### ESPP – DPP – NNP nutrient recovery technology catalogue <a href="http://www.phosphorusplatform.eu/techcatalogue">http://www.phosphorusplatform.eu/techcatalogue</a>

#### Disclaimer:

This document aims to provide an indicative overview, not technical information to support decision making. It is accurate to the best of our knowledge, but further information and updates should be sought from the indicated contacts. The information included has been discussed between ESPP and the technology suppliers, and in general validated by these companies. However, ESPP, DPP and NNP do not have resources necessary to audit information provided and information is included as provided by the companies. Inclusion in this document does not constitute any endorsement of technology(ies) by the nutrient platforms, nor validation of intellectual property nor commercial claims.

Sewage P-recovery: full scale plants of	perating or	CarboREM	14	Susphos	24
under permitting/construction	2	Other nutrient recovery TR6+	15	Spodofos (ThermusP)	24
Fertiliser industry – E.g. ICL, Borealis	2	N2-Applied	15	SIMPhos-process (Cirkel)	25
Ash2Phos (EasyMining)	3	Ash2Salt (EasyMining)	15	PHOSPHIX™ (Clean TeQ Water)	25
TetraPhos (Remondis)	4	Aqua2N (EasyMining)	16	Update not completed 2022 or underway	26
PAKU (Endev)	5	DUCTOR	16	HTCycle	26
EuPhore	6	Anuvia Plant Nutrients	17	HAIX ion exchange (LayneRT™)	26
TerraNova (HTC)	7	Hitachi Zosen	17	LYSTEK	27
Kubota	7	AMFER (Colsen)	17	Struvite enhanced: acid (MSE-mobile)	27
PHOS4Green (Glatt)	8	CCm	18	GENIUS (Nijhuis Saur Industries)	27
Metawater alkaline ash leaching	8	Agro America (VP Hobe)	18	RePeat (Nijhuis Saur Industries)	28
Struvite precipitation	9	Parforce	19	Byosis (Nijhuis Saur Industries)	28
Struvite enhanced	10	Slurry acidification	19	Varcor	28
Sludge lysis	10	STERCORE pyrolysis	20	Currently no longer under development (	
Renewable Nutrients	11	Pyreg (pyrolysis)	21	knowledge) - or integrated into other pro	cesses29
Sewage P-recovery: (TRL 6+)	12	Technologies at R&D scale	21	Extraphos	29
RAVITA (Helsinki HSY)	12	Prayon	21	RecoPhos thermal	29
Phos4Life (ZAR – Técnicas Reunidas)	12	AguaDB	22	BioEcoSim (Suez)	29
ViViMag® (WETSUS - Kemira)	13	Flashphos (Uni. Stuttgart, Italmatch)	22	ePhos (Fraunhofer IGB)	30
Kemira iron / aluminium phosphate	13	RSR (Green Sentinel)	23		
AshDec (Metso Outotec)	14	P-roc	23		







Process & contact | Input materials | Output products | Process description | Operating status | Photos

### Sewage P-recovery: full scale plants operating or under permitting/construction

# Fertiliser industry – E.g. ICL, Borealis ...

Members of ESPP / DPP / NNP

http://icl-groupsustainability.com/reports/pro ducing-fertilizers-withrecycled-phosphate/

Contact (ICL): anthony.zanelli@iclgroup.com Input: sewage sludge incineration ash, from sewage works using biological and/or chemica P-removal; animal byproduct ash (Cat 2, 3); recovered phosphate salts.

Standards mineral fertilisers.

Fertiliser production plant must have operating permit authorising to process waste. Recovered materials are mixed into the phosphate rock or phosphoric acid based fertiliser production process, either during acid attack of rock, or after this stage where product still has residual acidity (acidulation), so ensuring plant availability of P in ashes.

Contaminants in ash are diluted in final product. This is legal under EU regulation on condition that the ash is not classified as "Hazardous". Final fertiliser product is covered by EU Fertilising Products Regulation 'STRUBIAS' annexes as proposed.

Recovery rate (P in final product / P in input ash): c. 100%

Iron and aluminium in input ash are transferred into final product
Heavy metals are not removed.

ICL tested full scale and industrial installations now operation at ICL Netherlands (inaugurated March 2019, photo) and Germany (several hundred tonnes ash and struvite processed to date). Production from 100% ashes (without mixing with phosphate rock) is planned.

Use of ash in fertiliser production has also been tested at by Borealis, Austria and by Fertiberia Spain (MBM ash at lab scale)









## Ash2Phos (EasyMining)

#### Member ESPP, DPP

http://easymining.se/

Contact: <u>Jan.svard@ragnsells.com</u>

Updated 10/2021

Input: sewage sludge incineration ash, from sewage works using biological and/or chemical P-removal.

Calcium phosphate; can be converted into – superphosphate (SSP). - di-calcium phosphate (DCP).

- mono-ammonium

phosphate (MAP).

Product can be used as feed phosphates (subject to legal provisions): shown to be effective as a feed phosphate, soluble in citric acid and digestible for pigs and poultry.

Product can also be used as raw material for NPK fertilizers.

Ferric chloride as a coagulant for wastewater treatment

Aluminium hydroxide or Sodium aluminate as a raw material for coagulants and other industrial applications Sewage sludge ash is dissolved in hydrochloric acid (40°C, no pressure).

The residue of ash which is not dissolved in acid consists mainly of inorganic silicates, and after separation and washing can be used in the cement or concrete industries (Ottosen et al., 2021).

Phosphorus, iron and aluminium compounds are separated from the acid leachate and from each other by specific dissolution and precipitation reactions, in processes characterised by internal recirculation of chemicals.

The remaining acid solution is neutralized and treated to remove heavy metals.

Most heavy metals end up in a concentrated heavy metal cake (c. 30 kg DM cake per tonne ash input) that can be landfilled or used for further extraction of metals.

Recovery rate (P in final product / P in input ash): >85%

Recovery rates of iron and aluminium: will vary depending on ash, e.g. c. 20% for iron and c. 40% for aluminium. Further iron can be recovered from the silicates depending on demand and on silica sand colour requirements.

Removal of heavy metals: >96%

Pilots in Sweden: Uppsala, 50 kg ash/day ash and Helsingborg, 600 kg/day ash.

Full scale plants:

- 30 000 t/y ash, Helsingborg, Sweden (permit application submitted), with Kemira
- 30 000 t/y ash planned, Schkopau, (permit application ongoing)













## TetraPhos (Remondis)

#### Member of DPP

www.phosphorusplatform.eu/ Scope129

Contact;

industrie@remondis-aqua.de

**Updated 1/2022** 

Input: sewage sludge incineration ash, from sewage works using biological and/or chemical P-removal.

- phosphoric acid
- gypsum
- iron and aluminium salts
- mineral ash residues
- ash is leached using phosphoric acid, so solubilising phosphorus and calcium but not most of the iron or heavy metals
- 2) addition of sulphide to precipitate heavy metals and maximise the proportion of these which stay in the leached ash (solid fraction)
- 3) solid-liquid separation
- 4) from the liquid fraction (enriched phosphoric acid), calcium is precipitated as gypsum, by addition of sulphuric acid, and the gypsum is separated out by vacuum belt filter and water washing.
- 5) the resulting phosphoric acid is partially returned back to leaching process. The additional acid production is purified by ion-exchanger and optionally nano-filtration membrane
- 6) the resulting purified phosphoric acid is then concentrated (preferably using secondary heat, e.g. from a sludge incinerator)
- 7) regeneration of the ion-exchange resin produces (using hydrochloric acid) produces a metal salt solution, which can be potentially recycled to sewage works for phosphorus removal.

Pilot plant: Hamburg, Germany and Elverlingsen, Germany, 50 kg/h ash (photo)

Full scale plant: Hamburg, Germany (commissioning underway 2022) Throughput: 20,000 t/y ash.

Further full scale plants are in project phase in Kiel, (Germany) Lünen (Germany) and Moerdijk (The Netherlands).









#### PAKU (Endev)

#### Member of ESPP

www.endev.fi

Arttu Laasonen arttu.laasonen@endev.fi

**Updated 4/2021** 

Dewatered sewage sludge or dewatered sewage sludge digestate. Ash with low contaminant levels suitable for fertiliser use:

- Organic contaminants in sewage sludge are eliminated by incineration.
- Heavy metal levels in the fertiliser ash (indicative averages mg/kg DM): As 7; Cd 1.1; Cr 170; Cu 400; Hg <0.04; Ni 97; Pb 15; Zn 870.
- Phosphorus content in the fertiliser ash:4.7% P DM (average).

Phosphorous in the fertiliser ash is in slow-release form, plant availability approx. 65-70% (solubility at 1 M NAC).

Surplus energy for e.g. district heating: approx. 1 MWh heat per tonne of sludge treated (tonne sludge 25% DM).

Sludge disposal (incineration) with heat and nutrients recovery.

Dewatered sludge is thermally dried to >95% DM content, using secondary energy from a PAKU incineration unit. In a specific process, heat is transferred using hot sand. PAKU ensures incineration at 850°C without additional fuels.

