European Sustainable Phosphorus Platform

SCOPE NEWSLETTER

ESPP SCOPE Newsletter n°156 – April 2025

*This Newsletter summarises the ESPP workshop at Aquatech, 13th March 2025: **"The new EU Urban Waste Water Treatment Directive requirements for phosphorus removal and for reuse and recycling: interactions between tighter discharge consents, chemical P-removal coagulants, P-recovery"**, and the **Norwegian Water sewage sludge biochar workshop**, held in Oslo, 12th February 2025.*

ESPP workshop: P reuse and recycling rates

Summary of plenary presentations

- Iron and aluminium coagulants enable reliable phosphorus removal down to 0.5 mg P/l as required for larger wwtps under the recast EU UWWT Directive, or even 0.2 mg P/l as required by national authorities in some eutrophication risk catchments.
- Recovered iron/aluminium phosphates are currently excluded from the EU Fertilising Products Regulation (FPR): CMC12 (Precipitated phosphates and derivates) limits total iron + aluminium to < 10%/DM.
- The European Commission (JRC) is currently collecting information and data on phosphorus recycling technologies, sewage sludge and sludge management routes and relevant national policies, to prepare development of supporting analysis and regulatory proposals (Q2 – 2027) for the phosphorus "reuse and recycling" rates which must be fixed by end 2027.
- The cement industry wishes to continue and expand use of sewage sludge as an alternative fuel for cement kilns, so ensuring decontamination, and is interested to partner with research and testing of upstream P-recovery processes.

Another possible sewage P valorisation route is pyrolysis (sewage sludge biochar), but this is currently excluded from the EU Fertilising Products Regulation (FPR): CMC14 (but authorised under some national fertilisers regulations).

Parallel session summary

Iron / aluminium in sewage sludge and crop P availability

Rapporteur: Robert Van Spingelen, ESPP President

- Much of the phosphorus in soils is poorly plant available, leading to P accumulation in agricultural soils.
- Iron phosphates (in particular, iron phosphates in sewage sludges) are poorly plant available in soils, but long-term field trials show that they can be an effective P-supply for crops over time.
- Release of P from iron phosphates is strongly influenced by soil pH, with near neutral or alkaline soils showing slow P availability for crops.
- Iron phosphates in sewage sludge biochars tend to be poorly crop available, but potassium input to the pyrolysis process may mitigate this.

P-recovery from sewage / sludge upstream of combustion

Rapporteur: Laura Herrera Paiva, Aquacare

- Processes are today available to increase the % of total wwtp inflow phosphorus which can be recovered as struvite (e.g. iPhos, WASSTRIP), today achieving 50% or more recovery of wwtp total P inflow.
- Adsorbent and ion exchange systems (e.g. BiOPHREE, HAIX) can remove phosphorus, down to very low levels. Challenges to implementation may be longevity and robustness of the adsorbent or ion-exchange material in wastewater, and consumption and costs of chemicals needed for regeneration and then for P-recovery from the resulting regeneration liquor.

Impacts of iron / aluminium on P-recovery processes

Rapporteur: Ida Engan, COWI

- Technologies for phosphorus recovery from sewage sludge incineration ashes are today available, demonstrated, and in some cases with full-scale plants under construction.
- Iron in sewage sludge, and so in sludge incineration ash, poses more or less a problem to the different technologies. There are solutions, but iron content may result in lower P-recovery rates or higher operating costs.
- Other challenges are logistics (moving sewage sludge to mono-incinerators) and infrastructure (construction of mono-incinerators).
- Different EU legislations in the water and circular economy sectors, are not always aligned. This impacts phosphorous recovery, where for instance the use of biochar in agriculture is only legally approved in five countries. The technology is already in place, but the lack of legal approval is a barrier to further innovation and growth in the sector.
- There are also economical barriers in the form of logistics (moving sewage sludge to mono-incinerators), scalability and market saturation of certain products.

Possible recycling routes for recovered iron phosphates

Rapporteur: Willem Schipper, phosphorus industry consultant

- 50 – 60% of P in sewage sludge can realistically be recovered as 98% pure vivianite (iron(II) phosphate), by the magnetic ViviMag® process (<1% organics).
- There is a limited market as an iron fertiliser in regions of Southern Europe with calcareous soils, to combat crop iron deficiency (chlorosis).
- There may possibly be other markets in industrial applications, but quality, compatibility and supply logistics need to be demonstrated.
- An objective is to separate the iron and phosphorus in the recovered vivianite to a marketable P product and iron salts (for recycling in waste water P-removal).
- The SINFERT deoxychlorination process, currently at the laboratory development stage, seems to achieve this separation, producing organo-phosphorus chemical precursors which have potentially higher value than phosphoric acid or inorganic phosphates.

The revised Urban Waste Water Treatment Directive (UWWTD)

Robert Van Spingelen, ESPP President and Ostara, opened the workshop, welcomed participants and underlined the **major changes and challenges of the 'new' (completely recast) EU Urban Waste Water Treatment Directive** (UWWTD 2024/3019 November 2024). These include tighter phosphorus and nitrogen discharge limits for many waste water treatment works (wwtps) including for some sewage works today without N and P removal obligations today), "Quaternary Treatment" to remove organic contaminants, 'Extended Producer Responsibility' for pharmaceuticals and cosmetics (to cover quaternary treatment costs), "Integrated Water Management Plans" (storm overflows), energy neutrality requirement and greenhouse gas monitoring, measurement of microplastics …

The new EU UWWTD specifies that "**minimum combined reuse and recycling rates" for phosphorus will be fixed across the EU by January 2028**.

Details of provisions relevant to phosphorus and nutrients in the recast Urban Waste Water Treatment Directive (UWWTD): see ESPP eNews n°93.

Coagulants and phosphorus removal

Jean-Christophe Ades (ESPP Treasurer and Kemira), for INCOPA (the **European Inorganic Coagulants Producers Association**, an ESPP member), explained the role of coagulants in reliably removing phosphorus from wastewater down to low discharge concentrations ("chemical P removal"). Inorganic coagulants are iron or aluminium salts which precipitate soluble orthophosphates into insoluble phosphate salts (iron or aluminium phosphates). This precipitation process also agglomerates small organic particles into larger conglomerates which can be more easily removed from the water, so also reducing organics in discharge (BOD – Biological Oxygen Demand). As well as widespread use in sewage treatment, coagulants are also used in drinking water and industrial process water purification.

Appropriately managed coagulant dosage and particle removal (settling or filtration) can reliably achieve the new phosphorus discharge limits fixed by the revised UWWT Directive:

- **0.5 mg P/l for wwtps > 150 000 p.e.** (was 1 mg P/l under the previous 1991 UWWTD)
- **0.7 mg P/l for wwtps > 10 000 p.e**. (p.e. = population equivalent)

Photo: Regular verification of coagulant effectiveness in a wastewater sample (photo: Kemira).

Coagulants can also reliably achieve the significantly lower P discharge consents already required and implemented today in some regions. For example, 0.2 mg P/l in many wwtps in Finland, required to address Baltic Sea eutrophication. See further information in SCOPE Newsletter n°141.

A Life Cycle Analysis comparing chemical to biological P-removal, carried out by IVL for INCOPA (see SCOPE Newsletter n°138) indicates use of coagulants reduces carbon emissions, mainly because it improves organics removal, so transferring more organics into sewage sludge which is then used for anaerobic digestion methane production (renewable energy). Chemical P-removal uses less electricity than biological P-removal (the latter requires oxygenation) so facilitating the new UWWTD energy neutrality objectives (art. 11 of 2024/3019). INCOPA is now working with Carbon Minds (mandated by European Commission) to develop Environmental Footprint compliant Life Cycle Inventory (LCI) datasets for inorganic coagulants.

European Commission – UWWTD P reuse and recycling rates

Lukas Egle and Dries Huygens, European Commission Joint Research Centre, explained the preparatory work underway to support the definition by the European Commission of phosphorus "reuse and recycling rates", as required by recast Urban Waste Water Treatment Directive 2024/3019 (UWWTD).

