SCOPE NEWSLETTER

NUMBER 90

US agronomy and soil meeting

ASA – CSSA – SSSA - Cincinnati

Opportunities and limitations of P-reuse from manures

A symposium on phosphorus recycling, within the USA's biggest agronomy and agricultural research conference, showed that strong interest in P-recovery from manures, particularly in response to farmland application limits. Technologies for manure P-recovery are operational.

Public policy

Wastewater treatment

European Union renews pressure for sewage treatment conformity

The European Commission is pursuing actions against members states for inadequate treatment of sewage. Sweden is considering going further on nitrogen removal.

P food chain losses

EU retailers pledge action on food wastes

European retailers have promised actions on consumer food waste. This follows an EU Parliament motion of 19th January 2012 calling for actions to reduce food waste.

Algal biofuels

Nutrient recycling systems essential for biofuel production

The US NAS report on potential large-scale algae biofuel production identifies nutrient, water and land supplies as priority issues, and implementation of nutrient recycling systems as necessary.

P- recovery processes

Manitoba Canada

Struvite recovery from swine manures

Pilot scale trials show that a simple settling batch system with potassium hydroxide addition provided a system for P-recovery from digested piggery manures, appropriate for on-farm installation.

December 2012

Granular sludge process

Bacterial calcium phosphate accumulation

Bacteria in granular sludge in a sequencing batch reactor are used to induce calcium phosphate accumulation in the core of the sludge granules, and progressive transformation to hydroxylapatite.

Magnesium potassium phosphate

Potential for K- and P-recovery from urine

Laboratory experiments provide basic data concerning conditions for precipitation of MPP (magnesium potassium phosphate) from synthetic urine.

Swine manure

P-recovery as calcium phosphate

A process for recovering phosphorus as a calcium phosphate fertiliser from the liquid fraction of swine manure has now been demonstrated full-scale at several sites. A nitrification step avoids problems of pH buffering and carbonate precipitation interference.

China

Leaching ash for struvite recovery

Sewage sludge incineration ash was dissolved using acid, heavy metals removed, then the leachate used for struvite precipitation, as a possible route for P-recovery.

Future events

Brussels, 6-7 March 2013

European Sustainable Phosphorus Conference http://www.espc2013.org/

Monte Carlo, 25-27 March 2013

Phosphate industry conference

http://www.crugroup.com/events/phosphates/

Beijing, 18-20 June 2013

Global TraPs world conference

www.globaltraps.ch

Vancouver, 28-31 July 2013

International Nutrient Removal and Recovery 2013: trends in resource recovery and use.

http://www.wef.org/nutrients/ Call for abstracts open until 12th December 2012

ASA – CSSA – SSSA - Cincinnati **Opportunities and limitations of P-reuse** from manures

The annual combined meeting of the ASA (American Society of Agronomy), CSSA (Crop Science Society of America) and SSSA (Soil Science Society of America) brought together over 4000 scientists, regulators and industry representatives in Cincinnati, Ohio, 21st - 24th October 2012, under the theme "Visions for a Sustainable Planet". The symposium "Opportunities and limitations of phosphorus removal reuse from manures", organised by Dr. N. Nelson (Kansas State University) and Dr. A. Shober (University of Delaware) reflected this theme, and showed the awareness of phosphorus stewardship issues amongst agricultural scientists, the opportunities for new manure P management solutions opened by pressures on farmland spreading, and that P-recovery and P-recycling technologies are mature and have demonstrated their successful application in American pig, cattle and poultry farms.

Haygarth (Lancaster University P. UK *p.haygarth@lancaster.ac.uk*) explained that agronomists need to take into account the planetary phosphorus 'footprint' (pools and fluxes), including P in soils, geological P in rocks, human uses, waste streams and P in surface waters. He identified three challenges: to find better ways to use P already in soils (rather than continuing to apply P), for example enabling plants to better access organic P-esters in soils; preventing leakages of P to surface waters, and in some cases recovering P from eutrophied water bodies; recovery and recycling of phosphorus from all waste streams and all points of the use cycle. He emphasised that **P-management must be seen as part** of an overall sustainability approach, addressing contaminant limitation, climate change/energy and other nutrients.

University A&M S. Feagley (Texas USA sfeagley@ag.tamu.edu) presented the debate about phosphorus resource limitations and possible 'Peak **P'**, emphasising that whatever the horizon at which phosphorus scarcity might occur, geographical concentration of reserves is problematic and sustainable development requires that we act now to reduce reserve consumption, develop P stewardship and therefore implement phosphorus recycling.

He also presented potassium (K) resource status, indicating that there are some 100 known K rock reserve sites, and a further 100 brine K reserve sites worldwide, with just over 50% in Canada, but also sites in Russia, Belarussia, Germany, Israel, China, ... representing around 600 years of minable reserves, that is at least 6x longer horizon than for phosphorus reserves. Again, however, he emphasised that K stewardship should be engaged and that current use and loss patterns are not sustainable.

C. Thornton (Global Phosphate Forum *coordinator@phosphate-forum.org*) discussed the drivers for phosphorus stewardship and Precycling, including pressures on price, contaminants, geopolitical concentration of resources, leading to interest in regionally "closing the loop" for P. He indicated a number of recent national and international initiatives: UNEP, EU Green Paper, Global TraPs, Phosphorus Summits, national Nutrient Partnerships, etc. Farmland reuse of biosolids is ecologically optimal, enabling recycling of nitrogen, phosphorus and micronutrients, and return of organic carbon to the soil, but this is often not feasible because of regional nutrient excesses resulting from concentration of livestock production or urbanisation. Increasingly, the regulators and industry in Europe see sewage not as a 'waste' but as a 'resource' (for energy, nutrients, and water recycling), this paradigm change should also develop in manure management.

Phosphorus surpluses

Shober (University of Delaware USA A. ashober@udel.edu) explained that in certain regions of the USA, regional excesses of phosphorus are increasing. Soils that have been saturated with phosphorus, through years of spreading of manures to crop nitrogen needs (resulting in much higher loads than crop P needs) will take decades to return to normal phosphorus contents. There is therefore a clear need to develop and implement processes to dry, concentrate and so export manure, and in particular manure phosphorus.

(University **B**. Brown of Delaware USA *brown@udel.edu*) presented the considerable opportunities and challenges posed by poultry **manures** in the Delmarva Peninsula. The value of the nutrients in poultry manure is around US\$ 90 per tonne, but only to land where nutrients are needed. Because the region hosts some 6% of the USA's 9 billion broiler chicken annual production, soil has been saturated with phosphorus, and will take decades for P levels to come back down to levels where soils again have an agronomic need for phosphorus, particularly in the context of TDML implementation around the Chesapeake Bay (nutrient TDML: Total Maximum



Centre Européen d'Etudes des Polyphosphates

SCOPE Newsletter - nº 90 - December 2012 - Page 2 www.ceep-phosphates.org

Daily Loads, see SCOPE Newsletters n° 76 and 43). Although there has been some reduction in manure P content over recent years, because of the use of phytase to make P in chicken feed more available to the birds and because of improvements in chicken genetics to enhance feed efficiency, huge regional P excesses remain.

Challenges for recovered phosphates

B. Hall (Mosaic Fertilizers *bill.hall@mosaicco.com*) presented the massive scale, centralised organisation contract system of **fertiliser** and structured distribution logistics in the USA, and explained the difficulties this presents for marketing and distributing recycled P materials, which will be produced in (comparatively) small quantities and diffuse production sites. He also emphasised that recycled P fertilisers must respect fertiliser industry requirements for granulometry (eg. 2 – 4 mm granules), to enable compatibility with blending and spreading equipment.

At present, there are also a number of **issues with standards for labelling, nutrient availability assessment and efficacy verification**: existing methods are designed for water-soluble mineral fertilisers, and are not appropriate for many recovered materials (even though these may be plant available), posing problems for marketing and for conformity to regulations.