Thermal drying condensate undergoes nitrogen recovery (ammonia stripping) and energy recovery.

The ash from incineration is separated into a fertiliser fraction and a by-product fraction (<5% of total ash), which goes to landfill.

The NH₃ in condensed drying fumes is recovered through air stripping and absorption, with a concentrated solution of ammonium sulphate (AmS) as the end product (±25 % AmS).

Full-scale plant operating since early 2021 in Rovaniemi (Finland), located adjacent to the WWTP of Rovaniemi city (operating biological? chemical P-removal???). Capacity 10 000 tonnes sludge (25% DM) per year.











#### **EuPhore**

https://www.euphore.de
Contact:
siegfried.klose@euphore.de
frank.zepke@euphore.de
marianne.klose@euphore.de

Photo: Phos4You pilot plant, Dinslaken, 2020

Updated 28/12/2021

Dewatered sewage sludge.

Phosphorus rich biomass.

Phosphate-containing ash which can likely meet the heavy metal limits of German and EU fertilisers regulations, depending on input ash quality.

Carbon content of ash is < 1% (total carbon).

Iron content of output ash product at Dinslaken pilot is c. 7% Fe. Aluminium is pending analysis.

The EuPhoRe process uses a specifically-designed rotary kiln incinerator. Flue gas, either from e.g. separate solid waste incineration, or from the EuPhoRe process itself, is used to dry sewage sludge, and to ensure reducing conditions in part of the reactor. The kiln also has an oxidation zone at up to 1000°C (ensuring elimination of organic contaminants).

Depending on the input material quality, alkali- and/or earth-alkali chlorides may be added for reduction of part of the metals content and removal (as chlorides) into the gas phase.

Heavy metals are extracted from the flue gas to a waste stream. All other minerals, including iron and aluminium, remain in the ash.

> 95% of P in input sewage sludge is recovered in the final ash product.

70% – 90% of P in the ash product is soluble in 2% citric acid. Solubility in NAC not specified. In pot trials 80% fertiliser efficiency at soil pH of 7.4 compared to TSP

Selected indicative % removal of heavy metals, e.g.

- 20 40 % for Cu
- 35 65 % for Zn
- 90 95 % for Cd, Hg, Cr√l
- 85 95 % for Pb
- -45 65% for As

Pilot plant: Dinslaken Germany (Emschergenossenschaft): 100 kg dewatered sludge/hour wet weight input (photo)

Two industrial scale plant are planned in Germany at Offenbach (100 000 t/y) and Mannheim (135 000 t/y), plus several other projects are planned in Europe.









TerraNova (HTC)
Member of DPP
https://terranova- energy.com/umweltschut z/
Contact: erkan.yalcin@terranova- energy.com

Input: raw or digested sludge after dewatering, from wwtps operating biological and/or chemical P-removal.

Mass balance of iron and aluminium in input sludge: pending.

Calcium phosphate salt:

- phosphorus > 7% - Ca = 2 – 6 %
- N: 1-2 %
- K < 1 %
- Mg < 0.5 %

Organic carbon:  $C_{org} = c. 2\%$ 

#### Contaminants:

- Pb < 10 mg/kg</li>Cr<sub>-total</sub> <50 mg/kg</li>
- copper < 20 mg/kg - Zn < 200 mg/kg
- Hg ND
- etc.

All data above is / DM.

1) continuous hydrothermal hydrolysis carbonization process at 175°C, 20-25 bars, 2-5 hours

- 2) Acid treatment of hydrolysed sludge to dissolve P.
- 3) Mechanical separation into low-P solid "coal" and P-rich liquid filtrate.
- 4) Phosphate precipitation from the liquid using CSH (calcium silicate hydrate) to produce a calcium phosphate salt.

Full scale plant operating on dewatered sewage sludge in China (since 2016) input capacity 2 t/h

Demonstration plant at Ruhrverband/Duisburg Germany, input capacity 250 kg/h.



## Kubota

Kubota Surface Melting Furnace (KSMF)

**Updated 7/3/22** 

www.phosphorusplatform.eu/Scope125

contact:

hiroyuki.hara@kubota com

**Updated 12/2021** 

Input: dewatered sewage sludge with LHV (Lower Heating Value) 10 – 12 MJ/kg. A drier is included in the Kubota system, upstream of the furnace.

From sewage works with biological and/or chemical P-removal

P-containing slag, registered as a phosphate fertiliser in Japan.

Thermal treatment in a specific furnace with core temperature 1300°C. Iron oxide is added to retain phosphorus in the solid slag. Calcium hydroxide is added to improve phosphorus plant availability in the slag.

>90% of heavy metals (cadmium, lead, zinc ...) are volatilised and removed to fly ash.

90% of input P is in output slag product. The slag shows 95% P-solubility in 2% citric acid and, in pot trials at soil pH 5.5, 97% fertiliser efficiency relative to commercial phosphorus fertilisers.

Recovery rate (P in final product / P in input sludge): c. 90%.

Iron and aluminium in input sludge are transferred to final product, as are other minerals (Si, Ca, Mg).

Around 20 full-scale furnaces in operation in Japan, treating municipal solid waste incineration ash.

11 full-scale furnaces in Japan treating sewage sludge (1 – 10m diameter).

Of these, 4 furnaces (2 plants) in Toyama Prefecture, Japan are operating P-recovery since 2014: total 30 000 – 40 000 t/year dewatered sludge input, 1 200 – 1 500 t/year P-rich slag output.

At present (end 2021), the Prich slag is not sold as fertiliser, but is used as construction material as alternative to sand.











	PHOS4Green Glatt)
g	https://www.phos4greer latt.com/innovation- 8.html
_	Contact: an.kirchhof@glatt.com
L	Jpdated 10/2021
	Metawater alkaline ash
	eaching
	http://www.metawater.c

Sewage sludge incineration ash

P or NPK fertilisers

Ash is reacted with phosphoric acid to render the P-content of the ash more plant available. Other elements can be added in this suspension (N, K, Mg, S, trace elements). The resulting material is then granulated to produce fertiliser pellets.

Heavy metals, iron, aluminium, silica and other minerals present in the sewage sludge remain in the final product.

Recovery rate (P in final product / P in input ash): c. 100%

Iron and aluminium in input ash are transferred into final product

Heavy metals are not removed.

Lab and pilot scale plants tested in Glatt's Technology Center in Weimar. Pilot of up to 30 kg/h input ash operated continuously for a number of multi-day trials for different input materials.

Photo above: A full-scale plant (30 000 t/y ash) was commissioned June 2021 at Haldensleben (Germany) with Seraplant.

Photo below: Glatt experience in fertiliser granulation



http://www.metawater.co. jp/eng/product/plant/sew er/rin\_collection/

No update information available 2022

Sewage sludge incineration ash, from sewage works using chemical or biological P-removal.

Calcium phosphate (hydroxyapatite) for use as fertiliser or in fertiliser production.

Registered as a fertiliser in Japan (2009) and demonstrated in plant tests.

- 1) Dilute sodium hydroxide is used to leach sewage sludge incineration ash (90 minutes, 50-70°C)
- 2) After filtration, calcium phosphate is precipitated from the leachate, then separated (sedimentation) then dried.
- 3) Leaching of P in the ash is limited to around 30% in order to minimise leaching of heavy metals.
- 4) The remaining ash is treated with poly-ferric sulphate solution to immobilise remaining metals. This residual ash now has a slightly higher mass than initially (30% humidity) and is used as a construction material or soil amendment.

Recovery rate (P in final product / P in input ash): < 30% ?????

Approx. ???% of iron and aluminium (in input sewage sludge) remain in the final product

Approx. ???% of heavy metals remain in the final product, e.g.

- ??? % for Cu, Zn

Two full-scale plants operating in Japan: Gifu, since 2010, 5 t/day ash (taking ash from two monoincinerators treating sewage sludge from four wwtps, total 380 000 inhabitants), and Tottori, since 2014, 2t/day ash (taking ash from one mono-incinerator treating sludge from total 90 000 inhabitants).

Photo: Gifu plant, from "Phosphorus Recovery and Recycling", ed. Otake & Tsuneda, Springer; 2019









## Struvite precipitation

## Members of ESPP / DPP

Pearl (Ostara), producing CrystalGreen fertiliser product Contact: Matt Kuzma mkuzma@ostara.com

NuReSys www.nuresys.be Contact: wm@nuresys.com

Struvia (Veolia) Contact: cedric.mebarki@veolia.com

Phosphogreen (Suez)
Contact
thomas.bugge@suez.co
m

<u>AirPrex</u> (CNP) Contact: <u>bernhard.ortwein@cnp-</u> cvcles.de

PHORWater pilot plant, Calahorra wwtp Spain, 10 kg struvite/day, already operated for two years. Contact sofia.grau@damaguas.es

Parforce: https://parforcetechnologie.de Contact: info@parforcetechnologie.de Applicable on different liquids containing soluble phosphorus: sewage works, food processing, mining or industry, manure, biogas digestates, source-separated urine ...

