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# SCOPE NEWSLETTER

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NUMBER 55

May 2004

## Phosphorus recycling

### Belgium

#### **Phosphate recovery from starch wastewaters**

DHV is to install a Crystalactor® system to remove phosphorus from wastewaters from the Amylum Europe starch plant, Aalst, Belgium, recovering the phosphate as recyclable pellets.

### Struvite parameters

#### **Understanding struvite precipitation**

Experimental data for chemical and kinetic parameters for struvite precipitation at conditions comparable to wastewaters. Results show significant variations in crystal surface charge, crystal shape and precipitation rates as a function of pH, different counter ions, magnesium/phosphorus ratios.

### Treviso

#### **Full scale P-recovery process**

Results of operation of the full scale pilot phosphorus recovery plant at Treviso municipal sewage works (March 2001-October 2002) show successful phosphorus precipitation but difficulties with organics and suspended solids.

### Modelling

#### **Calcium phosphate precipitation**

The PHREEQC computer programme was used to thermodynamically model the effects of changes in solution conditions on calcium phosphate precipitation from pure solutions. Experiments show that carbonate concentrations can inhibit or enhance phosphate precipitation, but solution pH is still a dominant factor.

### Spain

#### **An industrial by-product magnesium source for struvite recovery**

Precipitation of struvite from pure chemical solutions and from real sewage treated liquors using a by-product of industrial magnesium oxide production, as magnesium source. The by-product proved effective used at higher dosages, but this

### Italy

#### **Improved REM-NUT for P-recovery**

A new phosphorus-selective ion exchange resin was tested, improving the REM NUT process for nutrient recovery as struvite.

### Poland

#### **Struvite crystal shape**

Laboratory studies and pilot reactor tests show formation of struvite with two different crystal forms (rhomboid and needle), influencing recovery possibilities. The authors suggest that this may depend on free ammonia levels.

### France

#### **Fate of phosphorus in manure treatment**

Three pig manure treatment plants were studied to assess under what conditions phosphates were found in solid or liquid fractions.

### Novaquatis

#### **Struvite precipitation from urine**

Ongoing R&D project addressing source separation of urine and recovery of nutrients, includes two workpackages on struvite precipitation

### North Carolina

#### **Struvite recovery from livestock lagoon liquor**

Operating parameters and reactor design for struvite precipitation were evaluated using first beaker tests, then a laboratory scale continuous mode crystalliser of novel fluidized bed design, and finally a continuous field scale reactor operated at an anaerobic pig slurry lagoon, sized to treat the wastewater flow to irrigation from 1,000 pigs.

## Sewage treatment

### Nutrient removal

#### **EU Commission report into Urban Waste Water Treatment (UWWT) Directive implementation**

EU Commission maintains pressure on Member States to implement adequate nutrient removal from sewage, and publishes Third Report on UWWT Directive 91/271 implementation.

## International Conference

### **STRUVITE: its role in phosphorus recovery and reuse**

Cranfield University, England,  
17-18 June 2004

[See page 2](#)

## International Conference

### **STRUVITE: its role in phosphorus recovery and reuse**

Cranfield University, England,  
17-18 June 2004

**Thursday 17<sup>th</sup> June 09:30 -17h00**

*Beneficial reuse of struvite : fertiliser value and acceptance*

*Practical Issues associated with P recovery : reactor design and operation experience*

**Friday 18<sup>th</sup> June 9h00-17h00**

*Struvite Chemistry & Crystallisation :  
modelling, supersaturation kinetics, interactions with other ions*

*Struvite recovery from sources other than sewage.*

### **Venue**

Cranfield University is situated north of London and midway between Oxford and Cambridge. It is close to thriving Milton Keynes, a new town with one of the largest covered shopping centres in Europe, and the historic riverside town of Bedford.

### **Registration**

The conference registration fee includes: a copy of the proceedings, the coffee breaks, lunch on Thursday and Friday. It also includes the banquet dinner on the Thursday night. Additional places are available for the dinner for accompanying persons.

### **Participant Fees**

Full delegate           UK£295  
Student participant    UK£200  
Accompanying Dinner   UK£40  
Accommodation inc. Breakfast – from approx UK£40/night

### **Information, registration and full programme details**

Tel: +44 (0)1234 754902 - Fax: +44 (0)1234 751671 - Email: [struvite@cranfield.ac.uk](mailto:struvite@cranfield.ac.uk)

<http://www.cranfield.ac.uk/sims/water/struvite/>

### Phosphorus recycling

#### Belgium

#### Phosphate recovery from starch wastewaters

DHV Water BV (Netherlands) are to install a Crystalactor® unit for phosphate recovery at the Amylum Europe starch and starch derivatives plant, Aalst, Belgium. The unit is located between the existing anaerobic and aerobic wastewater treatment installations.

This system crystallises magnesium phosphates onto sand seed particles in a fluidised bed reactor, induced by a pH increase (50% sodium hydroxide addition). Prior to the reactor, magnesium chloride is dosed, producing high-quality magnesium phosphate pellets which can be recycled into the phosphate industry.

The starch plant has an average wastewater flow of 155 m<sup>3</sup>/hour with up to 80 mg/l of phosphate. The Crystalactor® system will **bring the final effluent phosphate concentration down to 2 mg P/l**. The system is being delivered Autumn 2003 and should be operative by 2004.

The recovered phosphates pellets will be taken by *Thermphos International* (The Netherlands) and **used in their phosphorus production**, thus recycling the phosphates into industrial products. See <http://www.nhm.ac.uk/mineralogy/phos/Nordwijkerhout/Schipper1704.doc>

A Crystalactor® system has been running, recovering phosphates from a sidestream, at Geestmerambacht municipal sewage works since 1994, producing 200-300 tonnes of phosphate pellets per year from a 230,000 pe sewage flow. See: <http://www.naturalhistorymuseum.org.uk/mineralogy/phos/gaastr.htm>

Crystalactor® systems have also been previously installed for phosphorus removal and recovery (as struvite: magnesium ammonium phosphate) at the AVEBE potato starch factory, Veendam (The Netherlands) (150 m<sup>3</sup>/hour) and pilot units (calcium phosphate recovery) at Westerbork municipal sewage

works (12,000 pe) and Heemstede municipal sewage works (35,000 pe). See <http://www.nhm.ac.uk/mineralogy/phos/crystalact.htm>

The Crystalactor® system is also used for a number of other applications ranging from the chemical industry to drinking water purification.

