SCOPE NEWSLETTER

NUMBER 94

The Phosphorus Challenge

Call for partners

Take part in defining the European Sustainable Phosphorus Platform

The Platform is calling for partners to establish the Platform and define over the coming year its objectives, actions and mode of operation

P-REX

Market and regulatory aspects of P-recycling

The first P-REX workshop organised by the FHNW in Basel Switzerland discussed progress on analysing markets for recycled phosphate products and current legal requirements.

Washington USA

Phosphorus Sustainability Research Coordination Network (RCN)

The first meeting of the Phosphorus Sustainability RCN brought together stakeholders and researchers to discuss "Coordinating phosphorus research to create a sustainable food system".

P-recovery as struvite

Pot trials

Testing struvite as a fertiliser for maize

Struvite recovered from pig manure was compared to fused superphosphate + urea as fertiliser for maize in pot trials. Estimated atmospheric N_2O emissions were compared, based on measured nitrogen leaching.

New struvite reactor design

Recovering and purifying struvite from piggery waste

Struvite was precipitated from swine wastewater using a new design aerated two-zone reactor, heavy metal contents tested, and a purification stage proposed.

Israel

Membrane filtered seawater for struvite precipitation

A nanofiltration membrane was used to produce a magnesium concentrate from seawater, then used to precipitate struvite from sewage works sludge dewatering liquor.

June 2013

Aerated beakers

Struvite precipitation parameters with CO2 stripping

Pure chemical solutions were used to test parameters for struvite precipitation and reactor deposition using aeration to remove CO_2 .

Poland

Struvite precipitation from fertiliser industry wastewater

A laboratory scale (1.3 litre volume) continuously operated stirred crystalliser was used to precipitate struvite from phosphate-rich fertiliser industry wastewater.

Bacterial struvite formation

Biomineralisation

Bacterial processes contribute to struvite formation

Several papers show that bacteria and biomass exopolysaccharides can cause struvite precipitation or changes between potassium struvite and ammonium struvite

Bacterial chemistry

Struvite precipitation enhanced by bacteria

Bacteria can cause struvite precipitation by breaking down urea to ammonium, but can also increase the size of precipitated struvite crystals. This would accentuate urinary stone problems in the body, but may provide a route to improve struvite recovery processes for Precycling in wastewaters.

Conferences and meetings

Global TraPs

IWA Nutrient Removal and Recovery

Fertilisers Europe decadmiation meeting

Agenda: dates 2013-2014

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The Phosphorus Challenge

Call for partners

Take part in defining the European Sustainable Phosphorus Platform

The European Sustainable Phosphorus Platform initiative came out of the European Sustainable Phosphorus Conference, March 2013, at which more than 300 companies, knowledge institutes, governments and NGOs alongside European Commissioner for the Environment, Janez Potočnik, called for European-wide action on sustainable phosphorus and recycling.



The Platform is calling for partners to establish the Platform and define over the coming year its objectives, actions and mode of operation.

The goal of the platform is to speed up the transition towards sustainable phosphorus management and recycling, green jobs and the circular economy, by enabling knowledge and experience sharing, pre-competitive networking and collaboration, innovation, value chains, technologies and markets.

The European Sustainable Phosphorus Platform is now looking for partners, to further develop the network and support an interim action plan for the coming year. The goal of the interim period is to progress towards the establishment of the platform as a permanent structure in 2014.

Role of partners

The platform's partners are companies, industry associations, knowledge institutes, governments, national nutrient platforms and NGOs that contribute to funding the platform's interim action plan.

Centre Européen d'Etudes des Polyphosphates

Benefits for partners are:

- **Recognition as a front-runner** in sustainable phosphorus management in Europe
- Visibility in the platform's communications as a partner on the platform website, newsletter and other publications
- **Presentation of your actions** on the platform's website
- Access to and participation in the platform's network of experts, R&D projects, phosphorus user and management industries, regulators and other stakeholders
- Up-to-date information on technologies, projects, implementation, bench-marking and regulatory developments
- During the Platform's crucial set-up phase, direct participation in defining the permanent objectives, actions and organisation of the platform

Initial actions

In order not to lose the momentum of first European Sustainable Phosphorus Conference, actions will already be engaged during the interim period to move forward the platform's objectives, including network building, communications, technical meetings and supporting national/local value chain structuring. Interim period actions are detailed in the Call for Partners on www.phopshorusplatform.org.

The European Sustainable Phosphorus Platform is open for all organizations that support the development of the platform's network and activities for sustainable phosphorus management. The partner contribution for the interim period is

- €10,000 for national governments
- €6,000 for companies and industry associations*
- €2,000 for knowledge institutes*, NGOs*, small • businesses and other types of organizations*
- * in some cases, contribution in kind may be possible

To become a Platform partner, contact: info@phosphorusplatform.org www.phosphorusplatform.org



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P-REX

Market and regulatory aspects of Precycling

P-REX (see Scope Newsletter n°88) is a 3-year European Union funded programme (Autumn 2012 – 2015) bringing together 11 industry partners and 4 scientific institutes, which will demonstrate full-scale P-recovery, examine the technical and economic conditions of different phosphorus recovery and recycling routes operating in Europe, quantify market potential, and define strategies for fostering P-recovery from municipal wastewater in different conditions. This first project workshop discussed progress on work assessing the market structure for recycled phosphorus recovery and recycling.

The workshop on **market and regulatory aspects of P-recycling**, organised by the work area leader FHNW, brought together a number of P-REX's R&D participants, representatives of P-recycling technology suppliers and of regulatory authorities, and included a visit to the pilot phosphorus recovery plant at ARA Rhein municipal wastewater treatment plant near Basel.