In sewage works, only practicable in wwtps operating biological P removal, usually with sludge digestion (AD).

Only orthophosphate (dissolved PO<sub>4</sub> ions) in the liquid can be recovered.

Two options, with different constraints and resulting product quality:

downstream of sludge dewateringupstream of dewatering

Other phosphate salts can also be precipitated, e.g. (magnesium potassium phosphate) or calcium phosphates (e.g. brushite). For brushite, AirPrex has a 1 m³/h pilot (CalPrex)

Struvite, useable directly as a fertiliser.

Has added value as a slow-release, low leaching, non root-burning fertiliser.

Over 50 studies show that struvite is plant available an effective fertiliser (see SCOPE Newsletters n°s 43, 121, 122)

EU Fertilising Products Regulation criteria for recovered struvite proposed are under finalisation (see final STRUBIAS <u>report</u> Sept. 2019).

Recovered struvite <u>already has</u> End-of-Waste status and EU 2003/2003 fertiliser validation in a number of countries.

In bio-P wwtps, struvite precipitation.

Struvite (magnesium ammonium phosphate) is precipitated (or "crystallised") from a liquid stream, eg. sludge or sludge digestate, sidestream, dewatering liquors. pH is increased to above 7 (de-gassing, alkali dosing) and magnesium is often added. Wide range of different reactor types (fluidised bed, stirred ...).

Product quality and characteristics are extremely variable depending on process, operating conditions and input liquor (where the process is situated in the sewage works), with outputs ranging from 'sludge' (mix of struvite, organics, water ...) through to high quality, dry, size-sorted prills.

Only around 10-20% of sewage works inflow P is recovered in struvite applied to sludge liquor, but this can be increased by treating also sewage works sidestream return flows and/or by Prelease processes, see "Enhanced struvite precipitation" below.

Around 100 full scale struvite units operating in sewage works and other waste water treatment worldwide. Some units have been operating for > 10 years.

A list of struvite recovery installations operating worldwide is in the Inventory of operating P-recovery / -recycling installations worldwide (Christian Kabbe, P-REX Environment) here.

World's biggest installation to date is Ostara at Chicago Stickney wwtp, producing around 9 000 t/y of struvite.

Biggest plant in Nordic Europe is Phosphogreen at Marselisborg wwtp, Denmark. Capacity 200 t/y of struvite.

Veolia (Struvia) also offers compact installations, and has also demonstrated struvite recovery on a bio-P wwtp without AD (LET Brazil 2017).

Operating plants on other streams include NuReSys unit at Clarebout Potatoes (Belgium) producing around 440 t/y of struvite.

Photos (from the top)

Ostara.

Veolia Struvia compact fullscale installation Helsingør, Denmark (35 t struvite/year, operating since 2016).

NuReSys struvite recovery from urine ( 200 l/day); Kruitfabriek Vilvoorde, Begium.













Struvite enhanced  Members of ESPP / DPP  WASSTRIP (Ostara) https://ostara.com/nutrien t-management-solutions/ Phosphogreen (Suez). Phosforce (Veolia) Parforce	Only applicable to wwtps operating biological P removal, usually with sludge digestion (AD).	Struvite as above.	Sludge return streams or sidestreams in the biological treatment process are adapted to optimise soluble orthophosphorus release and to increase P available for struvite precipitation, enabling recovery of 20 – 35 % of sewage works inflow P as struvite. This rate can be further increased to 45 – 50 % by processes which hydrolyse sewage sludge to render the phosphorus soluble (see "Sludge lysis" below)	Ostara WAASTRIP (Crystal Green) is operating at 12 wwtps worldwide, recovering 45 – 50% of wwtp inflow P. Phosphogreen at Aarhus Åby, 70 000 p.e. since 2013: 45-50% recovery of wwtp inflow P is achieved so long as ferric dosing is not required in wwtp operation.  NuReSys (Apeldoorn Hybrid Unit): 30% recovery of wwtp inflow P.  Veolia Phosphogreen: pilot scale trials at 3 sites, demonstration scale planned Parforce: under construction (early 2022) at Wolfsburg, Germany, 150 000 p.e. (photo) up to 60 – 70% recovery of wwtp inflow P by struvite precipitation from hydrolysed sludge.	
Sludge lysis Cambi, Pondus, Haarslev, LysoTherm (Eliquo Stulz), Exelys (Veolia), Bio Thelys (Veolia),	Aqueous sewage liquors or sewage sludge	NOTE: Iysis is not itself a nutrient recovery process but breaks down organic matter and dissolves phosphorus, so making P available in solution for struvite or other precipitation processes.	Example: Cambi thermal hydrolysis operates typically at 150-170°C, 6 bars, 20-40 minutes. Breakdown of volatile solids improves methane production from anaerobic digesters and reduces required digester residence time, as well as releasing phosphorus into solution (ortho-phosphate).	55 full-scale Cambi plants operating across the world www.cambi.com - contact kine.svensson@cambi.com	







## Renewable Nutrients

Quick Wash®

www.renewablenutrients.

Contact:

info@renewablenutrients .com

\* = Szogi et al. 2020

\*\* = Bauer et al. 2007

**Updated 2/2022** 

Solid or liquid waste streams: poultry manure, pig or cattle manure or digestates, sewage sludge digestates, food processing wastes.

Final product is a stackable solid, typically c. 20% DM (can be dried to c. 90% DM), consisting of a mixture of amorphous calcium phosphate with organics (10 - 40 % DM organic carbon), and containing also organic nitrogen and minerals such as calcium, sulphur, magnesium.

P-content of final product: c. 0.25 – 0.55 % DM P for recovery from manures, digestates, wastewater or e.g. >6% for recovery from P-rich industrial stream.

The amorphous calcium phosphate has low water solubility (c. 1%\*) but high citrate solubility (>90%\*) and P fertiliser effectiveness\* \*\*. Data on NAC solubility (required for labelling as a "Mineral" fertiliser under the EU Regulation 2019/1009) are not provided.

Contaminants will depend on input stream treated. Typical levels of copper and zinc are 100 – 300 mg/kg DM The QuickWash process (SCOPE Newsletters <u>78</u>, <u>90</u>, <u>119</u>) consists of:

- 1) Solubilisation of phosphorus using acid at pH 3-5 (e.g. citric acid or hydrochloric aci).
- 2) Solid is then separated from the acid liquid by settling.
- 3) Precipitation of calcium phosphate from the acid solution by increasing to pH 8-10 by lime dosing
- 4) Recovery by settling. Anionic polyacrylamide polymer (at c. 7 mg/l) is dosed to enhance settling and recovery of the precipitated calcium phosphate.

Recovery of P from input material is generally > 95%.

This process enables recovery of a relatively pure amorphous calcium phosphate, but in operations today this is recovered along with organics, in order to provide organic carbon and other nutrients to farmers.

Over 20 pilot installations have been constructed and tested at sites including municipal wastewater treatment works, farms and industrial sites, treating up to c. 0.5 million litres/day (c. 1 500 t/y output product).

1.5 million litres/day installations are under planning in the UK and USA.







#### Sewage P-recovery: (TRL 6+)

#### RAVITA (Helsinki HSY)

Helsinki Region Environmental Services Authority

www.hsy.fi/ravita

Contact: kati.blomberg@hsy.fi

**Updated 2/2022** 

Chemical postprecipitation, then Precovery from the resulting P-rich sludge.

Also, nitrogen recovery from sewage sludge dewatering liquor. Phosphoric acid

Recovery of iron/aluminium chemicals for use as coagulants in wwtp P-removal.

Ammonium phosphate.

- 1) Tertiary post-precipitation, using iron or aluminium coagulants, then separation by e.g. disc filters, to generate a P-rich sludge. This can be installed in smaller wwtps, then the sludge transported to central processing. Heavy metals are low in this post-precipitation, so facilitating recovery.
- 2) Dissolution of this sludge in phosphoric acid.
- 3) Continuous solvent-solvent extraction to separate iron and aluminium salts in solution (can be recycled as coagulants) and phosphoric acid
- 4) Combination with nitrogen recovery (ammonia stripping from secondary sludge dewatering liquors) to produce ammonium phosphate.

Recovery rates (as % from the post-precipitation sludge): P >70%, Al > 90%

Because recovery is in wwtp effluent, heavy metals are low in the post-precipitation sludge, e.g. zinc 240, copper 57, nickel 13 (all mg/kg dw).

Post-precipitation: 1 000 p.e. pilot for tertiary P-removal operating since 2017 (achieving 0.4 mgP/l wwtp discharge).

P-recovery: 1 000 p.e. pilot under testing, started in 2020.



# Phos4Life (ZAR – Técnicas Reunidas)

#### Member of ESPP

See <u>SCOPE Newsletter</u> n°119

Técnicas Reunidas
Contact:
agalindoc@trsa.es

ZAR Foundation Contact: stefan.schlumberger@ke bag.ch Input: sewage sludge incineration ash, from sewage works using biological and/or chemical P-removal.

