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Recovering phosphorous from biogas fermentation residues indicates promising economic results

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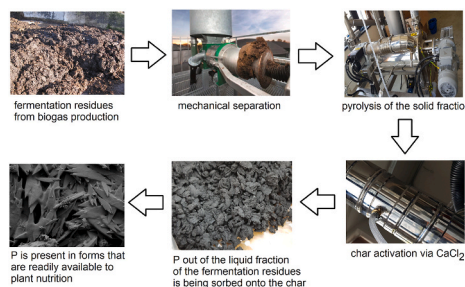
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HIGHLIGHTS

- There is urgent demand for nutrient recovery techniques worldwide.
- Fermentation residues are dewatered, charred and activated by calcium chloride.
- Phosphorus captured by this sorbent is readily available to plant nutrition.
- Novel methods outperforms established methods of phosphorus regeneration.

GRAPHICAL ABSTRACT



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ABSTRACT

The economics of producing energy-valuable gases by fermenting phytomass is deteriorated by the costs associated with waste management of highly diluted (typically 95% water) fermentation residues (FR). Previously, no better solution was known than to plough FR into the arable land and claim that it is an irrigation with soil improving and fertilizing effect. However, farmers soon realized that FR organic matter is of little agronomic value and nutrients are at agronomically insignificant levels. As FR watering has proved economically irrational in many countries the practice of separating water from the FR and using the solid fraction for energy purposes (such as charcoal) has dominated. However, most nutrients are lost in this way. For the first time it is proposed to activate the charred FR via calcium chloride (whose price is insignificant as it would be used for fertilization purposes anyway) and using the resulting sorbent to capture phosphorus (P) out of the FR's liquid fraction. It is reported for the first time that the activated char is capable of capturing $37.5 \pm 4.7 \text{ kg P t}^{-1}$ whereas the P availability for plant nutrition outperforms FR as well as struvite. In addition, the char demonstrates the potential to improve soil characteristics and the metabolism of soil biota. The cost breakdown and subsequent market analysis indicates that the novel fertilizer shows signs of competitiveness.

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1. Introduction

1.1. The current state of FR management

The number of biogas plants in the world has been sharply rising over the last three decades. The largest number of biogas plants (some 40 million) is located in China, and are mainly village projects that process post-harvest residues and manure (Wang et al., 2020). Scarlat et al. (2018) reported that there are only about 20 k biogas plants in the EU. However, their robustness and technological level imply that the EU biogas industry generates about half of the world's biogas production (electricity: 15 GW, heat 1 EJ, biomethane for vehicles: 1.3 G m³), (Maroušek et al., 2020a). Biogas production in the USA faces multiple barriers and is not expected to reach 3% of the domestic natural gas market by 2040 (Murray et al., 2017). The most widespread biogas plants in the world are agricultural (Kougiás and Angelidaki, 2018). Farm biogas plants often process various combinations of manure, post-harvest residues and purposefully grown phytomass which altogether results in a huge production of FR that contains from approximately 90% up to 98% water (Stehel et al., 2019). From a global perspective FR contains considerable amounts of nutrients (0.5% N; 0.1% P and 0.3% K on average) but its economic value is questioned because it is dissolved in a huge amount of ballast water (Valaskova et al., 2020). To reduce logistics costs, the FR is sometimes mechanically separated, where the higher the fibre content the higher the efficiency of separation (Dědina and Šánová, 2013). Bauer et al. (2009) report that ordinary commercially available separators divide FR into four fifths of liquid fraction (dry matter content of some 5%) and one fifth of solid fraction (up to 30% dry matter content). From an economic point of view, another problem is that most nutrients are present in organic forms (Stehel et al., 2018) that need to be firstly mineralized by soil biota to become valuable for plant nutrition (Maroušek et al., 2020b). Given the global energy prices (Vochozka et al., 2020), the solid fraction of the FR is either sun-dried for combustion (Judít et al., 2017) or charred via waste heat from biogas combustion engines (Mardoyan and Braun, 2015) instead of being used as a fertilizer (Kliestik et al., 2018). Provided that the dominant nutrient in FR is nitrogen (N) its recovery into ammonium nitrate (via nitric acid, HNO₃) and ammonium sulfate (sulfuric acid approach) is intensively investigated (Valentinuzzi et al., 2020). However, both techniques require costly setup of technology and external energy sources (Jandačka et al., 2017) which makes N regeneration from FR uncompetitive at the moment (Machová and Vrbka, 2018). With regards to P recovery, many methods can be traced in the literature (Muo and Azeez, 2019), but the Modified University of Cape Town process (Stávková and Maroušek, 2021) and precipitation into struvite (Bradford-Hartke et al., 2021) or vivianite (Wu et al., 2019) are the most frequently investigated (Blažková and Dvouletý, 2018). However, all these methods were originally designed to achieve the best cost effectiveness in terms of preventing P leakage from wastewater treatment plants into water systems rather than for turning P into forms readily available for plant nutrition (Li et al., 2019).