Pilot P-recovery plant

The participants of the P-Rex workshop had the possibility to visit a **pilot plant for phosphorus recovery from sewage sludge ash**. This is one of several pilot plants which are built and operated in the P-Rex project to support the technical assessment of process options for phosphorus recovery.



Site visit at the FHNW pilot sewage sludge ash extraction plant at ARA Rhein near Basel. Claudia Niewersch explains the function of the vacuum band filter and the dosage tanks shown

The pilot plant is being installed onsite a waste water treatment plant with mono-incineration (incineration of sewage sludge not mixed with other wastes). The pilot plant contains leaching and crystallisation reactors as well as a vacuum belt filter. The plant can be operated in batch mode with a capacity of 30 kg sewage sludge ash per batch.

Fertiliser market structure

P-REX R&D partners presented work underway categorising the phosphate fertiliser market structure in Switzerland, Czech Republic, Bulgaria, Germany and Spain. The project intends to identify and quantify the different companies importing, producing and supplying phosphate fertiliser in each of these countries, define phosphate flows and establish the market share of different supply chain routes (wholesale, retail) and of different uses (agriculture: crops, fruit and vegetables / hobby gardening, sports grounds and parks).

The project also intends to **identify the price for phosphate in fertiliser** at different levels of the supply chain in each of these countries (fertiliser producer, wholesaler, delivery at farm).

Ostara business model

Jim Hotchkies, Ostara, explained the Ostara / Crystal Green marketing model. Ostara installs the struvite recovery technology in sewage works and purchases the struvite at a guaranteed price from the water company. Ostara also has developed strong internal agronomic and commercial expertise, which allows them to market the recovered struvite, as Crystal Green, to target markets where **a premium price can be obtained because of the product's specific qualities:** dry, slow-release, closely defined and consistent mechanical and granulometry properties of the prills.

Ostara struvite from several US and Canada sewage works has been tested and consistently demonstrates **negligible levels of heavy metals, as well as pathogens and organic contaminants below detection limits**.

Integrating P-recycling into system trends in sewage treatment

Mr Hotchkies considers that development of phosphorus recovery and recycling in Europe will result from a **move towards biological phosphorus removal with accompanying anaerobic digestion**, which offers a long-term more economic and ecological solution than chemical P-removal (no inputs of iron or aluminium, reduced sludge disposal costs, energy recovery).



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In such configurations, phosphate release in the digester or in digester biosolids dewatering very often leads to struvite problems. **Struvite precipitation for P-recycling offers an effective solution**.

He also considers likely a trend towards sewage treatment where biosolids are input directly to anaerobic digestion (instead of the current activated sludge secondary biological treatment systems), in order to maximise energy recovery (avoid the loss of organic carbon and the energy consumption in mixing and aeration which occur in secondary biological treatment).

In this case, struvite recovery in the digester outflow liquor could enable **recycling of a high proportion of total sewage works phosphorus inflow** (c. 80%).

Drivers for phosphorus recovery

The market value of the struvite recovered by simple precipitation is not at present the main driver for phosphorus recovery and recycling (other drivers include resolving struvite precipitation problems, reducing sewage sludge production, sustainable development objectives). The chemical reagents necessary for struvite production (in particular magnesium chloride) cost more than the market value of phosphate fertiliser. In Ostara's case, it is the recovery of struvite in a size-controlled, slow release format that allows the company to realise a financial driver. Although the magnesium in struvite is a valuable micro-nutrient for plants, this is not a monetarised value.

Other P-recovery systems

It is noted that **other business models** are proposed different from Ostara's: for example:

- regionally available low-price magnesium sources may be used in place of magnesium chloride (magnesium industry by-products, desalination plant waste brine ...). These have been tested at lab-scale, but to date have not shown to produce a consistent, quality product sellable as a fertiliser;
- **produce a lower quality struvite** either integrated into sewage sludge which is valorised after composting, or sold to local markets where it is acceptable;
- produce a recovered phosphate product other than struvite, in a process not requiring magnesium input.
- recovery of phosphate from sewage sludge incineration ash

Regulatory context for recycled P products

P-REX is bringing together an analysis of some 50 different current national (Switzerland, Germany, Czech Republic, Spain) and EU **regulations impacting production and marketing of recycling phosphate products**: fertiliser regulations, organic farming standards, regulations/REACH, waste regulations, transport, production plant construction and operating rules ...

The project will develop an **advocacy summary**, **suggesting regulatory policies and tools to support P-recycling**.

Swiss fertiliser constraints

Markus Hardegger, Swiss Federal Agriculture Ministry, explained that Switzerland has more stringent rules for recycled phosphate fertilisers than for mineral fertilisers, for which the key relevant contaminant limit is cadmium < 50μ gCd/gP (for mineral fertilisers with >1%P). For recycled fertilisers, the limit for cadmium is lower at 1µgCd/g fertiliser (dry matter), but there are also limits for lead, zinc, copper, mercury, nickel ...

Because Switzerland incinerates 100% of sewage sludge, recycled phosphate would be produced from sewage sludge incineration ash. In this case, the lower limits for recycled present an additional hurdle at the moment, which might **make additional (costly) purification steps necessary**.

Regional Strategies

P-REX has defined as a working hypothesis **an objective of 80% phosphorus recycling from sewage.** The project will prepare strategies for achieving this for several different European regions (in Germany, Czech Republic, Spain, Switzerland).

These strategies will take into account sewage quantity and quality, water treatment technologies installed, biosolids uses and possible P-recycling technologies, regional agriculture and crop needs, legal conditions

A question to be addressed is how to account in such an objective **agricultural reuse of sewage biosolids** (after eg. digestion, composting ...), given that **in some cases it might be considered that spreading is not done to plant needs** (field disposal up to administrative limits) or that the phosphorus is not plant available (eg. if iron or aluminium are used for Premoval in sewage works), but such questions need to take into account crop use of phosphorus over several years.