B. Monley (Farm Pilot Project Coordination Tampa Florida USA Bob.monley@fppcinc.org) presented onfarm assessments of thermochemical processes for treating chicken manures, using torrefaction, pyrolysis, gasification and combustion processes. He noted that in addition to the use of energy recovered from such processes, the thermochemical processes can offer advantages by introducing a source of dry heat avoiding the moisture byproduct that is formed when propane is consumed for heating. The moisture from the propane combustion can promote ammonia formation in the chicken houses affecting bird health. Thermochemical processes can generate biochar, which can be utilised as an adsorbent material to collect pollutants and additionally the organic rich material in biochar can be returned to the soil to sequester carbon. Biochar is also being investigated as a soil amendment to rebuild and contribute to soil health by improving water retention and resilience to drought conditions and other climatic events. One disadvantage of thermochemical processes is that Nitrogen is not recovered and most is lost.

J. Ackerman (University of Manitoba Canada *joe.ackerman@gmail.com*) presented pot fertiliser trials using Canola (spring colza/rapeseed Brassica napus) in prairie soils (with pH 7.6). Pure laboratory

synthesised struvite and manure recovered struvite were compared to two commercial fertilisers (MAP mono ammonium phosphate, and polymer coated MAP) at control plus five different P doses.

The manure recovered struvite came from a **410 litre batch reactor operating on swine manure lagoon liquor**, with potassium hydroxide (KOH) added to increase pH and without magnesium dosing, resulting in phosphate precipitation. This product contained 4% potassium and 5-6% phosphorus, so may have been partly K-struvite (magnesium potassium phosphate) but there was no evidence of K-struvite in XRD analysis.

Results showed that the two struvites performed similarly, suggesting that it is not necessary in Precovery processes to ensure high purity of struvite. However, both struvites resulted in significantly lower final biomass production than did the commercial fertilisers, measured plant P-uptake being comparable. This result differs from other published literature which suggests that struvite performs as well as commercial fertiliser, this may be because of the soil pH > 7 used here, or it may be because the struvite gives a slow release of phosphorus so that commercial fertilisers enable faster initial biomass production which is never 'caught up' in the pot trials (biomass and P uptake were here measured at the end of growth only, further work is proposed to look at these parameters over the plant's growth cycle).

P-recovery processes

A. Szogi (US Dept. of Agriculture – Agricultural Research Service, Coastal Plains, Soil, Water, and Plant Research Center, Florence, South Carolina USA *Ariel.Szogi@ars.usda.gov*) presented two different processes for recycling phosphorus from manures. He notes that a key driver for implementation is the financial incentive from water quality credits, where these are in place.

The solid-liquid separation / calcium phosphate recovery process is presented in detail separately in this Newsletter – this is now operational full scale in two large swine farms in North Carolina.

Dr Szogi also presented the "QuickWash" Process (patent pending, commercialised by Renewable Nutrients LLC, Pinehurst, North Carolina), see SCOPE Newsletter n°78. Acid is applied to fresh swine manure (pH 3-5) releasing much of the phosphorus from solid to liquid fraction. After solid-liquid separation, lime and anionic PAM (polyacrylamide polymer) flocculant are added to the liquor (pH c. 8) resulting in a precipitant which is extracted in filter beds. The objective is to keep much of the nitrogen in the solid fraction, which is low in phosphorus, and thus has an



CEEP Centre Européen d'Etudes des Polyphosphates

SCOPE Newsletter - nº 90 - December 2012 - Page 3 www.ceep-phosphates.org N:P ratio close to plant needs, to optimise agronomic value for farmland spreading. The recovered precipitant has 5.5 - 6 % P dry weight (around 40 -50% of this P is in organic forms), dries readily by standing, and can be stored, transported and used as a fertiliser.

K. Heller (Kansas Environmental Management Associates USA *kheller@klaenviro.com*) presented the PHREDTM process for recovery of struvite from manure using ammonia injection. The process is operational full-scale for over 4 years at a beef feedlot near Liberal, Kansas. The process treats the liquor from solid-liquid separation (<1% suspended solids), by injecting ammonia, then precipitating phosphate (mainly struvite) in a fluidised bed reactor (a column, with a conical base for precipitate removal, and a wide, baffled settling zone at the top). The 38 litre/second (6m high, 2m diameter) full scale reactor was designed after successful testing of a 0.06 litre/second bench scale and 0.4 litre/second pilot scale reactors.

Ammonia injection into the base of the reactor is sufficient to ensure the pH increase necessary for phosphate precipitation, achieving c. 60% total P removal (25% of liquor P is organic). Ammonia is sold locally to farmers for fertiliser use, and is supplied under pressure so that reactor injection pumps are not necessary. The liquor after Pprecipitation is spread on fields, so the ammonia is used as nitrogen fertiliser. In tests using swine manure, magnesium injection was also needed to obtain struvite precipitation, but this was uneconomic in beef manure, where significant magnesium is available in the manure.

In a 70 000 head beef feedlot near Liberal, Kansas where the system is installed, it is estimated that the system's removal of phosphorus from the waste avoids the need to purchase an additional 103 hectares of land for manure application. The 250 – 300 000 US\$ installation costs are significantly cheaper than this land price.

When the system was modelled for a 18 000 head beef feedlot in Kansas, it was estimated that it would avoid the purchase of over 240 hectares land, otherwise necessary to enable spreading of the phosphorus removed from the liquor. The 250 - 300 000 US\$ installation costs would again be significantly cheaper than this land price.

The precipitated phosphate product contains 3 – 19 – 0.4 - 5 - 6 (N P K Ca Mg), dries to easily handled, stable pellets, and is marketed by TerraPhos www.kemallc.com It has been tested in greenhouse fallow pot trials and University field trials on corn, soybeans, and horticultural plants. Farmer trials have also been performed on these crops as well as grass

turf production. In all cases, it has proved comparable or better than commercial phosphorus fertilisers.

The results of these field trials on manurerecovered struvite are summarised in Ruiz Diaz et at. (2010). Four different application rates of recovered struvite, MAP (mono ammonium phosphate) and TSP (triple super phosphate) were tested on corn and soybean crops in the field in Scandia, Kansas, USA. The soil was low in phosphorus, with pH 6.3 to 6.7. Puptake, plant growth and final grain yield were analysed.

Phosphorus from agricultural drainage ditches

Penn (Oklahoma State University C. USA chad.penn@okstate.edu) presented field tests of different structures to remove phosphorus from agricultural drainage ditches or underground drains. The principal route for phosphorus loss from soils to surface waters is through transport of particulate phosphorus (P carried with soil particles lost through erosion and run-off). However, in soils which have repeatedly received P-rich manures, drains can carry typically 0.5 - 10 mg/litre of dissolved phosphate, which can pose significant eutrophication problems for receiving rivers or lakes. A number of different wastes were tested as Phosphorous Sorbing Materials (PSMs), in different in-drain filter-bed configurations. Performance showed to be the result of both sorption characteristics and hydraulic permeability, in that often significant quantities of P are carried in peak flow events and this will only be adsorbed if these high flows can move through the material bed. The materials were assessed for the cost of P removal, duration, availability. Total costs of 100 - 200 US\$/kg P removed were achieved, including material purchase and transport, structure, maintenance, material replacement when saturated with P and disposal.

USA C. Gill (University of Maryland cgillsoud@gmail.com) also presented work using Phosphorous Sorbing Materials (PSMs) in drainage ditches. Material beds were able to remove around 20% of total phosphorus inflow concentrations of c. 1 mgP/l. Materials tested included flue gas desulphuration gypsum (mainly calcium sulphate) and steel plant electric arc furnace slag (mainly calcium and iron oxides). The possible reuse of the Pcontaining materials was not considered, but this would not be possible for certain wastes, such as steel slags, which potentially contain contaminants.