1.2. The background of P recovery

P is currently the most expensive nutrient (Li et al., 2019) that plays an irreplaceable role in various life processes (both in plants and animals) and the vast majority of countries are dependent on imports (Rowland et al., 2019). Nine tenths of mined phosphorites are used for production of fertilizers such as superphosphates (Nätörp et al., 2017). The most well-known technique for capturing P is precipitation with iron salts into struvite. Shaddel et al. (2019) note that the main advantage of struvite is that it is composed of primary macronutrients (P and N) and a secondary macronutrient (magnesium, Mg). However, as demonstrated by Nätörp et al. (2017) pricing of iron salts (from 0.98 k USD up to 1.1 k USD t⁻¹) means that the specific cost for P (4 € kg⁻¹ at minimum) captured into struvite is roughly double that of conventional

P fertilizer (Khurshed et al., 2018). To make matters worse, struvite, hydroxylapatites or vivianite are minerals with complex nucleation and sturdy crystal morphology, therefore only limited fractions of precipitated P are readily available in terms of plant nutrition (Kulhánek et al., 2019). Suppliers of P-removal solutions for sewage treatment plants have for decades argued that all the extractable P (Pe) in these minerals is available for plant nutrition (Bradford-Hartke et al., 2021). However, the latest research has proved that only calcium phosphates (CaP, immediately available to plant nutrition) followed by aluminum phosphates (AlP, accessible in a few years) are valuable in terms of plant nutrition. The remaining iron phosphates (FeP) will take decades to become available for plant intake (Maroušek et al., 2020c). What is worse, the process of turning nutrients into mineral forms can be slowed down if not enough microorganisms are present or optimal soil moisture is not met. According to Kolář et al. (2009) the P management in soil is also driven by the type of clay minerals, the characteristics of soil organic matter (SOM), the presence of Fe; Al; and pH (strongly alkaline or acidic soils depress P bioavailability). Application of medium and slow-release P fertilizers is beneficial for growing trees because its natural source in soil is mainly organic matter (OM), which occurs most often in the upper layers of the topsoil (Van Geel et al., 2016). Given that trees receive P entirely through the roots (Yang et al., 2016), and the commercial growth of most fruit trees is about 20 years, it is advantageous if the P fertilizer is equally based on CaP (for the initial phase of growth) and AlP (for the gradual release of P to form fruits).

1.3. Urgency, economic justification and research hypothesis

P availability is a mandatory prerequisite for sustainable agricultural production (Kliestik et al., 2020). Several full (or pilot) scale technologies for P recovery from wastewater (mostly precipitation via iron salts) are in operation worldwide and many other technological alternatives have been demonstrated and are considered technically feasible (Hukari et al., 2016). Nevertheless, under current economic conditions, P regeneration is far from profitable and is carried out only as a necessary condition for compliance with wastewater discharge limits (Bashar et al., 2018). Even if the running costs of these P precipitation processes were reduced to compete with conventional mineral fertilizers it would not make a big difference. Despite many efforts carried out to reduce the size and morphology of the precipitated crystals, the P remains firmly locked and unavailable for plant nutrition (Kulhánek et al., 2019), making it unusable for agriculture without further modifications (Kratz et al., 2010).

Provided that only the CaP and AlP are readily available for plant intake, it is hypothesized that activation of charred FR via calcium chloride (CaCl₂) could result in the production of sorbent capable of capturing the most valuable forms of P. In addition, according to Smetanová et al. (2013) the char is expected to reduce soil density, increase pH and improve water retention.