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P-REX Next steps

Progress of the P-REX project on these different work areas will be presented at a **stakeholder workshop in Poděbrady, near Prague, 17th September 2013**. Analysis of market structure and current regulatory issues affecting phosphate recycling in selected countries will be presented. Stakeholders, including end-users, will be given a platform to interact in table discussions on demands and requirements for recycled phosphate products, innovation support and stakeholder cooperation.

Further information: www.p-rex.eu

P-REX stakeholder workshop, Poděbrady, 17th September 2013, programme: http://www.asio.cz/en/p-rex-workshop

Washington USA Phosphorus Sustainability Research Coordination Network

The first meeting of the Phosphorus Sustainability US Research Coordination Network - Science, Engineering and Education for Sustainability (RCN-SEES), funded by the US National Science Foundation, brought together stakeholders and researchers to discuss **"Coordinating phosphorus research to create a sustainable food system**". Summary video is available online (see below).

The US National Science Foundation RCN-SEES aim to "support investigators to share information and ideas, coordinate ongoing or planned research activities, foster synthesis and new collaborations, develop community standards, and in other ways advance science and education through communication and sharing of ideas".

The Phosphorus Sustainability RCN is a five year project with US National Science Foundation funding of US\$ 750,000.

The objective is to "envision, assess and communicate pathways to improve phosphorus efficiency and generate robust P recycling pathways".

A key principal is the close involvement of shareholders at all stages.

The Phosphorus Sustainability RCN is led by Arizona State University and University of Arizona, with a steering committee including the US Department of the Environment, the Stevens Institute and INPI (International Plant Nutrition Institute).

Three challenges

The Phosphorus Sustainability RCN theme, objectives, and initial topics build upon **broad agreement on key P sustainability challenges reached at the 3^{rd} Sustainable Phosphorus Summit 2012** (see SCOPE Newsletter n°85):

- Improving P efficiency in food production
- Developing robust pathways of P recycling
- Integrating efficiency and recycling to create a sustainable food system.

Launch meeting

The **first meeting of the phosphorus sustainability RCN** took place in Washington DC 12-14th May 2013, and brought together, either physically or by webinar, scientists, experts and stakeholders, farmers and growers, food processors, fertilizer producers, waste managers, water quality managers, regulators, legislators, and others.

James Elser, Arizona State University presented the aims of the RCN: to synthesise data, perspectives, and understanding of phosphorus management to envision solutions for phosphorus sustainability (the Phosphorus Challenge), informed by key stakeholders from relevant sectors of the global phosphorus system.

Arno Rosemarin, co-organizer of EcoSanRes and SuSanA and staff member of Stockholm Environment Institute, gave a keynote introduction presenting the need for sustainable phosphorus management, including geopolitical risks to food security of phosphate rock supply, contaminants in phosphate rock, environmental impacts of phosphorus dissemination, synergies with other drivers for sustainable sanitation and waste management and the specific challenges for developing countries.

Anita Street, Science and Technology Division, US Dept. of Energy, David Vaccari, Stevens Insitute of Technology, and Rob Mikkelsen, INPI (International Plant Nutrition Institute), presented specific aspects of phosphorus sustainability: economics and policy, urban systems and wastes, agriculture and ecosystems.

Tim Benton, UK Champion for Global Food Security, Chris Thornton, European Sustainable Phosphorus Platform, and Amit Pramanik, Water Environment Research Foundation summarised the relationship between phosphorus sustainability and long-term food security, developments in Europe and the potential for P-recovery and P-recycling from wastewaters.

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RCN workshop themes

Participants then split into workshops to discuss different themes, covering:

- agricultural P efficiency
- manure, sewage, and waste issues
- water quality and watershed management
- economics and policy
- communication.

Following from the meeting, the RCN intends to **further engage with stakeholders to gain feedback and input** and distil the project ideas into a list of research questions/topics that are feasible for the RCN to address.

The next meeting of the US Phosphorus Sustainability RCN is planned for Tempe, Arizona, $6^{th} - 10^{th}$ January 2014

US Research Coordination Network "Coordinating phosphorus research to create a sustainable food system": http://www.nsf.gov/awardsearch/showAward?AWD_ID=1230603 and http://sustainability.asu.edu/research/project.php?id=704

Jim Elser, Arizona State University, presents the Phosphorus Challenge and the RCN Network, on Arizona TV, TV 8 minutes: http://www.azpbs.org/arizonahorizon/detail.php?id=2161#Phospho rus

RCN launch meeting summary video: http://youtu.be/n3EL0odjHCo

P-recovery as struvite

Pot trials

Testing struvite as a fertiliser for maize

Struvite recovered from piggery waste in a laboratory scale reactor (Rahman et al. 2011, see SCOPE Newsletter n° 82) was used for pot trials of maize (Zea Mays, v. Barnali), with the pots placed outside in the field in South Korea. For comparison, fused superphosphate plus urea (FSP-U) were used as mineral fertilisers, with a control (no fertiliser) and two struvite dose levels and two corresponding mineral fertiliser doses (30 and 40 kgP/ha). In both the struvite and FSP-U pots, therefore, nitrogen was probably inadequate, because urea was dosed to the same nitrogen level as struvite, which has a 1:1 nitrogen phosphate ratio, significantly lower than crop needs ratio. Green biomass yield, leaf number, leaf area, plant height, stem circumference and plant nutrient composition were measured.

A total of 15 pots, each 28 cm deep and top diameter 22 cm, were used, with 1 maize plant in each pot. 8 kg of agricultural soil was placed in each pot, and the fertiliser mixed into the top 5 cm. Potassium fertiliser was added to all pots. Watering was by the natural rainfall.