Technical grade phosphoric acid.

Silica filter cake to cement/concrete industries

Heavy metal concentrate.

Fe-III-chloride solution as a coagulant for wastewater treatment

- 1) Leaching of ash in sulphuric acid to dissolve phosphorus, solid/liquid separation by filtration.
- 2) Separation of iron, aluminium and heavy metals by solvent extraction
- 3) Concentration of dilute acid to technical grade phosphoric acid by evaporation

Pilot tests carried out at Tecnicas Reunidas in Madrid Spain.

Full scale (30,000 t/y ash) implementation planned in Solothurn, Switzerland









ViViMag® (WETSUS - Kemira)
Members of ESPP
https://www.wetsus.nl/vivimag
www.kemira.com
Contacts: leon.korving@wetsus.nl Bengt.hansen@kemira.c om

Input: sewage sludge, before dewatering, from wwtp using iron salts for chemical P removal

works

Product: vivianite (iron(II) phosphate).

Can be used as an iron fertiliser to treat iron-chlorosis, of for instance olive trees.

Processes are under investigation to chemically split the vivianite into a P rich stream that could be used for P fertiliser production, and an iron rich stream that could be used to produce iron coagulants for use in WWTPs.

Phosphorus is removed from sewage by precipitation using an iron salt, in order to limit phosphorus discharge (prevent eutrophication and respect regulatory obligations). This generally results in precipitation of iron(III) phosphate.

Iron(III) phosphate is reduced to iron(II) phosphate, that is vivianite, in anaerobic conditions (e.g. in anaerobic digester).

The vivianite is then recovered by magnetic separation. The separated vivianite after washing has c. 10% P content and < 10% carbon content (%/DM)

Manual 1 m³/h pilot (photo) for magnetic separation of vivianite tested at Nieuwveer wwtp, NL.

Automatic continuousoperation 1 m³/h pilot is now in operational for longduration trials. First tests have been carried out in Germany, and further trials are planned in different wwtp configurations in Denmark, the Netherlands and elsewhere.

A demonstration scale plant is in the planning phase in The Netherlands.



# Kemira iron / aluminium phosphate

Updated 11/2022

Member of ESPP

www.kemira.com

Contact Outi.gronfors@kemira.co m

Updated 11/2021

Upgrading of existing Iron or aluminium phosphate sewage treatment

- Optimisation of primary and secondary (biological) treatment by polymer dosing with control algorithms giving increased biogas production
- 2) Tertiary P-removal with iron and/or aluminium coagulants, control algorithms, separation by settling and/or centrifugation to produce a P-rich sludge, <10% C-org, containing >50% of wwtp incoming P. Can be installed at smaller wwtps and recovered material treated at centralized treatment process.
- 3) Optionally further processing of the Prich sludge, by drying, granulation, or separation of P and Fe or Al to produce phosphoric acid, phosphate salt and Fe or Al coagulant for water treatment

Tested at full scale at two wwtps (63,000 pe and 130,000 pe) for 1-7 months, and at pilot scale at three wwtps for 2-3 months.

Iron phosphate granules have been tested as fertiliser in pot trials with rye grass.

Photos: NPK fertilizer granules including iron phosphate (left). Recovered aluminium phosphate pellets (right). Pilot set up for P-rich sludge separation in tertiary unit (bottom).









AshDec (Metso Outotec)  Member of ESPP  Contact: Tanja.Schaaf@mogroup.com Julian.Kunstler@mogroup.com Updated 1/2022	All ashes with P-content >7%	Modified Rhenania Phosphate (Calcium-Sodium-Phosphate) P <sub>nac</sub> solubility >80%; granular material with P <sub>2</sub> O <sub>5</sub> content of 15-25% (depending on input-ash); no organic matter; product is blendable with all other fertilising products.	Ash is mixed with a sodium carrier (Na <sub>2</sub> CO <sub>3</sub> or NaHCO <sub>3</sub> ) and heated to about 850-900°C in a rotary kiln to modify the P-compounds to neutral-ammonium-citrate soluble CaNaPO <sub>4</sub> (Rhenania Phosphate). Heavy metals are partly removed and captured in a baghouse filter from where they can be separated from the fertilising product as a small waste stream (3-5% of input material). Hence, > 97 % of phosphorus and other elements in input ash are recovered.	Pilot plant (300 kg/h, photo) operational for several years, Leoben, Austria. Continuous operating campaigns produced several hundred tons of product.  A full scale plant (30 000 t/y ash input) is planned in Altenstadt (Bavaria), with Enter GmbH and sePura GmbH, in the R-Rhenalia RePhoR project.	
			Recovery rate (P in final product / P in input ash): >95 %		
			Recovery rate (% from input ash in recycle stream) of iron, aluminium: >95%.		
			Removal of heavy metals (% from input ash in final phosphate product): Cd: ~ 50 % removal, Pb: ~10 %, As: ~ 50 %.		
			Cu, Zn and Ni are not removed from the ash with the standard process. Removal of Cu and Zn can be enhanced by use of chlorine additives.		
CarboREM	Digested dewatered sewage sludge (10-	Precipitated phosphate salts.	1) HTC (hydrothermal carbonisation) at c. 200°C for 1 hour.	Industrial-scale continuous HTC plant installed in 2019	
www.carborem.com Contact: info@carborem.com Updated 1/2022	15% DS).	Recovery rate (P in final product / P in input sludge): 90 – 91%.	2) solid-liquid separation of hydrochar, containing 95-96 wt.% of the P initially present in digested sludge.	and located in the wastewater treatment plant of Mezzocorona, Italy. Capacity: 1.4 t/h of wet	
			<ul><li>3) dissolution in acid (citric acid or HCl).</li><li>4) addition of alkali (NaOH) for</li></ul>	digested sewage sludge	S INTERES
			phosphate salt precipitation.  Metal removal from digestate after HTC and acid leaching of hydrochar:  - Cd~80%  - Pb~70-80%  - Zn~90%  - Cu~70%  - Cr VI~90%		







#### Other nutrient recovery TR6+

#### **N2-Applied**

Member of ESPP www.n2applied.com

Contact:

henk.aarts@n2.no

**Updated 3/2022** 

Manure slurry, biogas digestate (any inputs) Combines N fixed from air with N in manure/slurry to produce a stabilised, ammonium nitrate based, liquid fertiliser product, with a high nitrogen use efficiency and significantly reduced emissions of ammonia and methane during storage and field application.

Firstly, manure/slurry is solid/liquid separated. Most of the phosphorus stays in the solid fraction. The liquid fraction is then treated as follows.

Using renewable electricity and air, a plasma reactor fixes nitrogen from air, generating nitrogen oxides, which are mixed into manure/slurry and react to form nitrates and nitrites. This lowers the pH so that ammonium nitrogen is stabilised. After solid/liquid separation the liquid fraction of manure or digestate can be managed as a liquid nitrogen fertiliser and most of the phosphorus will remain in the solid fraction.

Farm scale pilots have been executed in 2021 and 2022 treating between 5 and 15 m³/day (of liquid fraction) on farms in UK, Norway, Sweden, Finland, Denmark and Netherlands, on fresh cow and pig manure and on a variety of biogas digestates (varying in feedstock). Pilot runs have included 24/7 for periods of months.

Commercial launch in 2022 with N2 Units treating between 5 and 15 m³ (of liquid fraction) per day, mostly depending on the ammonia-N content of the input material.



#### Ash2Salt (EasyMining)

Member ESPP, DPP

http://easymining.se/

Contact:

jan.svard@easyminin g.se

Updated 04/2022

Fly ash from municipal solid waste incineration (not bottom ash).

chloride and calcium chloride – as separate salts

Potassium chloride. Sodium

The salts are of high quality and suitable for industrial use and fertilises.

Ammonium sulphate (40% solution)

Fly ash is reacted with process water to dissolve calcium, sodium and potassium. Sulphates are then precipitated with calcium. The solid fraction is separated by vacuum filtration. The remaining solution is treated for heavy metal removal by precipitation, and soluble organics are removed by activated carbon. The treated brine is upconcentrated by recirculation, then undergoes ammonia removal and recovery (as ammonium sulphate). After removal of ammonia, the brine is evaporated and three different salts are recovered: potassium chloride (solid), sodium chloride (solid) and calcium chloride (solution). The salt separation is based on differences in the solubilities of the salts involved. The clean condensate water can be recycled in the process or be used for other purposes.

A first full scale plant is being built (photo March 2020) at Ragn-Sells waste management plant, Högbytorp, near Stockholm, Sweden. Planned start-up: late 2022. This plant will have a capacity of 130 000 ton fly ash per year, producing approx 3 500 t/y (dry) potassium chloride, 7 000 t/y (dry) sodium chloride and 32 000 t/y calcium chloride (36% solution).









Aqua2N
(EasyMining)

Member ESPP, DPP
http://easymining.se/
Contact:
anna.lundbom@easymining.se
Updated 04/2022

Liquors with a high ammonium concentration, e.g. wastewater treatment sludge dewatering liquors.

