., 2017); the manure-based P-Salt has already been found suitable for barley and faba bean (Ehmann et al., 2017). As horticulture is a business which is fairly location-independent, particularly the protected production in greenhouses, a dry and transportable form of recovered P fertilizers would be of considerable interest. The P-Salts and dried solids can be applied separately or in combination. The combined application of mineral and organic fertilizers in the form of TSP and compost was shown to result in increased plant P availability in a greenhouse study with maize (Muhammad et al., 2007). For this reason, we included a combination of P-Salt and dried solids in order to evaluate a potential synergy effect of these two components. This combined treatment may also provide a nutrient ratio more suited to the crops' requirements.

The recycled P fertilizers were tested in a greenhouse experiment with, sunflower (*Helianthus annuus* L.) and marigold (*Tagetes erecta* L.), both of which are among the most prominent ornamentals in Germany. Sunflower ranks fifth in sales of cut flowers in Germany, with a volume of 120 Mio. € per year (AMI, 2016). Marigold, a member of the family *Asteracaea* or *Compositae*, is an important commercial flower that is gaining popularity on account of its easy cultivation and wide adaptability (Asif, 2008). Both plants are marketed as cut flowers and potted flowers. Marigolds are often used for flower beds and for making garlands. Single sunflowers grown and sold in pots achieve high profit margins.

Chinese cabbage (*Brassica campestris* L. var. pekinensis Lour (Olson)) was included as a P sensitive indicator test crop. Symptoms of P deficiency are shown immediately through purpling of the leaves. Crops of the *Brassica* genus are among the ten most important vegetables in economic terms on global agricultural markets. Chinese cabbage is a cole crop plant and is an important fresh and processed vegetable, especially in Asian countries. In 2016, approx. 38.000 t were produced on 850 ha in Germany (Destatis, 2017).

The objectives of this study were: 1) to test the suitability as P fertilizers of P-Salts recovered from semi-liquid pig manure and biogas digestates and of dried solid fractions of non-treated digestates in two ornamentals and one vegetable; 2) to assess the competitiveness of these alternative P fertilizers compared to conventional superphosphate; 3) to determine whether the combined application of P-Salt and dried solid digestate improves the fertilizing performance through synergy effects; and 4) to assess the role of P fertilization in the flowering of sunflower and marigold, comparing the effect of the recycled fertilizers and commercial TSP.

Based on these objectives, the following hypotheses were set up:

- The effect of P-Salts recovered from semi-liquid manure and digestates, and the two dried solid fractions on biomass production and P concentration of sunflower, marigold and Chinese cabbage is equivalent to that of the conventional P fertilizer triple superphosphate (TSP).
- The combination of P-Salt and separated solids has a synergistic effect on plant growth.
- Recycled fertilizers enhance flowering in the same way as TSP and the level of P influences the number of flowers

These hypotheses were tested by means of a pot experiment with sunflower, marigold and Chinese cabbage.

2. Material and methods

2.1. Production of P-Salts and solids from pig manure and biogas digestate

The P-Salts were recovered from acidified semi-liquid pig manure (P-Salt_manure) and biogas digestate (P-Salt_digestate) as described by Bilbao et al. (2017). The dried solids were obtained from untreated digestate purely through solid-liquid separation. The solid fraction was dried either in warm air at 40 °C (air-dried solids) or with superheated steam at 120 °C (steam-dried solids).

The P-Salts had P concentrations approximately 5 times higher than the dried solids (Table 1). Soluble plant-available P fractions were determined for all products, Hedley fractionation was only performed for the digestate-based products (Tables 2 and 3). Both P-Salts are mixtures of magnesium ammonium phosphate (struvite) and calcium phosphates. The P-Salt_manure also contained 2.4% N, 1.3% K, 10.0% Ca and 4.8% Mg and the P-Salt_digestate 1.3% N, 1.0% K, 17.0% Ca and 5.0% Mg in the fresh matter.

Table 1

P concentration of the fertilizers.

Fertilizer	Acronym	Dry matter in % FM	P in % FM
P-Salt recovered from semi-liquid pig manure	P-Salt_manure	68.6	10.5
P-Salt recovered from digestate	P-Salt_digestate	69.7	10.7
Steam-dried separated solids from digestate	Steam-dried solids	91.6	2.3
Air-dried separated solids from digestate	Air-dried solids	95.4	2.1
Mineral P fertilizer as reference (Triple superphosphate)	TSP	–	19.0

FM, fresh matter; dry matter determined according to DIN EN 12880; P determined according to DIN EN ISO 11885.

Table 2
Total and soluble P, total organic carbon and pH of the fertilizers.

Property	P-Salt_manure	P-Salt_digestate	Steam-dried solids	Air-dried solids	Method
P_{total} in % FM	10.5	10.7	2.3	2.1	DIN EN 12880
Water-soluble P in % FM	0.28	0.13	0.35	0.35	VDLUFA II, 4.1.4
Neutral ammonium citrate-soluble P in % FM	11.47	10.42	0.90	1.06	VDLUFA II, 4.1.4
Formic acid-soluble P in % FM	11.38	10.12	0.92	1.06	VDLUFA II, 4.1.2
Citric acid-soluble P in % FM	11.42	10.38	0.92	1.07	VDLUFA II, 4.1.3
Mineral acid-soluble P in % FM	11.47	10.46	1.00	1.11	VDLUFA II, 4.1.1.4
Total organic carbon in % FM	1.43	3.58	38.90	38.40	VDLUFA II, 10.2
pH in $CaCl_2$	7.9	8.3	8.5	7.1	DIN EN 12176

Table 3
Total and fractionated P of the digestate-based products.

Variable	P-Salt_digestate	Steam-dried solids	Air-dried solids
P_{total} in % FM	10.7	2.3	2.1
Sequentially fractionated with … in mg P (g DM)$^{-1}$ *			
… $NaHCO_3$ (easily available P)	29.9	6.7	7.5
… NaOH	6.3	1.0	1.3
… H_2SO_4 (sparingly available P)	53.3	1.0	1.7

* Determined using a modified Hedley fractionation method (Hedley et al., 1982; Tiessen and Moir, 1993; Redel et al., 2013).

Table 5
Supply target of main nutrients N, P and K in the growing medium.

Nutrient	Sunflower	Marigold	Chinese cabbage
	mg L^{-1} growing medium		
Full supply of P (optimal)*	261.6	130.8	87.2
Reduced supply of P (low)*	130.8	65.4	43.6
Mineral N**	800.0	400.0	800.0
K	830.2	415.1	830.2

* P in growing medium not considered.
** CAT-extractable mineral N and K in growing medium considered (only difference applied).

2.2. Description of the pot experiment

A greenhouse pot experiment assessed the P fertilizing effect of 1) P-Salts recovered from biogas digestate and semi-liquid manure; 2) two different types of dried solids from the separated solid fraction of digestate; and 3) a combination of P-Salt_digestate and air-dried solids; each compared to a conventional mineral fertilizer.

The growing medium used was chosen specifically because it had the lowest P concentration of all media available (Table 4).

The growing medium was thoroughly mixed with varying amounts of P-Salts, solids, a combination of P-Salt and solids, or triple superphosphate (TSP) and filled into pots. The application rates of the P fertilizers were adapted to the optimal P supply for each of the three test species: sunflower, marigold and Chinese cabbage (Table 5). Phosphorus in the growing medium was not considered as it was very low. In the combined treatments, 50% of the P was applied as P-Salt_digestate and 50% as air-dried solids. In addition, a reduced P dose (50%) was included for all treatments to simulate P shortage and depict the dose-response effect of two P supply levels. A variant without any additional P was included as control.

Nitrogen and potassium were added to all pots including the control at the beginning of the experiment as solutions of NH_4NO_3 and K_2SO_4, respectively, in optimal amounts for each species (Table 5, Table S1). Additional N and K supply was corrected for the CAT-extractable fractions in the growing medium, whereas N and K in the recycled fertilizers were negligible and thus not considered. It was assumed that CAT extraction determined the total nitrate N quantitatively and the easily exchangeable ammonium N and K fractions, and that these were all in plant-available form.

Table 4
Characterization of the growing medium.

Raw material	95% upland peat (H3-H8), clay granules, quartz sand, lime, NPK fertilizer; pH 5.8 ($CaCl_2$), salinity 1.2 g L^{-1} (KCl), EC 1875 µS cm^{-1}
Manufacturer	ASB Grünland GmbH, Germany
Nutrients (CAT)	mg L^{-1}
Nitrogen (N)	200
Phosphorus (P)	21.8
Potassium (K)	124.5
Magnesium (Mg)	100

CAT, extraction with calcium chloride and DTPA (diethylenetriaminepentaacetic acid).

Plants were pre-cultivated in germination trays without any additional fertilizer for two weeks before transplanting into prepared pots: sunflower and Chinese cabbage in 0.6 L pots of 12-cm diameter and marigold in 0.4 L pots of 10-cm diameter.

The pots were set up on tables in a greenhouse in a randomized complete block design with four replications and ten plants per replication, resulting in a total of 1560 pots for 39 treatments. The pots were irrigated from above following good horticultural practice, redundant water was allowed to drain from the pots. Additional lighting was provided during the first two weeks only. The air temperature in the greenhouse was approx. 26 °C during the day and 16 °C at night. Temperature was controlled by automatically opening windows. On days with very high light intensity, the greenhouse was automatically shaded. The sunflower and marigold were treated once against thrips with abamectin (Agrimec Pro, Syngenta Agro GmbH). Yellow and blue adhesive panels were hung up during the entire cultivation period to control whitefly and thrips. The Chinese cabbage pots were moved further apart twice in order to provide sufficient space for growth.

The marigold and sunflower were harvested once they had reached the flowering stage after 5 and 8 weeks, respectively. The Chinese cabbage was harvested after nine weeks. SPAD readings were carried out on cabbage (youngest fully developed leaf) using a Konica Minolta SPAD-502Plus. In all three plants, the shoots were cut 0.5 cm above the surface. The fully developed flowers of sunflower and marigold were counted and separated from the shoot, both weighed and then dried at 60 °C. Chinese cabbage leaves were counted and dried at 60 °C. Dry weight was determined and dry matter content calculated. Samples of the growing medium were taken from all pots individually before and after the experiment.

2.3. Sample analyses

The dried shoots and flowers were ground to approx. 1 mm in a cutting mill (SM200; Retsch GmbH, Haan, Germany). Concentration of P was determined using microwave digestion followed by ICP-OES measurement (DIN EN ISO 11885). Plant P content was calculated from dry matter yield and P concentration. The growing medium samples were dried at 105 °C,

Table 6
N and K concentrations in biomass and pH in growing medium of four treatments.

	Treatment	N in plant biomass mg (g DM)$^{-1}$	K in plant biomass mg (g DM)$^{-1}$	pH in growing medium
Sunflower shoots (without flowers)	P-Salt_manure	50.2	53.2	4.5
	Steam-dried solids	68.4	56.9	5.0
	TSP	59.4	55.6	4.6
	Control	69.8	52.2	4.5
Sunflower flowers	P-Salt_manure	71.3	30.9	Same pots as sunflower shoots
	Steam-dried solids	81.8	30.2	
	TSP	84.4	32.1	
	Control	87.5	29.0	
Chinese cabbage (whole plant)	P-Salt_manure	53.4	24.3	5.1
	Steam-dried solids	69.2	21.2	5.1
	TSP	50.1	21.6	5.1
	Control	68.2	20.2	5.2

sieved to 2 mm and then analysed for plant-available P (P(CAL)) using calcium-acetate-lactate extraction followed by flame photometer measurement (OENORM L 1087:2012-1). Unfortunately, the samples of marigold from the harvest date could not be analysed, as they were mislaid.

In order to ensure a sound experimental approach with comparable conditions in all pots and to ensure that side effects of other main plant nutrients are kept to a minimum, the N and K concentrations in sunflower and Chinese cabbage plants of four treatments were analysed (DIN EN 13654-2 and DIN EN ISO 11885, respectively). In addition, the pH was determined in the corresponding samples of growing media taken after harvest. Results (Table 6) showed that differences in N and K in plants and pH of medium between treatments were moderate and also did not reveal any clear pattern.

2.4. Evaluation of synergistic effects and P use efficiency

To evaluate the effects of the combined application of P-Salt_digestate and air-dried solids, theoretical biomass production was calculated from P-Salt_digestate and air-dried solids applied alone using the following equation:

$$\text{Theoretical biomass production} = (\text{Biomass yield}_{\text{P-Salt\_digestate}} + \text{Biomass yield}_{\text{air-dried solids}}) / 2 \quad (1)$$

where Biomass yield$_{\text{P-Salt\_digestate}}$ is the biomass obtained with the application of P-Salt_digestate only and Biomass yield$_{\text{air-dried solids}}$ is the biomass obtained with the application of air-dried solids only.