Results for height and stem conference showed significant differences between the control plants and the fertilised plants, but no consistent differences between the struvite and the fused superphosphate + urea (FSP-U) fertilised plants. The control plants, for example, only reached < 2/3 the height of the fertilised plants after 56 days, and had not begun to make cobs by this time.

Performance of struvite

For the other factors measured (leaf number, leaf area, dry biomass yield) the fertilised plants performed significantly better than the controls, but also the **struvite fertilised plants performed significantly better than the FSP-U fertilised plants** for comparable doses.

Crude protein yield in the struvite plants was also significantly better than for the FSP-U plants.

The authors suggest that these positive comparison results may be due to **nitrogen insufficiency** in the FSP-U pots, as soluble urea was lost by leaching over time whereas struvite provides slow nitrogen release.

N₂O emissions

Atmospheric N_2O emissions (greenhouse gas), were estimated by measuring nitrogen leaching (measured in the water leaching out of the pots) and nitrogen uptake by the plants. This concluded that N_2O emissions were around 3x higher from the SFP-U fertilised pots than from the struvite fertilised pots. The authors suggest that this is related to the general risk of N_2O emissions from urea fertilisation, and the reduced emissions where slow release fertilisers are used.

The authors conclude that **struvite provides a performance alternative to traditional mineral fertilisers**, with potentially lower environmental impacts.

"Eco-friendly Production of Maize Using Struvite Recovered from Swine Wastewater as a Sustainable Fertilizer Source", Asian-Aust. J. Anim. Sci., vol. 24, n° 12, pages 1699 – 1705, 2011 www.ajas.info and http://dx.doi.org/10.5713/ajas.2011.11107

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New struvite reactor design

Recovering and purifying struvite from piggery waste

Real wastewater collected from a local pig farm (see Rahman et al. 2011, see SCOPE Newsletter n° 82) and a new design 18 litre precipitation reactor was used to recover struvite. Different operating parameters were tested in nine runs of duration 30 days each (with daily sampling). High copper and zinc in the piggery wastewater (from animal feed supplements) did not result in high levels in the precipitated struvite. A process of acid dissolution followed by alkali re-precipitation was tested to purify the recovered struvite.

The **piggery wastewater was very variable in composition**, with a pH of 7.8 - 8.9, with variable, soluble phosphate concentration of 25 - 608 mgP-PO₄/l, ammonia 1500 - 5300 mgN-NH₄/l. The wastewater was sieved to 0.5 mm resulting in suspended solids content of 0.3 - 38 gSS/l.

New reactor design

The struvite reactor used was a rectangular plexiglass column 21x8 cm, and 105 cm high. Within this column, a reaction zone compartment of 8x8 cm, 70 cm high, was separated, situated at the top of the reactor, with a perforated base (5mm holes) to allow precipitant solids to pass out downwards into the rest of the reactor, which acted as a settling zone. The reaction zone also contained five horizontal baffles (spigots) to facilitate decanting (see diagram in published paper).

Influent wastewater and magnesium chloride reagent were pumped into the top of the reaction compartment of the reactor, liquor flow was then down and through the base of the reaction compartment into the main settling zone of the reactor, with effluent taken out near the top of this main zone.

Hyrdaulic retention time was fixed at 4 hours, with the objective of enabling struvite particle size growth. Parameters tested were aeration rate (0 - 0.92 litres of air/litre of reaction zone volume/ minute) and magnesium chloride dosing (0 - 1.2:1 Mg:orthophosphate molar ratio).

Results showed that struvite precipitation increased with increasing aeration rate but even without aeration over 80% soluble phosphate precipitation was achieved (at Mg:P ratio = 1:1). Phosphate precipitation was however very low (<20%) without magnesium addition, increasing to over 90% at Mg:P ratio 1 or 1.2. **Potassium was also significantly removed** (from 2700 to 1400 ppm), as was calcium (180 to 72 ppm). These different removed elements were presumably being transferred to the struvite. Calcium and potassium would not be an issue for use of the struvite as a fertiliser, and in these relatively small quantities would probably not significantly modify the struvite's fertiliser properties.

Heavy metal transfer to struvite

Copper and zinc concentrations in the piggery wastewater were 6.4 and 8.8 ppm respectively, presumably because of dosing of these elements as micronutrients in animal feed supplements (used to promote the pig immune system and growth performance). These concentrations were reduced in the struvite precipitation reactor, to 4.0 and 5.0 ppm.

However, the levels of these metals (Cu, Zn) in the precipitated struvite were only 18 and 96 mg/kg (copper, zinc), that is round 4x and 10x lower than German fertiliser standards (Dungenmittelverordnung, 2003).

A struvite purification process was tested, by dissolving the collected struvite slurry from the precipitation reactor, centrifuging, then treating at pH with hydrochloric acid, before dosing to pH 10 with sodium hydroxide. This did not significantly reduce the metal concentrations (copper 16, zinc 81 mg/kg).

"Recovery of nitrogen and phosphorus by struvite crystallization from swine wastewater", Desalination, 277, pages 364–369, 2011 www.elsevier.com/locate/desal

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Israel

Membrane filtered seawater for struvite precipitation

Magnesium purchase is probably the highest operating cost in struvite precipitation processes. A relatively cheap magnesium source was produced by nanofiltrating seawater, using a commercial NF membrane. This concentrate was tested for struvite precipitation from real municipal sewage works sludge dewatering liquor, with added ammonium and phosphate to stabilise concentrations, in a laboratory fluidised bed reactor (12.6 litre total volume).



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The **magnesium concentrate** was generated using a laboratory scale, crossflow membrane cell, with 2.6 m^2 effective membrane area (commercial NF membrane, c. 0.45 nm pore radius), operating on Mediterranean seawater.