Innovative nutrient recovery

P. Sibrell (United States Geological Survey Kearneysville West Virginia USA *psibrell@usgs.gov*) tested Blue Valley acid mine drainage residuals for P-





SCOPE Newsletter - nº 90 - December 2012 - Page 4 www.ceep-phosphates.org

removal from wastewaters. This material contains mainly iron (hydr)oxides, with aluminium and calcium oxides. In this work, the material could be regenerated using 0.1 molar sodium hydroxide, recovering around ³⁄₄ of the adsorbed phosphorus in 12 hours, giving a 1000x more concentrated phosphate solution (0.1 mgP/l in the wastewater, up to 100 mgP/l in the regeneration solution). The system was operated for 6 months with weekly regeneration. From the regeneration liquor, hydroxylapatite (calcium phosphate) was precipitated, enabling P-recovery for recycling. Negligible water contamination by heavy metals from the waste material was confirmed.

Davis (Colorado J. State University *jessica.davis@colostate.edu*) presented an on-farm system for growing cyanobacteria algae in covered ponds, in order to fix atmospheric nitrogen for fertiliser. The cyanobacterial algae used were selected from local surface waters. The water containing the algae was used for irrigation, so spreading the nitrogen to crops without necessitating an algal harvest / filtration process. This could provide an interesting route to increase the N:P ratio in waste liquors, to be closer to crop needs, because the cyanobacteria can 'fix' nitrogen from the atmosphere, but need phosphate supply.

Conference website and proceedings (including video recording of all speakers): https://www.acsmeetings.org

"Phosphorus Recovered from Feedlot Manure as Fertilizer Source for Corn and Soybean", Kansas Fertiliser Research 2010 (Kansas State University), pages 8-11. D. Ruiz Diaz, N. Mueller, K. Heller, N. Nelson http://www.ksre.ksu.edu/library/crpsl2/srp1049.pdf

"Struvite precipitation in anaerobic swine lagoon liquid: effect of pH and Mg:P ratio and determination of rate constant", N. Nelson et al., in SCOPE Newsletter n°68

"Phosphorus Removal and Recovery from Hog Lagoon Supernatant Using a Gravity-Settled Batch Reactor and Increased pH", J. Ackerman, N. Cicek, Biological Engineering 4(4), pages 207-218, 2011 http://elibrary.asabe.org/

Public policy

Wastewater treatment European Union renews pressure for sewage treatment conformity

The European Commission is continuing its active policy of pressure on Member States to oblige full implementation of European wastewater treatment and water quality legislation, with actions concerning Germany, Greece, Italy, Portugal, the UK. At the same time, Sweden is considering implementing tradable certificates for nitrogen removal in sewage works, to try to achieve nitrogen emission reduction objectives needed to reduce eutrophication in the Baltic Sea.

See also SCOPE Newsletter n° 86 (action against France) for failure to implement the Nitrates Directive, and SCOPE 84 (Luxembourg) and SCOPE 79 (Spain) for failure to adequately implement phosphorus removal.

Sewage treatment obligations in Europe are fixed by the 1991 Urban Waste Water Treatment Directive (1991/271) which in particular requires phosphorus removal in all sewage works serving towns or groups of villages of > 10 000 person equivalents (generally around 6 000 population after taking into account industrial and commercial effluents) which discharge into potentially eutrophication sensitive areas.

This is reinforced by the **Water Framework Directive (2000/64)** which both confirms these obligations and adds the requirement to carry out any further measures necessary to achieve good ecological quality status, or equivalent, which may in some cases imply phosphorus removal in smaller sewage works.

The European Commission is maintaining pressure on Member States to fully implement these obligations:

European Commission actions

- IP/12/537 (31st May 2012) 'reasoned opinion' (first stage of a formal procedure which leads to European Court referral unless an adequate response is received) sent to **Greece concerning inadequate wastewater treatment of 12 areas**, including Prosotsani, Doxato, Eleftheroupoli and Vagia, where secondary biological treatment is not yet implemented
- IP/12/536 (31st May 2012) referral to the European Court of Germany because the full costs of water services are not charged to water users, contrary to the obligations of the Water Framework Directive (the objective being to encourage lower water consumption through the consumer – payer principle)
- IP/12/292 (22nd March 2012) 'reasoned opinion' sent to Italy concerning various issues with implementation of the Water Framework Directive: inadequate water basin management plans, inadequate monitoring, failure to put into place measures to achieve good water quality



Centre Européen d'Etudes des Polyphosphates SCOPE Newsletter - n° 90 - December 2012 - Page 5 WWW.ceep-phosphates.org

- IP/12/658 (21st June 2012) referral to the European Court of Italy for failure to implement the 1991 Urban Waste Water Treatment Directive obligations of nutrient removal from sewage works discharging into eutrophication sensitive areas (see above). The EU Commission considers that at least 50 Italian towns still do not satisfy these nutrient removal obligations.
- IP/12/662 (21st June 2012) 'reasoned opinion' to Portugal concerning inadequate treatment of sewage from small towns and groups of villages (2 000 – 10 000 p.e.). The EU Commission considers that 77 such conurbations still have inadequate sewage collection and treatment in Portugal.
- European Court Case c-301/10 (18th October 2012), condemnation of the UK, following referral by the EU Commission, for failure to adequately treat wastewater in the London and Sunderland in areas. In both cases, the European Court upheld the EU Commission's position that storm discharges were occurring regularly, corresponding to a failure to collect and treat sewage, and were not therefore acceptable under the Directive's obligation to collect and treat wastewater.

Obligation to collect and treat

In the UK case, the European Court has confirmed its previous jurisprudence finding "a failure to fulfil obligations in cases where the collection or treatment rate for urban waste water amounted to 80% or even 90% of the existing load".

The Court stated "the concept of 'sufficient performance' ... must be understood as meaning that, under usual climatic conditions and account being taken of seasonal variations, all urban waste water must be collected and treated" and considers that "an improvement of 0.3% in respect of the quality of the receiving waters ... (by avoiding storm overflows of) 20 discharges per year" does not represent a disproportionate cost.

This decision will place the UK under renewed pressure to move forward with the UK£ 4 billion 'super sewer' project passing under the tidal river Thames, to increase the capital's sewage collection capacity and avoid storm overflows in cases of heavy rainfall.

Sweden proposes nitrogen quotas

The Baltic Sea Action Plan (BSAP) fixes objectives for reductions of phosphorus and nitrogen emissions into the Baltic Sea. **Phosphorus goals have been met**, **but Sweden still has to reduce nitrogen emissions** **from sewage works** in southern Sweden by one third by 2021. In order to achieve this as rapidly and costeffectively as possible, the Swedish EPA has proposed a system of **tradable nitrogen certificates** ("CEASAR"), with progressively tighter total emissions floors for each of the three marine basins of the Baltic (Baltic proper, Sound, Kattegat).

Sweden EPA considers that this nitrogen quota system should be able to also address local EQS (Environmental Quality Standards) which apply to local nitrogen emissions to Baltic coastal areas (not to inland waters), in that the local differentiation in emissions objectives is not sufficient to justify a system with differing local targets.

Phosphorus reductions

Although BSAP (Baltic Sea Action Plan) reductions for sewage works phosphorus emissions have already been achieved in Sweden's sewage works, **EQS** (Environmental Quality Standards) for phosphorus for both inland (fresh) and coastal waters require further reductions in many local situations. These EQS are fixed at the river basin management plan level, with the aim of achieving Good Environmental Status (EU Water Framework Directive implementation).

Sweden EPA considers that a general system to reduce sewage works emissions (overall regulation, tradable quotas ...) would not be appropriate for phosphorus, because water basin local targets are very variable depending on local water system eutrophication sensitivity, water flow pathways, phosphorus retention patterns within water systems ...