Actually measured biomass yields that are higher than the calculated theoretical values indicate positive (synergistic) effects of the combined application and lower values indicate non-synergistic effects.

The P use efficiency was calculated using Equation [2] in order to compare the three species with regard to this parameter.

$$\text{P use efficiency} = ((\text{P content in fertilized plant} - \text{P content in untreated plant})/\text{P supply from fertilizer}) * 100 \quad (2)$$

2.5. Statistical analysis

Data analysis was performed using SAS software version 9.3 (SAS Institute Inc., Cary, NC, USA). Plant and growing medium data were subjected to a two-factor analysis of variance (ANOVA). Treatments and fertilization level were treated as fixed effects. Data were log-transformed where necessary. The graphs shown here were plotted with back-transformed data and simple means. Significance was determined at $P \leq 0.05$ using a multiple t-test, performed only on finding significant differences in the F-test. Significantly different means are indicated by different letters or mentioned in the text. The letters display the difference between marginal means of treatments across levels.

3. Results

3.1. Sunflower

The shoot biomass of sunflower was significantly higher in the treatments with TSP, steam-dried solids and the combination than in the control (Fig. 1). The flower biomass was higher for steam-dried solids than for TSP. There was no significant difference in flower biomass between TSP and the combination. By contrast, the shoot and flower biomass in the P-Salt treatments was comparable to that of the control. The two P-Salts performed similarly. The steam-dried solids induced significantly higher shoot and flower biomass than the air-dried solids. There were no relevant differences in shoot and flower biomass between the two fertilization levels. However, the number of

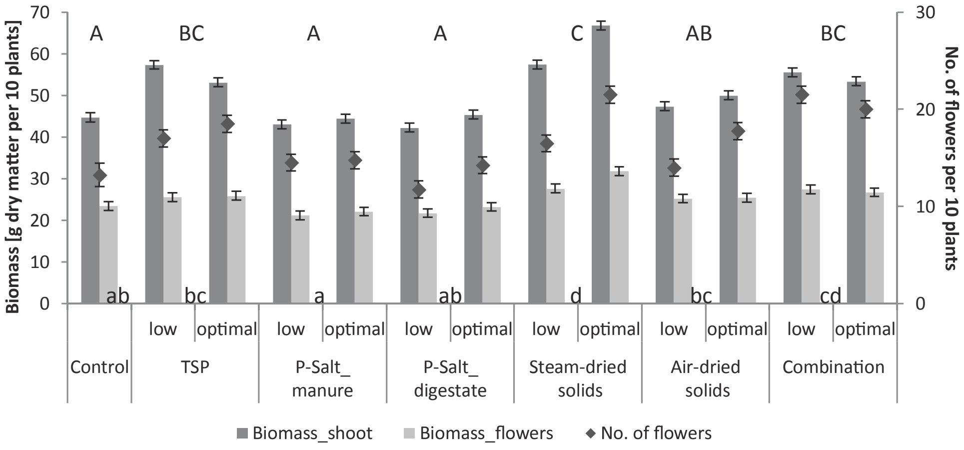

Fig. 1. Mean dry matter biomass of shoots (Biomass_shoot) and flowers (Biomass_flowers) and number of flowers of sunflower treated with P-Salts and dried solids compared to control and triple superphosphate (TSP). The error bars indicate pooled standard errors of the means; marginal means with identical letters are not significantly different from each other (upper-case for Biomass_shoot, lower-case for Biomass_flowers, $\alpha = 0.05$, $n = 4$) across the fertilization levels “low” and “optimal”. For explanation of treatments, see 2.2.

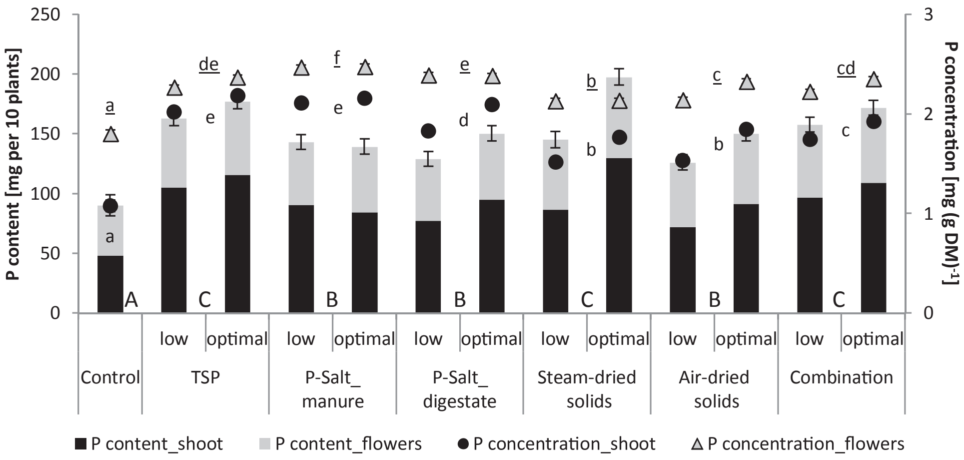

Fig. 2. P content and P concentration of sunflower shoots and flowers treated with P-Salts and dried solids compared to control and triple superphosphate (TSP). The error bars indicate pooled standard errors of the means; marginal means with identical letters are not significantly different from each other (upper-case for total P content (shoot + flower), lower-cases for shoot P concentration, underlined lower-case for flower P concentration, α = 0.05, n = 4) across the fertilization levels "low" and "optimal". For explanation of treatments, see 2.2.

flowers per 10 plants was significantly higher for the optimal fertilizer level in most treatments than for the low level and the control (Fig. 1, significant differences not indicated). The highest number of flowers was observed in plants treated with steam-dried solids and the combination, followed by TSP and air-dried solids. The difference between air-dried solids and steam-dried solids was significant. The P-Salt treatments led to a similar number of flowers as the control.

The P content of sunflower was significantly increased by the application of TSP, steam-dried solids and the combination compared to the other treatments (Fig. 2). The difference between the P-Salts was not significant. However, the steam-dried solids resulted in significantly higher P contents than the air-dried solids. As expected, both fertilization levels led to significant differences in P content with highest values for the optimal level, followed by the low level and then the control.

The shoot P concentration was significantly higher in the treatments with P-Salt_manure and TSP compared to the other treatments (Fig. 2). The P-Salt_digestate treatment showed lower shoot P concentrations than P-Salt_manure, but performed better than the combination and the solids. There was no significant difference between steam-dried and air-dried solids here. The highest flower P concentration was found for P-Salt_manure, followed by P-Salt_digestate, TSP, the combination and the solids. The air-dried solids led to a higher flower P concentration than the steam-dried solids. The fertilization level significantly influenced the P concentration of both shoot and flower, with highest P values for the optimal P level and lowest for the control.

3.2. Marigold

Significant differences in shoot biomass production of marigold were only found between the treatments with manure-based P-Salt and air-dried solids (Fig. 3). The flower biomass did not differ from the control in any of the treatments (Fig. 3). The same applies to the number of flowers (between 1 and 10 flowers per 10 plants, data not shown).

Shoot P concentration of marigold was higher in all treatments than in the control. The highest concentration in absolute numbers was found in plants fertilized with TSP, followed by the P-Salts, the combination, the steam-dried and finally the air-dried solids. The shoot P concentration of plants treated with the combination was comparable to that of plants receiving P-Salts or steam-dried solids, but significantly higher than that of plants treated with air-dried solids (Fig. 4).

The flower P concentration showed a different pattern to that of shoot P concentration, but without any clear trend. The P-Salts and the solids led to significantly higher values than the control. The combination and TSP had results in between these values and that of the control (Fig. 4).

Total P contents of both P-Salt treatments, steam-dried solids and TSP were significantly higher than for air-dried solids and the control. Total P content of the combined treatment was only higher than the control (Fig. 4).

The fertilization level only influenced the P concentration in shoots and flowers ($P < 0.0001$). The shoot P concentration differed significantly between the two levels and the control, with highest values at the optimal level. The flower P concentration was higher at the optimal level than at the low level and the control.

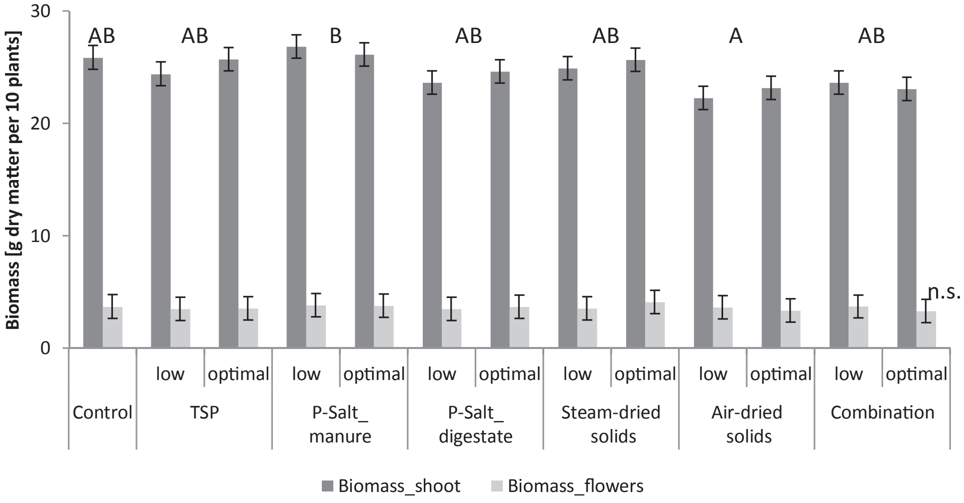

Fig. 3. Mean dry matter biomass production of shoots and flowers of marigold treated with P-Salts and dried solids compared to control and triple superphosphate (TSP). The error bars indicate pooled standard errors of the means; marginal means with identical letters are not significantly different from each other (upper-case for Biomass_shoot, not significant for Biomass_flowers, α = 0.05, n = 4) across the fertilization levels "low" and "optimal". For explanation of treatments, see 2.2.

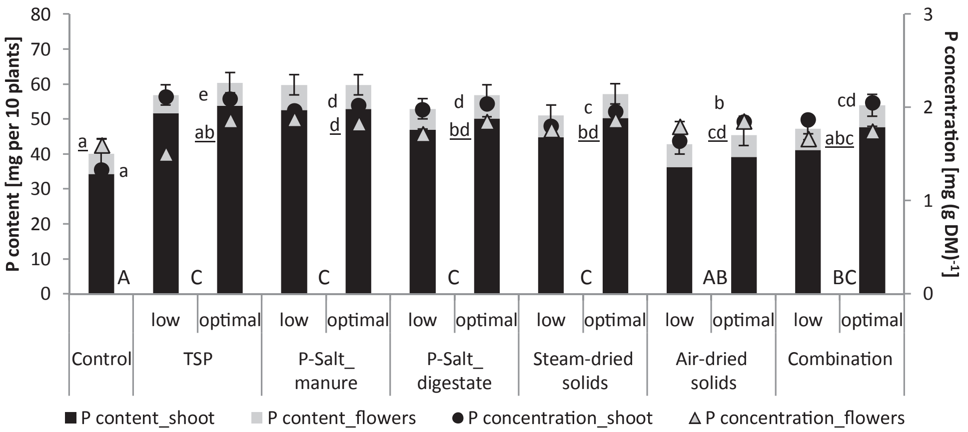

Fig. 4. P content and P concentration of marigold shoots and flowers treated with P-Salts and dried solids compared to control and triple superphosphate (TSP). The error bars indicate pooled standard errors of the means; marginal means with identical letters are not significantly different from each other (upper-case for total P content (shoot + flower), lower-case for shoot P concentration and underlined lower-case for flower P concentration, α = 0.05, n = 4) across the fertilization levels "low" and "optimal". For explanation of treatments, see 2.2.

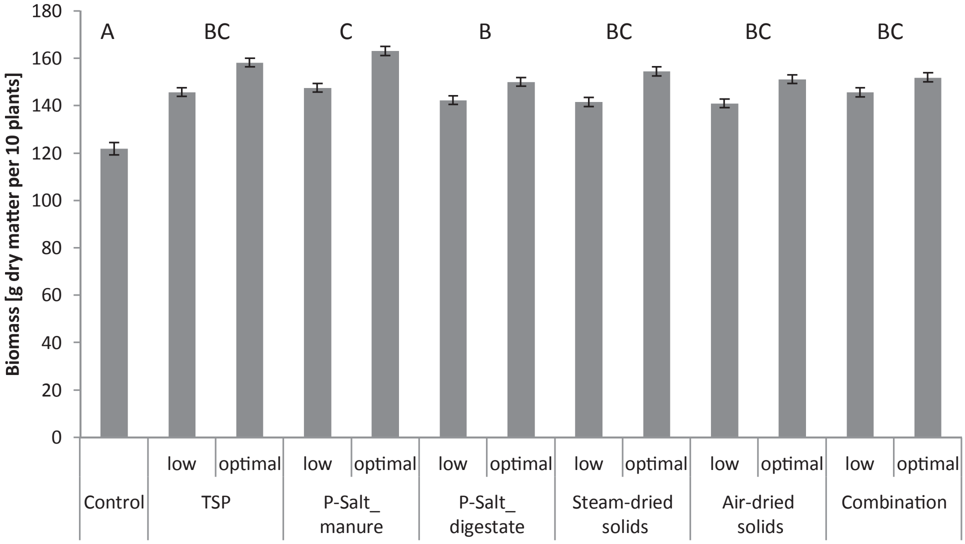

Fig. 5. Mean dry matter biomass production of Chinese cabbage treated with P-Salts and dried solids compared to control and triple superphosphate (TSP). The error bars indicate pooled standard errors of the means; marginal means with identical letters are not significantly different from each other (α = 0.05, n = 4) across the fertilization levels "low" and "optimal". For explanation of treatments, see 2.2.