Magnesium and other seawater ions

The membrane cell concentrated magnesium and sulphate ions from seawater, but also to some extent calcium, sodium, choride and potassium ions. For use for struvite precipitation, it is desirable to recover a concentrate with high magnesium concentration (in order to limit the dilution resulting from adding the concentrate to the sludge liquor) and with relatively low concentrations of other ions (susceptible to interfere with struvite precipitation, or generate impurities in the precipitated struvite).

The ratio between magnesium and other ions tended to improve, as well as higher magnesium and other ion concentrations, when the membrane cell was operated at **higher ion recovery rates** (achieved with higher pressures). A pressure of 17 bars (90% recovery rate) enabled concentration of magnesium by a factor of x6 compared to the feed seawater, reaching 8,000 mgMg/l, which resulted in a dilution of only c. 3% of the sludge liquor in ensuing struvite precipitation experiments. Sodium, chloride and potassium ions were only a little concentrated in the membrane cell at this pressure, calcium concentrated around 3x and sulphate concentrated similar to magnesium (around 6x).

Operation of the membrane cell at this high recovery rate, however, necessitated the **addition of an anti**scaling agent, to prevent mineral precipitation blocking the membrane (Genesys CAS at 30 mg/l).

Struvite precipitation

Sludge centrifuge supernatant from the Hadera municipal wastewater treatment plant, Northern Israel, was used for struvite precipitation testing in a laboratory scale fluidised bed reactor consisting of a 1.72m high, 3.8 litre volume fluidised bed compartment and a 0.53m high, 8.8 litre volume settling tank, with recycling pump (20 minutes hydraulic residence time) and run-times of 18-36 hours to enable stable conditions to be reached. Ammonium chloride and potassium phosphate were dosed to give required P and NH₄ concentrations in the sludge liquor, and sodium hydroxide was dosed to maintain pH around 8. Seawater concentrate was dosed to ensure a Mg:P ratio of 1:1 (giving a ratio of c. 1.15:1 considering the magnesium present in the sludge liquor).

Phosphate and ammonium concentrations were adjusted to test two scenarios considered typical of sewage works operating with and without biological nutrient removal: 310 or 100 mgP/l and 450 or 600 mgN/l.

Struvite precipitation results were very close to PHREEQC model predictions, with >90% phosphate precipitation at pH around 7.6 for the higher sludge liquor concentration scenario and pH around 8.1 for the lower concentration scenario.

XRD analysis showed only one crystalline form present (struvite). It was noted that amorphous minerals (calcium or magnesium phosphates) would not show up with XRD but chemical analysis showed these impurities to account for not more than 5% of the solid. The struvite crystals formed were small (mostly $60 - 250 \mu$ m). This may be a consequence of the antiscaling agent present, or just because the run times did not allow time for crystal growth or agglomeration.

Cost savings

Full scale cost of producing the magnesium concentrate was estimated, by comparison with reverse osmosis membrane plants operating desalination in Israel, at **0.25 US\$/kg magnesium**.

This is over 4 times cheaper than purchase of magnesium chemicals.

The authors consider this would **decrease total struvite precipitation costs by at least 25%** for sewage works located close to seawater. They consider that the membrane magnesium concentration is engineering feasible for full-scale operation.

Questions which need to be addressed are the possible implications of additional sodium (from the seawater concentrate) in the treated sludge liquor if this is used for irrigation, consequences of the anti-scaling agent on struvite precipitant size and form, and identification of membranes with more selective magnesium separation (compared to other ions).

"Struvite recovery from municipal-wastewater sludge centrifuge supernatant using seawater NF concentrate as a cheap Mg(II) source", 2013, Separation and Purification Technology, 108, 103-110, refs. SEPPUR 11046: http://dx.doi.org/10.1016/j.seppur.2013.02.002

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Aerated beakers

Struvite precipitation parameters with CO₂ stripping

Aeration by bubbling air at a fixed rate of 40Lmin⁻¹ through 600 ml of liquid (1 litre beakers) was used to precipitate struvite from supersaturated solutions of calcium carbonate (50 mgCaCO₃/l) with magnesium chloride. ammonium phosphate, potassium phosphate solutions, initially adjusted to pH6.5. Increasing and NH₄:P ratios different Mg:P had consequences on precipitation rates (P-removal), minerals formed and on deposit formation on reactor walls (as opposed to production of crystal particles suspended in the liquid).

A range of magnesium concentrations were tested at P-PO₄ concentrations of 130 mg/l, a range of phosphate concentrations at magnesium concentration of 92 mg/l, and ammonia concentrations over the range 18 - 227 mgNH₄/l.

Magnesium, phosphate and ammonium concentrations

At **Mg:P ratios** > **5**, minerals other than struvite were detected in the precipitate: newberyite MgHPO₄· $3H_2O$ and cattiite Mg₃(PO₄)₂· $22H_2O$.

As expected, **higher initial magnesium concentrations resulted in higher phosphorus removal rates** (precipitation) with phosphate removal of 25-92% for Mg:P ratios of 1-5, respectively. Magnesium concentration also affected precipitated crystal size.

For magnesium concentration 92 mg/l, with higher phosphate concentrations, phosphate removal rates decreased to 33 - 0 %. In particular, with Mg:P ratios of 1/3 - 1/5, no phosphate precipitation was observed.

Also as expected, **phosphate removal rates increased with higher ammonium concentrations**, eg. from 21 - 75% over the range NH_4 :P ratio from ¹/₄ to 3.

Reactor fouling

Reactor fouling levels showed considerable variation depending on magnesium and on ammonium concentrations, that is **precipitation of phosphates onto the reactor walls** rather than as suspended crystals in the liquor.

94% of precipitation was onto the reactor walls at Mg:P ratio of 1, compared to only 2% at Mg:P ratio of 5. Ammonium on the other hand appeared to have the contrary effect with 95 - 82% of phosphate

precipitating as suspended crystals at NH₄: P ratios of ¹/₄ to 3.