Sweden EPA concludes by emphasising that current policies will not enable Sweden to achieve the objective of 'No Eutrophication' because particularly of nutrient emissions from agriculture. This situation has been worsened by the **removal of the Swedish fertiliser tax in 2010** (approx.. 35 million Euros total tax collected annually, when operative). EPA suggests that the nutrient emissions quota system should be studied for possible extension to cover agriculture and industry.

EU Commission actions on waste water treatment: http://europa.eu/newsroom/press-releases/index_en.htm (enter number of press release as indicated in text above)

EU Court Case c-301/10 (UK):

http://curia.europa.eu/juris/document/document.jsf?text=Whitburn &docid=128650&pageIndex=0&doclang=EN&mode=req&dir=&o cc=first&part=1&cid=845517

Sweden EPA report – analysis of policy instruments for improving abatement of nutrients from municipal wastewater treatment plants, report 6521, October 2012, in Swedish with English summary:

http://www.naturvardsverket.se/Documents/publikationer6400/978 -91-620-6521-8.pdf





SCOPE Newsletter - nº 90 - December 2012 - Page 6 www.ceep-phosphates.org

P food chain losses

EU retailers pledge action on food wastes

Reducing food waste would save 20% of the world's food production. The agricultural land use economised could enable food production to feed all the world's hungry and reduce world net greenhouse gas emissions by 40%, as well as considerably world reducing phosphorus consumption (see SCOPE Newsletter n°81). This issue is now being taken on board by European regulators and commerce, with recent positions from the European Parliament, EU Commission and EuroCommerce, the association of supermarkets and retain chains.

EU Environment Commissioner, Janez Potocnik, October 2012, underlined that the EU generates 89 million tonnes of food waste per year (179 kg per person per year), contributing to "fuel and mining activity to make fertilizer, transport, refrigeration, packaging, toxic emissions to air and water, and the use of many other natural resources. ... Less food waste would lead to a more efficient land use, better water resource management, more sustainable use of phosphorous". He indicated that an EU Commission study suggests that the retail sector only contributes 4% of food waste, but can play a major role in helping consumers reduce household food waste.

The Commission's objectives, fixed in the **'Roadmap** to a Resource-Efficient Europe', include halving food waste and achieving near zero landfilling of food waste by 2020. The European Commission also intends to issue a Communication on Sustainable Food in 2013.

European Parliament resolution

The EU Parliament adopted a resolution on 19th January 2012, calling for action on food waste, and made the following proposals, amongst others:

- establish specific food waste prevention targets for Member States, as part of the waste prevention targets to be reached by Member States by 2014
- include specific initiatives targeting food waste in the EU "Resource-Efficient Europe" flagship initiative
- enable retailers to substantially reduce the price of fresh food to below the cost of production when it is close to its sell-before date, in order to reduce the amount of unsold food discarded
- **clarify the meaning of the date labels** ('best before', 'expiry date' and 'use by') in order to

reduce consumers' uncertainty regarding food edibility and to disseminate accurate information to the public, notably the understanding that the minimum durability 'best before' date is related to quality, while the 'use by' date is related to safety, in order to help consumers make informed choices;

• optimisation and efficient use of food packaging ... such as varying pack sizes to help consumers buy the right amount

The European retail sector, represented by EuroCommerce, has responded by promising to organise two major awareness-raising campaigns targeting consumer food waste before 2014. Euro-Commerce has also published a 'Retail agreement on waste'

Janez Potocnik speech, Retail Forum for Sustainability 2012, 9th October 2012 http://europa.eu/rapid/press-release_SPEECH-12-709_en.htm

European Commission food waste web page: http://ec.europa.eu/food/food/sustainability/index_en.htm

EU Parliament press release

http://www.europarl.europa.eu/pdfs/news/expert/infopress/201201 18IPR35648/20120118IPR35648_en.pdf and resolution: http://www.europarl.europa.eu/plenary/en/texts-adopted.html (then select: 19th January 2012)

EU press release: 'Retailers pledge action to reduce food waste' http://europa.eu/rapid/press-release_IP-12-1088_en.htm

EuroCommerce 'Retailers Environment Action Programme' http://ec.europa.eu/environment/industry/retail/pdf/reap_tor.pdf

Algal biofuels

Nutrient recycling systems essential for biofuel production

The US National Academy of Sciences has published a report on "Sustainable development of algal biofuels", concluding that further considerable innovation is necessary to achieve acceptable EROI (net energy production) and to reduce environmental impacts. Water, land and nutrient consumption are identified as high priority issues. The development of nutrient recycling and the reuse of wastewater nutrients are essential.

The report was prepared by a committee of 15 experts, convened by the US National Research Council on request of the Department of Energy. The committee was asked to identify and assess potential **sustainability concerns relating to large-scale development of biofuel production from algae** and to propose progress indicators and possible impact mitigation strategies.



CEEP Centre Européen d'Etudes des Polyphosphates

SCOPE Newsletter - nº 90 - December 2012 - Page 7 www.ceep-phosphates.org

Not an established industry

The committee underlines that algal biofuel production is still very much at the R&D stage, with different algae strains, production and biofuel/energy processing routes being proposed. Considerable improvements will have to be made in EROI before algal biofuels can possibly be sustainable (Energy Return on Investment, that is, the net energy contained in the biofuel produced compared to total energy consumed in the production and processing of the algae). Current EROI are in the range 0.13 - 3.3, which means that in some cases more energy is consumed than is contained in the produced biofuels, and in the best cases, a multiplying factor of only around 3 is achieved on total input energy. Considerably higher net energy production needs to be achieved.

This is also necessary to achieve corresponding improvements in net GHG emissions (green house gas).

Water energy and land

Beyond improving net energy production, the three priority sustainability concerns identified by the committee are consumption of water, land and nutrients.

Algal biofuel production will result in water consumption, through evaporation from algae ponds, consumption in chemical processing of fuels, and losses and leakages. Some 132 billion litres of water would be consumed to produce 39 billion litres of biofuels (that is 5% of US transportation fuel demand).

Land area demand is another priority concern. Land for algal production implies specific requirements for climate, topography, proximity to sustainable water supplies, proximity to processing installations. In order to use wastewater nutrient sources, proximity to urban areas or animal production is also necessary, and such land may be prohibitively Appropriate geographical siting of expensive. production must also take into account risks of pollution from algal pond overspill or process incidents.

Other potential issues need to be investigated, such as the implications of large areas of algae production ponds for biodiversity and possible development of mosquito-born diseases.

Nutrients

Nutrients are essential for algal production. 1 - 2million tonnes of phosphorus (P) and 6 – 15 million

tonnes of nitrogen (N) would be necessary annually to produce 39 billion litres of biofuels (that is 5% of US transportation fuel demand), if nutrients are not recycled. This is represents 20 – 51% of current total US phosphorus consumption and 44 – 107 % of current US nitrogen consumption.

Use of wastewater nutrients as system inputs, and the recycling of nutrients from the algal harvesting and biofuel production systems would therefore be essential, to ensure sustainability, reduce costs, and avoid (as for land use) competition with food production.

The NAS committee concludes that it does not consider any of these issues as a definitive barrier to sustainable development of algal biofuels, but that these concerns need to be addressed in an integrative approach through R&D into both improved algal production and processing systems and into impact management and mitigation strategies.

US National Academy of Sciences "Sustainable Development of Algal Biofuels in the United States", 2012 http://www8.nationalacademies.org/onpinews/newsitem.aspx?Reco rdID=13437

P-recovery processes

Manitoba Canada Struvite recovery from swine manures

A first paper (2010) presents beaker tests of struvite precipitation from anaerobically digested swine manure, collected from experimental digesters, then settled and centrifuged. This enabled identification of optimal conditions for pH, reaction and settling time and magnesium: phosphorus ratio. A second paper (2011) presents pilot trials using two unstirred 410 litre, conebased batch reactors operating on supernatant from the primary settling lagoon of a commercial piggery in Manitoba, Canada.