#### Information:

[http://www.dhv.nl/frameset.asp?mainpage=http://www.dhv.nl/Application.PerformanceManagerServlet/English/Groups/site/DHVProjects/Water/Crystalactor\\_unit\\_for\\_Amylum/Crystalactor\\_unit\\_for\\_Amylum.asp](http://www.dhv.nl/frameset.asp?mainpage=http://www.dhv.nl/Application.PerformanceManagerServlet/English/Groups/site/DHVProjects/Water/Crystalactor_unit_for_Amylum/Crystalactor_unit_for_Amylum.asp)

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### Struvite parameters

#### Understanding struvite precipitation

Despite considerable interest in struvite precipitation within different wastewaters, both because of nuisance deposit formation in installations, and as a route for phosphorus recovery for recycling, few studies are available of struvite precipitation and chemistry kinetics. F. Abbona *et al.* publications (1980's – early 1990's) remain references with some more recent applied chemistry studies (see eg. Parsons *et al* in Scope Newsletter n°54, n° 41 at [www.ceep-phosphates.org](http://www.ceep-phosphates.org)).

The two papers summarised here present detailed experimental work measuring, in closely controlled laboratory conditions, the induction time and precipitation rate of struvite from

- (A) **pure phosphate, magnesium and ammonium solutions** (plus sodium chloride for ionic strength) and
- (B) **synthetic wastewater** containing also organic carbon and certain counter ions (carbonate, sulphate).

In all cases unstable supersaturated solutions were used. The studies were carried out at pH8.5 or 9.0, comparable to pH susceptible to occur in waste waters by CO<sub>2</sub> degassing or to be developed in a phosphorus recovery reactor.

The studies used continuously stirred 0.25 litre beakers, water bath regulated to 25°C, held underneath a surface flow of water-vapour saturated nitrogen to avoid CO<sub>2</sub> intrusion and/or escape from the supersaturated solutions. pH and reagent concentration were automatically adjusted throughout the experiments by auto-titrators linked to pH and ion probes. pH was thus controlled to an accuracy of 0.05.

### Pure solution struvite chemistry

The first series of experiments (paper A) used magnesium chloride and ammonium phosphate solutions. Salt (NaCl) was used to adjust ionic strength to 0.015 molar. Precipitation of struvite resulted in a drop in pH, triggering reagent addition by the autotitrators.

Measurement of induction times as a function of supersaturation rates, enabled a **stability diagram** of the structure to be developed (log K for equilibrium of all reactions). In all cases, the only precipitant being formed was struvite. The **surface energy of the precipitating struvite** was calculated from the induction measurements as a function of the solution supersaturation. Newberyite (MgHPO<sub>4</sub>) was not formed at the experimental conditions examined. The struvite formed as **long needle like crystals**, as reported previously by various other authors. The precipitation rate showed a second order dependence on supersaturation, suggesting a surface diffusion mechanism.

**The precipitated struvite crystals showed a strong negative charge** which increased at higher pH but which was dependent on magnesium concentration. At low magnesium concentrations, the charge of the struvite particles was in fact slightly positive, and it became increasingly negative with increasing magnesium concentrations. Aggregation of precipitant particles may be favoured at higher magnesium concentrations.

At high pH 10.5, flocculation occurred. Small variations in pH caused significant changes in particle zeta potential, and this is likely to destabilise suspensions by aggregation.

### Organics and counter ions

The second paper (B) presents further experiments carried out under the same closely controlled conditions and experimental method, but this time in the **presence also of soluble organic molecules** (glucose at 100gCOD/l) and different counter ions: carbonate (NaHCO<sub>3</sub>), nitrate (NaNO<sub>3</sub>), sulphate (either by using magnesium sulphate as the magnesium source or from Na<sub>2</sub>SO<sub>4</sub>). Salt was used to ensure ionic strength. Phosphate : ammonium : magnesium ratios of 1:1:1 were used, and pH was adjusted using NaOH. Again, equilibrium log k was calculated for all reactions in the systems.

**Precipitation again showed to be a second order function of supersaturation rates.** The kinetics of spontaneous struvite precipitation showed, as expected, to be faster at pH9 than at pH 8.5. Further, the kinetics were faster for magnesium sulphate as magnesium source than for magnesium chloride.

**Very different shapes of struvite crystals were found**, with long needle like structures at pH8.5 and oblong rhomboid crystals at pH9 – see also on the question of struvite crystal shape summary of the report by Suschka *et al* in this Newsletter.

(A) “Nucleation and crystal growth of struvite in aqueous media. New perspectives in phosphorus recovery”. Paper presented at the WASIC Conference (Workshop on Advance in Sensing in Industrial Crystallization), June 18-20, 2003, Istanbul, Turkey.

A. Kofina, P. Koutsoukos, Dept. Chemical Engineering and Institute of Chemical Engineering and High Temperature Chemical Processes, University of Patras, PO Box 1414, 26500, Greece. [pgk@iceht.forth.gr](mailto:pgk@iceht.forth.gr)

(B) “Spontaneous precipitation of struvite from aqueous solutions”, *Journal of Crystal Growth*, 213 (2000) pages 381-388 [www.elsevier.nl/locate/jcrysgr](http://www.elsevier.nl/locate/jcrysgr)

### Treviso

#### Full scale P-recovery process

The full scale pilot phosphate recovery installation at Treviso municipal sewage works is presented in previous articles and in [Scope Newsletter n° 49](#). The unit is fed with liquor from the dewatering line from the anaerobic sludge digester at a now 70,000 pe biological nutrient removal sewage plant and aims to precipitate phosphorus by aeration only (CO<sub>2</sub> stripping leading to a pH increase) in robustly designed fluidised bed configuration. The published report covers results obtained in the plant from start up in March 2001 through to October 2002, as well as further preparatory bench ageing and supersaturation tests.

Previous work by Battistoni *et al.* used smaller semi-pilot installations and bench tests, using anaerobic supernatants from Ancona sewage works to model phosphorus precipitation kinetics and parameters.

Because of problems in the operation of the sewage plant itself (start up of new installations, water leakage into sewers resulting in diluted influent), it was necessary to add soluble phosphate to the digester supernatant to levels in the range 25-250 mgP-PO<sub>4</sub>/l.

#### Phosphorus removal

**A reasonably high level of phosphorus removal from the inflow liquor was achieved** in the phosphorus precipitation unit (average 54% removal of total phosphorus) and visible growth of the sand particles used as a seed in the fluidised bed reactor was seen.

Hydraulic conditions established in the fluidised bed reactor enabled the bed to remain homogenous and suspended, but **the bed showed a loss of sand** over the period. It was not clear whether this was the result of fixing of sand into the fluidised bed's gravel base, or of loss of sand carried out of the top of the reactor.

At the same time the density of the bed material decreased progressively during the operating period,

presumably because **organic material** is adhering to the seed sand as well as precipitated phosphates. This affected performance, as the lighter particles were then carried out of the top of the reactor and lost. An elevated level of nucleation instead of particle growth in the phosphate formation also resulted in the generation of fine particles, again partly lost over the top of the reactor in the outflow. A modification to the Dortmund settling device at the top of the reactor failed to resolve this. The presence of organics in the precipitant particles was also shown by chemical analysis.

Phosphate content of the bed material reached 8.1% and nitrogen content 0.4% by the end of the reported period.