2. Materials and methods

2.1. Novel sorption method

The FR originated from a farm biogas station (1.2 MW electricity and 1.1 MW heat) that processes maize silage (65%) and cow manure (35%) for 40 days at 42 °C (characteristics given in Table 1). The liquid fraction was mechanically removed using the SEPKOM separator (WAMGROUP, Italy). Subsequently, the solid fraction was dried at 60 °C for 40 min to reduce the water content to 10% using a Susicka400 tumble dryer (Votona, Czech Republic) where the drying medium was excess heat from the pyrolysis unit. The dried FR was charred (370 °C for 14 min) in the UHL-07 pyrolysis unit (AIVOTEC, Czech Republic) that is run by heat recovered from the JMS 416 biogas combustion unit (GE Jenbacher OG, Germany). Char characteristics are given in Table 2. The char was added to 15% CaCl₂ (CIECH, Poland) and slowly stirred (5 rpm) for 2 h

Table 1

Characteristics of FR, struvite and NF (n = 12; $\alpha = 0.1$) show that all products meet all the regulations for use as fertilizers, ULD = under limit of detection; NA = not applicable.

Parameter	FR	Struvite
RMP (m ³ t ⁻¹)	12.1 ± 4.3	NA
LP1 (%)	1	NA
LP2 (%)	26	NA
LP3 (%)	73	NA
BOD ₅ (g L ⁻¹)	0.4 ± 0.1	NA
COD _{Cr} (g L ⁻¹)	1.1 ± 0.1	NA
OM (kg t ⁻¹)	67.9 ± 14.4	NA
N (kg t ⁻¹)	2.4 ± 1.1	NA
K (kg t ⁻¹)	0.3 ± 0.1	NA
Pe (g t ⁻¹)	110.7 ± 62.3	15.1 ± 7.8
FeP (g t ⁻¹)	19.5 ± 9.0	10.1 ± 6.3
AIP (g t ⁻¹)	78.7 ± 31.6	3.9 ± 2.5
CaP (g t ⁻¹)	12.5 ± 8.2	1.1 ± 0.8
Ag (mg kg ⁻¹)	ULD	ULD
As (mg kg ⁻¹)	ULD	ULD
Cd (mg kg ⁻¹)	ULD	ULD
Co (mg kg ⁻¹)	ULD	ULD
Cr (mg kg ⁻¹)	0.2 ± 0.1	0.1 ± 0.1
Cu (mg kg ⁻¹)	0.1 ± 0.1	ULD
Hg (mg kg ⁻¹)	ULD	ULD
Mn (mg kg ⁻¹)	1.1 ± 0.6	0.3 ± 0.1
Ni (mg kg ⁻¹)	ULD	ULD
Pb (mg kg ⁻¹)	ULD	ULD
Sb (mg kg ⁻¹)	ULD	ULD
Se (mg kg ⁻¹)	ULD	ULD
Sn (mg kg ⁻¹)	ULD	ULD
Tl (mg kg ⁻¹)	ULD	ULD
Zn (mg kg ⁻¹)	3.5 ± 0.2	0.2 ± 0.1
Naphthalene (ppm)	ULD	ULD
Acenaphthylene (ppm)	ULD	ULD
Acenaphthene (ppm)	ULD	ULD
Fluorene (ppm)	ULD	ULD
Phenanthrene (ppm)	ULD	ULD
Anthracene (ppm)	ULD	ULD
Fluoranthrene (ppm)	ULD	ULD
Pyrene (ppm)	ULD	ULD
Benzo(a)anthracene (ppm)	ULD	ULD
Chrysene (ppm)	ULD	ULD
Benzo(b)fluoranthrene (ppm)	ULD	ULD
Benzo(a)pyrene (ppm)	ULD	ULD
Indeno(1,2,3-cd)pyrene (ppm)	ULD	ULD
Dibenzo(a,h)anthracene (ppm)	ULD	ULD
Benzo(g,h,i)perylene (ppm)	ULD	ULD
Benzene (ppm)	ULD	ULD
Toluene (ppm)	ULD	ULD
Ethylbenzene (ppm)	ULD	ULD
m-xylene (ppm)	ULD	ULD
p-xylene (ppm)	ULD	ULD
o-xylene (ppm)	ULD	ULD
Naphthalene (ppm)	ULD	ULD
EOH (ppm)	ULD	ULD
AHC (ppm)	ULD	ULD
ATF0.01 (%)	0	0
ATF0.1 (%)	0	3
ATF1 (%)	1	17
ATF10 (%)	0	65
ATD0.01 (%)	0	0
ATD0.1 (%)	0	9
ATD1 (%)	2	16
ATD10 (%)	0	33
ATA0.01 (%)	0	0
ATA0.1 (%)	0	5
ATA1 (%)	0	8
ATA10 (%)	0	26
ATS0.01 (%)	0	0
ATS0.1 (%)	0	0
ATS1 (%)	0	0
ATS10 (%)	0	0
BET (m ² g ⁻¹)	0.6 ± 0.5	0.2 ± 0.1
SP (m ² g ⁻¹)	0.2 ± 0.1	0.1 ± 0.1
L (m ² g ⁻¹)	0.1 ± 0.1	0.1 ± 0.1
MP (m ² g ⁻¹)	ULD	0.1 ± 0.1