The initial beaker tests looked at anaerobically digested pig manure effluents (after centrifuging to remove suspended solids) with soluble phosphorus concentrations 27 - 38 mg P-PO₄ /l, ammonium around 1300 mg NH₄/l, calcium around 95 mgCa/l and magnesium around 23 mgMg/l. Mg:P ratios from 1:1 to 1.6:1 and pH values from 7.5 to 9.5 were tested. Beakers were stirred for 10 minutes then settled for 24 hours. Precipitate was collected by centrifuging followed by filtration. ICP (Inductively Coupled Plasma) spectrometry and FIA (Flow Injection Analysis) were used to determine the effluent's basic





SCOPE Newsletter - nº 90 - December 2012 - Page 8 WWW.ceep-phosphates ord www.ceep-phosphates.org

constituents. Struvite precipitation was demonstrated by XRD (X-Ray Diffraction).

Maximum soluble phosphate removal was achieved at pH9 (80% P-removal from the effluent) but purest struvite was precipitated at pH7.5 (with calcium carbonate = calcite forming at higher pH). The struvite formation rate constant was calculated to be 1.55/hour at pH8.4.

Accessible P-recovery technology

Following the beaker experiments above, a pilot-scale system was tested with the **aim of developing a low energy consumption and low-investment phosphorus removal and recovery system for untreated liquid manure**, adapted to on-farm installation and operation.

Two 410 litre cone-bottomed tanks were installed at a commercial pig farm in Manitoba, Canada, to operate on primary manure settling lagoon supernatant. The effluent had pH of around 6.7, 60 - 220 mgP/l total phosphorus and around 95 mgP-PO₄/l soluble phosphate, around 142 mgMg/l magnesium and 470 mgCa/l calcium.

The tanks were **filled with effluent**, **dosed to pH8.5 using potassium hydroxide (KOH)**, **then allowed to settle** for 24 hours or 48 hours. The settled solids were then drawn off from the bottom of the tank cone base (approx. 20 litres withdrawn per batch), dried in the sun in shallow trays, milled, then analysed by FIA, ICP and by loss on ignition (for organic content).

The batch system reliably removed 70 - 80% of total phosphorus, with around 2/3 of this P-removal from soluble phosphate, and 1/3 from settling of particulate phosphorus forms. Typical influent suspended solids were reduced by over one half, from around 5 to around 2.2 gSS/1. Calcium was reduced from c. 470 to 335 mgCa/l and magnesium from c. 140 to 50 mgMg/l.

Analysis of the solids produced suggested that all magnesium present was in inorganics, whereas phosphorus and ammonia were present in both organics and inorganics. **Around half of the recovered solids content was organic**, with the inorganic part probably containing struvite, some calcium phosphates and calcium carbonate. The solids contained in total (of dry matter) around 5.6% P, 3.3% Mg, 3.8% K, 4.2% Ca and 6.3% N, that is less than 1/3 dry weight struvite.

Advantages of not stirring

The authors note that high calcium concentrations in the treated manure did not impede phosphorus removal and recovery, possibly because of the long settling times. They also suggest that the **absence of stirring may facilitate formation of flocs**, which then settle, whereas stirring breaks flocs into particles too small to settle. However, further work is necessary to improve the purity and characteristics of the recovered solid.

Cost estimate

The cost of such a system were estimated to treat manure digestion effluent for a 1000 pigs/year production unit, producing 3000 litres per day of manure. This would require two 4500 litre tanks (to offer redundancy and facilitate operation). The value of the recovered solids is calculated on the basis of the commercial fertiliser price for the nutrients present (US\$ 910 per tonne, of which US\$ 700 is for the potassium content). The potassium hydroxide purchase cost is estimated at US\$ 2600 /year /1000 pigs, of which US\$ 1780 is recovered through the fertiliser value of the potassium in the solids. The second largest cost item (after KOH) is labour, at US\$ 2200 per year.

Including investment depreciation, maintenance, and taking into account the recovered fertiliser values as above, overall net cost, is estimated at US\$ 4050 per year for the 1000 pig unit, that is US\$ 0.03 per kg live weight added to the pigs.

"Phosphorus removal from anaerobically digested swine wastewater through struvite precipitation", Water Science & Technology (WST), n° 61.12, pages 3228-3234, 2010

E. Jordaan, J. Ackerman, N. Cicek, Dept. Biosystems Engineering, University of Manitoba, 75 Chancellors Circle, Winnipeg, MB R3T 2N2, Canada joe.ackerman@gmail.com

"Phosphorus Removal and Recovery from Hog Lagoon Supernatant Using a Gravity-Settled Batch Reactor and Increased pH", Biological Engineering 4(4), pages 207-218, 2011 (ASABE ISSN 1934-2799). http://elibrary.asabe.org/ J. N. Ackerman, N. Cicek, as above.

Granular sludge process Bacterial calcium phosphate accumulation

Granular sludge processes offer potential for wastewater treatment because of the small footprint and the capacity to treat high organic loading rates and ensure simultaneous nitrogen and phosphorus removal. The dense structure of the granules results in specific local pH conditions, induced by the biological reactions, which can cause mineral precipitation in the core of the granules.



SCOPE Newsletter - n• 90 - December 2012 - Page 9 s www.ceep-phosphates.org



Specific management of the biological cycles can accentuate phosphate precipitation and facilitate transformation of precipitated amorphous calcium phosphate to more stable hydroxylapatite. Results with a laboratory scale sequencing batch granular sludge reactor showed that 40 - 75% of phosphorus removed was in the form of precipitated calcium phosphate.

Two identical 17 litre sequencing airlift batch reactors (SBAR) were used for tests, with air injection to ensure mixing during the aerobic phase and nitrogen during the anaerobic and anoxic phases. Synthetic wastewater was used containing glucose, acetate, propionic acid, ethanol, phosphate, calcium, magnesium, nitrate, ammonium, sulphate ions. A 2 litre reactor was also used to test precipitation at different concentrations of phosphate and other ions, with aerated sludge but without the biological cycling.

The principal results were obtained from one of the 17 litre biological reactors operated with a **4 hour cycle**: 20 mins. anoxic, 145 mins. aerobic, 30 mins. idle, 30 mins. emptying. Hydraulic residence time was 8.5 hours (50% exchange at emptying). The reactor was operated for 540 days, achieving 93 - 97% COD removal, 92 - 100% total nitrogen removal and 31 - 67% phosphate removal.

Phosphorus in sludge granules

Phosphorus content of the sludge at the end of operation was 5 - 6 % P. Raman analysis, SEM-EDX (scanning electron microscopy – energy dispersive X-ray detection) and XRD (X-ray diffraction) were used to identify the forms of phosphate present in the granules. This suggested that **only calcium phosphates were formed in the sludge granules**, with smaller amounts of other phosphate minerals (such as magnesium and potassium struvite) being formed in the sludge bulk.

Analysis showed that during the SBAR cycle, potassium, magnesium and phosphate were released during the anoxic phase (but no significant calcium release), whereas phosphate was taken up as expected during the aerobic phase. pH showed to decrease slightly during the anoxic phase, increase to pH 8.2 at the start of the aerobic phase (presumably because of CO_2 stripping, then decrease again at the end of COD consumption. This decrease is thought to be due to biological nitrification and to calcium phosphate precipitation.

Precipitation reactions were modelled using PHREEQC and a modified mineral database derived from NIST 2011. This modelling suggested that ACP (amorphous calcium phosphate) would initially be precipitated through the pH increase at the start of the aerobic phase. This could then progressively transform to more stable hydroxylapatite if retained in the sludge granules.

The authors suggest that the **bacteria in the sludge** granules facilitate calcium phosphate formation through three mechanisms:

- bio-aggregates serve as **nucleation sites** catalysing precipitation;
- biological reactions locally modify pH creating conditions for calcium phosphate precipitation;
- biological reactions **locally release soluble phosphates** so increasing local concentrations.