#### Suspended solids problems

The authors note that the results obtained so far in this pilot plant start up phase do not enable the theoretical and previous experimental results to be confirmed. Further work will look at growing the self-nucleating particles rather than using sand as a seed, thus increasing the potential P-content of the precipitated material and removing problems relating to sand release from the reactor which can pose problems for pumps downstream. Further work will look at systems to prevent carry over of small and light particles from the reactor (outflow at reactor top), for example a system to capture foams and froth.

Finally the **limitation of suspended solids** in the digester dewatering liquor used as feed appears as very important.

*“Phosphate crystallisation process for P-recovery applied at Treviso Municipal Wastewater Treatment Plant (Italy)”, report 2003*

F. Cecchi – University of Verona, Paolo Battistoni, Raffaella Boccadoro – Marche Polytechnical University, Italy [p.battistoni@univpm.it](mailto:p.battistoni@univpm.it)

Available for download at [www.nhm.ac.uk/mineralogy/phos/](http://www.nhm.ac.uk/mineralogy/phos/)

### Modelling

#### Calcium phosphate precipitation

The authors' 2002 paper used computer modelling to study the speciation and saturation index for the precipitation of calcium phosphates from pure chemical solutions under different conditions of ionic strength and temperature comparable to those found in wastewaters.

The **theoretical thermodynamic calculations** were carried out using the computer programme PHREEQC for precipitation of calcium phosphate as hydroxyapatite (HAP). Variable inputs were concentrations of phosphate,  $H^+$ , soluble calcium ion, total ionic strength (assumed addition of  $KNO_3$ ). Temperature ranges of 5-30 °C were considered.

Phosphate concentrations of 1-200 mgP/l were considered, and soluble calcium concentrations 1-10 x molar Ca:P ratio. The saturation index showed to increase logarithmically with phosphate and calcium concentrations, and polynomially with pH in the range 7-11. The saturation index, as can be expected, also decreases logarithmically with total ionic strength.

The authors conclude that adjusting soluble calcium concentrations and solution pH values should be two effective tools for controlling calcium phosphate precipitation, and in particular for enabling phosphates to be precipitated from wastewaters as HAP for recovery and recycling.

#### Effects of carbonate

Previous work by the same authors (2001) briefly reported in [Scope Newsletter n°41](#) showed that **calcium phosphate precipitation is significantly inhibited by dissolved carbonate**, particularly at pH below around 9. The authors suggested, on the basis of solution speciation calculations, that this is because of the formation of ion pairs between carbonate and calcium ions thus decreasing the concentrations of calcium ions, and so the thermodynamic forces for calcium phosphate precipitation.

This 2002 work used phosphate concentration of 20 mgP/l in solutions of total ionic strength comparable to

wastewaters at 0.02 mol/l, with carbonate concentrations up to 5 mmol/l.

In the absence of carbonate, at pH 8, phosphate precipitation showed firstly a rapid precipitation of around 10% of phosphates in around 10 minutes, then an "S" curve with a low rate of acceleration for around 4.5 hours, then a faster rate, achieving around 50% precipitation after 6 hours, then only very slow further precipitation. The acceleration at 4.5-6 hours is considered to be due to the formation of a new calcium phosphate species.

With 2.5 mmol/l carbonate addition at pH 8, initial phosphate precipitation was faster (around 20% in the first 10 minutes), but the time to the acceleration phase was delayed to around 7 hours. After 10 hours, around 50% of phosphate had been precipitated, comparable to the no-carbonate experiment after 7 hours.

With 5 mmol/l carbonate at pH 8, phosphate precipitation was significantly inhibited, with around 10% precipitation after 10 minutes, but only 20% after 12 hours.

At pH 9 also, the most efficient phosphate precipitation was recorded at 2.5 mmol/l carbonate, but again the precipitation was strongly inhibited at 5 mmol/l carbonate.

At pH 10 and 11, carbonate at 5 mmol/l inhibited phosphate precipitation only very marginally (by 1-2%).

Analysis of the precipitate, showed that at pH 9, 10 and 11 the phosphorus/calcium ratio of the precipitate was significantly lower for 5 mmol/l carbonate addition, compared to 2.5 or zero. This effect was more marked and the P/Ca ratio was also lower at all pH, when the pH was higher. P/Ca ratio of the precipitate was unchanged with carbonate addition (at around 0.8) at pH 8.

The authors conclude that the formation of ion pairs between carbonate and calcium ions decreases the concentrations of soluble calcium ions, thus the thermodynamic forces for calcium phosphate precipitation; calcium carbonate and calcium phosphate co-precipitate, thus enhancing the

precipitation of phosphate at certain solution conditions, resulting the decrease of the relative phosphate content in the precipitate, especially at higher pH values and higher calcium additions. Increasing solution pH value and the calcium addition properly are two approaches to overcome the inhibition of carbonate on the precipitation of phosphate.

*“Effects of solution conditions on the precipitation of phosphate for recovery a thermodynamic evaluation”*.  
*Chemosphere* 48, pages 1029-1034 (2002).

[www.sciencedirect.com/science/journal/00456535](http://www.sciencedirect.com/science/journal/00456535)

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Download PHREEQC:

[http://wwwbr.cr.usgs.gov/projects/GWC\\_coupled/phreeqc/](http://wwwbr.cr.usgs.gov/projects/GWC_coupled/phreeqc/)

*“The effect of carbonate on the precipitation of calcium phosphate”*, *Environmental Technology*, vol. 22, pages 207-215 (2002). [www.environtech.co.uk](http://www.environtech.co.uk)

Y. Song, H. Hahn, E. Hofmann, as above.

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## Spain

### An industrial by-product magnesium source for struvite recovery

One of the possible economic obstacles to phosphorus recovery from sewage for recycling via struvite precipitation (magnesium ammonium phosphate  $MgNH_4PO_4$ ), is the cost of adding magnesium which is generally present at significantly lower molar concentrations than phosphates. This paper examines the feasibility of using a by product of the Spanish magnesite mining and magnesium oxide (MgO) production industry, in effect low-grade MgO of low economic value.

Precipitation was tested in 1 litre beakers, stirred for up to 4 hours, using pure solutions of phosphate – magnesium and ammonium and using real wastewater,

with results for the industrial by-product being compared to those for pure MgO.

The wastewater liquor used was filtrate from dewatering of digested sludge from the Arroyo del Soto municipal wastewater treatment plant, Madrid, Spain, which uses and anaerobic/aerobic (A/O) biological phosphorus removal configuration.

The byproduct was analysed and showed to contain 68% (as MgO) of magnesium compounds with 10% (as CaO) of calcium compounds and significant traces (>2.5%) of iron oxide, sulphate and silicon compounds. Its particle size as supplied was 18% >0.1mm.