3.3. Chinese cabbage

The dry matter yield of cabbage was significantly higher in all treatments than in the control. The highest DMY in absolute terms was found with manure-based P-Salt, yet this was only significantly different from the treatment with P-Salt_digestate (Fig. 5).

The highest P concentration was found in the cabbage plants treated with the two P-Salts and TSP, followed by the combination and both solids. The control had the lowest P concentration (Fig. 6).

The P content of the cabbage plants followed the same pattern as the P concentration (Fig. 6).

The fertilization level clearly influenced the DMY, P concentration and P content: all three variables were higher at the optimal level than at the low level and lowest in the control ($P < 0.0001$).

The number of leaves was significantly higher in all treatments than in the control (data not shown). Plants treated with the manure-based P-Salt developed the most leaves. In contrast, the highest SPAD readings were recorded for the leaves of the control plants, followed by the solids and then the other treatments, as was expected. Both parameters were significantly influenced by the fertilization level.

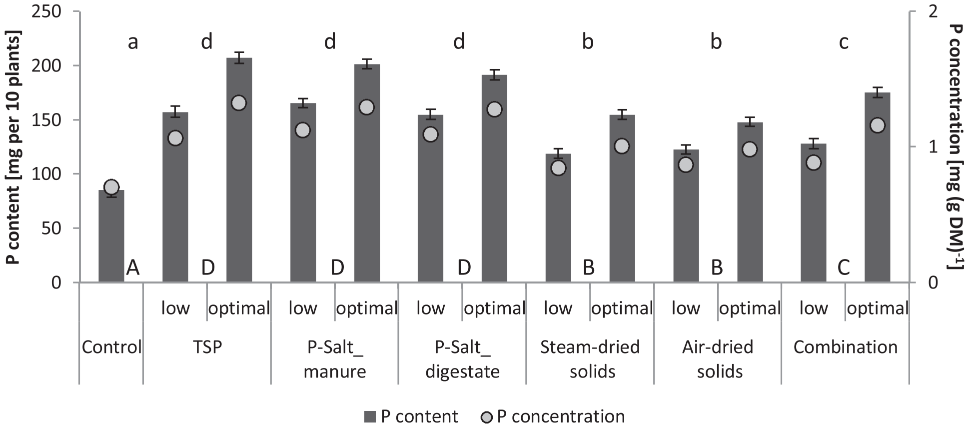

Fig. 6. P content and P concentration of Chinese cabbage treated with P-Salts and dried solids compared to control and triple superphosphate (TSP). The error bars indicate pooled standard errors of the means; marginal means with identical letters are not significantly different from each other (upper-case for P content, lower-case for P concentration, α = 0.05, n = 4) across the fertilization levels "low" and "optimal". For explanation of treatments, see 2.2.

Table 7
Evaluation of the synergistic effects of the combination treatment (P-Salt_digestate + air-dried solids). Theoretical biomass production was calculated using Equation 1 (Material and methods 2.4) and compared to the actual shoot and flower biomass in sunflower and marigold, and the actual total biomass in Chinese cabbage.

Plant species	Measurement	Combination			
		Low fertilization		Optimal fertilization	
		Shoot DM g per 10 plants	Flower DM	Shoot DM	Flower DM
Sunflower	Theoretical biomass production	44.81	23.49	47.70	24.35
	Biomass production measured	55.58	27.44	53.36	26.72
Marigold	Theoretical biomass production	22.96	3.57	23.89	3.51
	Biomass production measured	23.64	3.7	23.08	3.31
Chinese cabbage	Theoretical biomass production	141.69	–	150.59	–
	Biomass production measured	145.63	–	151.93	–

DM, dry matter.

3.4. Synergistic effects of the combined treatments

In sunflower, the actual shoot and flower biomass measured was higher than the theoretical biomass at both fertilization levels. In marigold, the same effect was visible, yet only for the low fertilization level. At the optimal level, the theoretical biomass was higher than the biomass measured. Chinese cabbage had higher actual biomass than theoretical biomass, at both fertilization levels (Table 7).

3.5. P use efficiency

The highest mean P use efficiency of the three crops was found in Chinese cabbage at 16% for the optimal and 19% for the low fertilization level. The percentages for sunflower (4% and 6% respectively) and marigold (2 and 3%), calculated for whole plants, were considerably lower (Table S2).

3.6. P concentration in growing medium

In sunflower, the highest P(CAL) contents at the beginning of the experiment were found in the pots fertilized with optimal levels of TSP and the P-Salts (Table 8). The P(CAL) contents in the treatments with P-Salt_digestate and both solids increased throughout the experiment. The P(CAL) was higher for steam-dried solids than for air-dried solids.

In Chinese cabbage, this was completely different. Here, the P(CAL) contents were higher at the beginning of the experiment than at the end in all treatments tested. The highest contents were found for the optimal levels of P-Salt_manure and the combination. The P(CAL) at the optimal level of P-Salt_manure (53.60 mg P (100 g medium)$^{-1}$) was considerably higher at the beginning of the experiment than at the optimal level of P-Salt_digestate (35.00 mg P (100 g medium)$^{-1}$).

For all treatments and all three crops, P(CAL) contents were mostly higher at the optimal levels than at the low levels.

4. Discussion

4.1. General comments

Overall, the yields and P concentrations of sunflower, marigold and Chinese cabbage observed in this study are comparable to the findings of related studies, including some using test soils low and high in P (Gunes et al., 2009).

In our study, the mean shoot P concentration of sunflower at 1.9 mg g^{-1} DM^{-1} can be seen as somewhat marginal (according to the classification by NSAC (2017) which rates P concentrations between 1.5 and 2.4 mg g^{-1} DM^{-1}as marginal), but not yet deficient. The NSAC classification levels refer to the top one to three most mature leaves collected at bud stage. Although we measured P in biomass of the entire shoot at flowering stage, our results seem comparable, as P concentration usually decreases over time. Vogel et al. (2015) tested struvite (magnesium ammonium phosphate) from wastewater compared to TSP in sunflower and found similar shoot P concentrations of 2.53 (struvite) and 2.11 mg g^{-1} DM^{-1} (TSP).

For marigold, the flower P concentration of all treated plants averaged 1.8 mg g^{-1} DM^{-1} in our study. A study by Naik (2015) found 2.6 mg P g^{-1} DM^{-1}, but the amount of P fertilizer was given on a per-hectare base and therefore not comparable. In contrast, Zeljković et al. (2013) found a much lower P concentration of only 0.53 mg P g^{-1} DM^{-1}in French marigold. Despite this low P concentration, which may be a consequence of lower P supply, they obtained biomass yields between 2.4–3.1 g DM per plant, which is comparable to those in our study.

In Chinese cabbage, the P concentration ranged from 0.84 mg g^{-1} DM^{-1}(steam-dried solids, low) to 1.32 mg g^{-1} DM^{-1}(TSP, high). Other studies have reported much higher values. For example, Li and Zhao (2003) found 5.2 mg g^{-1} DM^{-1}for struvite and 2.9 mg g^{-1} DM^{-1}for NP fertilizer and Ryu et al. (2012) found 5.4 mg g^{-1} DM^{-1}(struvite), 3.5 mg g^{-1} DM^{-1}(NPK fertilizer) and 1.8 mg g^{-1} DM^{-1}(compost). Chinese cabbage usually responds very sensitively to P deficiency.

Table 8
P(CAL) contents in the growing medium at the beginning and at the end of the experiment for the test crops and treatments.

		Control	TSP		P-Salt_ manure		P-Salt_ digestate		Steam-dried solids		Air-dried solids		Combination		LSD
			low	optimal	low	optimal	low	optimal	low	optimal	low	optimal	low	optimal	
		mg P (100 g medium)$^{-1}$													
Sunflower	start	15.20	63.80	122.80	73.60	120.80	70.20	131.80	31.80	48.60	21.60	33.20	64.80	80.00	–
	end	16.24	49.59	109.39	72.37	69.58	91.22	150.17	29.84	58.14	28.62	42.12	32.88	61.85	0.3993
Marigold	start	15.20	46.20	70.40	46.00	75.20	54.40	50.80	24.20	33.80	22.80	26.80	30.60	52.00	–
	end*	–	–	–	–	–	–	–	–	–	–	–	–	–	–
Chinese cabbage	start	15.20	30.00	46.00	32.60	53.60	28.60	35.00	19.20	38.00	17.40	25.00	24.40	54.00	–
	end	10.02	15.74	17.39	14.27	15.52	17.64	18.99	13.17	14.87	14.32	13.47	11.62	16.12	0.3551

LSD, least significant difference.

* Samples from marigold were not analysed at the end of the experiment.

Symptoms include colour change to a greyish green or purple and small, possibly deformed leaves. As this was not observed in our study, it can be concluded that the P supply was sufficient, at least until the time of harvest. The mean P use efficiency of Chinese cabbage observed here was rather high at 16% for the optimal and 19% for the low fertilization level, particularly with regard to the limited cultivation time and compared to the other two species.

4.2. Effect of P-Salts and solids on biomass production and P concentration

4.2.1. Steam-dried vs. air-dried solids

The results showed that the steam-dried solids performed significantly better than the air-dried solids in terms of biomass in sunflower and Chinese cabbage. This trend was also seen to a certain extent in marigold, but was not significant. In sunflower and marigold, the P content of the biomass was also higher for the steam-dried than for the air-dried solids.

This effect of the steam-dried solids was not expected, as it was assumed that the higher drying temperature might negatively influence the P availability. In addition, the proportions of easily extractable $NaHCO_3$-soluble P were found to be lower in the steam-dried solids than in the air-dried solids. Nevertheless, the actual plant availability of P was higher in the steam-dried solids: fertilization with steam-dried solids led to higher P(CAL) contents in the growing medium in all three crops and at both levels than with air-dried solids, despite application of the same amount of total P.

The P solubility may have been altered during the steam drying process, either through the heat or possibly also through condensation on the surface of the solids. As steam is often used to increase the turnover and digestibility of organic matter (Hendriks and Zeeman, 2009), it may also have enhanced the release of P in this case by breaking down certain chemical compounds. The more P is released from the organic matter, the more is expected to be subsequently mineralized.

Fertilizer P can often be rapidly immobilized after application to the soil (Smit et al., 2009). The different drying conditions could also have affected the process of P immobilization after mixing the solids with the growing medium.

However, although highly energy-efficient, drying with superheated steam is not (yet) a standard technology and only makes sense if available on-site. Where this is not the case, the easier implementation of air-drying using waste heat may compensate for the lower fertilizing effect of the solids.

4.2.2. Manure-based vs. digestate-based P-Salt

The results of this study confirmed that differences in the performance of the two P-Salts are minimal. This is to be expected due to the similarities in the production processes and the chemical composition. Both P-Salts had comparably low proportions of P in easily extractable form. This was clearly visible despite the different analytical approaches used to characterize the P availability of the two P-Salts.

4.2.3. P-Salts vs. TSP

Positive effects of recycled fertilizers such as struvites and calcium phosphates have been reported in the literature for many different crop types. Vogel et al. (2015) found higher P uptake in sunflower treated with struvite than with TSP, and also increased biomass yield following both treatments compared to the control. The biomass P concentrations reported by Vogel et al. (2015) were comparable to those presented in our study. Ryu et al. (2012) found that Chinese cabbage had higher fresh and dry weight and P content when treated with struvite than with organic fertilizers. Li and Zhao (2003) reported that struvite precipitated from landfill leachate is a suitable fertilizer for fast-growing vegetables including Chinese flowering cabbage (*Brassica parachinensis*) and Chinese chard (*Brassica rapa* var. *chinensis*).

In our study, the biomass P content of marigold and Chinese cabbage was the same after application of P-Salts and TSP. However, it has not yet been clarified why these recycled salts are as efficient as they are, given that their P water solubility is usually very low compared to TSP. One of the main reasons for the efficient P uptake from the P-Salts was very likely the low pH of the growing medium (5.8). In addition, it has frequently been hypothesized that plants are able to make use of P with low plant availability – as provided by P-Salts – by changing the conditions in the rhizosphere through the release of organic acids (Jones, 1998; Hinsinger, 2001).