Previous study

A previous study similarly looked at struvite precipitation from pure chemical solutions (MgCl₂, NH₄H₂PO₄, CaCO₃) at initial pH 6 by air bubbling to remove carbon dioxide in thermostatically controlled beakers. In this case, no precipitation was detected below pH approx. 8.

Struvite precipitation of nearly 80% soluble phosphorus removal was achieved with an air flow of 25 l/minute, but was lower for higher air flows of 30 – 40 l/min. Experiments using mechanical mixing showed no struvite precipitation, indicating that the precipitation was indeed the result of the aeration (carbon dioxide degassing).

"Phosphate recovery through struvite precipitation by CO₂ removal: Effect of magnesium; phosphate and ammonium concentrations", J. Hazardous Materials (Elsevier), vol. 186, issue n° 1, Feb. 2011, pages 602-613 http://www.sciencedirect.com/science/journal/03043894

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"Struvite precipitation by the dissolved CO2 degasification technique: Impact of the airflow rate and pH", H. Saidou, A. Korchef, S. Ben Moussa, M. Ben Amor, Chemosphere, n°74, pages 338–343, 2009 www.elsevier.com/locate/chemosphere

Poland

Struvite precipitation from fertiliser industry wastewater

Magnesium chloride, ammonium chloride and sodium hydroxide were used as reagents to precipitate struvite from fertiliser industry wastewater containing 1.5 g P-PO₄/l phosphorus, 0.3 g/l magnesium, 0.4 g/l calcium, 0.7 g/l sulphate and lower trace levels of aluminium, iron, and other elements, at pH 3.8.

leachate The wastewater was from a phosphogypsum heap at Z. Ch. POLICE SA, Poland. A lab-scale (1.3 litre total volume, of which 0.6 litre working volume) continuousstirred reactor was used. Different mode, operating conditions including mean residence time crystal suspension, of pH and magnesium:phosphate ratio were tested.



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The reactor used was computer controlled for stirrer speed, temperature, feed injection rates and pH (sodium hydroxide dosing). Upstream of the reactor, the **wastewater was mixed with the magnesium and ammonium reagents**, and this feed was then progressively injected into the stirred central zone of the reactor (stirring pushing water downwards) and the sodium hydroxide (pH adjustment) in the outer zone (upward moving).

Kinetic parameters of the continuous reaction crystallization process were modelled using the population density distribution of the product crystals and a simplified SIG kinetic model derived for a continuous MSMPR reactor (Randolph & Larson 1988).

Crystal size

The struvite precipitated showed to be mainly long rod-like crystals, with some smaller particles (see photos). **Higher pH (10) resulted in struvite crystals which were both smaller on average, and also more variable in size** (than at pH 8.5). Longer mean residence time of suspension in a crystallizer also resulted in larger overall crystal size.

Higher magnesium:phosphate ratios (excess magnesium) resulted in larger struvite crystals, and slightly less variation in crystal size. The higher magnesium doses also resulted, however, in coprecipitation of more impurities from the wastewater.

For example, the precipitated struvite contained up to 2000 ppm iron, 50 ppm aluminium, 20 ppm zinc, 0.1 ppm copper.

The largest size crystals were produced with a magnesium:phosphate ratio of 1.2:1, pH 9 and a residence time of 1 hour. Under these conditions, 98% of soluble phosphate was precipitated.

The authors conclude that the process is very effective for removing soluble phosphate from the phosphogypsum leachate wastewater, enabling recovery as struvite (magnesium ammonium phosphate hexahydrate). However, the impurities present in the wastewater are co-precipitated with the struvite which could pose potential issues for its use as a fertiliser.

Influence of other ions on struvite precipitation

In previous papers (2011a, 2011b, 2012), the authors used the same continuous reaction crystallization process installation to **test the effects of various other** ions on the precipitation of struvite: Al^{3+} , Ca^{2+} , Cu^{2+} , Fe^{2+} , Fe^{3+} , K^+ , Zn^{2+} , F^- , NO_3^- , SO_4^{2-} .

All these ions had effects on the form of the struvite crystals precipitated. Calcium, aluminium and the two different iron ions resulted in formation of hydroxides or phosphates of these ions, as generally very small particles, in some cases agglomerating onto the struvite crystals, making settling and separation more difficult.

The effects of calcium ions were specifically investigated, showing that **increasing calcium concentrations caused decreasing mean struvite crystal size**, with the presence of co-precipitated calcium phosphates. The calcium also tended to result in struvite formation as tubular crystals, with lengthwise cracks and irregular surfaces.

The effects of iron (II) ions were also specifically investigated, again showing that **iron decreased struvite crystal size**. Operating at pH 9, nearly all the iron present was co-precipitated as iron hydroxide, and so removed from solution.

The presence of **nitrate ions also resulted in smaller struvite crystal size**.

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Bacterial struvite formation

Biomineralisation

Bacterial processes contribute to struvite formation

Bacterial mineralisation has contributed to the formation of minerals since the beginnings of life on earth, yet our understanding of such reactions and how to induce or control them is very limited. Bacterial formation of struvite or other phosphates not only plays an important role in sewage treatment (phosphorus removal, ammonium removal) but could also potentially provide a costeffective route for phosphorus recovery for recycling.

Lin, Bassin & Loosdrecht (2012) present tests showing that sewage sludge can adsorb ammonium by struvite mineral changes, and that alginate like exopolysaccharides (EPS) extracted from the sludge biomass can induce struvite formation.

Potassium struvite and ammonium struvite

Sludge granules from a lab-scale EBPR sewage treatment reactor (enhanced biological phosphorus removal, with simultaneous nitrification / denitrification) could adsorb 1.5 mgNH₄N/g dry biosolids. This ammonium uptake was accompanied by potassium ion release.