### Preliminary experiments

**First struvite was precipitated from pure chemical solutions** of 1:1:1 molar phosphate, ammonium and magnesium ions (initial concentration 200 mgP/l) by raising the pH to 9 with NaOH.

**Then, the industrial by-product was tested in synthetic solutions** containing molar ratios of phosphate, ammonium and magnesium comparable to real wastewater, by adding up to 1.5, 2.0 and 2.6 x molar ratio to phosphate. Pure MgO was tested similarly for comparison. NaOH was not added (except for prior pH adjustment of the synthetic solutions to pH 7.5 similar to real wastewater) because the by-product or MgO caused a pH increase sufficient for struvite precipitation.

With pure MgO, the variation in Mg/P ratio from 1.5 to 2.6 did not improve precipitation efficiency, whereas the byproduct (particle size as delivered) required the highest Mg/P ratio as well as prolonged stirring time to achieve a phosphate precipitation efficiency of 80% (2 hours stirring) – 95% (4 hours).

X-ray diffraction analysis suggests that this is because **only a part of the magnesium ions in the by-product are “available”** to react and form struvite (periclase = MgO), with part being held as magnesite ( $MgCO_3$ ) and dolomite ( $CaCO_3/MgCO_3$ ), insoluble at the pH of the experiments.

Correspondingly, the phosphate content of the struvite precipitated in the experiments using the by-product was somewhat lower than when pure magnesium sources were used (9.8% - 10%, compared to 11 - 12.9%) because of the presence of other substances (in particular magnesite and dolomite from the by-product, which do not intervene in the reaction).

### Real wastewater

**Experiments were then carried out using real wastewater liquor**, previously aerated to increase its pH. In this case, three forms of the industrial by-product were tested: by-product as supplied ; ground to particle size <0.04mm ; and the fraction (47,5%) with particle size <0.04mm after sieving, and compared with pure MgO. Magnesium/phosphate ratios of 1.5 - 3.6 were tested.

**Finally, further experiments were carried out using the sieved <0.04mm by-product to confirm optimal magnesium/phosphate dosages.**

These experiments confirmed the effectiveness of the by-product in real wastewater at Mg/P ratios of 2.0 to 2.5. The sieved by-product (<0.04mm) was also effective at an Mg/P ratio of 1.6. In all cases, the precipitate contained >9% phosphorus and >3% nitrogen, important criteria for its value as a fertiliser.

**The authors conclude that the by-product can be an effective magnesium source for struvite precipitation**, for recovery of phosphorus from wastewaters in the form of a useful fertiliser. Higher Mg/P dosage ratios are necessary than with pure chemicals as a magnesium source, but this could be balanced because of the low cost of the by-product compared to the purchase of such chemicals. The sieving of the by-product to achieve particle size <0.04mm improved its effectiveness significantly, but only 47% of the product as supplied passes through a sieve of this size, so most of the supplied product would be wasted.

*“Use of a by-product of magnesium oxide production to precipitate phosphorus and nitrogen as struvite from wastewater treatment liquors”, Journal of Agricultural and Food Chemistry, vol. 52, n°2, pages 294-299, 2004.*  
<http://pubs.acs.org/journals/jafcau/>

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### Italy

#### Improved REM-NUT for P-recovery

The REM NUT process uses ion exchange to remove phosphorus and nitrogen from sewage works effluent. Resin regeneration then gives relatively clean and concentrated phosphate and ammonium solutions, which can be used to precipitate struvite, enabling nutrient recovery for recycling.

The REM NUT process was originally designed to remove all phosphorus and nitrogen from sewage works effluent as a third stage nutrient removal alternative to chemical or biological nutrient removal systems (see previous articles in *Water Research vol.13* and *vol. 20 issue 6*). This required an ammonium ion exchange line of 6-8x the capacity of the phosphate exchange line, reflecting the higher concentration of ammonium in wastewater effluent. Phosphate, as well as magnesium, addition was then necessary to precipitate the removed ammonium as struvite (magnesium ammonium phosphate with 1:1:1 molar ratio).

In 2001 the process configuration was revised to target phosphorus recovery (rather than removal of all effluent ammonium), with the ammonium ion exchange line being now of the same capacity as the phosphate line. This reduced investment costs, avoided phosphorus addition and reduced magnesium consumption requirements, thus reducing costs.

#### Interference problems

This work was presented at the 2<sup>nd</sup> International *Conference* on Phosphorus Recovery and Recycling, Noordwijkerhout, 2001 (see *conference paper, article* in *Environmental Technology* vol. 22, 2001, and summary including diagram in *SCOPE Newsletter*

[n°41](#)). A key problem identified however was the poor selectivity of the ion exchange resin available for phosphate and interference of  $\text{SO}_4^{2-}$ ,  $\text{Cl}^-$ ,  $\text{HCO}_3^-$  and  $\text{NO}_3^-$ .

Also at the 2<sup>nd</sup> Phosphorus Recovery and Recycling [Conference](#) 2001, A. Sengupta [presented](#) a new ion exchange resin developed at Lehigh University, PA, USA, with specific selectivity for phosphates. This new resin is based on the commercial weak base anion exchanger *Dowed M-4195 Dow Chemical USA*, converted to a copper form and with chelation of the metal species.

The 2003 paper reports results of extensive laboratory testing of application of this resin for phosphates removal from a tap water + phosphates solution and in a 0.1 m<sup>3</sup>/hour pilot REM NUT plant at the Grottaglie-Montieasi municipal sewage treatment works, South East Italy. The pilot plant was operated continuously using real sewage works effluent for 3 months.

### Laboratory results

The preliminary laboratory experiments, using a **tap water + phosphates solution** at concentrations comparable to waste water effluent, showed that under such conditions flow rates of up to 20 bed volumes(BV)/hour allowed phosphorus levels to be brought below 0.1 mgP/l and that up to 400 BV could be treated before resin regeneration. 95% of phosphate ions removed from solution were recovered during regeneration in <1 hour using 3 BV of slightly acidified (pH 4.3) brine (0.6 M NaCl). The regenerated phosphate solution thus had a >100x higher phosphate concentration than the original solution treated. The resin loading capacity was 15gP/litre.

### Performance

The pilot plant using real wastewater effluent showed significantly lower resin performance: 150BV between regenerations to achieve 0.1 mgP/l in the effluent and a resin loading capacity of only 1.2gP/l (**<10% of that found with tap water phosphate solutions in the laboratory**). However, no resin fouling or copper loss were noted after 3 months operation.

The authors conclude that the results are moderately encouraging as to resin performance in real wastewaters and that further work is needed to obtain a better understanding of the factors affecting this.

*“REM NUT ion exchange plus struvite precipitation process”*, *Environmental Technology*, [www.envirotechnol.co.uk](http://www.envirotechnol.co.uk) vol.22, pages 1313-1324, 2001. L. Liberti, D. Petruzzelli, L. De Florio. [Direct link to article](#).