4.2.4. P-Salts vs. solids

The P content of the two ornamentals was comparable or higher in the treatment with steam-dried solids than with P-Salts.

The volume of fertilizer applied was much higher for the solids than for the P-Salts because the solids have a lower P concentration and higher fraction of organic matter. This high volume may have slightly increased and stabilized the low pH of the growing medium, more so for the steam-dried solids (pH 8.5) than for the air-dried solids (pH 7.1). This buffer effect of organic matter has frequently been observed (Bot and Benites, 2005).

The texture of the product groups may be another reason for the differences in P plant availability. Both P-Salts were finely ground powders in contrast to the coarse structure of the solids, which more resembled wood shavings. It is assumed that the P-Salts were able to supply P right from the beginning of the experiment, whereas P from solids had to be mineralized first. This becomes obvious from the analysis of the growing medium at the beginning of the experiment: samples from pots treated with solids had much lower P(CAL) contents than those treated with the P-Salts (Table 8). Thus, P-Salts seem more suitable for crops with high P demand and short growth period.

In contrast to the ornamentals, a slightly different performance of the P-Salts and solids was observed in Chinese cabbage. This was doubtlessly a consequence of the higher biomass growth. The supply of P in form of P-Salts seemed to be adequate for marigold, as it had the shortest cultivation period and the five weeks until harvest were possibly not sufficient for the P from the solids to be converted into a plant-available form. However, species with a longer cultivation period such as sunflower and Chinese cabbage can use the P slowly released from the solids. They develop more biomass and have an accompanying higher P demand, which can be met by the P-Salts or solids. The combination of both allows the P demand to be met in the short term (quickly available P from P-Salts) and in the long term (slowly released P from solids).

4.3. The combination of P-Salt and separated solids has a synergistic effect on plant growth

The comparison between the separate application of P-Salt_digestate and air-dried solids and their combination was included to clarify differences in fertilization effects (synergistic effects) and obtain information on the suitability of the combination of these recycled products as alternative P fertilizers. Currently, there are no comparable products or product combinations known which can be used for a direct comparison.

In our study, P-Salt_digestate was precipitated from the separated liquid fraction of biogas digestates (Bilbao et al., 2017) and then dried. It can be used separately or in combination with air-dried solids depending on the needs of the specific plant and the conditions of the soil or growing medium. This allows as much of the permanently accumulating biogas digestates as possible to be used - but in the form of a dried, storable and transportable product - and the nutrient cycle to be closed by returning nutrients and organic matter to the fields.

The precipitation of calcium or magnesium phosphates or struvite crystallization from various organic resources (Le Corre et al., 2009; Wilsenach et al., 2007; Greaves et al., 2010) is already well known and partly also in practice. Risberg et al. (2017), Brod et al. (2015) and

Holm-Nielsen et al. (2009) reported the generally positive effects of similar organic materials used as fertilizer, improving soil quality and decreasing the need for inorganic fertilizers with limited availability. In addition, several studies have examined the P fertilization effect of organic amendments, pointing out that they provide plants with more P than unfertilized controls (e.g. Waldrip et al., 2011; Requejo and Eichler-Löbermann, 2014; Duong et al., 2012). However, none of these organic materials were subjected to a drying or precipitation process as the products in our study were. Thus, the fertilizers in the cited studies had the disadvantage that they were neither storable for longer periods of time nor easy to handle and transport due to their high water content.

The positive effect of drying products with warm air is commonly used to advantage in the food industry (Pronyk et al., 2004), but as yet not for biogas digestates. Air-drying of solids from separated digestates or manure can also potentially reduce volume, moisture and unpleasant odours as well as stabilize the nutrient content of the separated solids.

In our study, it was observed that the steam-dried solids had an equivalent effect to TSP on plant biomass production in all tested plants. The effect of the digestate-based P-Salt was also similar to that of TSP in marigold and Chinese cabbage. However, the combination of the two had better effects (Table 6) than the separate fertilization with P-Salt or air-dried solids in all plants. The actual biomass production measured tended to be higher than the calculated theoretical biomass production, indicating synergistic effects of the combined application.

One possible reason for positive synergy effects of the combination treatment might be that the air-dried solids contributed positively to the moisture content in the medium and thus promoted the P solubility of the P-Salt by increasing water flow and improving P uptake in the plant.

This is in line with Vanden Nest et al. (2015) who found that the addition of separated solid digestates stimulates increases in soil P availability. In a review of numerous studies, Möller (2015) reported that digestate application at field level enhances soil microbial activity, but that these are short-term effects. It would appear that in a pot experiment with limited duration the air-dried solids can stimulate microbial biomass which then – as suggested by Olander and Vitousek (2000) – mobilizes additional P in the soil or medium through an increase in phosphatase activity. Thus, the P availability in the growing medium is increased and plant uptake facilitated.

In addition, the easily plant-available P fraction (Hedley fractionation) of the air-dried solids was low compared to that of the P-Salt, which may have promoted the enzyme activity and in turn increased the P availability and may also explain a stimulation of the microbial biomass. The negative feedback mechanism of enzyme activity, as described by Olander and Vitousek (2000), implies that enzyme activity is induced and nutrients are mineralized when nutrient supply is low. By contrast, when nutrient supply is high, the enzymes are suppressed and mineralization stops. This may explain the lower yields of plants treated with air-dried solids compared to the combination, because the enzymatic mineralization of P induced a lag phase. The combination treatment can compensate for this lag phase through the direct P fertilization effect of the P-Salt. The low P availability of the air-dried solids in the growing medium was also shown by the low P(CAL) contents throughout the experiment.The higher P(CAL) in the combination treatment during the entire experiment compared to air-dried solids applied alone was thus a result of the P-Salt_digestate. In sunflower for example, the treatment with P-Salt_digestate led to the generally highest P(CAL) contents. Another aspect to be considered is that mineral P is often inorganically immobilized. The application of the P-Salt (mineral P) together with the solids (organic P) might have caused an organic immobilization which was then followed by a slow remineralisation of P.

The higher pH of the P-Salt_digestate (8.3) and the air-dried solids (7.1) already mentioned might have also affected the structure of the growing medium and influenced P mobilization by increasing the pH in the medium to a range more optimal for P uptake.

As P is a major prerequisite for flowering quality and quantity, there is a particularly high demand for P in horticulture. The combination treatment tested in this study can supply flowering ornamentals with both direct and long-term fertilization, thus reducing the need for an additional P application during the cultivation time or even rendering it superfluous. This can also help reduce working hours and costs for personnel and material.

4.4. Recycled fertilizers enhance flowering in the same way as TSP and the level of P influences the number of flowers

In our study, effects of the recycled fertilizers and their different P application levels on flowering were visible for sunflower, but not for marigold. In sunflower, both P-Salts led to the lowest number of flowers, but to the highest P concentration levels in the flower biomass. A higher, similar number of flowers was obtained with air-dried solids and TSP. Steam-dried solids increased the number of flowers further, but both types of solids led to lower flower P concentrations than TSP. The combination induced the highest number of flowers of all treatments, with a higher flower P concentration than with TSP. Thus, the use of recycled fertilizers can definitely reduce the need for chemical fertilizers in the production of flowering plants without any flowering losses.

In sunflower, a higher P fertilization level led to both a higher number of flowers and an increase in flower P concentration, but in marigold the effect was not significant. However, both ornamentals had fully developed healthy flowers with no visible P deficiency symptoms, even at the low P level. Marigold was probably better adapted to the prevailling greenhouse conditions and the low P fertilization level may have been sufficient for the given cultivation period. Furthermore, the P demand of marigold was overall lower than that of sunflower, as it developed less biomass.

The relationship between P fertilization and flowering of greenhouse plants has been described in contradictory ways in the literature. On the one hand, an increased level of P has been reported to result in higher fresh and dry matter yield, number of flowers, plant height and concentration of essential oils in marigold (Negahban et al., 2014). Naik (2015) found that marigold plants receiving the highest level of P developed more flower heads per plant and had a significantly extended duration of full flowering. The results of Negahban et al. (2014) are in line with the findings of Anuradha et al. (1990), whereas those of Naik (2015) are not. On the other hand, Dahiya et al. (1998) reported a reduced number of flowers in marigold treated with P and Polara et al. (2015) did not find any influence of P on growth. Thus, the relationship between P fertilization and flower quantity has not yet been clearly demonstrated.

Bergmann (1993) has described a P concentration status between 2 and 5 mg g^{-1} DM^{-1} as optimal for flowering plants. However, a differentiation between marigold and sunflower needs to be made here. Sunflower produces larger and later flowers (usually one large main flower) than marigold (several, smaller flowers). Although marigold developed a higher number of flowers in our study, the flower biomass production was still higher in sunflower. As such, the P demand of sunflower can be expected to be higher as well. The importance of P fertilization for flowering in sunflower was clearly shown in our study, as the reduced P fertilization level resulted in a lower number of flowers.

Thus, we conclude that the P demand of ornamentals depends on the size and number of flowers as well as on the time of flowering. This applies not only to the amount of P but also to the timeframe of its fertilizing effect.

These findings also suggest that the fertilization level we defined as “optimal” for sunflower here may not actually be the optimal level. In addition, the chosen P fertilization levels seemed too similar to obtain a more distinct dose-response effect. The plant development status at the time of harvest was good and the P concentration indicated healthy plants with sufficient P supply. There were no recognisable symptoms of a lack of any macro- or micronutrients (pictures → supplementary material). However, it should be mentioned that the fertilization status measured at this point in time may not be adequate for a marketable sunflower plant, as it may display symptoms of deficiency shortly after sale.

As consumers - mostly hobby gardeners - are not familiar with plant care requirements, it is of particular importance for the producer to prevent the plants showing deficiency symptoms later on by supplying a certain nutrient reserve. This could be ensured by the combination treatment tested, as it resulted in the highest numbers of flowers with the highest P concentration levels. The long-term fertilization effect of the dried solids seen in this study suggests that these can secure P supply for several weeks and provide a positive medium structure and moisture in the pot. The differing P demand of flowering plants needs to be considered. Ornamentals that flower steadily for several weeks fulfill customers' expectations and render follow-up purchases more likely.

5. Conclusion

An instant, or at least fast, P uptake into plants is of particular importance in the horticultural sector, due to the short production periods in comparison to vegetable and field crops. The fertilizer needs to be applied in a form that ensures high P plant availability. In general, this requirement was met by all the recycled products tested, with the exception of the air-dried solids. The two P-Salts can be fully recommended as powerful P fertilizers for the horticultural sector. The combination of P-Salt and air-dried solids with its synergistic effect ensures short- and long-term P supply, and is thus particularly advantageous for the production of ornamentals marketed as potted plants because it guarantees long-term product quality. The two types of solid tested can fulfil two functions: as a source of P and/or as a component of growing media. For the purpose of P fertilization, the steam-dried solids are the better choice; the air-dried solids would need to be supplemented with additional P. As the solids had to be applied in rather high volumes, they could also serve as growing medium component or possibly as a peat substitute, or at least a supplement, in order to produce peat-reduced growing media. For this purpose, the air-dried solids are more suitable. Naturally, important quality parameters including pH and salt content should be continuously monitored. This would suggest that a simple solid-liquid separation of digestates or manure is sufficient in some cases. However, in others, advanced P recovery technologies are advantageous and urgently needed, for example in countries where P field application has been restricted (P could be removed and remaining material applied) and in regions with excess manure and digestate (could be separated into transportable salts, organic matter and water). In both examples, the P is removed and then used in other regions. The origin of the recycled products can also be seen as a marketing advantage. Substituting synthetic by organic products in ornamental production as well as in fertilizers and growing media for hobby gardeners may be appealing for environmentally conscious consumers.

In this study, we were able to show that recycling P by means of chemical and thermal processes has high potential in reducing the dependency of horticulture and agriculture on P fertilizers derived from phosphate rock. The recycled fertilizers were adaptable to the requirements of different types of ornamentals and met the P demand as efficiently as commercial phosphate fertilizer.

Acknowledgements

This study received funding from the EC ("BioEcoSIM", grant no. 308637) and the Federal Ministry of Education and Research ("GOBi", grant no. 03EK3525A; "Humboldt reloaded", grant no. 01PL11003). Special thanks go to the Staatsschule für Gartenbau, Stuttgart-Hohenheim, for providing glasshouse facilities, watering service and support, in particular to Eberhard Weiss. We would also like to thank Jens Möhring for statistical advice and Nicole Gaudet for improving the language quality of the manuscript.

Appendix A. Supplementary data

Supplementary material related to this article can be found, in the online version, at doi:https://doi.org/10.1016/j.scienta.2018.08.052.