Also K-struvite (potassium struvite = magnesium potassium phosphate) was shown to be present in the sludge biomass by X-Ray Diffraction (visible as pyramidal crystals). The XRD was carried out after heating at 500°C to remove organics. Synthetic K-struvite adsorbed around 4 mgNH₄'N/g. Element analysis of ash confirmed that struvite-type minerals made up the majority of the inorganic content of the sludge granules.

Exopolysaccharide induced struvite formation

The authors deduce that the **ammonium adsorption** by the sludge biomass is probably due to K-struvite being modified to struvite (magnesium ammonium phosphate) in a biological ion-exchange process.

Alginate-like exopolysaccharides (EPS) were identified in the sludge biomass, and thought to be providing the support for this ion-exchange. This follows previous work by the same authors (Bassin et al 2011, Lin et al 2010) indicating that acidic extracellular polymers present in the sludge could be potential ion exchangers and isolating EPS. Around 125 mgEPS/gVSS was extracted from the sludge biosolids and tested, showing ammonium adsorption and potassium release.

Biomineralisation review : mechanisms of biomineral struvite formation

Gonzalez-Munoz et al. (2012) review knowledge and recent studies of bacterial biomineralisation, focusing on the genus Myxococcus. This genus can induce precipitation of phosphates, carbonates, sulphates, chlorides, oxalates and silicates.

Research has shown that bacteria can precipitate carbonates, calcites, iron oxides, hydroxides and sulphides, silicates, sulphates, calcium nitrate, chlorides (e.g. NaCl halite) and a range of magnesium and phosphate minerals (in particular calcium phosphate and struvite). Current knowledge is incomplete. For example, it is not certain whether bacterial mineral precipitation is genetically controlled, or simply occurs as a result of metabolic activity.

The **mechanisms of bacterial mineralisation include** generation of local ion concentrations (either inside the cell or around it) and nucleation (where the cell produces materials, often in the cell wall, which provide a template for precipitation of the mineral, often with specific local electrostatic conditions to facilitate precipitation).

Myxobacteria

Myxococcus are part of the myxobacteria family, gram-negative, heterophic bacteria with long rodlike cells 3-12 μ m, common in nature, for example in topsoils. They produce a range of enzymes and antibiotics with useful applications for man. Several species of the genus Myxococcus have shown biomineralisation capacities, producing carbonates, sulphates, calcium minerals, and various phosphates including struvite, hydroxylapatite, newberyite and schertelite.

Struvite precipitation has been shown to result from a combination of changes in pH and ion concentrations resulting from the bacterial metabolism, and physical presence of the bacteria (dead or living) providing heterogeneous nucleation media.

Bioengineering struvite crystal forms

Chen et al. (2010) demonstrate that a combination of bacteria (Proteus mirabilis) and a seed crystal of dittmarite [the monohydrate form of magnesium ammonium phosphate $(NH_4)Mg(PO_4)\bullet(H_2O)$], in urea solutions, can enable very specific and different forms of struvite crystals to be generated.



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Struvite [the hexahydrate form of magnesium ammonium phosphate $(NH_4)Mg(PO_4)\bullet 6(H_2O)$] usually forms in chemical solutions as rod-like or prism-like crystals. These customary forms are also produced in the presence of the bacteria if **different seed crystals are used** (magnesium phosphate), if no seed crystals are used, or if a solution other than urea is used (ammonium carbonate).

With the monohydrate seed crystal, the bacteria and urea solution, complex struvite superstructures are formed rather than rod or prism shaped crystals. With longer reaction time / ageing (up to 15 days), evolve from porous, nearly spherical these superstructures to either prismatic particles (size hundreds of µm) or porous tetragonal bipyramids (size tens of µm). The authors also demonstrate that the struvite superstructures can be used to support silver nanoparticles, concluding that this demonstrates the potential of biomineralisation processes for expanding the scope of crystal engineering and synthesising new and specific functional materials.

Carbonate and struvite biomineralisation

Delgado et al. (2008) showed that bacteria found in Spanish saline soil can precipitate both carbonate and phosphate minerals, including struvite, from solid culture media containing calcium and magnesium ions.

Bacteria were extracted from marine saltmarsh soil taken from near Alicante, Spain, and then cultured. The minerals precipitated with different concentrations of calcium and magnesium ions in the media were investigated. Calcite, magnesium-containing calcite (mangesian calcite) and struvite were shown to be precipitated, with the **development of struvite-precipitating bacterial colonies** or calcite-precipitating colonies being apparently influenced by the relative ion ratio Mg:Ca rather than absolute magnesium concentration.

The authors also found **struvite precipitation only occurred when the culture medium was enriched with organic materials**. They conclude that the fact that struvite is rarely found naturally in soils is not because of an absence of struvite-precipitating bacteria, but rather because struvite is precipitated only slowly (unless there is a continuous, rich supply of organic matter) and so redissolves as rapidly as it is formed.

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Bacterial chemistry

Struvite precipitation enhanced by bacteria

Work looking at links between bacteria and struvite kidney stone precipitation provides interesting information about the interactions between certain bacteria and struvite formation. The authors studied struvite precipitation in pure solutions of urea and of ammonium carbonate, plus magnesium and phosphate ions, with or without addition of the bacteria Proteus mirabilis.

Struvite is the main component of urinary infection calculi and **struvite stones** account for 10-15% of urinary tract stones. One contributing factor is thought to be bacteria which cause the breakdown of urea, to release free ammonium ions, which can then precipitate as struvite.

The authors carried out batch precipitation tests in 200 ml conical bottles using disodium hydrogen phosphate solution, magnesium sulphate solution and either urea



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or ammonium carbonate solution. 0.8g wet weight of the bacteria Proteus mirabilis was added or not in each case. pH was adjusted to 7.5 and the bottles were stirred at 5000 rpm, then incubated without stirring for 36 hours at 30°C. Precipitates were then separated by centrifugation, washed in distilled water, dried, then analysed by SEM and XRD.