*“A new phosphate-selective sorbent for the Rem Nut® process. Laboratory investigation and field experience at a medium size wastewater treatment plant”*, *Water Science and Technology* <http://www.iwaponline.com/wst/toc.htm> vol. 48,

N°1, pages 179-187, 2003

<http://www.iwaponline.com/wst/04801/wst048010179.htm>

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*“Water Research:*

[www.sciencedirect.com/science/journal/00431354](http://www.sciencedirect.com/science/journal/00431354)

### Poland

#### Struvite crystal shape

Laboratory beaker precipitation tests and experiments using two pilot reactors ( continuous flow fluidised bed, 10 and 100 litre capacity) were carried out using sludge digester supernatant from Zabrze and Komorowice municipal waste water treatment plants (wwtps), Poland. Magnesium was added and a combination of aeration and/or reagent addition used to increase pH, to obtain struvite precipitation.

The sewage sludge digester supernatants had the following characteristics. Zabrze wwtp is a Bardenpho type biological phosphorus removal plant, using also some iron dosing. Soluble phosphorus (P-PO<sub>4</sub>) in the sludge filtrate was 130-170 mg/l and ammonium N-NH<sub>4</sub> 800-1450 mg/l . Komorowice wwtp operates partial biological phosphorus removal, and the sludge centrate used had P-PO<sub>4</sub> at 65-106 mg/l and ammonium 990-1600 mg/l.

**Simple aeration in the laboratory resulted in pH increases** in these substrates from around 7.6-7.8 up to 8.6-8.8.

Laboratory preliminary tests in 3 litre beakers showed that 80% of soluble phosphorus could be precipitated from the wastewater by aeration combined with soluble magnesium addition to a 1/1 Mg/P ratio, and 90% at a 1.5/1 ratio, after 4 hours mixing.

#### Pilot reactors

The first reactor (see diagram) was 1m high and volume 10 litres. Partial recycling of the effluent flow to the reactor base enabled upflow rates in the reactor to be controlled for bed fluidisation. The reactor had a 50° slope conical base, then a vertical sided settling zone above the fluidised bed.

The second reactor, volume 100 litres, was a 1.8m high cone of slope 20°, with no recycling flow, again using a continuous flow fluidised bed design.

The two reactors were operated at the sewage works, with the first reactor at Zabrze and the second at Komorowice, for 85 and 67 days respectively continuous operation time.

#### Crystal shape

A number of experimental results and many photos of precipitated struvite crystals are provided in the published report. The crystals show two distinct shapes: wedge shaped (rhomboid) and long needle shaped. This confirms work by previous authors who have found one or other of these crystal forms:

- \* **needle shaped:** Schultze-Rettmer 2001, Stratful 2001, Abbona 1984, Hirose 1996
- \* **rhomboid:** Parsons 2001

Of previous authors, Hirasawa has suggested that the shape is dependent on Mg/P ratio (rhomboid for Mg/P=1, needle shaped for MG/P=4), Durrant 1999 suggested that the needle like form resulted from supersaturation with magnesium and nitrogen ammonia ions.

**The authors of this report suggest that the change in crystal shape could be related to levels of free ammonia** (NH<sub>3</sub> – as opposed to soluble nitrogen ammonium NH<sub>4</sub> ions) – with high levels of free ammonia resulting in the needle shaped crystal form. This is based both on the results of the different experiments and operating conditions tested by the authors (details experimental results published in the report), and with comparison of the operating conditions and crystal forms produced in the work of previous authors (see above).

Free ammonia will occur as a function of high soluble ammonium ion concentration if the pH is raised, but not under significant aeration which will drive the ammonia away with the air flow.

The authors emphasise that the form of struvite crystal being precipitated may be a key factor in successful phosphate recovery, in particular in influencing the **formation or not of “fines”** (susceptible to be lost from the reactor), the reaction speed of the precipitation, and in the mechanisms of aggregation of the crystals to form larger particles (which will then

settle in the reactor and can be recovered, and readily handled and recycled).

The pilot reactors in this work were operated to optimise production of larger particles, rather than complete removal of soluble phosphorus. After only 2 weeks of operation, aggregation of crystals into particles was noted in the reactors, with granules of around 1mm diameter being successfully formed.

*“Study of the effects of the reactor hydraulics on struvite precipitation at municipal sewage works”, Polish Academy of Sciences/ University of Bielsko-Biala research report, published at <http://www.nhm.ac.uk/mineralogy/P-recovery/> - direct link:*

<http://www.nhm.ac.uk/mineralogy/phos/Suschka2003rep.pdf> J. Suschka\*, E. Kowalski, S. Poplawski

\* Technical University of Lodz, Filial Bielsko Biala, Ul. Willowa 2 - 43-309 Bielsko Biala, Poland  
[jsuschka@ath.bielsko.pl](mailto:jsuschka@ath.bielsko.pl)

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### France

#### **Fate of phosphorus in manure treatment**

In France's main pig producing area, Brittany, limits to nitrogen loads in manure spreading (because of increasing nitrate levels in aquifers) are causing farmers to install aerobic/anaerobic treatment plants to achieve nitrification/denitrification of manure, transferring nitrogen content to atmospheric N<sub>2</sub>. Thirty such farm-scale treatment plants were already in operation in the area in 1999, with many more planned.

Such plants transfer phosphorus, and also potassium as well as metals such as copper and zinc, to the liquid plant effluent and/or to a solid fraction whose form can vary from liquid sludge to a dried, transportable compost or equivalent.

**This study presents mass balances for suspended solids, nitrogen, phosphorus, copper and zinc** for three such full-scale operational farm treatment plants, of similar size (7,200 – 7,800 – 8,300 weaned pigs per year, that is treatment capacities of 16-19 m<sup>3</sup>/day of slurry). The general principle of the three plants was similar: a reception/mixing/holding tank, followed by a

reactor tank subject to alternate aeration and anoxic (non aerated) periods (for nitrification/ denitrification respectively). However, **the configuration of the three plants varied significantly** in aspects such as: prior separation or not of the solid fraction before treatment, presence or not of a decanter, aeration/anoxic period lengths ...

#### **Liquid phosphates**

Only 25-30% of the initial manure phosphorus content was in the solid fraction, after the initial solid/liquid separation, using a screw press auger. This is significantly different from the figures given by Coillard 1998 (see *SCOPE Newsletter n°26*) who indicated 85-90% of pig manure phosphorus was in the solid fraction, after solid/liquid separation using centrifuges. This is because **much of the phosphate in pig manure is contained in small, dense particles**, which do not make up a large mass, but which are highly concentrated in phosphorus. These particles are separated from the liquid in a centrifuge, but not in a screw press auger. This is explained in a paper presented by the authors at the ISAAFPW Conference 2003 (see references).

The three plants studied each transferred around 60% of the total inflow phosphorus to the solid fraction after treatment, with an additional 20-30% (depending on the plant) being also in a solid form, but as suspended solids in the liquid effluent or sludge.