Results showing that the **mineral phosphate content was higher in the centre of the sludge granules than near their edges** suggests that local pH decreases resulting from biological activity during the aerobic phase could redissolve some amorphous calcium phosphate precipitated by nucleation on the outside of bacteria, whereas pH in the core of the granules may remain higher enabling reformation as hydroxyapatite.

"Biologically induced phosphorus precipitation in aerobic granular sludge process", Water Research n°45, pages 3776-3786, 2011 www.elsevier.com/locate/watres A. Manas (a,b,c,d), B. Biscans (a,b,c,d), M. Sperandio Mathieu (a,b,c). a = Université de Toulouse, INSA, UPS, INP, LISBP, 135 Avenue de Rangueil, F-31077 Toulouse, France. b = INRA, UMR792 Ingénierie des Systèmes Biologiques et des Procédés, F-31400 Toulouse, France. c = CNRS, UMR5504, F-31400 Toulouse, France. d = CNRS, UMR5503, 4, allée Emile Monso BP 84234, F-31432 Toulouse Cedex 4, France.

"Parameters influencing calcium phosphate precipitation in granular sludge", Chemical Engineering Science, n°77, pages 165-175, 2012 www.journals.elsevier.com/chemical-engineeringscience A. Manas (a,b,c,d), M. Poquet (a,b,c), B. Biscans (a,b,c,d), M. Sperandio Mathieu (a,b,c).

Magnesium potassium phosphate Potential for K- and P-recovery from urine

Wilsenach et al. 2007 studied the precipitation of struvite (magnesium ammonium phosphate MAP) but also "K-struvite" (magnesium potassium phosphate MPP, also referred to as potassium magnesium phosphate KMP or K-struvite) in laboratory batch tests and in a bench scale stirred reactor. Xu et al. 2011, 2012 provide further information concerning the conditions for MPP precipitation by testing different parameters in stirred beaker batch tests and in a lab-scale draft tube and baffle reactor (DTBR).





SCOPE Newsletter - n[•] 90 - December 2012 - Page 10 www.ceep-phosphates.org Both research teams used synthetic urine composed of calcium, sodium, chlorine, sulphate, potassium ions and creatonine. Urea and or ammonium were either included in the synthetic urine or specifically dosed to test impacts of different concentrations of these ions. Xu et al. 2012 also tested using real urine.

Magnesium dosing

Wilsenach et al. tested the addition of magnesium as McCl₂ and as MgO in stirred and unstirred batch beaker tests, at varying pH (adjusted using NaOH), and using synthetic urine with different concentrations of urea and ammonium. Specifically designed benchscale continuous flow, top-stirred reactors, including two different baffle designs to ensure upflow of liquid and settling / separation of particles were then used (30 cm high, 10 cm diameter, 0.8 l reaction zone and 0.8 l settling zone). Precipitated solids were analysed by XRD.

This work concludes, as have other studies, that it is relatively easy to precipitate struvite/K-struvite, but that fines tend to be formed and are difficult to prevent from being carried out of the reactor, and if separated are difficult to dewater. Also, problem struvite/K-struvite deposits tend to form on reactor walls, etc. The crystal form, density and characteristics of the K-struvite are found to be very similar to those of struvite crystals, with the same difficulties for recovery and nuisance deposit avoidance.

This study also concludes that in untreated urine, which has a very high ammonium:phosphate ratio (c. 43), compared to a K:P ratio of only 2, MAP struvite will precipitate. If urine is treated (eg. by nitrification denitrification or by air stripping) leaving low levels of ammonium, then part struvite / part K-struvite will be formed.

pH and K:P ratio

Xu et al., 2011, assessed the effects of pH, of different residual concentrations of ammonium. and of molar K:P ratios. Batch stirred beaker tests were carried out, using MgCl₂ to dose magnesium, NaHPO₄ to adjust phosphate and NaOH for pH.

This work confirmed the role of pH, with pH adjustment giving good levels of phosphate precipitation, improving slightly up to pH 11. Less than 50% of potassium was removed from solution at pH 10 with 1:1 molar ratio of Mg and P to K, compared to c. 98% with Mg and P at 2x molar ratio of Κ.

Ammonium concentrations from 40 to 500 mgN/l were tested, showing that increasing ammonium resulted in lower potassium removal, without significantly modifying P removal. Thus, if ammonium is present, it struvite tends to preferentially be formed (rather than K-struvite), so that potassium recovery is reduced. Even a small % of urine ammonium remaining after treatment would significantly reduce potassium recovery (with MAP struvite being formed instead).

This work also showed that some magnesium sodium phosphate (MSP) was precipitated in place of magnesium potassium phosphate. The synthetic urine has a molar Na:K ratio of c. 7.5 so that this may also be an obstacle to efficient potassium recovery from urine.

DTBR reactor experiment with real urine

Xu et al., 2012, tested precipitation of magnesium potassium phosphate (MPP) from real urine in a bench scale draft tube and baffle reactor (DTBR). The reactor was 25 cm high with diameter 13 cm (total 2.5 l), including a central stirred zone (0.5 l) with a variable speed impeller, top settling outflow zone, and a conical crystal collection zone at the base. Magnesium (Mg Cl₂) and NaOH (to increase pH) were dosed.

Operating parameters (mixing speed, pH, hydraulic residence time HRT, Mg:K:P ratio) were first studied using synthetic urine, then a 182 hour experiment was carried out using real urine (diluted 5 times and ammonia stripped).

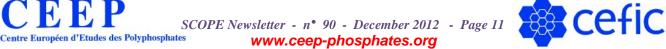
The synthetic urine tests showed that a reactor speed of at least around 100 rpm was necessary for efficient reaction, but that above around 250 rpm significant fine crystals were being lost in the reactor supernatant.

Potassium removal efficiency increased slightly with higher HRT (from 2.5 to 12.5 hours), whereas phosphate removal efficiency decreased significantly, again probably related to fine crystal loss. An optimal mixing speed of 150 rpm and HRT of 10 hours were identified.

Crystal Retention Time (CRT) was calculated as slurry discharge time (extraction of precipitates) / slurry volume. Longer CRT resulted in increased crystal concentration of MPP (rather than MAP), but too high CRT resulted in accumulation of slurry in the reactor base, and consequent crystal loss.

The diluted, ammonia stripped real urine used for the DTBR test had 63 mg/l COD, 32 mgN/l ammonium, 150 mgP/l phosphate and 480 mg/l potassium. Average removal of 73% ammonium, 76% potassium and 68% phosphate were achieved, and also 23% of COD was removed to the crystal slurry. The crystal slurry collected had a 98% water content





for a CRT of 11 hours. Its Zeta potential was 12 to 14 mV, which probably prevented the agglomeration of struvite particles. XRD showed the solids to be a mixture of MAP (struvite), MPP (K-struvite) and MSP (magnesium sodium phosphate). Around 9g of crystals were produced per litre of diluted urine (45g per litre of recovered product from pure urine).

"Phosphate and potassium recovery from source separated urine through struvite precipitation", Water Research 41, pages 458 – 466, 2007 www.elsevier.com/locate/watres

J. Wilsenach, C. Schuurbiers, M. van Loosdrecht, Delft University of Technology, Dept. Biotechnology, Julianalaan 67, 2628BC, Delft, The Netherlands. JWilsenach@csir.co.za

"Simultaneous removal of phosphorus and potassium from synthetic urine through the precipitation of magnesium potassium phosphate hexahydrate", Chemosphere 84, pages 207–212, 2011 www.elsevier.com/locate/chemosphere

K. Xu, C. Wang, Y. Qian, School of Environment, Tsinghua University, Beijing 100084, PR China. H. Liu, School of Civil and Environmental Engineering, University of Science and Technology Beijing, Beijing 100083, PR China wangcw@tsinghua.edu.cn

"Laboratory experiments on simultaneous removal of K and P from synthetic and real urine for nutrient recycle by crystallization of magnesium–potassium–phosphate–hexahydrate in a draft tube and baffle reactor", Chemosphere 88, pages 219–223, 2012, www.elsevier.com/locate/chemosphere

K. Xu, C. Wang, X. Wang, Y. Qian, as above.

Swine manure P-recovery as calcium phosphate

Traditional pig manure treatment in the USA widely uses anaerobic lagoons. These pose a number of issues including ammonia emissions, odour, pathogens, space consumption and loss of pollutants to surface waters. Agreements were made between North Carolina authorities and the state's two largest hog producers in 2000 to develop Environmentally Superior Technologies (EST).