Art. 20 of the recast UWWTD away states: "*The Commission is empowered to adopt delegated acts … specifying a combined minimum reuse and recycling rate for phosphorus from sludge and from urban wastewater not reused…, taking into account available technologies, resources and the economic viability of phosphorus recovery as well as the phosphorus content of the sludge and the level of saturation of the national market with organic phosphorus from other sources while ensuring that there is safe sludge management and no adverse impact on the environment or human health. The Commission shall adopt those delegated acts by 2 January 2028*".

Recital 28 indicates that the minimum combined reuse and recycling rate "*should be defined at Union level giving Member States the flexibility to choose whether to reuse and/or to recycle the urban wastewater and/or sludge to recover phosphorus. This combined minimum reuse and recycling rate should take into account … Member States should, while taking into account … take measures to encourage the production and purchasing of recovered nutrients from urban wastewater and sludge*."

Recital (42) indicates that this shall "*contribute to the strategic autonomy of the Union fertiliser industry*".

JRC will therefore collect data particularly concerning:

- phosphorus recycling technologies available and under development,
- phosphorus contents of sewage sludges,
- other organic phosphorus sources, in particular manures,

- environmental impacts, in particular sewage sludge contaminants and related risks.

This work will involve research and data collection by JRC with structured stakeholder dialogue, engaging Member States, industry, NGOs and experts, as well as possibly the organisation of workshops.

JRC's tentative timeline for deliverables from this work is:

- Q3 / Q4 - 2025: overview of existing phosphorus recycling technologies and current sewage sludge management routes (including agricultural valorisation, mono-incineration, composting, relevant national phosphorus recycling regulations);
- Q2 - 2026: data on market viability of recycled phosphorus products (taking into consideration the Waste Hierarchy), cost-assessment of sewage sludge management routes, scenario analysis;
- Q4 - 2026: assessment of impacts on human health and environmental protection from selected priority contaminants in sludge and derived materials;
- Q2 - 2027: integrated draft study report including technical proposals.

This work will build on two published JRC studies: 1) Screening risk assessment of organic pollutants and environmental impacts from sewage sludge management (see ESPP eNews n°71), 2) Feasibility study in support of future policy developments of the Sewage Sludge Directive (see ESPP eNews n°81).

At present, JRC is collating data on wwtps (number, capacity, treatment methods, ingoing and outgoing phosphorus, and other parameters, sludge production), current sewage sludge management routes, sludge nutrient content, as well as on P-recycling technologies, national P-recycling legislations and other applicable EU regulations (e.g. Waste, Landfill).

Knowledge gaps identified include:

- Type of P-removal at wwtps (chemical, biological or a combination of both? This may impact possible phosphorus recycling processes and affects future scenarios on P recycling.
- Management routes of sludge where destination in data is "other" (more than 1/3 of EU sludge) ?
- What is the final destination of composted sludge ?
- What proportion of sludge goes to anaerobic digestion ?
- Expected increases in sludge mono-incineration ?
- TRL and state of full-scale implementation roll-out for different P-recycling technologies ?
- Member State P-recycling strategies?

Sewage sludge valorisation in the cement industry

Sussan Pasuki, Heidelberg Materials and CEMBUREAU (European Cement Association) explained that Europe has around 200 cement plants, with the capacity to valorise a total of some 2 MtDM/y, that is over one fifth of EU sewage sludge. Use of sewage sludge as fuel in cement kilns converts the organic carbon content to energy - contributing to the cement industry's objective to reach 60% non-fossil fuels by 2030 and 95% by 2050, and simultaneously valorises calcium, silicium, iron and aluminium into the cement, immobilises heavy metals and fully eliminates all organic pollutants including PFAS (cement kilns operate at > 1450°C, significantly higher than the EU Waste Framework Directive incineration requirements of 850°C, and with longer residence times). Phosphorus in sewage sludge is a limiting factor for cement kiln valorisation (phosphorus retards setting) so there is synergy between phosphate recovery upstream from sewage/sludge and valorisation of sludge in the cement industry. The cement industry is ready to work with research and the water industry to develop such upstream P-recovery systems and to test these locally with cement factories. The aim is to promote synergies and regional circular economy solutions, including using potential excess heat from cement kilns for drying the sludge

Photo Sewage sludge drying, Heidelberg Materials cement plant, Geseke, North Rhine-Westphalia, Germany

Parallel session – Iron / aluminium in sewage sludge and crop P availability

José-Marie Gomez, Biomasa Peninsular and EFAR (European Federation for Agricultural Recycling), set the context: the EU currently produces some 9 million t DM/y of sewage sludge (50 Mt WW/y) containing 4 Mt/y organic matter, 450 000 tN/y (nitrogen) and 270 000 tP/y (phosphorus). Of this, currently nearly 50% goes to agriculture after stabilisation and sanitisation by anaerobic digestion and/or composting. EFAR represents 19 members in 8 EU countries, managing 4 million t/y of biosolids (stabilised sewage sludge), used by around 5 000 farmers.

Average phosphorus (P) content of stabilised sewage sludge ("biosolids") is around 2.5% P/DM, of which 25 – 65 % is organic and 50 – 75 % mineral (mainly phosphates of Ca, Mg, Fe). Literature papers show widely varying levels of P availability in sewage sludges, from 20 to 150% compared to mineral phosphate fertilisers.

EFAR, Eureau and Aqua Publica are establishing a "Biosolids Observatory" to analyse sewage sludges and collate and update data from across Europe. This will address sewage sludge nutrient content and organic contaminants (including PFAS, PAH, PBDE (one family of brominated flame retardants), pharmaceuticals, microplastics … To participate and to supply sewage sludge for analysis, contact biosolidsobservatory@gmail.com

Kasper Reitzel, University of Southern Denmark (SDU), presented soil incubation, barley pot trials and rye grass field trials of the phosphorus fertiliser value of vivianite (iron(II) phosphate), iron(III) phosphate, and lake sediment with differing iron content or processed sediment (e.g. HTC). The lake sediment was from suction dredging, carried out to restore the eutrophied Lake Ormstrum (Denmark) and contained around 3 mg/PDW total P, of which over 2/3 as iron phosphates. The sediment was tested as dredged and after iron depletion (dithionide extract, leaving mainly the P fraction not bound to iron). In the pot trials, the iron-containing materials showed fertiliser replacement value of 20 – 60% compared to TSP (triple super phosphate). In the field trials, however, the iron-rich sediment showed fertiliser value comparable to TSP with lower risk of phosphorus leaching. This may be because of benefits of the organic matter in the sediment.

Ruben Sakrabani, Cranfield University UK, summarised soil phosphorus chemistry and equilibria between readily available and less available P. He explained that the 'push and pull' between pools makes the soil solution P the smallest pool which is plant available. Overall calcium phosphates are more plant available than aluminium phosphates than are iron phosphates. There are more than a dozen methods for measuring plant availability of P in soil: none is perfect, the NAC method (neutral ammonium citrate) adopted in the EU Fertilising Products Regulation is a reasonably good indicator. However, low measured phosphorus availability does not mean that a material will not function as a fertiliser as plant roots and associated rhizobacteria and fungi can solubilise recalcitrant forms of phosphorus.

Anders Finnson, Svensktvatten (Swedish Water), based on data from Håkan Jönsson, SLU (Swedish Agriculture University), presented results from long-term field trials (since 1981) in Scania, southern Sweden, comparing fertiliser value of sewage sludge to mineral fertiliser. The sewage sludge was from sewage works using iron and aluminium salts for chemical P-removal and was applied at 0, 4 or 12 tDM/ha every four years. Results show that sewage sludge cannot provide immediately available P in the same way as mineral fertiliser (e.g. application in crop initial growth period), but that P in sewage sludge progressively similarly available to P from mineral fertiliser over time. The application of sewage sludge has shown to increase soil organic carbon levels.

He underlined the importance of prevention at source for reducing contaminants in sewage sludges. Heavy metal levels have been considerably decreased in sewage sludges over recent decades, in particular cadmium and mercury, which are generally today not an issue. Nickel, zinc and copper levels have not decreased, but these are plant micronutrients and not toxic if applied at controlled rates. Past use of sewage sludge on fields has shown to increase soil heavy metal contents, but not impact levels in crops.