Ammonium sulphate ((NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>). The demonstration plant is producing 10-25% solution, but upconcentration or making a dry product are being investigated.

Contaminations in the ammonium sulphate are well below fertilizer requirements.

In the first step, the ammonium nitrogen is precipitated with a specific chemical.

In the second step, the precipitant is regenerated and sent back to the first step, and acid is used to convert the nitrogen to a form usable either directly in fertilisers or in fertiliser production.

The processes is currently (early 2022) being demonstrated in a continuous demonstration plant (photos) of capacity 4 m³/h inflow. This installation will be tested December 2021-March 2022 at Ragn-Sells' waste management plant Högbytorp in Bro, Sweden, and in April-September 2022 at BIOFOS' municipal wastewater treatment plant Lynetten in Copenhagen, Denmark.

The demonstration plant is built within the EU LIFE cofinanced project RE-Fertilize.

LIFE RE-Fertilize (easymining.se)





#### **DUCTOR**

Member of ESPP

www.ductor.com

m

Contact: leonie.boller@ductor.co

Updated 03/2022

Nitrogen-rich manure, in particular poultry manure Liquid nitrogen fertilizer with 5% nitrogen as soluble ammoniac form and  $C_{\text{org}}$  = c. 10% of dry solids (see HERE)

Solid fraction of digestate, dried and pelletised as NPK fertilizer or soil improver with high organic content

The liquid N fertilizer has obtained USDA Organic Farming compliance.

The Ductor process integrates anaerobic digestion (biogas production) and fertiliser production. The nitrogen recovery upstream of the digester improves biogas production by reducing inhibition by ammonia in the digester. The resulting fertiliser products are adapted to farmers' needs.

One full-scale installation operating in Jalisco, Mexico, since 2019, treating c. 3 000 tonnes/year of chicken manure (30% DM).

One installation under commissioning in Haren (Emsland, Lower Saxony, Germany) treating c. 8 000 tonnes/y chicken manure (40% DM)..

Projects in planning stage in Sweden and in the USA.









Anuvia Plant Nutrients https://www.anuviaplantn utrients.com/ Contact: Hugh MacGillivray hmacgillivray@anuvianut rients.com	Secondary organic materials, including crop wastes, animal manures, food processing wastes and waste-water organics, are combined with mineral nutrients.	Granulated organo-mineral fertilisers (SymTRX).	The organic materials are processed to create an organic matrix with +ve and -ve sites onto which mineral nutrient ions can be fixed. The final product is granulated.  Less than 5% of the nutrients of the final product come from the organic substrates used, most are added as minerals through the manufacturing process.  Input materials ensure stable final product analysis, eg. SymTRX20S (17-1-0-20S) or SymTRX10S (14-24-0-10S).	After 5 years development and over 450 field trials, and following the opening of a new production facility in Plant City, Florida, Anuvia can produce up to 1.2 million tonnes / year.	
Hitachi Zosen https://www.hitachizosen. co.jp/english/products/pr oducts006.html  Contact: ueda k@hitachizosen.co .jp  Updated 2/2022	Manure, solid/liquid separated and dried to 60% DM.	Stabilised and sanitised biochar, rich in phosphorus and can be used as organic fertiliser and/or soil improver	Manure is pyrolyzed at 400-500°C under oxygen-limiting conditions in the reactor.  The reactor is kept at a slight negative pressure to prevent leakage of pyrolysis gas.  Energy efficient pyrolysis can be operated without requiring energy input if input is drier than 60%DM / 40% water	Pilot scale plants operating in Japan on manure (capacity 720 kg/day, operating since 2012)  Demonstration plant (5 t/day) contracted for 2022, Shenyang, Chin. Full scale plant (18 t/day) under study.	
AMFER (Colsen) www.colsen.nl Contact: Jan Willem Bijnagte jw.bijnagte@colsen.nl or info@colsen.nl	Nitrogen recovery from ammonia rich stream such as fresh manure/slurry or digestate (unseparated or liquid fraction)	Solution of ammonium sulphate (AS) or ammonium nitrate (AN) Concentration is up to 75 g/l for AS and 150 g/l for AN (50:50 NH <sub>4</sub> :NO <sub>3</sub> ). The mineral nitrogen compound solution can be used as a commodity chemical or as a mineral N fertiliser.	Ammonia is removed from the liquid to a gaseous stream pH increase achieved heat (60-70°C) and by CO <sub>2</sub> stripping and/or chemical dosing pressure, then recovered by contact with sulphuric or nitric acid.  Ammonia removal can facilitate use of manure or digestate (lower N content), avoid atmospheric ammonia losses, and improve aerobic treatment or biogas production (if applied upstream of treatment or digester).  Around 50% - 80% of ammonia in substrate is recovered.  Contaminant levels in the recovered N	Three full scale plants are operating since 2021:  - Van Alphen – Axel NL(30 000 t/y ww digestate)  - Bio-gas Veendam – Veendam NL (30 000 t/y ww digestate)  - Green Create W2V Kent – Kent UK (170 000 t/y ww digestate).  Containerised installations are available treating up to 43 000 t/y ww.	MR 1 1 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2







product are not analysed to date.

#### CCm

CCm Technologies
Carbon Capture and
Utilisation

https://ccmtechnologies.c o.uk

Contact:

alexander.hammond@cc mtechnologies.co.uk

**Updated 2/2022** 

Digestates of sewage sludge, food waste, manure or other biowastes.

In some cases, also other secondary materials e.g. wood chips, organic fibres, biomass ash,

Offgas CO<sub>2</sub>

Pelletised organo-mineral fertiliser, containing stabilised N and P.

Field tests of the fertiliser product show compatibility of the pellets with existing farm fertiliser equipment: rotating discs up to 36m wide spreading radius), crop performance comparable to commercial mineral fertilisers and positive impacts on soil bioflora, water retention, soil carbon and reduced nutrient runoff.

Ammonia captured from digestate is used to capture (as carbonate) CO<sub>2</sub> from digester biogas (mixed off-gas or separated CO<sub>2</sub> stream from biomethane). This is then combined with organics in digestate cakes, further stabilising the nitrogen and carbon. The product is then dried and pelletised, to produce a stable organo-mineral fertiliser (OMF).

Additionally, CCm are operating a pilot unit Pilot (4 m³/day) at Yorkshire Water Caldervale site, UK, to remove and recover phosphorus as struvite from Prich sludge dewatering streams, using magnesium and ammonia. The output integrates the phosphorus into the stable organo-mineral fertiliser pellets. Aim: 75% P reduction from liquors. Startup planned Q2 2022.

Industrial demonstrator at Kew Technology Sustainable Energy Centre, UK, to produce OMF fertiliser. Output: 500 t/yr. Inputs: biochar, digestate, recovered CO2 from enhanced thermal conversion technology. Operational in Q1, 2022

Industrial demonstrator at Severn Trent Water Minworth site, UK (*photo*). Output 10 000 t/yr OMF fertiliser pellets. Input capability: sewage sludge, biomass ash, recovered CO2, recovered ammonia. Operational from 2021.

Full scale plant at Walkers potato processing plant (Pepsico, Leicester, UK). Outputs: 12 000 t/yr OMF fertiliser pellets. Inputs: food waste digestate, recovered CO2. Operation start Q2, 2022.



#### Agro America (VP Hobe)

Member of NNP

www.agroamerica.nl
Contact: h.willems@vp-

Updated 10/2021

systems.nl

Liquid or dry pig manure, or digestate of manure and/or other organic wastes Biochar.

Ammonia solution.

Potassium as Liquid concentrate

Clean water returning to surface waters

Pig manure is solid-liquid separated by belt-press, most P remains in solid fraction. Liquid fraction is spread locally to fields.

Solid fraction is dried then pyrolysed (with heat energy recycling).

Process can also treat manure digestates.

Ammonia can be recovered by evaporation then recovery into concentrated ammonia solution.

Potassium can be recovered as liquid concentrate

Several manure treatment installations operating 24/7 for significant periods (depending on manure supply, permitting ...) in:

- 2015: Horst-Venlo (NL),
   250 000 t manure / year
- 2019: Oirschot (NL)\*+, 35 000 t
- 2020: Storg (BE)\*, 45 000 t
- 2021: Uden (NL)\*+, 200 000 t
- 2021: Sterksel (NL), 125 000 + 80 000 t
- Underway 2022\*+\$: Almelo (NL), 200 000 t
- \* with N-recovery. + with K-recovery. \$ with P-recovery.









#### **Parforce**

#### **Member of DPP**

www.parforcetechnologie.de

Contact: <u>info@ptc-</u> <u>parforce.de</u>

**Updated 3/2022** 

Sewage sludge incineration ash, other ashes, phosphate rock or other secondary materials.

Struvite can be used as raw material after calcining (prior to step 1) to remove organic contaminants, with ammonia recovery.

Phosphoric acid

By-products or waste streams depending on process design and input material.

% of P in input material recovered in phosphoric acid: > 80% for sewage sludge incineration ash, higher for other input materials.