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5 General Discussion

The primary objective of this thesis was to find out whether recycled fertilisers from organic residues are comparable to mineral fertilisers and can serve as a suitable substitution. This was evaluated with different crops under field and greenhouse conditions. In **Chapter 2,** recommendations are provided on how to integrate separated biogas digestates in biomass production systems. The combined application of digestates and mineral fertiliser was considered the best approach to meet the multiple demands, including high yields, low-cost farming and minimal negative environmental impacts. The study in **Chapter 3** formed the basis for integrating novel fertilisers into efficient fertilisation strategies. The P-Salt recycled from manure had similar or even better effects than mineral fertiliser on the yield of the test crops under the given conditions. In **Chapter 4**, the findings from Chapter 3 were transferred to an application in the horticultural sector. All tested recycled products were generally able to meet the plants' P demand as efficiently as triple superphosphate (TSP). The P-Salts showed a better suitability for short-term and the steam-dried solids for long-term P supply. A comprehensive discussion of the results was incorporated into each scientific publication.

The general discussion links the findings presented in Chapter 2 to 4. **Chapter 5.1** starts with the agronomic efficiency of residues and recycled fertilisers, because this was the main focus of this work. This is examined based on the plant availability of recycled nutrients, which is essential for the recycled fertilisers' effect on crop yield. Integrating recycled fertilisers into agricultural practice will only work in the long term when the nutrient supply of crops is at least comparable to that of mineral fertilisers. In **Chapter 5.2,** the implications of the findings are discussed from an agronomic, a practical, an ecological and an economic perspective.

5.1 Agronomic efficiency of residues and recycled fertilisers

The adoption of recycled fertilisers by agricultural practice will only be successful in the long term when the nutrient supply of crops is at least comparable to that of mineral fertilisers. The studies presented in Chapter 3 and 4 have shown that the nutrient uptake from P-Salts was comparable to that of commercial TSP. While the study in Chapter 2 does not include explicit data on nitrogen uptake, the yield results suggested that the crops were sufficiently supplied.

The presented experiments had their focus on N and P. However, a principal differentiation is required between N and P, as their dynamics differ. The N demand needs to be met in a timely manner, when the crops need it. Thus, N fertilisation is carried out every year. In addition, it is often split into several applications in order to minimise the risk of losses. Main pathways of N losses are via gaseous emissions (NH_3, N_2O) and leaching (NO_3^-) (Schilling 2000).

In contrast, P fertiliser can be basically applied anytime. In many cases, it is required only once every few years, depending on the soil supply status and the amount of removal with crops. The acute risk of P losses is low, as it tends to remain in the layer of soil where it was introduced with fertilisers and manure (Eghball et al. 1996, Simard et al. 1995). However, the (temporary) P immobilisation following application in soil represents a challenge.
P occurs in soil in different forms: it is basically always present as phosphate, consisting of organic P which comprises 30-70% of the phosphate in soils; esters, microbial and humic P from dead organic matter; and inorganic P of which the most common form is apatite (Frossard et al. 1995). Depending on the soil type and pH, P may form e.g. Al and Fe complexes in acidic soils, and Ca complexes in calcareous soils, resulting in a reduced plant availability.

The soil P concentrations (also known as "P stocks" or "legacy P") have increased in many areas (Sharpley et al. 1994) and led to high positive P imbalances in parts of EU27 (van Dijk et al. 2016). This is due to extensive P fertilisation in previous decades and also due to the common practice of applying manure and digestate based only on their N concentration while P is neglected (Maltais-Landry et al. 2016). Although these excessive P stocks are mostly not plant-available (Johnston et al. 2014), they are still exposed to the risk of loss mainly through soil erosion and surface run-off (Addiscott and Thomas 2000; Sharpley et al. 1994). Gaseous emissions are uncommon (Reid et al. 2018). Leaching depends on soil type and soil P content, e.g. it can be low despite high P applications when the P sorption capacity in the subsoil is high (Djodjic et al. 2004).

The farmers' main interest is the reliable and exact supply of their crops. To achieve this, the recycled fertilisers need to be integrated in a suitable fertilising strategy so that the nutrients are plant-available preferably at the time of demand. In-depth knowledge of the behaviour of recycled N and P fertilisers in soil – right after application, during the first vegetation period, and in the following years -, supported by suitable prediction methods, is therefore an important prerequisite for their adoption in practice. This work contributes by providing knowledge on the fertilising effect of selected recycled fertilisers, as discussed in this chapter.

5.1.1 Plant availability of N and P

For both N and P, the nutrient uptake depends on the plant availability (PA). The PA of nutrients in turn depends on the mineralisation rate of organic forms, which vary e.g. with soil conditions, including temperature and moisture. In addition, the PA depends on interactions with the soil.

The immediate PA of N in liquid digestates is high, because more than half of the total N is present in form of NH_4^+-N (Gutser et al. 2005). Assuming ideal conditions, mineral fertiliser may be completely substituted with liquid digestates, as NH_4^+-N can be directly and quickly taken up by the crops (Tambone et al. 2017). In contrast, the immediate PA of N in solid digestates is low or medium, thus, their fertilising effect must be considered rather under the long-term aspect of the gradual mineralisation of organic nitrogen (N_{org}). In practice, where ideal conditions are rare and the weather in the course of the vegetation period is not foreseeable, a combination of separated digestates and mineral fertiliser helps to secure a certain yield level.
The methods for determination of plant N availability in digestates are established and predictions on the behaviour of the N fractions in the soil are relatively reliable.

The PA of P in P-Salts depends on various factors, soil characteristics (e.g. soil type, temperature, moisture, pH, microbial and enzymatic activity), crop type (e.g. crops with the ability to release organic acids or legumes can mobilise otherwise unavailable P; crops with extensive root systems have an advantage), and the properties of the product (e.g. particle size, raw material, production process). The influence of factors relevant for the experiments has been adequately discussed in Chapter 3 and 4, supported with respective references. The comparison of two P-Salts in Chapter 4 showed that both products had a similar chemical composition and a very similar fertilising performance despite being recycled from different raw materials with slightly different techniques.

Based on chemical analyses, a low PA of P in P-Salts was expected, as the share of water-soluble P was very low. However, the actually observed fertilising efficiency and plant uptake were comparable to water-soluble TSP, indicating that the PA must be higher than expected. This was observed in numerous studies (Johnston and Richards 2003; Römer 2006; Wollmann et al. 2017) including our own work. Thus, the solubility of P in water is not necessarily a suitable indicator. We explain the unexpectedly high PA of the P-Salts with inappropriateness of the analytical approach.

5.1.2 Prediction of P plant availability

The prediction of plant availability (PA) of P or of the behaviour of a recycled P fertiliser in soil with available analytical methods was poor at the time when the experiments were carried out. Usually applied extractants included water, citric acid, formic acid, mineral acid, and neutral ammonium citrate (EC 2003/2003). Sequential P fractionation applied to the digestate-based P-Salt in Chapter 4 also gave limited additional information. However, the mentioned methods were developed for standard conventional P fertilisers, not for highly heterogeneous recycled fertilisers. Results were thus considered unreliable or had to be at least interpreted with caution. This emphasised the importance of actual crop growth tests as best approach to give reliable results, although they are time-consuming and thus expensive. There was an urgent research need to develop reliable, easy, quick and low-cost methods, to refine existing methods, or at least foster the standardisation of growth experiments (Kratz et al. 2018).

Meanwhile, a universal method for reliable determination is still lacking, but alternative methods have been studied. Predicting the PA of P using advanced methods, e.g. DGT (diffusive gradients in thin films) or iron bag method, partly overcome the mentioned limitations as they represent continuous, sink-based P extraction methods. The DGT method is actually used for testing P availability in agricultural soils (Six et al. 2013) and was not specifically developed for a use directly on fertilisers. It simulates P uptake into plant roots by constant removal of P from the soil solution (Kratz et al. 2019). However, the DGT is not exactly a quick or easy method and its suitability could be limited in soils with very high P concentrations (Christel et al. 2016a). The iron bag method is also an infinite-sink extraction using ferrihydrite slurry filled into dialysis membrane tubes. In a study with 13 contrasting recycled and mineral fertilisers, the iron bag method resulted in high correlations with the plant P uptake (Duboc et al. 2017). Suitability for routine analysis is still questionable.

Kratz et al. (2019) conclude their review with the call for intensive testing of alternative methods, including sequential fractionation, or extraction of incubated soil and fertiliser mixtures with standard soil extractants or with P sink methods, as alternative options to predict the P availability of recycled fertilisers. Using non-standardised extraction methods was subject of a recent study by Duboc et al. (2021) who suggested $NaHCO_3$ extraction as a cost-effective and reliable method to predict P availability in routine analysis. In addition, Duboc et al. (2021) raised the question if a “universal, one-method-to-fit-all-conditions approach” is necessarily the best option and recommend their own approach of using contrasting extraction mechanisms instead. The evaluation and comparison of methods to predict the P availability of recycled fertilisers is one subject of the current project LEX4BIO.

5.1.3 Mineral fertiliser replacement value

All the experiments presented in Chapter 2 to 4 were carried out assuming a mineral fertiliser equivalent or mineral fertiliser replacement value of the recycled fertilisers of 100%.

For N, this may seem uncommon as the literature reports nitrogen fertiliser replacement values such as 50-70% of total N for liquid and 30-50% of total N for solid digestates in the year of application (Möller et al. 2009; Baumgärtel and Backes 2020). The current minimum efficiency in Germany is 60% of total N for liquid and 30% of total N for solid digestates in the year of application (DüV 2021), when applied to arable land. These values basically correspond with the contents of NH_4^+-N, provided that gaseous losses are minimised (Möller et al. 2009). The study in Chapter 2 only considered the total N concentration of digestates, mainly for simplicity reasons as e.g. no differentiation between cropping systems was necessary. In addition, it was shown that the digestate treatments were mostly able to maintain a satisfying yield level despite the factual underdosing of N.

5.1.4 Effect of combination treatments

All three studies presented in this thesis included combination treatments. Such combinations can fulfil several purposes. One purpose can be to ensure a short-term and a long-term nutrient supply. Another purpose can be to combine one product that mainly supplies nutrients and another that functions as soil conditioner, e.g. increases soil organic matter, nutrient or water retention capacity. This was tested by combining solid digestates and mineral N fertiliser in Chapter 2, and P-Salts and dried solids in Chapter 4. These two combinations fulfilled both purposes. The effect of biochar is discussed separately in Subchapter 5.1.5.

The first purpose described above could be tested with the chosen experimental setups and both respective treatments were evaluated to be effective.
The second purpose was not specifically evaluated in the study in Chapter 2. However, the soil organic carbon contents at both sites were assessed in 2014 and 2015 by Schabel (2016). In that context, higher soil organic carbon contents were more often observed in plots receiving solid digestates (pure and in combination with mineral N fertiliser) than in the other plots. This build-up in soil organic matter contributed positively to the consistently good dry matter yield (DMY) in the annual and intercropping system.

The dried solids tested in the study with ornamentals in Chapter 4 were originally intended to serve as P source. However, as they were applied in relatively high volumes, they represented a not negligible fraction of the growing medium. Building on the experiment in Chapter 3, we wanted to quantify a potential effect of combined treatments. Thus, a simple equation was introduced to calculate a theoretical DMY of the combination based on the DMY of the single treatments. For sunflower, Chinese cabbage and partly for marigold, a positive effect of combined application of dried solids and P-Salt was verified.
The same equation was subsequently applied to the results of Chapter 3, namely to the combination of P-Salt and biochar. The actually measured DMY clearly exceeded the calculated DMY, which confirms the synergistic effect of the P-Salt and biochar (0.1%) combination. Considering the example of spring barley grown in sand soil, the measured DMY was 2.4 times higher than calculated.

Bach et al. (2021) reported that the combined application of P-Salt and dried solids performed equally well as TSP in a low fertility soil and assigned this to the soil conditioning effect of solids. An increased plant P availability may be another reasonable factor. This was not exactly found in our work, probably due to the short growth period. However, the combination of organic (compost) and mineral (TSP) fertiliser led to higher P supply than separate application in a 92-days experiment with maize (Muhammad et al. 2007).

Solids – either in form of separated digestates, biochar or dried solids – contribute to the soil organic matter, can thus positively influence soil water retention capacity and soil moisture, which fosters in turn the soil microbial activity and this finally results in an enhanced additional mobilisation of P (Möller 2015; Olander and Vitousek 2000). This sequential process has been discussed in more detail in Chapter 4, but this may serve as general explanation of the good performance of the combination treatments in all studies presented within this thesis. In the end, it remains an individual decision if and how the application of combination treatments is practicable. At pot-scale, e.g. for horticultural purposes, products can be mixed with the growing medium before filling the pots. A later application to planted pots is certainly less favourable. An implementation in agriculture at field-scale depends on the cropping system, the type of product, and requires appropriate integrability into work processes on a farm.