SM Ashekuzzaman, MTU Munster Technological University, Ireland, presented phosphorus fertiliser replacement value (P-FRV) of short-term pot and 3-year field trials (grassland, low P soil) of sewage sludge incineration ash and poultry litter incineration ashes, calcium stabilised dairy sludge, aluminium P-precipitated dairy sludge, and two recovered struvites. The calcium treated sludge and the ashes showed significantly lower first-year crop P availability (25 – 39%) than mineral fertiliser, whereas P-FRV increased to 74 – 103% after 2-3 years for Ca-sludge and poultry litter ash, and up to 57% for sewage sludge ash. The first-year P-FRV of struvite products was between 137 and 141%, increasing to 150–160% over 2 and 3 years of cumulative P-FRV. The P-FRV of aluminium treated dairy sludge on the 1st harvest was 50% increased to 106% over the four harvests after 1st-year, and remained between 146 and 162% after 3 years of field trial. See also Elizabeth O'Carroll, TEAGASC, in SCOPE Newsletter n°149.

See: "Differing Phosphorus Crop Availability of Aluminium and Calcium Precipitated Dairy Processing Sludge Potential Recycled Alternatives to Mineral Phosphorus Fertiliser", Ashekuzzaman et al., Agronomy 2021, 11, 427. DOI.

Naeimeh Vali, University of Borås, Sweden, presented a study on the impacts of aluminium and potassium on mineral speciation in sewage sludge-derived biochars. Thermodynamics equilibrium calculations revealed that aluminium, iron and silicate-bound phosphates were the predominant phosphorus species in the resulting biochar, accounting for over

70% of the total phosphorus, largely due to the prior use of aluminium salts for chemical phosphorus removal at the wastewater treatment plant. When wheat straw or bakery waste, rich in potassium, were introduced into the pyrolysis feedstock, the biochar produced at 650 °C exhibited a significant transformation in phosphorus speciation. Potassium-, calcium-potassium-, and magnesium-potassium phosphates became predominant, together constituting over 90% of the total phosphorus in the biochar. This shift may enhance the plant availability of phosphorus, although plant trials have not yet been conducted to verify this potential.

Rouven Metz, Norwegian University of Life Sciences (NMBU), presented experimental results showing the biochemical mechanisms impacting bioavailability of phosphorus (P) and iron (Fe) in vivianite ($Fe(II)_3(PO_4)_2\ 8H_2O$). Vivianite can act as both an Fe and P source and sink; depending on the prevailing redox conditions. Under anoxic conditions, dissolution rates are fast, structure-defect driven and controlled by solution saturation. Thus, under anoxic, slightly acidic conditions, vivianite may be a suitable P source for plants, since solubility and hence dissolution rates, increase strongly with decreasing pH. However, under oxic conditions, structural Fe(II) oxidizes rapidly to Fe(III), forming an amorphous Fe(III)-phosphate surface shell, which strongly decreases vivianite dissolution, and thus the availability of P and Fe. The oxidised vivianite surface, however, does not only hinder vivianite dissolution, but also prevents further oxidation, preserving an unoxidized vivianite core which may then be detected as “vivianite” with common measuring methods. These findings may explain some contradicting results about the suitability of vivianite as a fertiliser.

See: “Effect of Oxidation on Vivianite Dissolution Rates and Mechanism”, R. Metz et al., Environ. Sci. Technol. 2024, 58, 15321−15332, DOI.

Parallel session – P-recovery from sewage / sludge upstream of combustion

Joachim Clemens, SF Soepenberg, presented the iPHOS® process:

- reduction by a chemical process with addition of sulfide,
- acidification to pH 4,
- separative precipitation of heavy metals,
- flocculation (polymer dosing) and dewatering,
- phosphate precipitation from dewatering liquor and settling, as $KMgPO_4$, $NaMgPO_4$, NH_4MgPO_4 (struvite).

Phosphate release from sewage sludges (from sewage works using iron flocculants) of up to 75% (soluble P after the flocculation / dewatering stage) are achieved in laboratory conditions with around 15-20 hours for the reduction process. P-release is considerably less effective from aluminium precipitated P. A mobile 1 – 2 m^3/h inflow pilot unit has been tested at Gifhorn wwtp, Germany, continuous operation for three months, achieving nearly 70% P-release. Soepenberg is constructing a mobile plant that will start operation in summer 2025 at interested wwtps.

Dirk Herold, Xylem, presented optimisation of struvite P-recovery in sewage works by the Ostara WASSTRIP® return and release stream process. The Ostara Pearl® struvite precipitation process improves biological P-removal in sewage works by avoiding pipe and pump blockages by struvite deposits and by avoiding return of soluble phosphorus from sludge processing to the P-removal stage. WASSTRIP further improves this by deliberately releasing soluble phosphorus from secondary clarification sludge (by dosing of volatile fatty acids or 5-10 % primary sludge in specifically controlled anaerobic conditions) and sending this to the struvite precipitation unit. This can double the % of wwtp inflow P recovered as struvite and avoid struvite deposit problems in anaerobic digesters. With around 12 – 24 hours WASSTRIP residence time, 50% of more of phosphorus can be released (from biological P-removal sludge). Combined with P-release in the anaerobic digester, this enables overall recovery of around 55% of wwtp input P_{total} in struvite.

Carlo Belloni, Wetsus, Netherlands, presented pilot tests of P-removal and then release (to a soluble P stream) using iron oxide adsorbents (BiOPhree process, see SCOPE Newsletter n°138 and n°132) and discussed work underway to improve regeneration of the adsorbent and on process scale-up. A 3 m^3/h pilot operated by Aquacare and Royal HaskoningDHV for more than one year at Dronten wwtp The Netherlands (biological P-removal combined with aluminium coagulant dosing). The BiOPhree unit treated the wwtp final effluent after secondary clarification, as a polishing step. BioPhree showed consistent P-reduction from unit influent 0.7 – 10 mg P_{total}/l (average 1-2 mg P_{total}/l) to below 0.1 mg P_{total}/l, but with ‘breakthroughs’ when influent P was high. The iron oxide adsorbent was regenerated using acid (to release Ca minerals) followed by alkali (to release P), resulting in a soluble phosphorus solution of 200 - 500 mg P/l from which phosphorus could be recovered by e.g. precipitation.

Ana Soares, Cranfield University UK, updated on ion exchange processes for P-removal and recovery. HAIX (hybrid ion exchange) see ESPP nutrient recycling technology catalogue shows high selectivity for phosphorus and can be regenerated using sodium hydroxide. A 10 m^3/day pilot operated for 2 ½ years showed outflow of < 0.1 mg $P_{soluble}$/l. Phosphate is recovered from the regeneration solution by precipitation of calcium phosphate. Two full scale installations are currently planned in the UK plus a demonstration pilot in The Netherlands.

Parallel session – Impacts of iron / aluminium on P-recovery processes from ash

All these processes are presented in the ESPP online nutrient recycling technology catalogue (process description, input materials, output product, operating status) technology catalogue

Christian Kabbe, EasyMining, presented **Ash2Phos**, indicated that priorities in defining P-recycling routes should be a product with real demand, which depends on quality and reliable production supply security, and volume.

- Ash2Phos output: dry solid calcium phosphate RevoCaP^{TM} (animal feed grade), with 16.5% P content (38% P_2O_5).
- P-recycling rate: >90% of P in ash is recovered.
- Operating status: two 30 000 t/y ash input plants are under construction or permitting in Sweden and in Germany, and will each recycle > 2 500 tP/y.
- 96 - 99 % of heavy metals from the ash are transferred to a concentrated depollution cake for disposal
- Fate of iron and aluminium in ash will depend on levels in input ash and Ash2Phos process configuration:
  - c. 20% of iron in ash is recovered as 40% aqueous solution in the basic Ash2Phos configuration, increasing to >95% iron recovery with the 'Sahara' module, c. 0.1 % Fe/DM in output calcium phosphate,
  - c. 60 – 80 % of aluminium in ash is recovered as 38% aqueous solution or solid ATH (aluminium tri hydrate), c. 0.1 – 0.2 % Al/DM in output calcium phosphate.

Photo: EasyMining Ash2Phos pilot, 600 kg/day ash input, Helsingborg

Mohamed Takhim, TTBS, presented the **RubiPhos** technology to recover phosphorus from ashes, based on selective acid digestion and membrane purification.