Approx. 5 - 35% of iron and 40 - 55% of aluminium in ash are leached by acid in step (1). Iron must then be removed in step (3) to protect the electrodialysis of step (4).

>99% removal of leached heavy metals, copper, zinc can be achieved in the phosphoric acid purification, step (4).

- 1) Acid digestion using HCl or HNO<sub>3</sub>, to generate raw phosphoric acid
- 2) solid-liquid separation (filtration)
- 3) if the input material is sewage sludge ashes, then iron and aluminium are extracted (prior to electrodialysis) by either ion exchange or solvent extraction
- 4) membrane electrodialysis to separate metal cations (especially Ca, Mg and heavy metals) to a concentrated solution.
- 5) concentration of the remaining phosphoric acid
- 6) the metal Ca, Mg, heavy metals etc solution (separated from the phosphoric acid in step 4) is treated with lime. This precipitates the metals to a waste stream leaving a salt solution which can possibly processed to road salt.

In pilot trials, some of the phosphorus passed the electrodialysis membranes. so that a return stream was required. This is resolved in larger scale trials where continuous electrodialysis offers better selectivity.

Batch pilot, capacity 150 -250 kg ash per batch and semi-continuous acid purification, tested for several different materials in since 2018 at Freiberg University of Mining and Technology, Germany.

An automated demonstration plant is now planned (2022) for Bottrop, Germany, capacity 1 000 t/y ash. Continuous campaigns will test different ash inputs.



## Slurry acidification

BioCover / Vogelsang GmbH http://www.biocover.dk/ https://www.vogelsang.in fo/

contact BioCover: mt@biocover.dk vanja.cobec@vogelsang. info

**Updated 2/2022** 

On-farm system for improved manure management, using technology integrated into farmers' existing slurry storage and spreading

Input: organic slurry from livestock or from AD digestors

equipment.

Conversion of NH3 (ammonia) to NH4 (ammonium)

Acidification process also converts calcium phosphate to plant available P.

Sulphuric acid is converted into sulphate – SO<sub>4</sub>.

Sulphuric acid is injected into the slurry flow during application to the field. The process results in foaming of the slurry before reaching the field and lowers the pH to below pH 6.4. This reduces the ammonia emission by up to 70 % and increases the plant available ammonium.

There are no unwanted by-products from the process.

System has EU BAT\* status for manure storage and spreading and VERA certification

154 units operational in 8 EU member states. Treats c..20% of volume of slurry in Denmark.

\* EU BAT BREF "Intensive rearing of poultry or pigs" 2017/302









## STERCORE pyrolysis

STERCORE HOLDING BV

WEB: www.stercore.nl
Email: info@stercore.nl

Updated 10/2021

Manure, digestates, crop and food industry wastes: 25%+ dry matter content.

#### **Biomethane**

Liquid CO<sub>2</sub>

Heat: 5MW heat/hour

**Bio-Based Carbon** (enhanced biochar), for fertiliser and soil improver uses.

Specifications depending on input materials:

- Form: solid, as pellets, powder or granules
- Water content 2-12%
- Organic carbon 49-52%
- C/N 12-18.

Contaminant levels:

- typical heavy metals within regulations.
- PAK16:0.01-0.05 mg/kg
- Thallium: 0.09 mg/kg
- ndl-PCB: 0.0 mg/kg
- PCDD/F 0.38 mg/kg
- Pharmaceuticals: ND
- Pathogens: ND

Typical nutrient contents (on demand)

- N: 0.3 0.4 %
- P: 0.1 0.4 %
- K: 0.2 0.6 %
- Mg: 0.2 0.4%
- Ca: 1 2 %

- 1. Bio-active composting ensures predrying to 80% DM and pathogen removal (5 days @ average 70°C).
- 2. Continuous 24/24 pyrolysis system with variable temp and time (proprietary information). The input compost is separated to 20% water, 40% gas and 40% solids. All solids remain / fixed in de Bio-Based Carbon
- 3A. Syngas treatment system with specifically designed methane process.
- 3B. Bio-Based Carbon processing, mixing/adding client specific NPK/micro nutrients/organic matter, etc.

The whole system is modular build.

A first full-scale installation is under construction, expected to be operational end 2023:

- capacity (input raw material) 350 – 400 kt/y (25%+DM)

-outputs 23 million m<sup>3</sup> / y biomethane 30 kt/y liquid CO<sub>2</sub> 85 kt/y Bio-Based Carbon 5MW 70°C heat/h.

Two demo/pilot installations, batch and continuous, have been operated for a total time of c.15 months.

Photos: rotary drum and predrying installation designed by suppliers.







#### Pyreg (pyrolysis)

Member of DPP

https://www.pyreg.de/
Contact: info@pyreg.de
Updated 2/2022

Sewage sludge (minimum calorific value 10 MJ/kg, that is around 80% DS). Biomass materials. Output biochar, from sewage sludge as input, has (as % of DM, and depending on the input sludge characteristics):

- c. 25% (15 35%) organic carbon,
- 6 7% P content (of which c. 80% is NAC-soluble),
- just over 1% N
- more than 10% K.

Pyreg biochar (from sewage sludge) registered as a fertiliser in Sweden (PYREGphos). However, sewage sludge biochar is not included in EU Fertilising Products Regulation CMC14.

Twin screw carbonisation reactor operated at 500 – 800 °C.

This temperature results in a biochar with labile organic carbon content < 0.4%.

Nearly 50 full scale units in operation today, of which 7 using sewage sludge (below: tonnage = input capacity):

- Unkel, Germany (1200 tDS/y, since 2015)
- Homburg, Germany (1200 tDS/y, since 2016)
- Redwood, California (1200 tDS/y, since 2016)
- Hammenhög, Sweden (1200 tDS/y, since 2016)
- Trutnov, Czech Republic (1.200t DS/y, since 2020)
- Lorsbach, Germany (1.500 t DS/y, since 2021)
- Kleve, Germany (1.600 t DS/y, in commissioning)

Also: Basel, Switzerland (11 000 t DS/y food waste) and three further plants using sewage sludge under construction in the USA.





### Technologies at R&D scale

#### **Prayon**

https://technologies.pray on.com/ourprocesses/fly-ash/

Contact:

hhalleux@prayon.com

And

MSonveaux@prayon.co m

Updated 11/2021

Input: phosphorscontaining incineration ashes or low-grade phosphate rock.

DCP (Di Calcium Phosphate) or phosphoric acid.

Prayon is developing three processes:

GetMore P: attack with dilute sulphuric acid, production of DCP.

Ecophos DCP: attack with hydrochloric acid, production of DCP.

Ecophos H<sub>3</sub>PO<sub>4</sub> (process acquired from Ecophos): attack with hydrochloric acid, purification (using ion exchange) and production of phosphoric acid. Aluminium chloride can be recovered for recycling as a sewage treatment coagulant.

Semi-industrial pilot plant in Varna, Bulgaria (photo), 200 kg/h input ash, can operate the three processes (acquired from Ecophos, see www.phosphorusplatform.eu/ Scope120)









AguaDB www.aguadb.com Contact: Mike Waite mike.waite@aguadb.com Updated 11/2021	Nutrient recovery from drinking water treatment.  Adaptation possible for tertiary N-removal in sewage plants.	Solution of nitrate with K, S, Ca and Mg for local fertigation.	lon-exchange is today widely used to remove nitrates from drinking water, but uses salt for regeneration. This generates a phytotoxic sodium nitrate brine, which has to be disposed. The Agua DB process uses water quality potash (KCI) for regeneration, instead of salt, in significantly lower quantities, so generating liquors rich in sulphate, nitrate and potassium, which can be used for fertigation in local agriculture. These can partially replace synthetic fertilisers and reduce use of potash by farmers, so reducing salination (Cl input) to farmland.	A three months pilot project with Affinity Water (a UK drinking water company supplying 3.6 million people), showed effective nitrate removal down to 5 mgN/l. The resulting fertigation solution was demonstrated to be effective for use in hydroponics.	
Flashphos (Uni. Stuttgart, Italmatch)  Italmatch: Member ESPP  Project summary on EU CORDIS website.  Contact: Matthias Rapf matthias.rapf@iswa.uni- stuttgart.de  Updated 9/2021	Dewatered sewage sludge or other organic wastes containing P.	P <sub>4</sub> white phosphorus	Input materials are dried and ground, then flash gasified at high temperatures with CaO (lime) as slagging agent to produce elemental phosphorus (P4).  The process claims to also produce a cement material and a valorisable iron metal alloy (so recovering iron salts used in wastewater phosphorus removal).  FlashPhos presentation at ESPP's PERM4 meeting, 2nd June 2021.  Project summary on EU CORDIS website.  University of Stuttgart press release 7th June 2021.	FlashPhos is based on different technologies of project partners will develop and unify to best standards. The process will be integrated into existing industrial infrastructure (cement plants).  12 M€ funding under Horizon 2020 announced June 2021 to construct and test a c. 2 tonnes/day dry matter input pilot plant	