5.1.5 Effect of biochar application

Biochar addition showed potential as soil improver in the sand soil, where it resulted in increased DMY in combination with P-Salt. In the clay soil, the effect was not significant. The different expression of this effect depending on soil type can be assigned to the organic matter content and the water and nutrient retention capacity which were all very low in the sand soil. Enhanced fertiliser efficiency through biochar application in sandy soils has been reported before (van Zwieten et al. 2010; Schulz and Glaser 2012).

Biochar addition was found to reduce the PA of N in the study in Chapter 3. The applied N was at least temporarily immobilised, visible in lower soil N_{min} in the respective treatments.
The observed N immobilisation can be positive or negative in practice. Immobilised N is protected from losses via leaching, particularly in light soils. If the N is released later, it can be used by plants. However, the effect becomes negative when the crops cannot meet their need due to N sorption to biochar.

The general fertilising effect of the P included in biochar can be hardly predicted or controlled as too many mechanisms are involved. The biochar applied in our work was pyrolysed at 450 °C, a temperature range which indicates a high immediate PA of P (Bruun et al. 2017). Plant-available soil P in biochar treatments significantly increased towards the end of our study which is consistent with findings by Christel et al. (2016b) and Lehmann et al. (2003).

In general, controversial results have been published whether biochar application has beneficial (Lehmann et al. 2011; Jeffery et al. 2011), adverse, or just indifferent effects (Reents and Levin 2013; Spokas et al. 2012). The interest in biochar and related products has considerably increased at national and global scale in the recent decades, evidenced by the rising number of scientific publications and the research effort (Holweg 2011). Muskolus (2021) divided publications on fertilising and soil effects of biochar in two groups: most often, positive effects were found in pot experiments, whereas in field experiments no or even negative effects occurred, e.g. growth depression of maize due to nutrient deficiency. This is applicable for the study in Chapter 3, in which a positive effect of biochar applied to sand soil was monitored at pot-scale. The same P-Salt and biochar combination was tested again in an extended experimental setup with maize, spring wheat and rapeseed and resulted in increased biomass yields and harvest indices (Reetz and Franzke 2018).

The fact is that a reliable measurement of biochar stability in soil is challenging. Its estimated persistence in soil varies widely from few years to almost four millennia (Gurwick et al. 2013; Kuzyakov et al. 2014). The raw materials and biochar properties are crucial for its stability, furthermore the climate, soil type and soil management. It is also known that in order to have a significant effect on physical soil characteristics, large quantities need to be applied. For instance, Bruun et al. (2014) concluded that biochar applied at rates of 1 to 2% by mass improved the quality of a sandy subsoil. These percentages correspond to 100 to 200 t of biochar per hectare.

An imaginable application of biochar with practical potential would be as ingredient of recycled fertiliser mixtures, with the benefits of supporting the formulation process, and adding carbon and structure to the product.

5.1.6 Validation of results from pot experiments in the field

Naturally, the findings from the greenhouse experiments with P-Salts (Chapter 3) need validation in field experiments. This was realised in 2016 with winter wheat, maize and sunflower at three field sites in Spain and two field sites in Germany. P-Salt and crystallised ammonium sulfate (also obtained from pig manure using the BioEcoSIM technology, see Chapter 1) were applied individually and in various combinations and compared to mineral fertilisers (CAN, ASN, TSP).

This paragraph summarises the findings of the experiments described in Ehmann et al. (2016, unpublished) and Ehmann et al. (2017): In Germany, almost all treatments resulted in higher DMY and better yield quality of wheat and maize grown at both sites than the control. The protein content of wheat was increased by all treatments compared to the control; grains of almost all treatments were classified as quality wheat (protein ≥13.5%). Furthermore, with regard to sedimentation value and falling number, even elite wheat quality was achieved with all treatments. Two different application techniques (broadcast vs. root-zone) of P fertilisers both resulted in a similar maize DMY and P concentration. In Spain, treatments with the manure-based fertilisers significantly increased the DMY of all test crops. Germany and Spain represented regions with contrasting climate and soil conditions within Europe. Thus, results are expected to be transferable to other regions. The recycled fertilisers proved to be as effective as comparable commercial products, yet with an environmentally more benign performance.

Two findings from the experimental sites in Germany were interesting with regard to practical implementation. The combined application of P-Salt with different synthetic nitrogen forms (CAN or ASN) did not influence the fertilising effect. Thus, the P-Salt may be flexibly used for compiling customised fertiliser products. No significant differences were found in DMY and P concentration of maize treated with P-Salt or TSP, and this was independent of broadcast or root-zone application. From these results, we conclude that the P-Salt has a good competitiveness with TSP also under field conditions and is suitable for different climatic regions.

5.2 Implications of presented findings

It is notable that reasonable DMY results were achieved with the tested recycled fertilisers and combinations, and that they were adaptable to all three different experimental setups. In this chapter, the conditions for the use of these fertilisers in agricultural practice along with environmental and economic implications are discussed. Several aspects could be assigned to more than one subchapter though, as they are closely interconnected and the boundaries between agronomic, environmental and economic implications are blurred. This underlines the high complexity of agricultural systems.

5.2.1 Agronomic and practical implications

Untreated digestates serve as a multi-nutrient fertiliser and soil improver. Separated digestate fractions fulfil the same purpose but with a different nutrient ratio, and the solid fraction has more potential as soil improver than the liquid fraction. Depending on the pre-treatment, the dried solids can serve as slow-release P fertiliser, soil improver, or component of growing medium. The P-Salts represent more or less single-nutrient fertilisers, particularly, since the recovery technology has been refined in a follow-up project, where the N content of the P-Salt has been successfully reduced (from 8.1 to 0.6% N in DM) and the P content increased (from 5.0 to 9.5% P in DM). This avoids accompanying N application and increases the flexibility of the use of P-Salt as proposed in the conclusion of Chapter 3.

The practical implementation of using recycled fertilisers must be devised starting from farm-level. Farmers need to diversify their fertilisation strategy, because one solution for all fields is no longer sustainable. This increases the requirements for farmers' skills, as fertilisation is turning from a formerly simple "to do" into a more and more strategic task. The fertilisation practice has to be adapted to the respective types of recycled fertilisers in terms of logistics, timing and machinery.

Separated digestates, dried solids and P-Salts have differing suitability for application nearby, in a medium or in a long distance. Separated digestates are– or at least should be – ideally applied near an anaerobic digestion plant. Dried solids have a higher transportability compared to only separated material. The P-Salts are in a compact form and can be transported over long distances if necessary. In terms of timing, farmers must consider if fertilisers contain nutrients in readily available form or if they function as "slow-release" fertilisers. Fertilisers that provide nutrients in not immediately plant-available form require earlier and more sophisticated planning of the application. Indeed, the uncertainty in nutrient content and higher effort for planning and utilisation were identified as main barriers to organic fertiliser use in a survey among Danish farmers (Case et al. 2017). The findings from the present study can contribute to overcoming these concerns.

Farmers aim at harvesting stable crop yields with high product quality. The economic situation of most farms usually does not allow experimenting with fertilisation and putting yields, i.e. income, at risk. Consequently, their acceptance of alternative fertilisers is currently fairly low. Here, a gradual substitution of mineral with recycled fertilisers may seem a safer approach for farmers and an option to get them convinced. In the study presented in Chapter 2, combinations of mineral fertiliser and separated digestates performed as well as mineral fertiliser alone in most of the cropping systems, sites and years; but with 66% less mineral fertiliser input. To facilitate the transition, a farmer may start with a lower share of recycled fertilisers and then increase it step by step. Equipment for spreading mineral fertilisers as well as for liquid or solid organic residues is usually available on farms. The combination of recycled with mineral fertilisers is recommended to ensure optimal nutrient supply in certain cases, e.g. when unfavourable conditions are anticipated or for crops with a particularly high demand.

Another important practical aspect is the physical form of recycled fertilisers because this determines the mode of field application. In our experiments, P-Salt was provided as fine powder, which is easy to apply at pot-scale, but more than challenging at field-scale. For a reasonable on-farm use, P-Salts – or any products that are produced from them, pure or in combination with solids or something else – must be brought into spreadable form. Granulated or pelletised recycled fertilisers can be applied with standard machinery usually available on farms.

The application of organic fertilisers is being more and more facilitated by the progress in new precision farming technologies and smart applications. For example, sensors which allow real-time nutrient measurement in manure and digestate, or of soil N demand during application. Examples for available commercial systems include solutions which measure dry matter, total N, NH_4^+-N; P_2O_5, and K_2O in liquid manures and digestates in real-time during filling, stirring, or spreading, using near infrared sensors (John Deere 2021; Zunhammer 2021). The use of outdated and inaccurate spreading techniques has already been and is going to be further restricted by regulations (DüV 2021). Site-specific maps and GPS-controlled track guidance systems enable a more targeted and accurate application. This may be supported by drone- or even satellite-based remote sensing and spectral imaging of crops. The further development and widespread use of the described appliances in the coming years can help to estimate the spatial variation of plant nutrient demand and optimise the targeted fertiliser application. Such advanced technologies play an important role in the context of increasing the agricultural productivity without negative environmental impacts in the future (Lewandowski 2015). Maximising the nutrient utilisation by crops reduces losses into the environment.

Practical example: implementation of residue treatment in the Hohenlohe region

The Hohenlohe region, located in the Northeast of Baden-Württemberg, is characterised by arable farming and intensive animal husbandry, accompanied by anaerobic digestion. In consequence, the region has to deal with resulting nutrients in manure and biogas digestates that exceed the amounts needed for fertilising the fields, particularly for P. This renders the treatment of the residues very useful for agronomic, ecological and economic reasons. Thus, the implementation of advanced digestate treatment in practice is subject of the current research project Agriplus Hohenlohe (EIP-AGRI 212018). The project aims to improve the regional nutrient management and nutrient flows by upgrading biogas digestates with a full-scale nutrient recovery plant. Four farmers integrated the fertilisers (P-Salts, liquid ammonium sulfate) recycled from biogas digestates into their common crop rotations to demonstrate the practical feasibility in a large on-farm field trial. These treatments were compared with the usual farm practices, i.e. application of untreated manure and biogas digestate, and mineral fertilisation. Preliminary results from two vegetation periods indicate that the products have fertilising potential, but that the application technique (injection of liquid ammonium sulfate into deeper soil layers) and product formulation (P-Salt must be granulated or pelletised) require optimisation (Müller et al. 2021).

Suitability of recycled organic residues for organic farming

For organic farms, limited nutrient availability is an increasing challenge (Reimer et al. 2020), particularly for farms without livestock. Options for N fertilisers with high solubility are limited and external P inputs are restricted (Cooper et al. 2018). Thus, biogas digestates are valuable fertilisers for organic farming on condition that requirements regarding the biogas substrates fed into biogas plants are considered (EC 2021/1165). The liquid and solid digestate fraction may both be of interest, as the high $NH4^+$-N availability in the liquid and the slower N release in the solid fraction complement each other ideally (Tambone et al. 2017) and the P supply is a positive side effect. P-Salts recycled from organic residues may provide an equally valuable P source for organic farming, but their use requires an entry in Annex II of Regulation EC 2021/1165 and this is still awaited.

5.2.2 Legal framework

Another important prerequisite for their use in practice is of course that the advanced recycled fertilisers, such as P-Salts from manure or digestates, are explicitly covered by legislation. The current legal framework for manure- and digestate derived fertilisers is complex, because they are covered in several regulations (e.g. Fertiliser Product Regulation (FPR; EC 2019/1009), Nitrates Directive (EC 91/676/EEC), Animal by-products regulation (EC 1069/2009). In addition, the implementation of these regulations happens country-specific and poses administrative barriers, slowing down innovative approaches for manure and digestate valorisation. The new FPR states conditions regarding the raw material (Component Material Category, CMC), treatment technologies, the fertiliser type including nutrient concentrations (Product Function Category, PFC), thresholds for potential pollutants, sanitation and product labelling. The relevant legal framework must be flexible enough to be timely adapted to technical progress in nutrient recovery technologies and to additional residue streams. This opportunity seems to be given through the CMC and PFC concept in the new FPR. However, the legal aspects concerning recycled fertilisers are deliberately not considered in more detail in this work.

5.2.3 Environmental implications

The greatest environmental benefits of an intensified use of organic residues and recycled fertilisers are certainly the reductions in mineral fertiliser production and lower emissions during residue storage, handling and spreading when residues undergo treatment. This was already mentioned before in this study. However, there are some more advantages worth pointing out.

A sound integration of separated biogas digestates into cropping systems allows - or aims to achieve - that the nutrients, mainly N in this case, are plant-available preferably at the time of the crop demand. When N and P are as completely as possible used by crops, then losses are kept at a minimum. Soil organic matter is positively influenced by digestate application and this increases the soil's ability to absorb nutrients and water (Nabel et al. 2017). In addition, applied quantities of solid digestates and biochar are usually in the range of several tonnes per hectare. The C mineralisation of solid digestates is slow and the long-term stability of biochar for decades is significant (as discussed in Chapter 5.1.5). Therefore, the application and incorporation of these solids is an opportunity to sequester high carbon amounts in the soil (Polifka et al. 2018; Egene et al. 2021), an aspect that cannot be highlighted enough in view of the current efforts to protect the climate.