The levels of phosphorus in the liquid effluent from these plants were sufficient, according to the authors, to raise concerns about their possible contribution to eutrophication, given the ready availability and susceptibility to run-off of phosphorus applied to fields in such effluent, particularly if application rates are calculated on the basis of the (low) residual nitrogen content in these liquids.

**The authors suggest that the phosphorus can be orientated towards either the solid or the liquid fraction from a plant**, by modifying the aerobic (tends to fix phosphorus in organic solids) or anoxic (tends to cause phosphorus release to solution) final phase of the treatment.

In this case, plant operators could choose to either optimise phosphorus transfer to solids, where these are in a transportable form (dry compost or similar) which can be moved away from the farm area, or concentrate phosphorus in the liquid fraction then operate a phosphorus recovery process. They suggest that phosphorus could be recovered either by struvite precipitation (magnesium ammonium phosphate) or possibly as potassium struvite (potassium magnesium phosphate) where inadequate ammonium was available following the nitrification/denitrification treatment.

*“Fate of phosphorus from biological aerobic treatment of pig slurry. By-products characterization and recovery”*  
*Environmental Technology*, vol. 24, pages 1323-1330.

<http://www.envirotechnol.co.uk/>

M-L. Daumer, F. Beline, F. Guiziou, CEMAGREF  
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A preparatory version of this paper was presented at the  
2<sup>nd</sup> International Conference on Phosphorus Recovery and  
Recycling, Noordwijkerhout 2001,

<http://www.nhm.ac.uk/mineralogy/P-recovery/> and is  
available at:

<http://www.nhm.ac.uk/mineralogy/phos/Nordwijkerhout/Daumer.doc>

*“The effect of pig diet and solid/liquid separation of pig slurry on phosphorus fractionation”*. Proceedings of the  
9th International symposium on animal, agricultural and  
food processing wastes (ISAAFPW), Research Triangle  
Park, NC, USA, 2003 pages 463-471

<http://www.asae.org/meetings/index.html>

Direct link:

<http://asae.frymulti.com/request2.asp?JID=1&AID=15284&CID=fpw2003&T=3>

M-L Daumer, F. Beline, M. Sperandio - as above.

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### Novaquatis

#### Struvite precipitation from urine

The Novaquatis research package underway at EAWAG Switzerland is looking at routes and technologies for recovery of nutrients from human excrements in wastewater. Urine typically contains 80% of nitrogen, 50% of phosphorus and 90% of potassium in human excrements. Source separation toilets save 80% of water use and would enable recovery of nutrients from urine by struvite precipitation.

Two workpackages in this overall R&D project are looking specifically at struvite precipitation.

A PhD thesis (March 2002-December 2005) is addressing biological and chemical interactions affecting phosphorus precipitation in high strength media.

A second workpackage is researching the thermodynamic and kinetic properties of struvite precipitation in real and artificial urine, in particular looking at the effects of inorganic complexes present in urine. First results suggest that organic complexing agents do not influence struvite solubility, and that struvite precipitation rates are controlled by surface reaction.

Novaquatis web site:

<http://www.internal.eawag.ch/~maurer/Nova/nova.htm>

### North Carolina

#### Struvite recovery from livestock lagoon liquor

Keith E. Bower's thesis, published by North Carolina State University 2002, presents three years of work investigating parameters and testing reactor design for phosphorus precipitation as struvite from anaerobic livestock slurry lagoon effluents. The published thesis includes an initial literature review, results of laboratory beaker tests, reactor modelling, design and laboratory tests of a continuous mode struvite crystalliser, and finally design and operation of a field-scale reactor at a hog farm sized to treat the wastewater flow to irrigation from 1,000 pigs (11 litre bed volume) and achieving peak total phosphorus and orthophosphate phosphorus removal of 82% and 87%, respectively. All of the experimental work used real pig manure lagoon liquors.

A preliminary literature review concluded that struvite precipitation offered the potential to remove phosphorus from North Carolina pig manure lagoon effluent in a recoverable form. It also provided thermodynamic and kinetic information regarding struvite precipitation, necessary for reactor design.

#### Initial beaker tests

**Initial parameter testing experiments were carried out using lagoon effluents from two local pig farms:** one covered, ambient temperature in-ground digester (pH 7.8, total phosphorus [TP] 62 ppm, total ammoniacal nitrogen [TAN] 30 ppm, magnesium [Mg] 30 ppm); one anaerobic lagoon (pH 7.6, TP 47 ppm, TAN 1087 ppm, Mg 19 ppm).

49% and 73% respectively of the TP was present as soluble orthophosphate, and thus potentially available for struvite precipitation.

Magnesium bicarbonate and ammonia were used to provide magnesium ions and increase the pH for struvite precipitation. Beakers were stirred for 20 minutes then stood overnight.

As expected, phosphate precipitation increased with magnesium addition and/or ammonia addition.

#### Magnesium availability

Literature data showed that magnesium solubility is higher in solutions containing species which will lose protons when the pH increases and before the pH reaches 10, for example ammonium. This effect can be accentuated by bubbling CO<sub>2</sub> through the solution and this was experimentally demonstrated. **Ammonia addition was also shown to be effective in precipitating struvite**, at addition rates as low as 40 ppm, with the advantage of adding no "foreign" ion not already present in the liquor.

**Calcined magnesium carbonate was identified as an optimal magnesium source** when rendered soluble by CO<sub>2</sub> addition. This magnesium product is readily available (used as a livestock feed supplement). CO<sub>2</sub> could in theory be collected from lagoon digester biogas.

#### Reactor design

From the beaker experiment results, it was concluded that the struvite precipitation reaction was sufficiently rapid for a continuous mode fluidised bed reactor to be effective. Reactor design needs to prevent the loss of fine particles with the fluid flow out of the top of the reactor, so a cone shaped reactor was used.

Reactor operation was modelled using three hypothesis: **MLMB** = mixed liquid, mixed bed (liquid perfectly mixed and particle sizes mixed throughout the bed); **PLMB** = plug flow (liquid moves up the reactor linearly without mixing vertically), mixed bed; **PLCB** = plug flow, classified bed (that is, the particles in the bed are stratified by size).

#### Laboratory test reactor

The laboratory scale test reactor was a 40 cm high, transparent plastic cone, with 1.27 cm internal diameter at the base (reactant and fluid injection zone), 10.2 cm at the top (the bed top generally stayed at around two-thirds of the cone height, thus forming a bed occupying about 0.45 litres). Liquid throughput rate used in the reactor was 34 – 57 litres/hour.

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**Fifty different reactor runs of 1.5 – 5 hours duration were carried out in the lab**, using real lagoon liquors as indicated above, and testing ammonia addition rates of 0 – 200 ppm TAN, magnesium addition rates of 0 – 60 ppm Mg, with and without seeding. Results are presented in detail in the published thesis available online, including statistical analysis.