Table 1 (continued)

Parameter	FR	Struvite
E (m ² g ⁻¹)	0.1 ± 0.1	0.1 ± 0.1
pH	7.7 ± 0.4	7.7 ± 0.3
EC (mS cm ⁻¹)	22.9 ± 15.0	NA
CEC (mol H ⁺ kg ⁻¹)	3.1 ± 2.9	NA

Table 2

Interactions of FR and NF with soil structure and activity of soil biota represented as % change (0 t ha⁻¹ = 100%, average value from 6 samples).

	5 t ha ⁻¹		10 t ha ⁻¹		15 t ha ⁻¹		20 t ha ⁻¹	
	FR	NF	FR	NF	FR	NF	FR	NF
TP (%)	0	0	-1	0	-2	3	-4	7
AFP (%)	0	0	0	2	-1	5	-3	8
WR (%)	-1	0	-2	1	-4	0	-5	7
BR (%)	1	0	1	3	2	7	1	12

12 cm thick permeable mattresses were filled with the activated char and immersed for another 2 h in a slowly stirred liquid fraction of FR that had been previously filtered through a 4.8 m³ char filter (to reduce the content of OM). This product is hereinafter called 'novel fertilizer' (NF). All reactants are of industrial purity.

2.2. Conventional precipitation method (struvite)

The filtered liquid fraction of FR was poured into an intensively oxygenated reactor (200 L O₂ minute⁻¹ m⁻³) and slowly stirred (20 h at 5 rpm). Magnesium chloride hexahydrate (MgCl₂ × 6H₂O, PENTA, Czech Republic) was added to obtain a Mg:P ratio of 1.3:1 (molar weight). Subsequently, ammonia (NH₃, PENTA, Czech Republic) was added to establish a ratio of N:P to 3:1 (molar weight). The precipitate was separated by a centrifuge (450 rpm for 1 h) followed by 24 h of drying at 40 °C. All reactants are of industrial purity.