A rainfall simulation experiment with incubated soils from three European field sites showed that several bio-based P fertilisers had a lower leaching potential than mineral P fertilisers or separated pig manure (Ylivainio 2021, unpublished). Therefore, it is likely that the P-Salts tested in our studies show a similar behaviour, which is an additional ecological advantage. The environmental impacts of fertilisers are usually evaluated using the life cycle assessment (LCA) methodology. A standardised LCA framework for a comparable evaluation of advanced recycled fertilisers is not yet available, but currently under development (Tanzer 2021). This will facilitate comparing advanced recycled fertilisers for non-experts in the future.

The unfavourable N:P ratios in manure and digestate, that rarely match the crops' requirements, are no longer a limiting factor for application when the residues are treated accordingly. Exctracting P in form of P-Salts or struvites from organic residues allows a targeted P application, only when it is actually needed by crops. Simultaneously, the remaining fractions of residues that are free of P can be either field-applied or further treated. Reduced P concentrations in treated residues considerably increase their suitability for many European soils (Egene et al. 2021). This approach takes account of the high soil P stocks in many regions and avoids their further increase.

Awiszus et al. (2019) assessed the environmental impacts of nutrient recovery from biogas digestate using a model. They conclude that only excess digestates need to be subject to nutrient recovery to make reasonable use of the required energy and additives. Furthermore, the complete nutrient recovery is considered mainly applicable in hotspot regions with high nutrient and residue surplus and limited agricultural land (Awiszus et al. 2019). Before surplus P recovered from organic residues is redistributed from a hotspot region ("donor") to a region with P deficit ("recipient"), detailed knowledge of regional nutrient availability is required, as emphasised by Hanserud et al. (2017). Such data is currently being compiled by researchers within the LEX4BIO project. Hanserud et al. (2017) assessed the environmental impacts of P redistribution from manure. They conclude that results are specific for case study regions and promote the inclusion of region-specific parameters in respective LCA studies.

These considerations emphasise once more the importance of adapting treatment approaches to farm-specific and/or local requirements. In fact, the best or most suitable solution must be determined on a case-by-case basis. Trade-offs are inevitable. The "best" strategy can be different depending on the point of view. Willeghems et al. (2016) conclude that transport of raw manure is the most economically advantageous strategy as it is cheaper than processing it, whereas manure separation is the most advantageous strategy from a greenhouse gas perspective. Ten Hoeve et al. (2014) summarise that none of the pig slurry treatment options they assessed (screw press; screw-press + composting; centrifuge; centrifuge + ammonia stripping) was clearly superior regarding potential environmental impacts.

5.2.4 Economic implications

The economic situation of many farms can be described as tense, particularly in pig farming (BMEL 2021). Most farmers will therefore mainly implement changes in their routine when they are economically advantageous.

During phases with low fertiliser prices, the ambition to promote nutrient recycling and use of residues is moderate. Ambition for changes is commonly led by economic or environmental pressure. Research and investment in advanced treatment technologies will pay off at the latest when the current practices are no longer feasible, e.g. when regional nutrient surpluses become too high, when imported P is becoming too polluted to be applied, or when N and P fertilisers become too expensive or simply undeliverable. Change is also driven by regulations that further restrict field application of untreated manure and digestate or applicable nutrient charges which will in the end basically result in unsustainable costs for residue disposal. Treatment as simple as solid-liquid separation of digestate followed by application saves costs already now, because less mineral fertilizer has to be bought in addition and less storage capacity for digestates is required (Möller and Müller 2012).

Fast forward to November 2021 and the fertiliser prices have soared. For example, German sales prices for CAN as important N fertiliser were three times higher than at the beginning of the year; for P fertilisers DAP twice as high (top agrar 2021). This was primarily a consequence of increased gas prices which led to a limited production of fertilisers, particularly of energy-intensively synthesised N. At the same time, the global demand has risen and the situation with the already insufficient fertiliser supply has become increasingly tense due to disrupted global supply chains. In this situation the local availability of recycled fertilisers (e.g. digestates) or the option for locally manufacturing them from residues (P-Salts) is a considerable advantage in terms of reliable supply.

Fast forward to 2021 again and there are many more nutrient recovery technologies available than 2012 when the work on this study commenced. Technologies have been refined and scaled up, new approaches were developed and have reached market maturity. In addition, the list of recycled fertilisers on the market has grown.

Local implementation of nutrient recovery technologies can create additional income for farmers. New jobs are created as the treatment and recovery installations have to be built and maintained. Recovered nutrients are converted into marketable products and sold. Beyond fertilisers for agriculture, organic residues and recycled nutrients can be used for creating products with higher profit margins, i.e. for amateur gardeners (Herbes et al. 2019). Nutrient-free solids can serve as peat-free growing media. There are plenty of ideas for biorefinery concepts on upgrading organic residues and beyond, and this opens up enormous opportunities (not only) for rural areas.

In sum, residue and nutrient management contribute significantly to the EU Circular Economy Package and the EU Circular Economy Action Plan that aim to accelerate recycling and reuse, with benefits for both the environment and the economy. The revision of the fertiliser regulation was actually initiated very early in the EU Circular Economy Package, emphasising the ambition to promote organic and residue-based fertilisers across the EU. Moreover, the topic of this study addresses several of the objectives stated in the EU Farm-to-Fork Strategy that is at the core of the EU Green Deal, including resilient food supply, reducing excessive fertilisation and losses, increasing the use efficiency of

N and P. The Green Deal strives for improving the economy in rural areas, eliminating pollution, and fostering a bio-based industry. The priority of efficient nutrient recycling from residues is gaining ever more importance in view of the increasing global fertiliser demand resulting from the need to supply a growing world population with food and the emerging bio-based economies with biomass. The knowledge and the opportunities for a bio-based circular economy, including nutrient recycling, are available and are being refined every day through efforts by researchers and innovators. Now it is time for implementation.

5.3 Conclusion

This study has shown that recycled organic residues from animal husbandry and bioenergy production are suitable for use as fertilisers.

Five specific objectives were explored. The specific objectives (1) and (2) addressed the competitiveness of separated biogas digestates with mineral fertiliser and their suitability for different biomass production systems. It is concluded that separated biogas digestates can substitute mineral fertiliser in perennial and intercropping systems, even on marginal sites. Solid digestates are ideally applied in cropping systems with regular soil tillage where an incorporation is possible. For perennial grassland, liquid digestates are better than solids in terms of workload and application. Intercropping proved to be the most stable system that gives constantly high biomass yields and can be maintained with either solid or liquid digestates. However for annual crops such as maize, a combined application of digestates and mineral fertiliser is recommended to ensure sufficient nutrient supply.

The specific objective (3) focused on comparing the fertilising effect of recycled P-Salts with commercial phosphate fertilisers. The P-Salt recycled from manure was found to have the same or even better effects than TSP on growth of spring barley and faba bean in two test soils. In the experiment with sunflower, marigold and Chinese cabbage, the two P-Salts recycled from manure and digestate had more or less the same effect as TSP on biomass production. The good performance in cereals, legumes, ornamentals and vegetables affirms the versatile applicability of the P-Salts for a broad range of crop types. From these results, it is concluded that both P-Salts have an equivalent fertilisation effect to TSP and can thus replace it as mineral fertiliser.

For addressing the specific objective (4), potential interaction effects of recycled P-Salts with biochar and dried solid digestates were examined. Biochar in combination with P-Salt enhances the fertilising effect of the latter, especially on poor soils with low organic matter. A general recommendation of biochar application cannot be drawn from the results, mainly because of the N immobilising effect and the large application quantities (>100 t ha^{-1}) needed for a significant soil effect. The combination of P-Salt and air-dried solids resulted in measurable synergistic effects on biomass production of all test crops. These effects are attributed to the short- and long-term P supply of the two fertilisers and the soil conditioning effect of the solids. In conclusion, this combination is suitable for potted plants because it provides a P reserve for prolonged plant quality and spares the consumers plant care measures.

Finally, the specific objective (5) dealt with differences in the uptake efficiency of recycled and mineral fertilisers. The two P-Salts recycled from manure and digestate can meet the P demand of sunflower, marigold and Chinese cabbage as efficiently as TSP. The P-Salts have a better suitability for short-term and the steam-dried solids for long-term P supply. Thus, they can be flexibly adapted to different purposes, e.g. P-Salt ensures a fast P uptake which is important for horticulture due to short production cycles, and the dried solids provide a slow-release P source for crops with longer growth periods. The combination matches both purposes.

Enhancing the use efficiency of nutrients represents a challenge to be faced independently of whether organic or inorganic fertilisers are applied. A better utilisation of N and P already available on farms is necessary to reduce dependency on both N fertilisers synthesised with high energy input and imported P fertilisers derived from phosphate rock, as well as to mitigate negative environmental impacts of current practice. By reusing residues, nutrient cycles at farm-level can be further closed. Completely closed cycles are not to be realised as long as products for sale, e.g. animal products, food and feed, leave the farms. Returning these nutrients would require recovering them from urban waste and human excreta. The system approach still fails here and agriculture is not yet thought of as part of a societal circular economy. It is therefore all the more essential that all farms with residues from animal husbandry and/or biogas production rethink and optimise their residue management and make their own contribution to nutrient (re)cycling. Residue treatment approaches are adaptable to farm-specific and/or local requirements, whether directly on-farm, in collaboration with other farmers, at regional or even industrial scale. From basic solid-liquid separation, to P and/or N removal only with field-application of the remaining material, and up to complete processing into single nutrients, organic matter and water: suitable options can be found for every farm, as this work has demonstrated. Naturally, the highest environmental benefits of nutrient recycling and residue treatment using advanced recovery technologies can be realised on farms with high or excess residue amounts and limited arable land nearby. It is therefore highly recommended that these farms consequently implement one or more of the described treatment measures with priority. Additionally, the use of customised fertilisers that contain recycled nutrients in site- or crop-specific ratios will contribute to the production of better crops with higher quality while minimising the environmental footprint. It is obvious that sound residue management demands strategic anticipatory actions and capital investments from farmers and companies, but it is a crucial step towards the sustainable intensification of national and European cropping systems and towards resilient future agriculture. This work provides a small but essential piece of the puzzle. Consequently, the successful implementation of the presented measures must be accelerated through supplementary grants mainly for farmers. In addition, a reliable and clear legal framework is necessary for the production and utilisation of recycled fertilisers, supported by coherent and science-based political decisions.

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6 Curriculum Vitae

Personal details

Name: Andrea Bauerle (born Ehmann)
Date of birth: 13 July 1983
Place of birth: Stuttgart
Nationality: German

Professional experience

02/2012 - now

University of Hohenheim, Stuttgart
Institute of Crop Science, Department Biobased Resources in the Bioeconomy (340b)
Research Assistant, Doctoral Student

- PhD Thesis: „Suitability of recycled organic residues from animal husbandry and bioenergy production for use as fertilizers“
  Supported by a grant from State Graduate Funding (05/2012-04/2014)
- Research: Nutrient recycling from organic residues; lab, greenhouse and field experiments within research projects BioEcoSIM, GOBi, KuFe Performance, LEX4BIO, Agriplus Hohenlohe; scientific publications, writing project proposals, project management
- Assistant supervision of B.Sc. and M.Sc. theses
- Teaching: B.Sc. module „Produktionsverfahren und Stoffeigenschaften von Nachwachsenden Rohstoffen“; occasional lectures in M.Sc. modules

10/2016 – 06/2019

University of Hohenheim, Stuttgart
Institute of Landscape and Plant Ecology, Department of Plant Ecology and Ecotoxicology (320b)
Research Assistant
Innovation group APV-Resola

10/2011 – 02/2012

Winegrowing farm Ralf Bauerle, Fellbach
Winegrower

08/2010 – 09/2011

AgrarKontakte International (AKI) e. V., Stuttgart
Project manager
Agricultural internship programmes with Russia and Ukraine

05 – 07/2010	**Kuratorium für Technik und Bauwesen in der Landwirtschaft (KTBL), Darmstadt** **Research Assistant** EU-funded projects: AgriXchange, Transparent Food
10/2006 – 04/2010	**University of Hohenheim, Stuttgart** **Institute of Farm Management, Department Management in Agribusiness (410c)** **Student assistant**
08/2008 - 12/2009	**University of Hohenheim, Stuttgart** **Institute of Crop Science, Department Agronomy (340a)** **Student assistant**
08/2007	Internship LTZ Augustenberg, Department Organic Crop Protection, Stuttgart
03/2007	Internship Rebschule Tutzer, Bozen, Italy