Of five ESTs selected from a proposal assessment, only one **treated the whole manure waste stream onfarm** (the others only addressed the manure solids fraction). This process, developed by USDA-ARS (US Dept. of Agriculture – Agricultural Research Service) and licenced to Terra Blue Inc. (Clinton, North Carolina, USA *www.terrablueinc.com*) involves a **solid-liquid separation** by filters enhanced by PAM (polyacramide) coagulants, followed by **aerobic composting of the solids**, and **nitrification then calcium phosphate (CaP) precipitation of the liquid stream**. The denitrification – CaP precipitation process has since been **successfully tested also on anaerobic lagoon liquors**. Also, the CaP precipitate has been tested in greenhouse pot trials, and its plant availability shown to be around 99%.

Since 2007, North Carolina requires by law all new swine farm developments to use alternative manure treatment and not lagoons.

Ammonia, carbonate, buffering and pH

Other processes have met difficulties in precipitating calcium phosphate from waste waters, because of the need for prior carbon stripping to avoid bicarbonate interference with calcium phosphate precipitation. For example, by first taking the pH down to pH6 to remove bicarbonate, then up to pH9 to precipitate calcium phosphate (see Geestmerambacht experience, SCOPE Newsletter n°41). This results in high consumption of chemical reagents.

Animal wastewater contains **high levels of carbonate**, **because this is generated as urea is decomposed** to ammonium. The treatment of the liquor by nitrification uses a fluidised bacterial bed on polymer pellets and bubble aeration. In a more economical version, a high performance nitrifying sludge replaced the polymer pellets (case study $n^{\circ}3$ in 2012-A). The biological process converts ammonium to NO_3^- and $2H^+$ (acidity) which reacts with the bicarbonate to produce CO_2 , which is removed to air with the aeration.

As a result, calcium phosphate precipitation can occur when calcium is dosed, without interference from bicarbonate (calcium carbonate precipitation) and the liquor's pH buffering is considerably reduced, so that a rapid pH increase to pH 10.5 - 11 can be achieved by adding calcium hydroxide (lime), thus facilitating calcium phosphate precipitation. The recovered phosphate showed a P content of around 9%

Demonstrations full scale

A first pilot plant was tested using swine manure lagoon liquor, with a 1.8 m³ total capacity biological COD removal and nitrification reactor, and a 279 litre calcium phosphate precipitation rector (case study 1 in 2012-A). The process has now been demonstrated full-scale at two North Carolina swine farms: one at Goshen Ridge Farm, Duplin County, NC, 4360 pigs (case study 2 in 2012-A), one at Tyndall Farm, Sampson County, NC, 5145 pigs (case study 3 in 2012-A), treating 28 to 39 m³/day manure. Installations were operated successfully 24 hours/day for more than 2 years.

93% suspended solids removal was achieved in the solid – liquid separation step.



CEREP Centre Européen d'Etudes des Polyphosphates WWW.CE

SCOPE Newsletter - n° 90 - December 2012 - Page 12 www.ceep-phosphates.org The nitrification process resulted in up to 97% ammonia-N removal, producing very little biosolids sludge (<2% of solids generated from the initial solid separation). The system showed to remove over 90% of soluble phosphorus in the calcium phosphate precipitation. Because the liquor after calcium phosphate precipitation is very poorly buffered, 2 hours of bubble aeration was sufficient to restore pH to neutral, and natural aeration by standing would probably be adequate.

Greenhouse fertiliser tests

70% of the total phosphorus in the manure is transferred to the solid fraction compost, and 25% recovered from the liquid fraction as calcium phosphate, around 5% remains in the final effluent. The compost produced has c. 5.3% N, 4.0% P, 0.5% K and 2.1% Mg.

The precipitated phosphate was recovered as a wet sludge, and dewatered in filter bags. It was tested (2007-A) in **greenhouse pot trials using rye grass** (Lolium multiflorum). Two different particle sizes of the recovered calcium phosphate (obtained by grinding and sieving) were compared to commercial triple super phosphate (TSP) at five different phosphorus application rates (including control = zero P addition). Ryegrass biomass development and leaf phosphorus content were measured.

With the larger particle size recovered phosphate (2-4 mm), little or no fertiliser effect was noted. With smaller particle size (0.5 - 1 mm), the recovered phosphate gave results approximately half to two-thirds those of TSP. The authors conclude that the phosphorus in the recovered material is 99% plant available, but that **release depends strongly on particle size**. Larger particles of the recovered calcium phosphate could be used as a slow release fertiliser.

Field fertiliser tests

In further studies (2012-B), the recovered calcium phosphate (in two particle sizes as above) was compared to commercial diammonium phosphate (DAP) fertiliser and to a control (no fertiliser) in 4m x 10m plot field trials using cotton (Gossypium hirsutum) grown after winter rye (Secale cereale) with conservation tillage. The experiment included four replicates per plot and lasted two years (two harvests of each crop). Ca(NO₃)₂ was applied to ensure the same nitrogen fertilisation on all plots.

The recovered phosphate was processed by International Fertilizer Development Corporation (IFDC) in Mussel Shoals, Alabama, and proved **easy to compact and granulate** at 20% moisture content, achievable by air-drying. Phosphorus uptake and biomass development was not increased for the rye by the recovered phosphate, but did increase with DAP, whereas for the cotton no increases were noted for any of the fertilisers compared to the control, presumably because the soil already contained adequate available phosphorus without fertilisation. Analysis of the soil available phosphorus during the trial suggested, as above (2007), that grinding to small particle size is necessary to render the recovered calcium phosphate plant available.

Contact emails: Matias.Vanotti@ars.usda.gov Ariel.Szogi@ars.usda.gov and website: www.terrablueinc.com

2007-A: "Agronomic effectiveness of calcium phosphate recovered from liquid swine manure", Agron. J. 99, pages 1352–1356 (2007) https://www.agronomy.org/publications/aj P. Bauer, A. Szogi, M. Vanotti

2012-B: "Phosphorus recovered from swine wastewater as a fertilizer for cotton grown with conservation tillage", Journal of Cotton Science 16, pages 97–104 (2012) http://journal.cotton.org P. Bauer, A. Szogi, J. Novak, M. Vanotti

"Technology for recovery of phosphorus from animal wastewater through calcium phosphate precipitation", International Conference on Nutrient Recovery from Wastewater Streams, Vancouver 2009, ISBN: 9781843392323, published by IWA, M. Vanotti and A. Szogi

http://www.iwapublishing.com/template.cfm?name=isbn18433957 70

"Removal of phosphorus from livestock effluents", J. Environ. Qual. 38, pages 576–586 (2009) https://www.soils.org/publications/jeq A. Szogi, M. Vanotti

2012-A: "Method for Recovery of Phosphorus from Animal Wastewater", Presentation at WEFTEC 2012, 85th Annual Water Environment Federation Technical Exhibition and Conference, New Orleans, Oct. 3, 2012 www.weftec.org and https://www.ars.usda.gov/SP2UserFiles/Place/66570000/FY13/wef tec_2012_vanotti.pdf

M. Vanotti (1), M. Garcia (2), A. Szogi (1), P. Hunt (1), P. Millner (3). 1: USDA-ARS, Coastal Plains Soil, Water and Plant Research Center, Florence, SC. 2: Agriculture Technological Institute ITACyL, Valladolid, Spain. 3: USDA-ARS, Environmental Microbial and Food Safety Lab, Beltsville, MD.