2.3. Analytical procedures

The humidity level was evaluated using an AS 310.3Y automatic analytical balances with internal calibration (RADWAG, Czech Republic) and by drying samples for 72 h at 105 °C in a DUROCELL dryer that is coated with a chemically resistant layer (BMT Medical Technology, Czech Republic). Residual methane (CH₄) production (RMP, converted to methane at 0 °C and 101.3 Pa) was measured for 20 days at 24 °C by a battery of Stix-03 automatically monitored semi-continuous batch reactors (Stix Ltd., Czech Republic). Labile (LP₁); semi-labile (LP₂) and resistant (LP₃) pools of carbon (C) were assessed via resistance to acid hydrolysis at elevated temperatures (sulfuric acid approach) according to Shirato and Yokozawa (2006) using an EA5100 elemental analyzer (Analytik Jena AG, Germany). Biological and chemical oxygen demands (BOD₅ and COD_{Cr}) were analyzed as stated in Vochozka et al. (2016a). Total macronutrients (potassium, K; nitrogen, N and phosphorus, P); heavy metals suspected of adverse biological effects (silver, Ag; arsenic, As; cadmium, Cd; cobalt, Co, chromium, Cr; copper, Cu; mercury, Hg; manganese Mn; nickel, Ni; lead, Pb; antimony, Sb; selenium Se; tin, Sn; thallium, Tl and zinc, Zn); polycyclic aromatic hydrocarbons (PAH) (naphthalene; acenaphthylene; acenaphthene; fluorene; phenanthrene; anthracene; fluoranthrene; pyrene; benzo(a)anthracene; chrysene; benzo(b)fluoranthrene; benzo(k)fluoranthrene; benzo(a)pyrene; indeno (1,2,3-cd)pyrene; dibenzo(a,h)anthracene; benzo(g,h,i)perylene; benzene; toluene; ethylbenzene; m-xylene; p-xylene; o-xylene; naphthalene); extractable organic halogens (EOH), and aromatic hydrocarbon compounds containing 10–40 hydrocarbon atoms (AHC) were analyzed according to Vochozka et al. (2016b) using a 8860 series gas chromatograph equipped with a quadrupole 5977B series mass-selective detector (both Agilent, USA). Acute biotoxicity (at 0.01; 0.1 and 1%

concentration) to fish *Poecilia reticulata* (ATF); daphnia *Daphnia subspicatus* (ATD); freshwater algae *Desmodesmus subspicatus* (ATA); and seeds *Sinapis alba* L. (ATS) was measured as stated in Vochozka et al. (2016c). Microporosity was analyzed using a 3H-2000PM1 pore size and surface analyzer (Tmax BE, China). Brunauer-Emmette-Teller surface area (BET); single-point surface area (partial vapor pressure of helium in equilibrium with the surface)/(saturated pressure of helium) = 0.1 (SP); Langmuir surface area (L); micropore area (MP) and external surface area (E) were analyzed after 10 h of degassing at 400 °C. pH and electrical conductivity (EC) was investigated via a Seven2GoPro multimeter (Mettler-Toledo, Switzerland). Cation-exchange-capacity (CEC) was measured in agreement with the procedure suggested by Munera-Echeverri et al. (2018). The sum of mineral and mineralizable nitrogen (N) was determined according to Figueroa-Barrera et al. (2012). The ammonium lactate soluble potassium (K₂O) content was assessed by a PinAAcle 500 flame atomic absorption spectrometer (Perkin Elmer, USA) with continuum source background correction according to the method specified by Jakab (2020). P readily available for plant nutrition (Pa refers to sum of CaP1; CaP2; CaP3 and AlP), was analyzed according to Stávková and Maroušek (2021). The level of OM was analyzed according to Kolář et al. (2009). The novel fertilizer was scanned by a JSM-7401F field emission scanning electron microscope (JEOL, Japan) with a cold cathode field emission gun at 4 kV. In an effort to prevent charging of the sorbent, samples were coated with approximately 7 nm of white gold using a JFC-1600 sputtering device (JEOL, Japan) before scanning. Interactions with the soil structure were investigated for different application doses (0; 5; 10; 15 and 20 t ha⁻¹) after one growing season by analyzing total porosity (TP); air filled porosity (AFP) and water retention after 48 h of free drainage (WR), all measured as described in Maroušek et al. (2016). Activity of soil biota was investigated by analyzing basal respiration (BR). 41 EU respondents took part in the survey. Cost breakdown was performed as suggested by Hašková (2017), all the costs are rounded up. All reactants are of analytical purity.