Education

10/2008 – 04/2010	**University of Hohenheim, Stuttgart** **Master Programme Agricultural Sciences, major Crop Sciences, specialisation Phytomedicine** Supported by a grant from Südwestbank AG (10/2008 – 9/2009) Master‘s thesis: „Vergleich verschiedener Stickstoffdünger in ihrer Auswirkung auf Ertrag und N-Versorgung bei Silomais“ Degree: Master of Science, Grade: very good
10/2005 - 09/2008	**University of Hohenheim, Stuttgart** **Bachelor Programme Agricultural Sciences, major Crop Sciences** Bachelor‘s thesis: „Der Einfluss der Kalium- und Wasserversorgung auf den Gaswechsel der Rebe während der Reifephase“ Degree: Bachelor of Science, Grade: very good
09/2003 – 08/2005	**Weingut Wöhrwag, Stuttgart-Untertürkheim** **Apprenticeship** Certificate: Winzerin (Winemaker)
09/1994 – 07/2003	**Wirtemberg-Gymnasium, Stuttgart-Untertürkheim** Certificate: Abitur (General qualification for university entrance)

Skills

Language:	German: Native English: Fluent French: Basic Russian: Basic
Computing:	MS Office, SAS, R, SigmaPlot

Interview Seminar, NAWIK, Karlsruhe (03/2018)
Certificate of competence for crop protection (Sachkundenachweis Pflanzenschutz)
Certificate for instruction of apprentices (Ausbildereignungsprüfung)
Paramedic training German Red Cross

Publications

Trommsdorff, M., Kang, J., Reise, C., Schindele, S., Bopp, G., **Ehmann, A.**, Weselek, A., Högy, P. and Obergfell, T. (2021): Combining food and energy production: Design of an agrivoltaic system applied in arable and vegetable farming in Germany. *Renewable and Sustainable Energy Reviews* 140, 110694. doi: 10.1016/j.rser.2020.110694

Weselek, A., **Bauerle, A.**, Hartung, J., Zikeli, S., Lewandowski, I. and Högy, P. (2021) Agrivoltaic system impacts on microclimate and yield of different crops within an organic crop rotation in a temperate climate. *Agronomy for Sustainable Development* 41:59. doi: 10.1007/s13593-021-00714-y

Weselek, A., **Bauerle, A.**, Zikeli, S., Lewandowski, I. and Högy, P. (2021) Effects on Crop Development, Yields and Chemical Composition of Celeriac (*Apium graveolens* L. var. rapaceum) Cultivated Underneath an Agrivoltaic System. *Agronomy*, 11, 733. Doi: 10.3390/agronomy11040733

Schindele, S., Trommsdorff, M., Schlaak, A., Obergfell, T., Bopp, G., Reise, C., Braun, C., Weselek, A., **Bauerle, A.**, Högy, P., Goetzberger, A. and Weber, E. (2020): Implementation of agrophotovoltaics: Techno-economic analysis of the price-performance ratio and its policy implications. *Applied Energy* 265 (7263), 114737. doi: 10.1016/j.apenergy.2020.114737

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Weselek, A., **Ehmann, A.**, Zikeli, S., Lewandowski, I., Schindele, S. and Högy, P. (2019) Agrophotovoltaic systems: applications, challenges, and opportunities. A review. *Agronomy for Sustainable Development* 39:35. doi: 10.1007/s13593-019-0581-3

Ehmann, A., Thumm, U. and Lewandowski, I. (2018) Fertilizing potential of separated biogas digestates in annual and perennial biomass production systems. *Front. Sustain. Food Syst.* 2:12. doi: 10.3389/fsufs.2018.00012

Ehmann, A., Bach, I.-M., Laopeamthong, S., Bilbao, J. and Lewandowski, I. (2017) Can phosphate salts recovered from manure replace conventional phosphate fertilizer? *Agriculture* 7:1. doi: 10.3390/agriculture7010001

Conference contributions

Oral presentations

4. Berliner Klärschlammkonferenz, Berlin, Germany, 15.-16.11.2021
A. Bauerle, B. Müller, I. Lewandowski, T. Müller, K. Ylivainio
„EU-Projekt LEX4BIO – Projektvorstellung und Ergebnisse aus dem ersten Versuchsjahr mit biobasierten Düngern“

EBU Scientific Forum, online, 22.-23.09.2021
A. Bauerle, B. Müller, T. Karle, T. Müller, I. Lewandowski
“Efficiency Enhancement in Arable Farming in Hohenlohe based on Nutrient Recovery from Farm Manure: Agriplus Hohenlohe”

27th European Biomass Conference and Exhibition, Lisbon, Portugal, 27.-30.05.2019
M. Wagner, M. Zhumagulova, A. Weselek, P. Högy, S. Schindele, I. Lewandowski, **A. Ehmann**
“Agrophotovoltaics: combining the production of biomass and solar energy seen from a GHG perspective”

Seminar für Biogasanlagenbetreiber, DLR Eifel, Bitburg, Germany, 19.02.2019
A. Ehmann
„Düngepotential separierter Biogasgärreste in ein- und mehrjährigen Biomasse-Anbausystemen“

Fortschritt Gülle und Gärrest, Schwäbisch Hall, Germany, 17.10.2018
A. Ehmann, U. Thumm, I. Lewandowski
“The fertilising potential of separated biogas digestates in annual and perennial biomass production systems”

61. Tagung der Gesellschaft für Pflanzenbauwissenschaften, Kiel, Germany, 25.-27.09.2018
A. Ehmann, A. Weselek, S. Zikeli, I. Lewandowski, T. Schmid, F. Reyer, P. Högy
„Agrophotovoltaik – Auswirkung der Beschattung durch Solarmodule auf Bestandesentwicklung und Ertrag von Kulturpflanzen“

3rd ManuREsource Conference, Eindhoven, Netherlands, 27.-29.11.2017
A. Ehmann, I. Lewandowski, M. Calvo, J. Bilbao
“Validating the fertilizing performance of P and N salts recovered from pig manure in on-farm field trials in Germany and Spain”

17th RAMIRAN Conference, Wexford, Ireland, 04.-06.09.2017
A. Ehmann, I. Lewandowski, M. Calvo, J. Bilbao
“Validating the fertilizing performance of P and N salts recovered from pig manure in on-farm field trials in Germany and Spain”

16th RAMIRAN Conference, Hamburg, Germany, 08.-10.09.2015
A. Ehmann, I. Lewandowski
"Effect of manure-based P fertilizer and biochar on biomass yield of spring barley and faba bean in comparison to conventional fertilizer"

Progress in Biogas, Stuttgart, Germany, 10.-12.09.2014
A. Ehmann, U. Thumm, I. Lewandowski
"Fertilising potential of separated biogas digestates applied to annual and perennial biomass production systems"

56. Tagung der Gesellschaft für Pflanzenbauwissenschaften, Weihenstephan, Germany, 04.-06.09.2013
A. Ehmann, U. Thumm, I. Lewandowski
„Düngeeffekt separierter Biogasgärreste auf den Ertrag von Silomais"

Poster presentations

63. Tagung der Gesellschaft für Pflanzenbauwissenschaften, Rostock, Germany, 28.-30.09.2021
B. Müller, T. Müller, I. Lewandowski, **A. Bauerle**
„On-Farm Versuch mit Düngern aus der Aufbereitung von Biogasgärrückständen im Kreis Hohenlohe"

FNR-Fachtagung, online, 15.09.2020
L. Mack, B. Müller, T. Müller, I. Lewandowski, **A. Bauerle**
„Agriplus – Effizienzsteigerung im Ackerbau in der Region Hohenlohe durch Nährstoffrückgewinnung aus Wirtschaftsdüngern"

9th International Phosphorus Workshop, Zürich, Switzerland,08.-12.07. 2019
K. Ylivainio, E. Bünemann, J. Santner, **A. Ehmann**, A. Delgado
„Bio-based fertilisers for securing crop P requirement in the EU"

15. Wissenschaftstagung Ökologischer Landbau, Kassel, Germany, 05.-08.03.2019
A. Weselek, **A. Ehmann**, S. Zikeli, I. Lewandowski, P. Högy
„Agrophotovoltaik – Auswirkungen der Beschattung durch Solarmodule auf landwirtschaftliche Erträge"

23rd European Biomass Conference and Exhibition, Vienna, Austria, 01.-04.06.2015
A. Ehmann, I. Lewandowski
"Effect of manure-based ammonium sulfate and biochar on biomass yield and soil properties"

15th RAMIRAN Conference, Versailles, France, 03.-05.06.2013
A. Ehmann, I. Lewandowski
"Biomass ashes: characteristics and potential for use as fertilizer"

53. Tagung der Gesellschaft für Pflanzenbauwissenschaften, Stuttgart-Hohenheim, Germany, 28.-30.09.2010
A. Ehmann, S. Graeff-Hönninger, W. Claupein
„Vergleich verschiedener Stickstoffdünger in ihrer Auswirkung auf Ertrag und N-Versorgung bei Silomais"

Fellbach, 13.12.2021

Andrea Bauerle

7 Acknowledgement | Påskønnelse | Danksagung

First of all, I would like to thank Prof. Dr. Iris Lewandowski for being my supervisor and mentor and for giving me the chance to start this adventure of obtaining a PhD at the department. I cannot emphasise enough how much I appreciate your continuous support, your trust in me and your always open door despite your overflowing schedule. Your talent for finding the thread in manuscripts is unique and your way of "system thinking" has been a great inspiration. Thank you so much for everything!

Tak til Prof. Dr. Lars Stoumann Jensen for at biddrage som medvejleder på min afhandling. Din ekspertise på feltet er imponerende og jeg har lært en masse af dig. Tak for din indsats og opbakning.

I would also like to thank Prof. Dr. Torsten Müller for being part of my thesis advisory committee. Your enthusiasm for teaching and science is remarkable. Every meeting with you ends with constructive suggestions and new food for thought.

The research resulting in this thesis was partly performed in the BioEcoSIM project and received funding from the EU Seventh Framework Programme under grant agreement no. 308637. I am also grateful for the grant from State Graduate Funding (05/2012-04/2014).

Thanks to all my colleagues at 340b, for your company during all the years, for encouragement, for sharing expertise and many cakes. I am particularly grateful to my PhD colleagues Moritz Wagner, Andreas Kiesel, Yasir Iqbal, Anja Mangold and Elena Magenau, for the great office atmosphere, for our conversations on every imaginable topic and for many shared moments in and outside the office. Thanks to Laura Mack for the perfect management of my projects during my maternity leave. Thanks to Benedikt Müller for being part of the nutrient group and for taking over all my work tasks during the final phase of thesis writing. Special thanks to Jan Lask for patiently answering my countless questions before the submission of this thesis.

Many thanks to Nicole Gaudet for unlimited motivation in improving my English, teaching me the really important phrases, for revising my manuscripts and for advice in all aspects of life.

Ein ganz besonderer Dank gilt den Mitarbeitern der Versuchsstation Lindenhöfe, auf denen meine Feldversuche stattgefunden haben. Ganz besonders herzlicher Dank gebührt hierbei Peter Weckherlin vom Oberen Lindenhof, für Deinen unermüdlichen Einsatz und Deine große Zuverlässigkeit. Bei Dir waren meine Versuche jederzeit in den besten Händen und sind es immer noch.

Danke an das ganze Laborteam für die Analyse meiner unzähligen Proben und die Organisation drumherum. Vielen herzlichen Dank an Dagmar Mezger, dass ich immer mit allem zu Dir kommen konnte, für Deine große Hilfsbereitschaft und für die vielen Süßigkeiten in stressigen Zeiten. Ein weiterer Dank geht an Martin Zahner vom Ihinger Hof und sein Team, ebenfalls für die Analyse von Proben.

Herzlichen Dank an Thomas Ruopp für allerlei Materialien, Werkzeug und das zuverlässige Lösen meiner Computer-Probleme.

Danke an Nicole Janke und Andrea Serce aus dem Geschäftszimmer für die Hilfe bei allen organisatorischen Anliegen.

Danke an Jens Hartung für die Unterstützung bei der statistischen Auswertung meiner Ergebnisse.

Danke an alle Hiwis für den tatkräftigen Einsatz auf dem Feld, im Gewächshaus und im Labor, vor allem an Anja Neuberdt und Sven Schabel.

Die Promotion war ein langer Weg, der zum Glück mehr Höhen als Tiefen mit sich brachte. Ich bin froh und dankbar, dass ich auf diesem Weg nie alleine war und immer auf meine Freunde und meine Familie zählen konnte. Mein größter und ganz besonderer Dank gilt meiner Familie. Danke Papa, für Deine bedingungslose Unterstützung in all den Jahren und dass Du immer hinter meinen Entscheidungen gestanden hast. Danke Lotta, dass Du zuletzt so oft auf mich verzichtet hast. Danke Ralf, für Deinen Rückhalt und die Motivation während der ganzen Doktorandenzeit, aber besonders während der Schreibphase am Ende. Ohne Dich wäre das alles nicht möglich gewesen!