References from 2012-A by case study:

Case 1 (anaerobic effluent)

Vanotti, M. B., Szogi, A.A., and Hunt, P.G. (2003) Extraction of soluble phosphorus from swine wastewater. Trans. ASAE 46(6):1665-1674.

Vanotti, M.B., Szogi, A.A., and Hunt, P.G. (2005) Wastewater treatment system. US Patent No. 6,893,567, Issued May 17, 2005. U.S. Patent & Trademark Office, Washington, D.C., USA.

Case 2 (Goshen Ridge farm)

Vanotti M.B., Szogi, A.A., Hunt, P.G., Millner, P.D., and Humenik, F.J. (2007) Development of environmentally superior treatment system to replace anaerobic swine lagoons in the USA. Bioresour. Technol. 98(17):3184-3194.



Centre Européen d'Etudes des Polyphosphates SCOPE Newsletter - n° 90 - December 2012 - Page 13 WWW.ceep-phosphates.org Vanotti, M.B. and Szogi, A.A. (2008) Water quality improvements of CAFO wastewater after advanced treatment. J. Environ. Qual. 37:S86-S96.

Vanotti, M.B., Millner, P.D., Hunt, P.G., and Ellison, A.Q. (2005) Removal of pathogen and indicator microorganisms from liquid swine manure in multi-step biological and chemical treatment. Biores. Technol. 96:209-214.

Szogi, A.A., Vanotti, M.B., and Hunt, P.G. (2006) Dewatering of phosphorus extracted from liquid swine waste. Bioresour. Technol. 97(1):183-190.

Case 3 (Tyndall farm)

Vanotti, M.B., Szogi, A.A., Millner, P.D. and Loughrin, J.H. (2009) Development of a second-generation environmentally superior technology for treatment of swine manure in the USA. Biores. Technol. 100(22):5406-5416.

Vanotti, M.B., Szogi, A.A., and Fetterman, L. (2010) Wastewater treatment system with simultaneous separation of phosphorus sludge and manure solids. U.S. Patent No. 7,674,379 B2. U.S. Patent & Trademark Office, Washington, D.C., USA.

Garcia, M.C., Vanotti, M.B., and Szogi, A.A. (2007) Simultaneous separation of phosphorus sludge and manure solids with polymers. Trans. ASABE 50(6):2205-2215.

China Leaching ash for struvite recovery

The authors tested different methods for leaching phosphate from sewage sludge incineration ash from two local sewage treatment plants in China. Cation exchange resin was then used to remove heavy metals from the leachate, before adding magnesium and ammonium and adjusting pH to precipitate struvite, enabling phosphate recycling as fertiliser.

The ash tested came from two different sewage works, one with conventional treatment (no phosphate removal process) and 7% industrial wastewater, one with biological nutrient removal and 40% industrial wastewater.

The sewage sludge incineration ashes showed phosphorus contents of 6.5% and 11.8% P (15.2% and 27.4% P₂O₅) (as % dry matter), but also significant levels of heavy metals such as zinc, copper, nickel and lead.

Acid extraction

Ash extraction was tested in laboratory flask studies, using hydrochloric acid and sodium hydroxide (alkali) at varying concentrations and varying amounts added (L/S - liquid to solid ratio). Flasks were shaken for 2 hours, then the leachate separated by filtration.

As could be expected, phosphorus extraction efficiency increased with concentration of acid or alkali and with the quantity added, up to ceilings with 0.2 - 0.5 mol/l HCl and 50 ml/g L/S (98% P extraction). P extraction by NaOH was limited (<30%).

Heavy metals

The authors note that the residue after acid extraction is corrosive and requires safe disposal.

The heavy metals present in the ash were also largely leached out by the acid P extraction, and could then precipitate with struvite, contaminating this fertiliser product. Therefore, cation exchange resin (CER) was added to the leachate, a dosage of 0.04 g CER/l proving optimal for heavy metal removal. No phosphorus was removed by the CER. The separated CER/heavy metal complex would also need safe disposal.

Struvite

Struvite was precipitated from the leachate by adding magnesium chloride and ammonium chloride, as magnesium and ammonium sources (the leachate does not contain ammonium, because nitrogen in the sewage sludge is lost during incineration), plus sodium hydroxide to increase pH. 97% of phosphate was precipitated at pH 10 with magnesium and ammonium addition at 1.4:1 ratio to phosphate.

The struvite contained <0.8 mg/g of zinc, <0.35 mg/g of copper, < 0.2 mg/g lead, < 0.06 mg/g nickel and <0.00025mg/g mercury, that is lower than legal limit values for fertilisers for all countries considered (Austria, China, Switzerland and Turkey).

The authors calculate struvite production of 0.2 - 0.4kg/kg ash, HCl consumption of 10-25 mol/kg ash and CER consumption of 2 kg/kg ash. Consumption of other reagents (magnesium, ammonium, sodium hydroxide) is not indicated. Profit is estimated at > 5USD/kg sludge on the basis of fertiliser value of struvite (considered to be 20 US\$/kg). The costs of the other reagents is not considered because these can be reused.

"Recovery of phosphorus as struvite from sewage sludge ash", Journal of Environmental Sciences 24(8), pages 1533–1538, 2012 www.jesc.ac.cn

H.Xu, P. He, W. Gu, G. Wang, L. Shao, State Key Laboratory of Pollution Control and Resource Reuse, College of Environmental Science and Engineering, Tongji University, Shanghai 200092, China solidwaste@tongji.edu.cn





SCOPE Newsletter - nº 90 - December 2012 - Page 14 www.ceep-phosphates.org

Brussels, 6-7 March 2013 **European Sustainable Phosphorus** Conference

http://www.espc2013.org/

Frontrunners from several European Member States have joined forces to raise awareness about the more sustainable phosphorus necessitv for management. The ambition is to create a clear and coherent legislative framework for eco-innovation, a sustainable European market for secondary phosphorus and more efficient phosphorus use.

- Raise awareness about the necessity for more sustainable phosphorus management within the context of a **Resource Efficient Europe**.
- Create support for a clear and coherent **legislative** . framework, to create an enabling environment for eco-innovation, a sustainable European market for secondary phosphorus and more efficient phosphorus use.
- The further development of **sustainable nutrient** chains within Europe. Connecting different nutrient waste flows and market possibilities between stakeholders (private sector throughout different sectors, knowledge institutes, government and NGO's) within the EU.

Phosphates 2013 CRU

Fertilizers • Industrial • Feed phosphates 25-27 March 2013, Fairmont Monte Carlo, Monaco www.phosphatesconference.com

Monte Carlo, 25-27 March 2013 Phosphates 2013

http://www.crugroup.com/events/phosphates/

The CRU Phosphates conferences are the only global meeting for the worldwide phosphate industry (rock production, fertiliser, animal feeds, food, detergents, other industrial uses).

These conferences bring together over 500 delegates from tens of countries worldwide, including senior industry executives and organisations that define phosphate supply and demand. Phosphates 2013 will provide a macro view of historical and current markets, supply, demand and prices to better understand the context for future trends.

Earlybird booking conditions available until 30th November 2012

Centre Européen d'Etudes des Polyphosphates

Beijing, 18-20 June 2013 Global TraPs world conference

www.globaltraps.ch

Global Transdisciplinary Processes The for Sustainable Phosphorus Management (Global TraPs) project is studying phosphorus use. management and sustainability from a supply chain perspective involving academia, industry, governments, NGOs and other concerned parties (see SCOPE Newsletter n° 86).

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 in the future.

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.

The Conference 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.

Call for abstracts (for papers and posters) is open **until 12th December 2012** on categories including:

nutrient recovery processes

www.ceep-phosphates.org

- 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 International Federation the water quality people* Water Association