- Output: phosphoric acid $\geq$20 % P_2O_5 before concentration, 62% P_2O_5 after concentration, containing <1% of ash input heavy metals.
- P-recycling rate: >95% of P in ash is recovered.
- Operating status: 12.5 kg/h pilot operated for two hundred hours.
- Fate of iron and aluminium in ash:
  - 40-50 % of iron in ash is recovered as 5-8% aqueous solution, < 5 ppm Fe in output concentrated phosphoric acid,
  - 60-70 % of aluminium in ash is recovered as 2.5-5 % aqueous solution, < 1 ppm Al in output concentrated phosphoric acid.

Marga Breeuwsma, SusPhos B.V, presented the company's process in which acid is reacted with the ash then a proprietary inorganic solvent is used to extract phosphoric acid from the resulting wet solid. Phosphate salts can be recovered from the solvent without an aqueous phase.

- Output: Phosphate salts.
- P-recycling rate: >80% of P in ash is recovered.
- Operating status: 1 kg/h pilot operated for two years, full scale plant planned.
- Fate of iron and aluminium in ash: both iron and aluminium will end up around half in the phosphate salts and around half in the process residue, which is intended for valorisation in cement.

Hubert Halleux & Marc Sonveaux, Prayon, presented the company’s **PELP (Prayon Ecophos LooP Process),** using hydrochloric acid, filtration and ion exchange to recover phosphorus from ashes as phosphoric acid.

- Output: phosphoric acid ≥85 % P_2O_5 after concentration.
- P-recycling rate: >90% of P in ash is recovered.
- Operating status: 200 kg/h ash input industrial pilot operated for several months (photo).
- Fate of iron and aluminium in ash:
  - 50 – 60 % of iron in ash is recovered as 40% $FeCl_3$ aqueous solution. The remaining 40-50% goes to the solid residue. < 5 ppm Fe in output product (purified phosphoric acid).
  - 60 - 70% of aluminium in ash is recovered as 30% $AlCl_3$ aqueous solution. The remaining 30-40% goes to the solid residue. < 5 ppm Al in output product (purified phosphoric acid).

Matthias Rapf, Stuttgart University, presented the **Flashphos** project for recovery of elemental phosphorus (P_4) from sewage sludge and/or sludge incineration ash, by thermal reduction.

- Output: P_4.
- P-recycling rate: objective >85% of P in ash recovered.
- Operating status: project pilot plant is being designed for a) 50 kg/h wet sewage sludge input in the dryer-grinder, and b) 250 kg/h dry sludge input in the Flash reactor + refiner. The project builds on the experience of the 10 kg/h RECOPHOS Leoben pilot (EU FP7, see ESPP SCOPE Newsletter n°136)
- Fate of iron and aluminium in ash:
  - most iron is expected to come out as ferrophos Fe-P alloy, with low market value
  - aluminium is expected to come out as slag
  - iron and aluminium levels in recovered P_4 expected to be near zero.

Frans Horstink, ThermusP, presented the **Spodofos** project for recovery of elemental phosphorus (P_4) from sewage sludge incineration ash or other ashes, by thermal reduction using secondary aluminium metals as reducing agent to avoid consumption of primary energy (see ESPP eNews n°64)

- Output: P4.
- P-recycling rate: pilot results >90% of P in ash is recovered.
- Operating status: tested to date at laboratory TRL5 kg scale (photo). Pilot TRL6 (100 kg ash/h) is under construction.
- Fate of iron and aluminium in ash:
  - all iron in ash is expected to come out as low-P ferrophos Fe-P-Si alloy. ThermusP consider that this has potential market replacing ferrosilicon in Dense Medium Separation (separation of minerals),
  - all aluminium in ash comes out with the secondary aluminium metal feed as high-alumina slag. ThermusP consider that this has potential market as hydraulic active cement replacement or refractory granulate,
  - iron and aluminium levels in recovered P_4 are near zero

Other P-recovery processes from sewage sludge incineration ash, not presented at this workshop, include the following (see ESPP nutrient recycling technology catalogue). The information below was provided by the technology supplier.

SINFERT – see below in this document

Remondis (Tetraphos):

- Output: phosphoric acid c. 55 % P_2O_5 (75% H_3PO_4) after concentration.
- P-recycling rate: 80 – 95 % of P in ash is recovered.

- Operating status: one full-scale plant, Hamburg, Germany, started operation, today undergoing process optimisation.
- Fate of iron and aluminium in ash:
  - the ash is leached using phosphoric acid, with the primary goal of phosphorus recovery, so solubilising phosphorus and calcium but not most of the iron, aluminium, nor heavy metals
  - iron and aluminium leached from the ash are recovered as aqueous solution (concentration not specified). The % of iron and aluminium recovered and % remaining in leached ash are variable.
  - the output RePacid® (phosphoric acid) contains 0,1 - 1,0 % of aluminium and iron in total.

Técnicas Reunidas (Phos4life):

- Output: phosphoric acid c. 54 % P_2O_5 (c. 13 % P_2O_5 before concentration).
- P-recycling rate: >80% of P in ash is recovered.
- Operating status: 1 kg/h ash input pilot operated continuously 24/7 for seven campaigns, each of 2 – 4 weeks.
- Fate of iron and aluminium in ash: the iron and aluminium are transferred to the solid leaching residue intended for recycling in cement production (c. 15% Fe and c. 2.5 % Al content) and to a gypsum by-product (c. 3% Fe, 3-4% Al).
- Output phosphoric acid contains 5 ppm iron (aluminium not reported).

Impacts of iron / aluminium on P-recovery via biochars

Helmut Gerber, Pyreg, indicated that the company has today over 50 commercial pyrolysis units in operation producing biochar, of which seven processing sewage sludge. Sewage sludge biochar is authorised as a fertiliser in the Czech Republic, Denmark, Estonia, Finland and Sweden, but is to date excluded from the EU Fertilising Products Regulation (FPR CMC14) following questions expressed by the EU-JRC STRUBIAS work group 2019 as to whether organic contaminants are eliminated. Pyreg considers that studies since then show elimination of organic contaminants if pyrolysis takes place at >600°C with a residence time >10 min. An advantage of pyrolysis compared to sewage sludge incineration is that installations can be smaller*. Pot trials suggest that sewage sludge biochar offers P fertiliser effectiveness comparable to mineral fertilisers, however solubility of phosphorus may not be sufficient for classification as a P-fertiliser under the EU FPR (>80% NAC required). Tests of the effects of iron or aluminium in sewage sludge suggest that P-solubility or P-crop availability is somewhat reduced by iron.

Andrea Salimbeni, ReCord, presented Investigations into extraction of iron and phosphorus from sewage sludge biochars, by sequential leaching and precipitation (using specific organic acids and alkalis): an update of the Charlene process, see ESPP nutrient recycling technology catalogue. With the developed process, the phosphorus is extracted from a biochar, whereas heavy metals remain. The so obtained biocoal (leached biochar) can be used in the metallurgy sector. With the previously tested Charlene process, over 95% of P in sewage sludge was recovered as phosphate salts, while 50% of Al and 20% of Fe are left in the char and the rest is transferred into the recovered phosphate salt. However, with the updated two-steps leaching process, 92-95% of phosphorus is recovered, while 90% of Zn and about 50% of Fe and Al are left in the biochar, enabling to obtain a higher quality phosphate salt. This output can achieve the EU FPR CMC12 criteria (P_2O_5 > 16%/DM and (Fe+Al) < 10%/DM) but today is not included in the FPR because, according to the CMC12, 'Precipitated Phosphates' can be recovered from sewage sludge, but not from sewage sludge biochar.

Parallel session – Possible recycling routes for recovered iron phosphates

Martijn van Leusden, Royal Haskoning DHV, argues that there will be a need to be able to influence the P-content of sludge already at wwtps to maintain control over sludge disposal costs.

Further, he updated on development and testing of the ViviMag® vivianite recovery process This process uses a magnetic field to extract vivianite (iron(II) phosphate) from sewage/sludge liquors. It is particularly effective in sewage works using iron salts as coagulants for phosphorus removal (increases vivianite content of sludge) and after anaerobic conditions or anaerobic sludge digestion (such conditions tend to result in iron phosphate being reduced from iron(III) phosphate to vivianite).

A 1 m^3/h inflow, fully-automated pilot has been tested for several months operating time at four wwtps in Germany, Denmark and the Netherlands, using both anaerobically digested sludge and non-digested sludge (in both cases, before dewatering). See ESPP nutrient recycling technology catalogue.