3. Results and discussion

3.1. Fermentation residues

The use of raw FR from biogas stations as fertilizer is gradually falling, yet this practice is common in countries which have limited access to capital and know-how (Insam et al., 2015). The average long-term biogas yield of the biogas station under investigation is 130.5 m³ t⁻¹ which (related to dry weight of feedstock and after conversion to CH₄ at 0 °C and 101.3 Pa) is 5% lower than reported by Samarappuli and Berti (2018) and some 6% lower than reviewed by Bőjti et al. (2017). The reason may be that the fermentation time of 40 days is quite short. The same finding can be made based on the survey carried out by Bauer et al. (2009), who report average hydraulic retention times between 45 and 83.7 days. Reduced performance is also indirectly indicated by analysis of the FR (Table 1) which reveals that after 40 days in the biogas station the FR are still able to release almost 12.1 m³ t⁻¹ of additional CH₄ over a further 20 days, which is almost 15% of the original yield. Such a result is in good agreement with the analysis of the amount of unhydrolyzed OM (LP₁ and LP₂) which is almost twice that reported by Vochozka et al. (2016b). Both BOD₅ and COD_{Cr} are roughly 9% higher than FR investigated by Kolář et al. (2009) which is another argument to support the assumption that the hydraulic retention time of 40 days may not be sufficient, and its prolongation could be reconsidered. The level of N is roughly 8% higher than reported by Bauer et al. (2009), whereas levels of P and K are average. This may be due to a slightly higher proportion of manure that is rich in N. In comparison to Insam et al. (2015), levels of heavy metals remain low, mostly under the limit of detection (ULD). Nevertheless, the findings of Ungerman et al. (2018) suggest that given their long evolutionary history, the anaerobic consortia are inhibited only by high concentrations of heavy metals.

Levels of organic pollutants (PAH; EOH and AHC) are also low, mostly ULD. It can be assumed that agricultural biogas plants generally have lower levels of organic pollutants than biogas plants that treat various types of communal waste (Dědina and Šánová, 2013). It is therefore not surprising that the FR showed negligible acute biotoxicity in some cases even in 10% concentration (Prokopchuk et al., 2018). In summary, the characteristics of the FR indicate that there are still some challenges in optimizing the processing parameters of the biogas plant. However, the results obtained comply with established standards and do not deviate from data found in the reviewed literature. Therefore, the FR under investigation can be understood as a representative sample and can be used to generate broad generalizations for the commercial practice of agricultural biogas plants.

Table 1 also confirms that the application of FR adds a considerable amount of OM to the soil. However, Kolář et al. (2009) point out that most of these substances are stabilized (with a higher degree of aromaticity) and hard to biodegrade (slowly mineralizable by soil biota), not providing soil microorganisms enough easily available C as an energy source. As knowledge has grown over recent decades, there is a growing consensus that the benefits from this OM are worthless, or even negative, because it can form hard-to-permeate layers on the soil surface that prevent water infiltration (Cepel, 2019). Table 2 presents analyses of the soil structure and the activity of the soil biota.

The experiment over one vegetation season indicated that increasing higher doses of FR tends to worsen soil structure characteristics and the metabolisms of soil microorganisms as well. It should be admitted that the experiment is quite short, and it would be better to process a longer time series. Nevertheless, according to Kolář et al. (2009) the mechanism is clear (repeatedly and independently confirmed), provided the most readily available organic C was already hydrolyzed in the biogas station during the anaerobic fermentation process. Volatile fatty acids are broken down by more than 90%, hemicellulose more than 80%, cellulose more than 50%, while lignin is not decomposed by anaerobic consortia at all (Kolář et al., 2009). According to Insam et al. (2015) the OM (including the nutrients it contains) of the FR is further mineralized in the soil, but to a much lesser extent than after the application of manure (approximately 4 times less than cattle manure). With the delayed mineralization of organic C in the FR, the activity of soil microorganisms (e.g. ammonification and denitrification bacteria) also decreases and intensive application of FR tends to reduce the content of SOM by consuming readily available C from the soil (Kolář et al., 2009). In other words, imperfect processes in biogas plants result in FR that have more easily mineralizable OM and are therefore more valuable from an agronomic point of view. The amount of ammonium lactate soluble K, mineral and mineralizable N and plant-accessible forms of P is similar to that found in the reviewed literature (Vo, 2020).

3.2. Struvite precipitation

Table 1 indicates that the properties of the struvite precipitated from the liquid fraction of the FR are similar to commercially available struvite products obtained from comparable liquid biowaste (Bradford-Hartke et al., 2021). Literature confirms that this recognized precipitation technique allows negligible levels of critical heavy metals and organic pollutants to be maintained even from sludge water (Chen et al., 2020) so it is no surprise that the struvite obtained from the FR shows only negligible traces of inhibitors (Table 1). Nevertheless, there are some signs of acute biotoxicity to water organisms. However, these are in line with other reports (Tansel et al., 2018) and occur only at high concentrations. Generally speaking, these numbers are similar to commercially available struvite-based fertilizers and meet all the corresponding regulations (Nättorp et al., 2017). Most importantly, no inhibition to plant growth was detected. Contrary to the claims of suppliers of struvite crystallization technologies, it shows that the availability of P from struvite crystals for plant nutrition is low (only one third of Pe is from CaP and AlP that is readily available for plant intake).