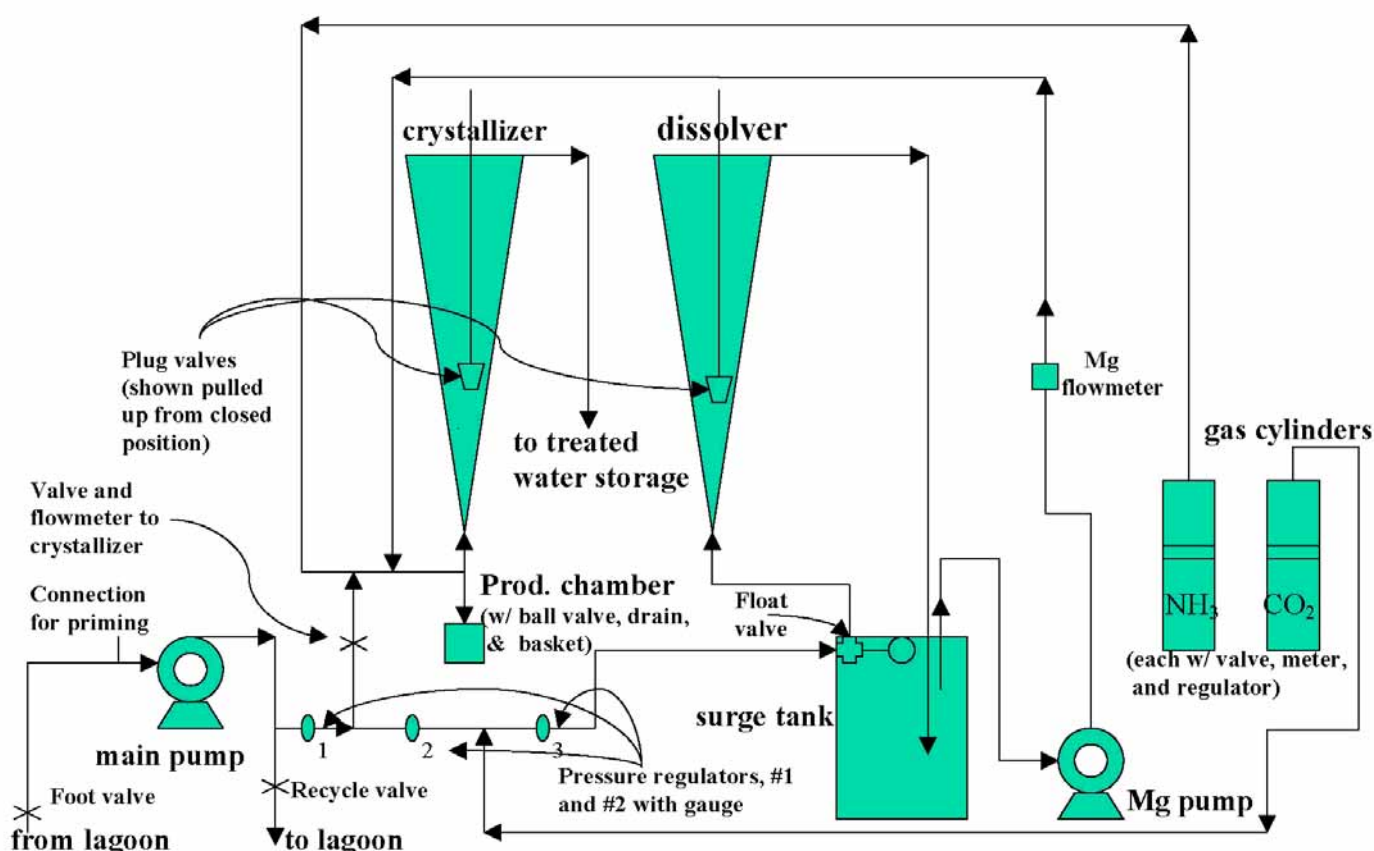
Reductions in total phosphorus (TP) and orthophosphate phosphorus (OP) of 58% and 74% were achieved in the second set of runs.

The PLMB reactor operation model (see above) showed to best fit to the reactor behaviour. Results also suggested a surface-area specific rate constant in the range of 10-15 dm/hour.

### Field scale reactor

The successfully tested laboratory reactor design was scaled up to 455 litres/hour, to accommodate irrigation water throughput, corresponding with 1,000 hogs averaging 58 kg each. Channelling of fluid flow (instead of mixing) had been observed in the lab scale reactor, and to reduce this effect the field scale reactor was built with a more gradual upward increase in diameter (steeper cone sides): height 152 cm; internal top and bottom diameters 25.4 cm and 3.8 cm (the bed top generally stayed at around two-thirds of the cone height, thus forming a bed occupying about 11 litres). Two identical cones were used: one as the struvite precipitation fluid bed crystalliser, the other for dissolving magnesium oxide (magnesium chloride was used in later runs to achieve the control on Mg concentration needed for factorial experiments). Ammonia was injected into the base of the reactor from a pressurised supply, alongside the magnesium solution

### Scheme of field reactor (U.S. patent application no. 10/659,239)



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Fifteen initial runs of 0.5 – 30 hours were carried out achieving OP reductions of 22-58% when ammonia injection was operating. Three further runs were then carried out, each testing 18 different operating parameters combinations (in a randomised order). In these runs, TP and OP removal averaged 70 and 77% when ammonia and magnesium were added, and 82% and 87% at optimum conditions.

The author concludes that the reactor performed at least as well in the field as had been initially envisaged at the start of the work. Phosphorus removal was higher than for theoretical calculations of struvite precipitation (other phosphate compounds were maybe also being precipitated to a small extent). The system “shows promise for application in treating North Carolina livestock waste lagoon effluent and removing most phosphorus”.

*“Development of a struvite crystallizer for reducing phosphorus in effluent from livestock waste lagoons”, PhD Dissertation, North Carolina State University, Biological and Agricultural Engineering.*

K. Bowers [keithebowers@yahoo.com](mailto:keithebowers@yahoo.com)

Full text available online (174 pages) at:  
<http://www.lib.ncsu.edu/theses/available/etd-11042002-164459/>

## Sewage treatment

### Nutrient removal

### EU Commission report into Urban Waste Water Treatment (UWWT) Directive implementation

The EU Commission’s previous reports into implementation of the 1991/271UWWT Directive underlined the failure of many Member States to have introduced adequate sewage treatment collection and treatment within the required deadlines, and particularly the failure to implement nutrient removal by 1/1/1998 as required by the Directive for any agglomeration of 10,000 person equivalents or more discharging into waters potentially susceptible to

eutrophication (“Sensitive Areas”). As indicated in [Scope Newsletter n°45](#) the Commission ran an active media campaign in 2001-2002 (“*Name and shame*”) denouncing the non implementation of the Directive, and in particular the failure of Member States to adequately designate Sensitive Areas. The European Parliament also voted a *motion* on 14<sup>th</sup> March 2002 underlining the importance of implementation of this Directive.