See below, preliminary results of ViviMag process development published by Prot et al. 2019, Wijdeveld et al 2022, Nguyen et al. 2024.

Around 40-80% of total P in undigested sludge is present as vivianite and 40-90% from digested sludge. With vivianite recovery rates of up to 80%, this represents up to 50% of total P in wwtp inflow. The recovery rate can be increased by increasing iron dosing. Work now starting in the EU-LIFE

Phos4EU project (9 m^3/h unit) aims to achieve recovery of 60% of wwtp inflow P by converting 80% of inflow P to vivianite in sludge, and recovery of 80% of this vivianite from the sludge.

Valorisation of vivianite is under investigation. Possible routes include separation of vivianite into iron and phosphate compounds, or to use it as an anaerobic digestion additive (reduction of H_2S) or to identify possible industrial applications.

Martijn Bovee, Aquaminerals, The Netherlands, further discussed possible valorisation routes for vivianite towards products with markets.

After washing (using dissolved air flotation, so separating organics) and air drying, the recovered vivianite from the ViviMag® pilot is today around 98% purity (98% vivianite / total dry matter). Most of the remaining 2% is other minerals and organic carbon is < 1%.

Possible markets identified to date are:

- Iron micronutrient fertiliser, to address chlorosis, important for some crops in particular on calcareous, alkaline and low-iron soils, or phosphate fertiliser in some specific conditions (but for both these applications, currently not authorised under the EU Fertilising Products Regulation, see below),
- Raw material for possible industrial applications such as wildfire fire retardants (currently ammonia phosphate is widely used), batteries,
- Use for sulphur binding in anaerobic digesters or water treatment (avoid H_2S),
- Electro/chemical processing to separate the iron from the phosphorus and recover marketable iron and phosphorus compounds.

ViviMag® magnetically recovered vivianite today achieves 98% purity

Aquaminerals (see above) confirm that the **recovered vivianite from the ViviMag® pilot today, after washing, can be air dried and has organic carbon <1%.**

Previous information, showing lower purity, concerned non-washed recovered vivianite, partly because the initial R&D targeted primarily phosphorus removal from the sewage sludge, rather than investigation of the recovered material quality:

- Initial results with the ViviMag® 1 m^3/h pilot indicated that the recovered vivianite, after washing, contains with 5 – 10% C_{org} organic carbon (communicated by Korving and Wilfert, Wetsus, in SCOPE Newsletter n°138, page 18).
- Prot et al. 2019, in first pilot results, showed recovered material with 52 – 62 % vivianite content and 20% organic matter (that is maybe 7 -10 % C_{org} assuming an Organic Matter / Organic Carbon ratio of 2-3).
- Wijdeveld et al. 2022 (see below) indicates that the recovered material is 80% vivianite, with 10% total carbon content.
- Nguyen et al. 2024 (see below) indicate that the recovered vivianite has 70-86 % purity and Prot et al. 2020 (see below) indicate 55 – 80 %, but it is unclear what this means (% vivianite/DM ?) and it is not clarified what are the remaining %.

Recovered vivianite and the EU Fertilising Products Regulation (FPR) 2019/2009

- ➢ **Organic carbon** content: the current EU Fertilising Products Regulation criteria for precipitated phosphates (CMC12) specify a limit of max. 3% organic carbon (C_{org}/DM). This applies to the phosphates as precipitated, that is irrespective of processing (not to ‘derivates’).
- ➢ **Iron and aluminium: v**ivianite is in any case at present excluded from the FPR by CMC12 because these criteria limit the sum of aluminium and iron to max. 10% (Al + Fe / DM), including after possible processing (that is, also in ‘derivates’).

Published studies presenting the ViviMag® process development

Prot et al. 2019 published results of a first proof-of-concept lab-scale (1 litre capacity, batch) trials using anaerobically digested sewage sludges from two municipal wwtps using iron salt coagulants for phosphorus removal (Dokhaven, Netherlands – Espoo, Finland). The magnetically recovered material, after washing, was 50 -65% vivianite, c. 20% organics, nearly 10% quartz, with also significant calcium and carbonates (1 - 2 % Ca) and traces of sulphur and of other minerals (magnesium, potassium … <1%). Over 80% of the phosphorus in the magnetically recovered material could be solubilised using 7.5M sodium hydroxide (alkali), with an OH:Fe ratio of 5 – 10x.

“Magnetic separation and characterization of vivianite from digested sewage sludge”, T. Prot et al., Separation and Purification Technology 224 (2019) 564–579, DOI.

“Full-scale increased iron dosage to stimulate the formation of vivianite and its recovery from digested sewage sludge”, Water Research 182 (2020) 115911, DOI.

Wijdeveld et al. 2022 and Prot et al. 2022 published first results of trials with the 1 m^3/h continuous-flow Vivimag® pilot at wwtp Nieuwveer The Netherlands Netherlands and effects on vivianite formation of increasing the iron coagulant dosing in the wwtp. The pilot plant was fed anaerobically digested sludge from the wwtp. 34 operating runs were carried out over 9 months. The wwtp was using iron salts (Fe((II)), dosed in the aerated section, as phosphorus removal coagulants throughout the trials (but with nearly one third of the digester input sludge also coming from other wwtps) and the iron dosing was doubled for four months at the Nieuwveer wwtp plant to assess impact of this on vivianite in the digested sludge (from c. 0.5 to close to 1 molar Fe:P), as presented in Prot et al. 2020. This caused the proportion of vivianite (as % of iron phosphate) in the digested sludge to increase from 20% to 50%. It also increased P removal to the sludge and decreased H_2S in biogas. The ViviMag® pilot achieved recovery of over 80% when operated with three pass recirculation (that is: 80% of the vivianite present in the sludge), that is 5 – 20 % of the total phosphorus present in the sludge (fig. 4). The magnetically recovered material was identified to be up to 80% vivianite (10% P), with 10% carbon (as % of DM). Further tests of this 1 m^3/h continuous-flow pilot have now been carried out in three other sewage works, confirming and improving these initial results (see Martijn van Leusden, Royal Haskoning DHV presentation above, Nguyen et al. below, and SCOPE Newsletter n°138, page 18).

"Pilot-scale magnetic recovery of vivianite from digested sewage sludge", W. Wijdeveld, T. Prot et al., Water Research 212 (2022) 118131, DOI.

Nguyen et al. 2024 published further results of pilot trials over two weeks with the 1 m^3/h continuous-flow Vivimag® pilot at Schönebeck wwtp Germany, operating on digested sewage sludge. The P is biologically removed at this WWTP (EBPT) but for the duration of the piloting, a high quantity of iron (Fe/P ~0.8-1.2) was added to promote vivianite formation. During the pilot trials, the sludge was engineered to modify its viscosity and vivianite particle size, and separately study the effect of these parameters on vivianite recovery rates. Conclusions are that the ViviMag vivianite extraction system efficiency was unaffected by sludge viscosity (1.8 - 4% DM tested). Recovery rates remained identical for different vivianite particle sizes, with a decreased efficiency only for particle sizes below 10µm. The bulk of vivianite in sludge is typically 20-200 µm, suggesting that the technology should be robust in real wastewater. In addition, it was observed that recovery rates increased with increasing vivianite concentrations, likely due to the mechanism of magnetic flocculation, when vivianite particles under a magnetic field behave as mini-magnets themselves.

"Robust magnetic vivianite recovery from digested sewage sludge: Evaluating resilience to sludge dry matter and particle size variations", H. Nguyen, T. Prot et al., Water Research 266 (2024) 122407, DOI.

See also details in "Experiences from Phosphorus Recovery Trials with the ViviMag® Technology", O. Grönfors et al., Klärschlammkonferenz Berlin, Verwertung von Klärschlamm 5, 14-15/11/2022 LINK.