RSR (Green Sentinel) (Recovered Sludge Resources) https://green-sentinel.at/en/recovered-sludge-resources/ Contact: office@green-sentinel.at  Updated 9/2021	Dewatered sewage sludge (after e.g. filter-press or centrifuge)	PecuPhos®: a mixed aqueous solution of minerals, including phosphates with c. 75% water / 25% minerals w/w, which can be used as an input to mineral fertiliser production.  The solution is pH 3 and contains: - 1.5 - 4 % P - 2-4 % Ca - 0.5 - 1.5 % K - sulphates, chlorides, etc.  Iron is < 1.3%,  Heavy metals are low, e.g. Cd, Hg, Cr < 13ppm, Cu and Zn < 50-500ppm.  Organic carbon is < 30ppm  PecuPower® = alternative fuel - comparable to pellets (water content < 10%, thermal value 4 800 MWh/t) or PecuGrow® = a refined sludge which can be composted	The RSR module uses a special solvent to adjust pH-value and ensure high dewatering of sludge (PecuLeach®) to extract up to 75% of the phosphorus (and of other minerals) from the dewatered sewage sludge into an aqueous solution (PecuPhos®).  The solvent is recycled to enrich the aqueous solution with phosphorus and minerals.  Optionally, solid phosphate can be precipitated from this solution (as a mixture of brushite, struvite).  Heavy metals are also extracted by the solvent, then precipitated to a separate solid fraction for disposal (c. 60-80% of e.g. Hg, Cu, Zn, Fe in dewatered sludge are separated out to this fraction).  The remaining organic fraction can be further processed either by drying to produce fuel (PecuPower®) or is processed to compost (PecuGrow®)	Pilot installations have been tested at 8 kg dewatered sludge input per hour scale for 2 weeks.  A first semi-full scale installation is contracted for Wels municipal sewage works, Austria (165 000 p.e., operating chemical Premoval), commissioning planned mid 2022. This will intially treat 2 500 t/y wet weight dewatered sludge input, that is c. 30% of the sewage from the works. Extension to treat 100% of the works' sludge is planned as a second stage.	
P-roc Member of DPP Website: https://www.cmm.kit.edu/ english/297.php Contact: anke.ehbrecht@kit.edu Updated 2020, no new information in 2021-2022	Sewage sludge dewatering liquor. Liquid manure.	Highly disordered and microcrystalline phosphate salts (hydroxyapatite, struvite, K-struvite).	Crystallization by means of Calcium-Silicate-Hydrate	Mobile pilot plant: 300 litres/hour.	







#### Susphos

P-recycling from ashes and precipitates

https://vnci.nl/chemiemagazine/actueel/artikel ?newsitemid=586163814

Contact: sales@susphos.com

**Updated 2/2022** 

Tested to date: sewage sludge incineration ash, struvite.

Planned: other phosphate-rich materials with low levels of organics, e.g. vivianite. or flame retardant quality).

Iron / aluminium salts as aqueous solution: can be recycled to sewage works for P-removal.

diammonium phosphate (fertilizer

Phosphoric acid, or mono- or

Solid magnesium sulphate salts (if struvite is input), recycled by sewage works for struvite precipitation.

Inert mineral material stream (containing sand, gypsum, iron oxides) can be used in the construction industry. The input materials are attacked with concentrated sulphuric acid.

The resulting phosphoric acid is purified using a proprietary organic solvent extraction process, without requiring ion-exchange or membrane filters. The acid has low levels of impurities and concentration >50% P2O5 (from ash), because the only water input is in the sulphuric acid.

Iron and aluminium are c. 99% removed from the phosphoric acid, and partly recovered for recycling, partly fixed in the insoluble minerals stream.

Mono- or diammonium phosphate can be precipitated from the phosphoric acid by reaction with ammonia gas.

Heavy metals are largely removed and rendered inert in insoluble calcium minerals: e.g. >95% of Cd, Hg, Cu, Zn ...

25 kg/day pilot operated in Leeuwarden (NL) for 8 months for struvite and tests with sewage sludge incineration ash are underway since May 2021.

Full scale plant of 50 000 t/y is planned in The Netherlands, with objective of operation in 2023-2024.



## Spodofos (ThermusP)

www.thermusP.com
Contact:
info@thermusa.com

**Updated 2/2022** 

Ashes from sewage sludge incineration and bone meal, precipitated phosphate salts (struvite, calcium phosphate ...).

STOWA report on Spodofos 5/1/2022 "Spodofos: witte-fosforproductie uit slibverbrandingsasse n. Eerste evaluatie van de duurzaamheid en technologische en economische haalbaarheid"

P4 (white phosphorus).

High aluminium-content slag (may find specific uses).

Ferrophosphorus (low value by-product).

Volatiles (lead, zinc, other heavy metals) to recycling or disposal.

Secondary aluminium (post-consumer, low quality) is heated to 600°C with the ash (or other input materials), resulting in a solid-solid, exothermic thermite reaction, raising the temperature to > 1800°C.

Unlike in conventional P4 reducing furnaces (using coke and electricity), pre-sintering of the input materials is not necessary, carbon-monoxide is not generated.

External heat energy is only needed for preheating the input materials, because of the intrinsic energy content of the secondary aluminium.

Laboratory experiments at 100g scale to date (end 2021) and thermodynamic modelling.

Pilot development now underway









#### SIMPhosprocess (Cirkel)

**Member of DPP** 

Website:

https://www.cirkel.de/en /multifunctionalminerals/simpur

Contact:

Katja.kolodzi@cirkel.de Maria.becker@cirkel.de

**Updated 2/2022** 

Dewatering liquor from sewage sludge digestate, containing soluble phosphate. optionally after redissolution of phosphates.

90 - 95 % of soluble phosphate (orthophosphate) is removed and recovered in final product

Granular calcium phosphate.

Final product contains up to 13% P DM.

Water content of final product is around 50 - 60% after standing to dry (dries easily due to pore structure).

Organic carbon content of final product not yet available and depends on input stream.

Plant availability of P has been demonstrated in pot trials but P-solubility in NAC as specified in EU Fertilising Products Regulation is not yet available.

Crystallisation of calcium phosphate from dewatering liquor by means of SIMPur, a specific calcium silicate hydrate (CSH), which releases SiO<sub>2</sub> / silicon compounds and takes up phosphorus, with 7:1 ratio of input consumed SIMPur CSH:P in final product.

SIMPur is produced by processing the natural CSH mineral tobermorite to tailored granulometry.

Contaminant levels in SIMPur are low. Contaminant levels in the in final product depend on input stream, with the following results from tests to date:

- Cu, Hg, Cd: not detectable
- Zn: 10 mg/kg DM
- Fe: 2 mg/g DM
- Al: 1 mg/kg DM

Operational test in 2016: mobile unit, inflow 0.5 m<sup>3</sup>/h, operated continuously for c. 3 months using real sewage sludge digestate.

Planned operation in 2022 with capacity 7.5 m<sup>3</sup>/h treating part of the sludge digestate centrate at the wwtp Neuburg an der Donau, Germany (67 000 p.e).



#### **PHOSPHIX**<sup>TM</sup> (Clean TeQ Water)

https://www.cleanteqwa ter.com/technology/pho sphix/

Contact: sales@cleanteqwater.c <u>om</u>

**Updated 2/2022** 

Liquor stream with soluble phosphorus < 100 mg/I P-PO4 (including very low P concentrations) and pH in the range 6-8.

Calcium phosphate (hydroxyapatite) for use as slowrelease fertiliser or in fertiliser production

Treated water with <0.1 mg/l P-PO4

Phosphate is selectively removed to < 0.1 mg/L using proprietary continuous ion exchange.

Phosphate is desorbed using calcium-rich sodium hydroxide into a concentrated stream

Hydroxyapatite is crystallised from this stream.

The recovered regenerant stream is reused for desorption, limiting chemical consumption and waste production

Laboratory scale testing complete, using synthetic feeds simulating sewage treatment works (MBBR) effluent. Pilot plant of capacity c. 2 m<sup>3</sup>/h being prepared to remove P from river water

Full scale (12,000 m<sup>3</sup>/d) installation of similar technology under construction in Ordos. China, to remove nitrate to <1 mg/l N-NO3 mg/l from clarified mine water, producing nitrogen gas.