This may be yet another independent result which proves that the dark side of struvite precipitation in wastewater treatment plants has been deliberately downplayed for decades. The results obtained are in agreement with latest literature (Stávková and Maroušek, 2021) and it can be inferred that the low availability of P for plant nutrition is caused by struvite being a hexahydrate which crystallizes in an orthorhombic structure (straight prisms with a rectangular base) that is subject to biological decay over long periods (Shaddel et al., 2019). Being an orthophosphate fertilizer levels of P, Mg and NH₄ in identical molar concentrations. All things considered, it can be argued that levels of other nutrients in the struvite produced from FR are in good agreement with Bradford-Hartke et al. (2021), only the efficiency of its production is lower, because the presence of P in FR is lower than in sludge waters. Soil acidification should not be a problem provided the pH is comparable to other struvite products on the market (Tansel et al., 2018).

3.3. Novel fertilizer (NF)

As far as charring of the dewatered FR, the energetic balance of the material flows did not allow the operating temperature of the pyrolysis chamber to be increased steadily over 370 °C and the hydraulic retention time of the pyrolytic process to be prolonged over 14 min. Analyses of microporosity show values that are half to one third that of char produced from various agricultural wastes (Bencsik et al., 2018). It is worth mentioning that the microporosity characteristics are strongly predetermined by the temperature dynamics (in particular the highest temperature reached) during the pyrolysis process. Such a problem could be solved by using an external energy source (Jandačka et al., 2017). However, this would significantly affect the production cost. Nevertheless, even such reduced porosity was sufficient to improve the soil structure and the metabolism of soil biota (Table 2). A closer look at Table 1 reveals that the levels of heavy metals slightly increased during the pyrolysis process. This is due to the concentration of the material (Mardooyan and Braun, 2015) as a result of the pyrolysis process. However, all the levels of critical heavy metals remain well within the level for safe use in agronomic operations (Nefzi, 2018). Similarly, levels of organic pollutants (PAH; EOH and AHC) slightly increased during charring but remain fully in accordance with established agricultural standards and safety regulations (Kliestik et al., 2020). Charred FR shows no acute biotoxicity. In comparison to chars obtained from agricultural biowaste the pH is normal (Smetanová et al., 2013). Results from EC and CEC are also in agreement with existing literature (Gabhi et al., 2020). In brief, we can say that charred FR is just a little worse than chars applied in commercial practice and results from subsequent experiments are widely transferable. The porosity of the charred FR

(Fig. 1) allowed char to be activated with 1.2–2 kg of CaCl₂, which allowed the capture of 37.5 ± 4.7 P kg t⁻¹ (~261.5 g Pe t⁻¹). Most importantly, Table 1 shows that a large proportion of the P captured is present in forms readily available for plant nutrition (CaP + AIP).

3.4. Economic considerations

Capturing P from FR is in intensive demand from farmers because its application has been repeatedly and independently shown to be loss-making (Streimikiene, 2021). Getting FR ready for soil application only requires large storage tanks which have a lifespan of 15 years and usually cost 150 k€. Production of struvite is affected mainly by the costs of the reactants (Hnatyshyn, 2018). The precipitation technology costs roughly 0.2 M € and is expected to serve for 10 years. Provided that the vast majority of energy needed for NF production is obtained from waste hot flue gases and pricing of the CaCl₂ is irrelevant as it would be used for fertilization purposes anyway, the cost of the NF is driven mainly by depreciation of the pyrolysis unit (lifetime 10 years). However, production cost is only part of the story (Nwaiwu, 2018). The complexity of the phenomena can be understood from Table 3, which reveals that the low availability of nutrients present in the FR is not the only obstacle (Androniceanu et al., 2020). It is also the high dilution which significantly increases the application cost and worsens the cost competitiveness of FR against fertilizers with higher concentrations of nutrients (Nguyen, 2020). This was indirectly confirmed by a questionnaire on the willingness to pay, which confirmed that no farmer intended to accept FR even for free. On the other hand, it was also revealed that the production costs of the struvite obtained are only 2% lower than the average market price. This can be explained by 2 simultaneously acting factors. First, the experimental technology cannot compete with mass production in terms of efficiency, therefore the experimental production costs must be higher (Svazas et al., 2019). Nevertheless, the difference is so

Table 3
Financial analysis.