Vivianite precipitation instead of struvite ? A number of recent research publications from China (see e.g. reviews by J. Zhang et al. 2022 and C. Zhang et al. 2022) suggest that vivianite precipitation from wwtp liquors is more interesting than struvite precipitation. This research interest seems to be based on the fact that vivianite can be precipitated by adding iron salts (cheaper than magnesium salts for struvite), with high P-removal efficiencies without control of conditions (e.g. pH), but also on the idea that recovered vivianite has a very high value, apparently based on a price of 10 000 €/t in Wu et al. 2019, this being based on the website Alibaba. However, the price for public retail quantity and laboratory quality of a chemical bears no real relation to the price for industrial quantities, taking into account water content, purity, quality, supply and logistics. Vivianite precipitation would require the dosage of divalent iron and because of the low solubility of vivianite compared to struvite there are challenges to obtain large crystals. The P-recovery percentage via this approach is, like in the case of struvite precipitation, limited by the amount of phosphate that can be released to the liquid phase. Vivianite formation directly in the sludge and subsequent recovery via magnetic separation shows potentially higher recovery percentages because other mineral phosphate species are recrystallised into vivianite.

Photos: Kemira ViviMag® continuous-flow 1m^3 mobile pilot vivianite recovery plant

Kirill Nikitin, University College Dublin, summarised results to date of recovery of organic phosphate esters from vivianite in the SINFERT process (see ESPP nutrient recycling technology catalogue and SCOPE Newsletter n°153). These P-compounds can be used for fire safety and other industrial applications, that can otherwise only be accessed via thermal-route P_4 (P_4 is specifically listed as an EU Critical Raw Material, separately and in addition to Phosphate Rock). The process is non-aqueous so generates water-free outputs, which is important for applications such as flame retardants for polymers. The process is currently at laboratory scale-up from 1g to 100g. Tests to date suggest that 95% of the P in vivianite can be extracted to phosphate esters. DAP fertiliser generated via SINFERT contains (ICP results) alkali and alkali earth metals below detection, heavy metals in the 0-3 ppm range.

Maria Cinta Cazador Ruiz, Fertiberia: agronomic value and market for vivianite as an iron and phosphorus fertiliser. Iron is an essential element for both plants and animals. One third of European soils are calcareous (elevated calcium carbonate) and soil alkalinity restricts the bioavailability of iron. Iron availability to plants is impacted by pH, oxidation, temperature, other metals, bicarbonates, organic matter. Plants have various mechanisms for mining iron from soil (e.g. Vélez-Bermúdez et al. 2023). Iron chelates are used to correct iron deficiency in some Mediterranean soils, providing rapidly plant available free Fe^{3+}, with a total market value of around 75 M€. Tests have shown that in calcareous soils vivianite can be progressively oxidised, releasing phosphorus and converting to iron forms which are plant available, such as (poorly crystallised lepidocrocite and ferrihydrite (Roldan et al. 2002). Iron sulphate on the other hand tends to oxidise to less plant available iron forms (geothite, crystalline lepidocrocite). 35-day pot trials (cucumber, calcareous soil) comparing vivianite to iron chelate (Fe-EDDHA) and to iron chloride showed good results with vivianite, with optimal dosing or around 1 kg vivianite / kg soil. An obstacle is that recovered iron phosphate compounds are today excluded from the EU Fertilising Products Regulation (CMC12).

Extraction of phosphorus from vivianite

Laboratory studies have looked at release of phosphorus from vivianite (iron(II) phosphate) and from ferric phosphate (iron(III) phosphate) using **alkali** (Prot et al 2019, see above), **acids** (see below) and **sulphide** (see below).

Release of phosphorus and iron from ViviMag® sewage-recovered vivianite slurry. Zhao et al. 2024 tested at lab scale (1g batch) extraction of phosphorus from sewage-recovered vivianite slurry, using five different acids (hydrochloric, sulphuric, phosphoric, oxalic, citric). Vivianite was from the ViviMag® pilot operated at Schönbeck wwtp, Germany (wwtp using iron salts for chemical P-removal, ViviMag operated on digested sewage sludge). The recovered vivianite (slurry) contained over 70% water. After drying, dried vivianite contained c. 13% organics and 21% water of crystallisation. Based on stoichiometric ratio Fe:P, around 65% of the phosphorus in the material was vivianite, but around 80% was bound in some form to iron or aluminium, with also around 7% in organic form, and some magnesium and calcium minerals. Hydrochloric and sulphuric acid both extracted 90% of the phosphorus from vivianite slurry at an H^+/P ratio of 2.5 for 15 minutes. The authors noted that a higher ratio of 3 is necessary to extract phosphorus from sewage sludge incineration ash. However, these acids also extract iron from the vivianite sludge, so a further processing step would then be necessary to separate, in the acid solution, in phosphorus from the iron. Oxalic acid showed to also extract the phosphorus to solution, whereas in this case the iron was precipitated as insoluble ferrous oxalate, so enabling one-step recovery of the phosphorus. Oxalic acid is high cost, but ferrous oxalate has a commercial market. However, in these tests organic and other impurities were precipitated in the ferrous oxalate. The authors suggest that purer ferrous oxalate could perhaps be achieved by first pre-leaching the vivianite slurry with phosphoric acid to remove organics and other impurities.

Hydrochloric acid P extraction from synthetic iron slag. Du et al. 2022 tested lab-scale hydrochloric acid (pH 3, 2.5g/l solid/acid) to extract phosphorus from synthetic iron slag (produced by heating iron(III) oxide Fe_2O_3 with calcium and silicon minerals at 1500°C. Hydrochloric acid pH3 extracted 25 – 50% of the phosphorus from the slag. Higher calcium content in the slag (basicity) resulted in higher P extraction. Calcium phosphate could be recovered from the acid extraction solution by increasing pH to 9.

Hydrochloric acid P extraction from sewage sludge incineration ash (SSIA). Hong et al. 2022 tested lab-scale hydrochloric acid to extract phosphorus from SSIA at high solid/acid ratios (>200g/l). With 2 mol HCl, over 90% of phosphorus was extracted after ten minutes, resulting in a 23 mgP/l phosphate solution. Significant leaching (>25%) of heavy and other metals also occurred in these conditions (As, Ca, Cd, Cu, Hg, K, Mg, Mn, Pb), resulting in significant consumption of acid. Release of iron was however low (<10% of iron content of ash). The authors note that with lower HCl concentrations, phosphorus and metals are initially released but then re-adsorb back onto the ash particles.

Sulfide P-release from iron phosphate and drinking water sludge. Mejia Likosova et al. 2013 tested lab-scale extraction of phosphorus from pure synthetic iron(III) phosphate (ferric phosphate) suspension at 0.1 molar (comparable to levels in sewage sludge). Sodium sulfide (Na_2S) was dissolved in water to 0.8 molar then stirred into the ferric suspension, with HCL dosing to adjust to pH 3 to pH 7, for 10 – 100 minutes. Acid dosing to pH 4 was necessary because otherwise the generated iron sulphide particles did not settle – the particles appeared to remain small or colloidal, but sedimented at pH4 because of agglomeration to form larger particles. Phosphorus extraction, to phosphate solution, of around 70% (at S/Fe molar ratio 1.5) to 90% (S/Fe = 2.5) was achieved. Further trials with drinking water treatment sludge with 10.6 g P/l and 0.7 g Fe/l, confirmed these results, with near zero P extraction at S/Fe molar ratio of 1 up to around 75% P extraction at S/FE molar ratio of 1.75.

Sulphide P-release from vivianite, sewage sludge and sewage sludge incineration ash. Wilfert et al. 2020 (see also ESPP SCOPE Newsletter n°138) lab-tested release of phosphorus from iron using Na_2S in different media: synthetic ferric iron(III) phosphate, synthetic iron(II) phosphate = vivianite, digested sewage sludge from sewage works using mainly biological (Leeuwarden, Amersfoort) or chemical P-removal using iron salts (Nieuwveer, Dokhaven) and sewage sludge incineration ash (SNB). The digested sludges showed P/Fe molar ratios of c. 1 to 2, and the ash a P/Fe molar ratio of 2 (P content of ash = c. 8%P). For P release tests, pH was maintained at around 7.5 by NaOH dosing. Results with synthetic phosphates showed 92% P-release from vivianite (after one hour) but only 55 – 75% P-release from iron(III) phosphate, with reduced effectiveness partly due to rebinding of P to iron(II) phosphate. From the sewage sludges with higher iron content, only up to 30% of P was released and insignificant P-release was shown with sewage sludge incineration ash. The authors note that sulphide is a readily available waste from oil refining. Challenges to resolve are the low sulfide P-release in sewage sludge and apparent deterioration in sludge dewaterability. These problems could maybe be avoided if phosphorus was extracted from the sewage sludge as vivianite and then sulphide P-release applied.