Results show that, both with urea and with ammonium carbonate, the **struvite particles formed are significantly larger in the presence of bacteria** (see SEM images in the published paper).

Also, in the presence of bacteria, the struvite particles seem to be more amorphous rather than clearly shaped crystals, suggesting a process of aggregation as well as crystal growth.

Results for quantities of struvite produced or for % phosphorus or magnesium removal from solution are not given (the latter would in any case be difficult to assess because of possible uptake or release from the bacteria).

Urease and cell wall properties

The medium in the bacteria-containing bottles was analysed using SDS-PAGE and UV-vis spectrum, showing the presence of proteins. These, it is hypothesised, could partly be urease.

It is also hypothesised that the **bacterial cells may** enhance struvite precipitation by adsorption of positively charged Mg^{2+} ions onto specific sites on the cell walls, thus locally increasing the concentration of magnesium and so the struvite precipitation rate. The bacteria may thus accentuate the nucleation, growth and aggregation of struvite crystals.

This **increase in struvite particle size** would be a factor for increased risk of medical problems with urinary stones in the human body, but could provide a route for using biological processes to enhance struvite recovery from wastewaters.

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Conferences and meetings

Beijing, 18-20 June 2013 Global TraPs world conference



www.globaltraps.ch

The Transdisciplinary Processes Global for Sustainable Phosphorus Management (Global project TraPs) is studying phosphorus use, management and sustainability from a supply chain involving perspective academia, industry, governments, NGOs and other concerned parties.

The conference theme is "Learning from Case Studies – Exploring Policy Options." with the objective of assessing specific areas for policy intervention to ensure sustainable phosphorus use.

The conference will be co-hosted by China Agricultural University, Ministry of Agriculture, Chinese Ministry of Education, Phosphorus Fertilizer Industry of China, National Science Foundation of China, IFDC, Fraunhofer Institute and other Institutes and will coincide with the **5th International UNEP Global Platform Nutrient Management Symposium**

Vancouver, 28-31 July 2013 International Nutrient Removal and Recovery Conference

http://www.wef.org/nutrients/

Combined WEF and IWA-NRR conference: Nutrient removal and recovery 2013 – trends in resource recovery and use.

- nutrient recovery processes
- nutrient recovery from source-separated urine and agricultural effluents
- nutrient management of biosolids

Conference organised by WEF (Water Environment Federation), IWA (International Water Association), WERF (Water Environment Research Foundation) and British Columbia Water & Waste Association.



Water Environment Federation the water quality people*



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Brussels, 3 October 2013

Decadmiation workshop

Organised by Fertilizers Europe



With the new European Sustainable Phosphorus Platform being launched, the new Fertilisers Regulation being drafted by the European Commission and the Green Paper on Phosphorus becoming available in the near future, phosphorus has become the centre of attention and feeds many discussions. Together with the increased focus on phosphorus (P), came the attention for cadmium (Cd). Cadmium is a heavy metal that naturally occurs in phosphate rocks, albeit at different concentrations depending on the origin of the phosphate rock. Several cadmium removal (decadmiation) technologies exist but none is used at industrial scale for fertilizer production.

The objectives of this decadmiation workshop are to:

- bring together technology providers, companies active in phosphate fertilizer production or the agricultural sector, knowledge institutes and regulators (both at European as national level)
- provide information on the state of the art in decadmiation technologies and give the platform to P fertilizer producers to demonstrate their developments in this area.
- give an update of the current and future Cd balance in European agricultural soils

To participate or for more information, contact Laetitia Six Laetitia@fertilizerseurope.com +32 2 663 31 49.

Agenda

Dates 2013 - 2014

- 18-19 June, Strasbourg: Efficient Sludge Treatment (VDI = Ass. German Engineers) http://www.vdi.eu/media-services/article/efficientsewage-sludge-treatment/
- 18-20 June, Beijing: Global Traps www.globaltraps.ch

Centre Européen d'Etudes des Polyphosphates

 25-28 June, Santiago de Compostela, Spain: 13° world congress on anaerobic digestion – Recovering (bio) resources for the world www.adl3.org

- 28 31 July, Vancouver: IWA Nutrient Removal and Recovery http://www.wef.org/nutrients/
- 9-13 September, Uppsala, Sweden: 7th International Phosphorus Workshop http://www-conference.slu.se/ipw7
- 17th September, Poděbrady near Prague
 P-REX stakeholder workshop
 Markets and leglistation www.P-REX.eu
 and http://www.asio.cz/en/p-rex-workshop
- 3rd October, Brussels,
 Fertilisers Europe decadmiation meeting www.fertilizerseurope.com
- 27-31 October, Berlin
 Global Soil Week "Losing Ground?"
 www.globalsoilweek.org
- 3-8 November, Tampa, Florida ASA/CSSA/SSSA + Canada SA agronomy meetings + SERA17: Water, food, energy and innovation for a sustainable world www.acsmeetings.org and http://www.sera17.ext.vt.edu/
- ✤ 6-7 November, Braunschweig, Germany Re-Water www.re-water-braunschweig.de
- 5-6 December 2013, Bruges: ManuResource 2013 (manure management and valorisation) http://www.manuresource2013.org/registration
- 7-10 January 2014, Phoenix Arizona 2nd Sustainable Phosphorus US Research Coordination Network meeting. http://sustainability.asu.edu/research/project.php?id=704
- ✤ March 2014: Phosphates 2014 (CRU)
- ✤ May 2014, Morocco: SYMPHOS
- 25 Aug 3 September (to be defined), Montpellier, France
 4th world Sustainable Phosphorus Summit http://sustainablepsummit.net/

Nutrient Platforms

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