	FR	Struvite	NF
Energy (€ 1 kg Pe ⁻¹)	0	23	6
Reactants (€ 1 kg Pe ⁻¹)	0	5991	0
Labor (€ 1 kg Pe ⁻¹)	0	265	15
Depreciation and overheads (€ 1 kg Pe ⁻¹)	103	860	198
Production cost (€ 1 kg Pe ⁻¹)	103	7139	213
Cost (€) per application of 1 kg Pe ha ⁻¹	594	7199	273
Willingness to pay (€ 1 kg Pe ⁻¹)	0.0 ±	7043.2 ±	221.1 ±
	0.0	75.5	80.5

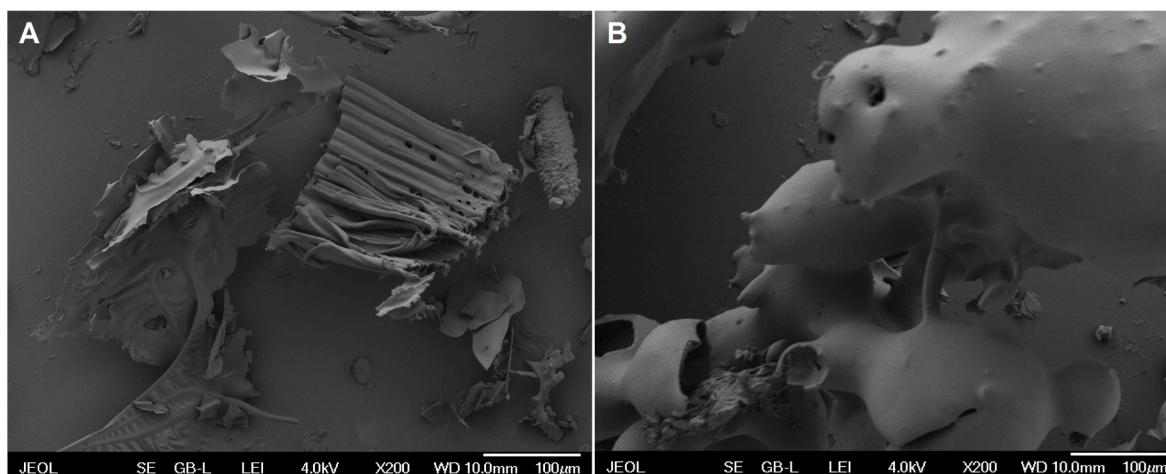


Fig. 1. Scans from field emission scanning electron microscope show that charred FR (A) is mainly represented by porous plant fibres, whereas after CaCl₂ activation and sorption of phosphates (B) its morphology is significantly changed and is accompanied by a reduction of microporosity.

low that it can be inferred that the seller does not reflect the full production cost. This is due to the fact that a significant part of these costs is paid by the municipality for water treatment which puts the regeneration of P from FR at a disadvantage. However, only until then are soil-affecting properties like water retention taken into account. Farmers showed a good awareness of the low quality of the OM in FR and declared an interest in NF that is based on char.

4. Conclusion

It was firstly demonstrated that charred fermentation residues from a biogas station activated by CaCl_2 (a) allows the capture of some 117.9 g CaP and 134.2 g ALP per 1t of its liquid fraction and (b) are capable of improving soil properties (at 20 t ha⁻¹) as follows: total porosity +7%; air filled porosity + 8%; water retention + 7%; basal respiration + 12%. Most importantly, the production cost (213 € 1 kg Pe⁻¹) is some 3% lower than the amount that the farmers are willing to pay for such a product.

Author contribution statement

Josef Maroušek: Writing – original draft, Conceptualization, Findings, Reviewing and Data curation. Beata Gavurová: Editing, reviewing and Data curation.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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