Sulphide P-release from ferric phosphate. Kato et al. 2006 also tested sulphide (NaHS) for phosphorus and iron solubilisation from several sewage sludges (with differing iron contents) and from pure ferric phosphate (iron(III) phosphate, $FePO_4$). This study showed up to 44% release of P from iron-dosed sludges but over 98% release of iron, and close to 100% release of P from pure ferric phosphate, at an S:Fe ratio of 1. The authors conclude that the lower P release from the sewage sludges is because H_2S is only releasing P from iron compounds, not from organic phosphorus forms or from other phosphate minerals.

"Acid leaching of vivianite separated from sewage sludge for recovering phosphorus and iron", Y. Zhao et al., Water Research 266 (2024) 122361, DOI.

"Efficient extraction of phosphate from dephosphorization slag by hydrochloric acid leaching", C-M. Du et al., J. Cleaner Production 332 (2022) 130087 DOI.

"Phosphorus Extraction from Sludge Incinerated Bottom Ash with Hydrochloric Acid at Low Liquid-Solid Ratio", H. Hong et al., Env. Engineering Science, vol. 39, n°2, 2022 DOI.

"Understanding colloidal FeSx formation from iron phosphate precipitation sludge for optimal phosphorus recovery", E. Mejia Likosova et al., J. Colloid and Interface Science 403 (2013) 16–21, DOI.

"Sulfide induced phosphate release from iron phosphates and its potential for phosphate recovery", P. Wilfert et al., Water Research 171 (2020) 115389, DOI.

"Extraction efficiency of phosphate from pre-coagulated sludge with NaHS", F. Kato et al., Water Science & Technology Vol 54 No 5 pp 119–129, DOI.

Sewage sludge biochar workshop

Norwegian Water sewage sludge biochar workshop

Over 100 people joined this workshop in Oslo, 12th February. ESPP underlined the need for the biochar industry to propose consensus proposals for sewage sludge biochar and data to show safety and phosphorus availability at these specified conditions.

Questions raised: Sewage sludge is currently excluded from use as an input material in CMC14 ("Pyrolysis and gasification materials") of the EU Fertilising Products Regulation 1009/2019. **ESPP considers that acceptance of sewage sludge as an input could be reconsidered by the European Commission if industry were to propose consensus agreed criteria (for processing conditions, contaminants, quality criteria** (e.g. H/C_{org}) – beyond the 'minimum' conditions of CMC14 which were designed for inputs such as clean biomass). Data must be provided showing that, under these specified conditions, contaminants of concern in sewage sludge (microplastics, pharmaceuticals, industrial organic chemicals in particular PFAS) are reduced to very low levels.

Data is also needed to show that contaminants (in particular PFAS) are not being transferred to offgas, converted to non-analysed metabolites (e.g. HCF_3 in offgas?), or 'stuck' into the biochar matrix such that they are not detected by analytical methods. If part of contaminants (PFAS, heavy metals) are transferred to offgas, then regulators may require gas cleaning systems for sewage sludge biochar plants. Depending on what gas cleaning is required, this could add considerable cost, and so question the model of small decentralised sludge pyrolysis. Companies present indicated that the EBI (European Biochar Industry Consortium) sludge biochar working group is preparing an update to its 2024 position paper (see ESPP eNews n°88) which aims to address these points.

Another question is **how might sewage biochar fit into the "phosphorus reuse and recycling rates" requirement of the revised Urban Wastewater Treatment Directive** 2024/3019? If the biochar does not have EU or national fertiliser 'product' status (not 'waste') or if the phosphorus is not plant available (under the FPR: 75% NAC soluble) then it risks not being considered recycling.

A route of interest is **addition of minerals to sewage sludge pyrolysis to improve plant availability of the resulting biochar,** and possibly also improve heavy metal or contaminant removal.

Chris Thornton, ESPP, summarised the current regulatory framework for biochars and hydrochars produced from sewage sludge or manure, at the EU level and in several EU Member States, and actions needed to open a market for sewage sludge biochar.

Arne Haarr, Norway Water (national water industry federation), organiser of this workshop, explained that Norway produces around 125 000 tDM/y of sewage sludge, of which somewhat over half is currently used on farmland. The new Norway regulations 2024 (see below) will however pose challenges to agricultural use. Norway Water considers that the heavy metal limits will be generally achievable, because of Norway's longstanding action to reduce inputs upstream. The organic contaminants may cause challenges in some places which have known point sources, in particular for PFAS: landfill leachates, airport runoff, fire service training sites (PFAS have been used for many years in firefighting foams). Norway Water considers that permits for discharge of such point sources into municipal sewers are often too lax.

At the same time, the revised Urban Waste Water Treatment Directive (UWWT brings significant challenges for Norway, with tighter requirements for nutrient removal in some sewage works, and nutrient removal becoming obligatory in coastal works where it is not today required (Western and Northern Norway). This adds to national pressure to reduce nitrogen discharges into coastal waters, to address ongoing eutrophication problems, in particular the Oslo Fjord. Tighter nutrient removal requirements will result in increased sewage sludge production.

The revised UWWT Directive "phosphorus reuse and recycling rates" pose a challenge of geographic nutrient misbalance in Norway. Western Norway has significant livestock manure and fish sludge, but much grassland, so low demand for sewage sludge, whereas Eastern Norway, with grain production on clay soil, has demand for sewage sludge. Overall however, livestock, aquaculture and sewage contain more phosphorus than Norway's agriculture needs, so there is a need to recover phosphorus in concentrated forms which can be stored and transported.

Anne Bøen, Norwegian Food Safety Authority, presented the recently revised national regulations for fertilising products and regulations for spreading and use of fertilising materials. Sewage sludge application will be limited to 250 kg-phosphorus/ha/ten-years, but also to crop phosphorus requirements. Under the In the previous regulations, sludge application was restricted only by heavy metal content which often limited application to 20 tonnes dry matter/ha/ten-years. This dry-matter restriction based on heavy metal content still apply, but usually the new phosphorus restriction (250 kg P/ha/ten-years) will be the most restrictive and will decide how much sludge can be applied. For many sludges this may result in approximately a 50 % reduction in application rate. For the crop P requirements, only plant-available phosphorus will be counted. Tighter requirements will apply in specific nutrient-sensitive catchments. Limit values for some organic contaminants are introduced: DEHP (di(2-ethylhexyl) phthalate, used in plastics), PFAS+PFOA and PCB7.

Under these revised regulations, sewage sludge biochar can be authorised with contaminant limits similar to those of the EU Fertilising Products Regulation CMC14, but remains under 'waste' status. Installations taking sewage sludge as an input material for biochar production will require a specific permit. Sewage sludge biochar will be subject to the same application limits and traceability obligations as for land application of sewage sludge.

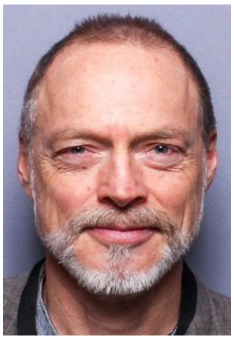
Trine Eggen, NIBIO/VKM presented the currently underway updated risk assessment of sewage sludge use in agriculture in Norway, carried out by the Norwegian Scientific Committee for food and environment (VKM), commissioned by Norway Food Safety Agency (NFSA), see ESPP eNews n°92. This risk assessment will include looking at which contaminants in sewage sludge are relevant to include in the risk assessment (a screening priority step), including in sewage sludge derived struvite, biochars, ash, and how different sludge treatment methods (including hygienisation by thermal hydrolysis), sludge application methods and rates can reduce contaminant risks and open possible new uses of sewage sludge. There is a need for more data concerning fate and transfer of contaminants to different materials, and possible uptake from fields into the food chain.