### Update not completed 2022 or underway

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Part of IPI group (International Power Invest AG) https://htcycle.ag/ Contact: m.steinfurth@ipi.ag	Sewage sludge with 25-30% dry matter.	Activated carbon (HTC coal) produced can be used in the wastewater treatment processes to remove micro plastic, hormones and pharmaceutical residues.  Phosphorus can be leached from the activated carbon, either before or after its use for water purification, using sulphuric acid, to produce phosphoric acid.  Ammonia stripped from off-gases can be either reacted with the phosphorus to produce struvite and/or used to produce ammonium sulphate.	The patented HTCycle process is a continuous hydrothermal carbonisation technology, using steam as the reaction medium at around 220°C, 24 bars for 3-5 hours.	??? pilot installations , capacity 8 000 or 16 000 t/y (input material, wet weight???) capacity have been operated for a total of more than ten years in countries ????	Photos of operational pilot plant please
HAIX ion exchange (LayneRT <sup>TM</sup> ) Contact: Cranfield University UK, Ana Soares a.soares@cranfield.ac.uk No update information provided in 2021-2022	Secondary treated sewage (discharge after biological treatment)	Orthophosphate solution from which calcium phosphate is precipitated by adding lime.	Selective ion exchange using HAIX (hybrid ion exchange) of hydrated ferric nanoparticles in a strong base anion (quaternary ammonium) resin, using a LayneRT resin base (this is a commercially available arsenic removal media in beads form).  Sodium hydroxide is used to release P from the HAIX iron sites, so generating orthophosphate solution, plus water washing to regenerate the resin.	10 m³/day (58 litres, 1.6m high column) demonstration plant operated continuously for 2 ½ years (60 regeneration cycles), with automated bed regeneration, using real wwtp secondary effluent, Cranfield, UK, 2019 -2021.  Inflow 0.7 mgPtotal/I was reduced to below 0.1 mgPpo4+/I.  See SCOPE Newsletter n°141.	Marity







LYSTEK www.lystek.com jdunbar@lystek.com  No update information provided in 2021-2022	Dewatered sewage sludge (prior to anaerobic digestion) or other organic waste streams such as food waste.	Hydrolysed sludge can be:  - used directly on fields, and contains 20 – 40% C-org/DM and around 5-2-4 N-P-K. Depending on local regulations, can be used as USA Class A biosolids or as a fertiliser product;  - or partly returned to anaerobic digestion, with biogas production c. 25% increased;  - or used to provide available carbon to feed enhanced biological nutrient removal.	Lystek THP® is low temperature thermal hydrolysis: steam injection @ 75°C, alkali addition and physical action for shearing (ensures sensitisation).	Lystek treats nearly 200 000 t DM / yearof sewage sludge in the USA (containing over 3 000 tonnes of phosphorus).  Recent installations by Lystek include St. Thomas - Ontario, Innisfil – Ontario, St. Cloud – Minnesota, Fairfield- Suisun – California, Goleta – California (food waste).  Installations in Europe ???	
Struvite enhanced: acid (MSE- mobile)  Member of DPP  https://www.mse- mobile.de/Dienstleistun g/PhosphorRecycling/ contact: r.turek@mse- mobile.de  No update information provided in 2021- 2022	Sewage sludge before dewatering, from sewage works using chemical or biological P- removal.	Struvite Phosphorus-depleted dewatered sewage sludge (filter cake)	1) Sulphuric acid is added to sewage sludge, to dissolve phosphorus 2) Solid/liquid separation by filter press 3) The phosphorus depleted filter cake contains <2%P/DM 4) The acid liquor is treated with citric acid (to complex and remove metal ions) then neutralised with sodium hydroxide and magnesium oxide is added to precipitate struvite 40-60 % of P in the inflow sewage sludge is recovered in the struvite.	One mobile (two containers) pilot-scale installation has been operated since 2016 at 7 sewage works. Capacity 50 t/day inflow sewage sludge (wet weight before dewatering).	
GENIUS (Nijhuis Saur Industries)  Member of NNP https://www.nijhuisindustr ies.com/de/solutions/dige sters/nijhuis-geniaal/ Contact: thijs.wolbrink@nijhuisind ustries.com  Pending	Liquid manure digestate	N-K sulphate fertiliser solution, used by farmers locally (1 - 1,5 % N wet weight and approx. 1% K wet weight). This solution contains c. ???% C-org wet weight.  P-rich solid organic fertiliser (1 - 3 % P dry weight)  Clean water.	solid/liquid separation by decanter, with polymer use     two-stage membrane filtration with sulphuric acid dosing to produce N-K sulphate fertiliser solution and purified water     the solid fraction is phosphoruscontaining organic fertiliser	Full scale plant operating since 2019 at Groot Zevert Digestion, Beltrum, The Netherlands (100 000 t/y cattle ??? manure wet weight)	







#### RePeat (Nijhuis Solid fraction of Calcium phosphate with Sulphuric acid treatment to Pilot at Groot Zevert Digestion, organic carbon ??? - ???? % dissolve phosphorus then lime Beltrum. The Netherlands. Saur Industries) solid/liquid separated manure digestate dry weight. dosing to precipitate calcium - Continuous or batch? Member of NNP phosphate. Low P organic soil Input capacity (t/h) ? https://www.groenemineralence - Operating since (date)? conditioner. ntrale.nl/english Contacts: steven.nieuwboer@niihuisindus thijs.wolbrink@nijhuisindustries. geo.smith@nijhuisindustries.co **Pendina Byosis (Nijhuis** Liquid fraction of Approximately how many full-scale Ammonium sulphate (ASL unseparated liquid streams organic wastes. 40% solution) or ammonium can be processed but installations operating today Saur Industries) worldwide? Total annual N manure or digestate: nitrate (ANL 50%) or preferential set-up is with prior Member of NNP sewage sludge, ammonium bicarbonate (0.5% removal of solids, e.g. by recovery? manure or other solution) or ammonia water centrifuge, cloth filter, filter https://byosis.com/systems/byof - Tully Centralised Anaerobic biowastes. (20%)press. Digestion Plant, Northern Ireland: Design avoids clogging Ammonia stripping reduces the liquid is heated and 40.000 t/y of poultry litter, production of ASL. and input liquor can toxicity so increasing biogas ammonia is stripped (off-Contacts: contain up to 15% dry production or improving gassed) from the liquid by a steven.nieuwboer@nijhuisindus - Bio Energy Coevorden matter and/or with biological treatment operation, proprietary chemical process, tries.com Netherlands: digestion of 300.000 particles up to 12 mm. depending on site and recovered t/y of manure and waste streams, thijs.wolbrink@nijhuisindustries configuration. production of ASL. - heat exchanger enables energy recovery .vandenbroek@byosis.com - ZWAR, Bergen auf Rügen, 70 – 85 % recovery of Germany - please specify size of ammonia-N can offer optimum sewage works (p.e.): recovery of **Pending** OPEX – CAPEX, but higher N >80% of ammonium from works' recovery rates are possible. sludge digestate and from sludge dryer condensate. Hardenberg, Netherlands - please specify ... manure? Sludge? and capacity ?: recovery of ammonia from waste streams as ammonium bicarbonate for further processing to fertilisers. Varcor No information provided https://www.sedron.com/varcor/ Contact: nfo@sedron.com







Extraphos	Input: digested sewage sludge	Calcium phosphate	1) Liquefied CO <sub>2</sub> (standard gas product) is used to partially dissolve phosphorus in liquid sludge to soluble form (ambient temperature, operating pressure c. 10 bars).  2) solid/liquid separation	Process initially developed by Budenheim with a mobile pilot plant: 1 m³ volume. Technology now owned by <b>Remondis.</b>	
			3) the liquid fraction is used for P-recovery, by pressure release and some lime addition, resulting in calcium phosphate precipitation		
			Iron, aluminium, heavy metals and organic carbon remain mainly in the solid fraction. Tests of contaminant removal using acid acidulation are underway.		
RecoPhos thermal Now integrated into he Flasphos project above.	Input: sewage sludge incineration ash, from sewage works using biological and/or chemical P-removal.	White phosphorus (elemental phosphorus P4), Critical Raw Material for electronics and organophosphorus chemical industry	Initial patent Patent SGLCarbon, acquired by Italmatch.  Electrical induction heated InduCarb reactor, heats ash with coke or graphite to 1500°C. P4 (elemental P) is released in gas form, which can be reacted to PCI <sub>3</sub> (chemical vector for industrial organic phosphorus chemistry) or to high purity phosphoric acid (electronics grade).	10 kg/h input pilot was tested at Leoben, Austria in 2015 (EU FP7 project)	Recophos Project by Italmatch Chemicals
BioEcoSim (Suez)	Liquid manure or liquid digestate, with dry matter content of at least 35%	Recovery of precipitated phosphate salts, ammonium sulphate, organic soil amendment (biochar)	<ul> <li>mineral acids are used to dissolve nutrients into liquid fraction</li> <li>solid-liquid separation</li> <li>optionally: superheated steam drying, then pyrolysis</li> <li>phosphate precipitation from liquor</li> </ul>	Process developed by Fraunhofer IGB and transferred to Suez. A 1.2 t/day pig manure pilot was operated by Fraunhofer IGB 2017-2018 at Kupferzell,	







stripping

gas permeable membrane ammonia

palletisation of solid biochar

Germany.

A pilot plant (10 000 t/y) is operating since July 2019 in

Zorbau, Germany. Industrial scale in preparation. ePhos (Fraunhofer IGB) Contact: siegfried.egner@igb.frau nhofer.de

Sewage sludge dewatering liquor Struvite or K-struvite (magnesium ammonium phosphate)

sium Electro

Electrochemical struvite precipitation, using sacrificial magnesium anode and no chemical inputs

Full-scale installation 2017 OVIVO (USA) ????