Erik Joner, NIBIO (Norwegian Institute of Bioeconomy Research), discussed sewage sludge use in agriculture. In his opinion, only a few % of phosphorus in sewage sludge is plant available where chemical P-removal has been used in sewage works, but evidence shows that sewage sludge has positive impacts on soil health (biota). Heavy metals have been largely reduced at source and are today at low levels in Norway's sewage sludge. Pathogens and anti-microbial resistance (AMR) are largely removed in the sewage works and sludge processing and organic contaminants present in sewage sludge are largely decomposed in soil or bound to soil particles and there is no evidence that microplastics from sewage sludge pose risks in the food system. PFAS however remain a problem. Sewage sludge biochar may be a solution, and can capture around one third of carbon in sewage sludge. Questions remain however on the interest of biochar for soil health (carbon is not biota-available), on nutrient value (N is lost in pyrolysis, P in sewage sludge biochar may not be crop available) and on contaminants in offgases (e.g. metabolites of PFAS).

Egil Hoen, Norgen Bondelag (Norway farmers' federation), sees biochar as a potential significant contributor to the farmers' federation commitment with Government to reduce carbon emissions. Biogas from agricultural residues (in particular manure,: target of 30% of manure to anaerobic digestion, generating biogas and reducing climate emissions in storage and spreading) will contribute 280 kt/y CO$_2$-equivalent carbon emissions reductions from soil carbon storage (cover crops, biochar, agronomic practices) an estimated 320 kt/y CO$_2$-equivalent by carbon storage in soil. The federation is cooperating closely with municipalities biowaste management installations. The federation has developed a digitalised climate calculated to enable Norwegian farmers to estimate their climate emissions and compare to other farming system choices. The commitment with Government also targets agriculture adaptation to climate change.

Michał Sposób, Rambøll, indicated that there are around 170 biochar plants operating in Europe. Denmark has around 10 pyrolysis plants, for which the main objective is a combination of biochar production together with syngas and biooil production to maximize revenue. Currently, the biggest pyrolysis projects in Denmark are connected to already existing biogas plants such as Stiesdal SkyClean at Agri Energy Vrå, which will produce 15 100 tons of biochar per year. A number of smaller plants (<1.0 MW) take sewage sludge as an input. In 2024, the Danish government developed a strategy for development of pyrolysis in Denmark aiming to clarify regulations and strengthen incentives and methodologies for climate impact evaluation. Currently, biochar use in Danish agriculture is authorised up to 7 t/10-years, with specified limits for some contaminants, and subject to waste regulation (environmental permit required for spreading). Mr Sposób considers that challenges to development are the regulatory framework, public acceptance and reduction of operating costs.

Kari Anne Sølvernes, Municipality of Oslo, presented field trials (autumn oats) using a liquid slurry mixture of biochar (produced from spruce wood) with concentrated digestate from anaerobic digestion of food waste (biomethane production). Tests showed that the biochar is effective in fixing the nutrients in the digestate, so reducing nitrogen loss and smell. Biochar particle size is a key criteria for farmer use (large particles block spreading equipment). Results to date show higher grain protein content when using the digestate compared to mineral fertiliser, with no clear effect of the biochar. The biochar does however appear to improve drought and water saturation resistance.



Ketil Stoknes, Lindum, a company owned by the municipality of Drammen, presented 5 years of R&D and testing of biochar applications (e.g. as an absorbent for contaminants, as reduction agent,) and of pyrolysis (e.g. for plastic removal from organic wastes, of sludge or digestate, co-pyrolysis with green waste). Biochars from sludge showed to be effective adsorbents of PFAS. Tests of sewage sludge pyrolysis show that upstream drying is needed and is energy-expensive, reducing economic feasibility. The sewage sludge biochar has low carbon content (>70% ash) when chemical P precipitation is used in the waste water treatment works. Pyrolysis of other dry wastes (e.g. waste wood, woody fraction of green waste) is more feasible for integration with AD in terms of energy balance. Better definitions for material recycling are needed.

Anna Fagerheim, Rå Biopark, presented this joint project in Northern Norway, currently at the planning stage. 41 municipalities will deliver biowaste for processing by anaerobic digestion (biogas) and pyrolysis. Challenges are that the region has little arable farming, so the nutrient-rich biochar will have to be transported significant distances. Also, regulatory status is unclear: biochar from biowaste is currently

excluded from the EU Fertilising Products Regulation (because food waste is an Animal By-Product), however it may be allowed if the anaerobic digestion upstream achieves the Animal By-Product Regulation End-Point (to be clarified). The situation under Norwegian fertiliser regulations is also unclear.

Hans Peter Arp, Norwegian Geotechnical Institute (NGI), presented detailed flow analysis for sewage sludge in Norway (different processes and destinations) and for contaminants in sludge. He summarised life cycle analysis comparisons between sewage sludge land application, pyrolysis and incineration. Data shows that pyrolysis eliminates 90-99% of PFAS in sewage sludge, with only long-chain PFAS tending to remain in the biochar.

Erlend Sørmo, Norwegian Geological Institute (NGI), discussed use of biochars to trap PFAS. Pyrolysis at 600°C or higher can eliminate over 95% of PFAS in contaminated organic wastes. Losses in offgas are variable depending on process, maybe up to 3 mg PFAS per tonne of biochar (see Sørmo et al. 2023 in ESPP eNews n°88). Offgas emissions can be reduced using gas scrubbers. Wood biochar can be 'activated' by high temperature steam treatment (>800°C) resulting in an activated carbon like material which can be used for contaminant removal from wastewaters, or for contaminant immobilisation in soils. Sewage sludge biochar without activation is nearly as effective for contaminant trapping as activated wood biochar. Such biochars are very effective at removal of long-chain PFAS, but less effective for short-chain PFAS. Short-chain PFAS can be taken up from soil be some plants, then pyrolysed to eliminate the PFAS and produce a biochar for application back to the soil.

Maria Estevez Rego, COWI Aquateam, presented results from the RenCARBio project. Sewage sludge biochar from a pilot pyrolysis unit (20 kg/h input), operating at 400°C or 600°C with residence time one hour (5x longer than commercial pyrolysis plants) showed low phosphorus plant availability as measured by P-$_{\text{AL}}$ (ammonium acetate lactate extractable P = 10 – 45 %) and Olsen-P (sodium bicarbonate extraction = 0.5 – 15 %). Phosphorus availability was higher with biological P-removal sewage sludge biochar than with sludge from sewage works using chemical P-removal (iron or aluminium salts). PFOS and most pharmaceuticals (not all) were removed (>95% reduction) at both 400°C and 600°C (one hour) but other PFAS were not measured. PFAS were not measured in offgas. A challenge is dust from dried sewage sludge which has to be pelletised to enable handling. Conclusions were that phosphorus availability in the biochar was higher at the lower temperature (400°C) but that PAH was generated in the process at this temperature. Pot trial testing of the biochars produced is underway.

Pål Jahre Nilsen, VOW Scanship, presented the company's pyrolysis technology, developed for treating sewage sludge and food waste on cruise ships, with 30 pyrolysis reactors sold, and capacity up to 50 000 p.e. sewage plus 4-6 tDS/d of other organic waste for large cruise ships. Larger on-land plants are under construction (20 000 t biochar/year). The current destination of this biochar will be as a carbon reducing agent for metallurgy. VOW also offers a steam heat pressure treatment, for e.g. sewage sludge or food waste (raw or digestate), resulting in a granulated material with 50% dry matter (compared to around 20-25% for filter press or centrifuge drying). Current costs of sludge disposal in Europe and US can reach 150 – 200 €/t-DM. Limits to sewage sludge spreading under the new Norwegian regulations (see above) will mean some sewage works must find alternatives. Sewage sludge biochar could be attractive if the carbon storage in soil is monetised or if concerns about persistent pollutants eliminate the land application route. VOW sees a future for sewage sludge pyrolysis, equipped with off gas cleaning and ammonia recovery. Added value would be generated for wastewater operators if part of the sludge biochar is used for quaternary organic contaminant removal from sewage works discharge, then recycled to the pyrolysis unit to eliminate these contaminants.

ESPP stay informed

SCOPE Newsletter: *https://www.phosphorusplatform.eu/SCOPEnewsletter*

eNews newsletter: *www.phosphorusplatform.eu/eNewshome*

If you do not already receive ESPP's SCOPE Newsletter and eNews: *https://www.phosphorusplatform.eu/subscribe*

LinkedIn: *https://www.linkedin.com/company/phosphorusplatform*

Slideshare presentations: *https://www.slideshare.net/phosphorusplatform*

YouTube: *https://www.youtube.com/user/phosphorusplatform*

ESPP members