The Commission’s third report into implementation of this Directive, [COM\(2004\)248](#), covering the period up to 2003, is therefore of considerable political significance. This report confirms the continuing failure of Member States to implement in particular the nutrient removal requirements of this Directive.

**As of 1<sup>st</sup> January 2002, some 42% (by organic load) of wastewater from the 5,500 agglomerations identified by Member States as discharging into “Sensitive Areas”, was still not subject to adequate treatment (nutrient removal). 91% of the wastewater from these agglomerations was being effectively collected by 2002.**

### Failure to designate “Sensitive Areas”

The Commission particularly criticises the failure of certain Member States to correctly designate as “Sensitive Areas” water bodies which are in fact subject to eutrophication or potentially so. The Commission employed ERM Consultants from 1998-2000 to verify the Sensitive Area designation by Member States and [this audit](#) revealed a number of shortcomings. These had already been emphasised in the IEEP report for CEEP (see [SCOPE Newsletter n°34](#)) and in *Scope Newsletter n°s 52, 45 and 46*.

As well as inland waters inadequately taken into account by Member States, the Commission emphasises that major coastal areas including the Baltic Sea, the Adriatic and the North Sea (from Northern France to Sweden) are in effect “Sensitive”.

The Commission indicates that certain Member States (in particular Spain, for the Ebro and Guadalquivir rivers) and Italy (for the Po) have failed to plan nutrient removal for agglomerations in the (upstream)

catchment of Sensitive Areas. The Commission points out that since nutrients are carried downstream, designation of part of a catchment must inevitably mean effective designation (and nutrient removal requirements) for all agglomerations upstream of this area.

The Commission indicates that the legal actions it has brought against certain Member States has been effective in leading to improvements in Sensitive Area designation and that most member states now plan to achieve conformity with the nutrient removal requirements of the Directive by 2005 – 2008.

**Germany and The Netherlands have achieved respectively 90% and 79% total removal of phosphorus from sewage** (in conformity with the Directive) and 74% and 66% removal of nitrogen (not conform). These States have designated some time ago their whole territory as Sensitive Areas

### **Inadequate sewage treatment**

Particularly poor performing Member States, according to the Commission's report, include **Belgium**, where a number of large agglomerations (including Brussels, Liège) do not even have adequate sewage collection systems, and only 22% of sewage flowing into Sensitive Areas is treated adequately (the whole of Belgium has now been classified as a Sensitive Area).

**Greece** achieves only 10% adequate treatment of discharges into Sensitive Areas.

**Spain** has still not adequately designated Sensitive Areas (the Commission has identified 44 areas which should be but have not yet been designated). Spain also failed to provide performance monitoring results regarding treatment, but the Commission estimates that only 25% of wastewater flowing into Spain's Sensitive Areas (as designated to date) is treated adequately.

**France** is also pinpointed as not having adequately identified a number of Sensitive Areas, and is currently facing legal action from the Commission on this issue. France also failed to provide adequate monitoring data. The Commission estimates that only 36% of

wastewater flowing into French Sensitive Areas (as designated to date) is treated adequately.

**Italy**, as indicated above, is criticised for inadequate designation of Sensitive Areas and all catchments of the Po and the Adriatic will need to be designated. Cities currently without adequate sewage treatment or appropriate nutrient removal include Milano (under construction), Venice, Rimini, Ravenna, Cagliari, Como, Trieste and Florence.

### **Legal pressure**

The Commission is backing up its report with **legal actions against Member States**, announcing actions against 8 Member States (Commission [Press Release](#) 13<sup>th</sup> January 2004, ref. IP/04/39). The legal actions are against **Belgium, France, Germany, Greece, Ireland, The Netherlands, Portugal and Spain** for non-compliance with different EU water regulations including dangerous substances discharged to water, drinking water, beach pollution, shellfish waters, the nitrates directive.

In particular, **France is being referred to the European Court of Justice** for failure to provide information concerning implementation of the Urban Waste Water Directive, and in particular concerning designation of Sensitive Areas. **Spain** has received a final written warning for contravening this Directive and the Bathing Water Directive in the region of Valencia with inadequate sewage treatment leading to beach water pollution.

On 25<sup>th</sup> March 2004, the [conclusions](#) of the Advocate General to the European Court of Justice to the European Court suggested that **France should indeed be condemned for failure to designate as Sensitive areas which are subject to eutrophication** in the Seine-Normandy, Artois-Picardie and Rhône-Mediterranean-Corsica basin authority areas, and consequently for failure to ensure adequate treatment of sewage in these areas. This follows condemnation of France in the European Court in a separate procedure for failure to take account of nitrates contribution to eutrophication (see [SCOPE Newsletter n°46](#)).

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### Sweden and Finland

However, the Commission is also pointing to Member States usually reputed for the quality of their water treatment, and has sent Final Written Warnings to both **Sweden and Finland**, indicating that they are failing to ensure adequate nitrogen removal in sewage works discharging in the Baltic Sea (Commission [Press Release](#) 2<sup>nd</sup> April 2004, refs. IP/04/449).

The Commission has also published a [summary](#) of **recent European legal judgements** regarding environmental issues, which provides information on a number of different legal actions brought by the Commission against Member States for non compliance with different EU Directives concerning water quality and sewage treatment.

*EU Commission Third Report into implementation of the Urban Wastewater Treatment Directive 91/271, document refs. COM(2004)248 : [http://europa.eu.int/eur-lex/en/com/rpt/2004/com2004\\_0248en01.pdf](http://europa.eu.int/eur-lex/en/com/rpt/2004/com2004_0248en01.pdf)*

*EU Parliament Resolution March 2002:*

[http://www.europarl.eu.int/plenary/default\\_en.htm](http://www.europarl.eu.int/plenary/default_en.htm) then go to "Texts adopted by Parliament" -> "By date" -> March 14<sup>th</sup> 2002 or by number A5-0459 or P5\_TA(2002)0122

[http://www3.europarl.eu.int/omk/omnsapir.so/calendar?AP=P=PDF&TYPE=PV2&FILE=P5\\_TA\(20020314\)0122en.pdf&LANGUE=EN](http://www3.europarl.eu.int/omk/omnsapir.so/calendar?AP=P=PDF&TYPE=PV2&FILE=P5_TA(20020314)0122en.pdf&LANGUE=EN)

*EU sewage treatment "name and shame" press release*

[http://europa.eu.int/comm/environment/nsf/city\\_sewage.htm](http://europa.eu.int/comm/environment/nsf/city_sewage.htm)

*Commission permanent update page summarising infringement procedures:*

[http://europa.eu.int/comm/secretariat\\_general/sgb/droit\\_com/infractions](http://europa.eu.int/comm/secretariat_general/sgb/droit_com/infractions)

*Commission summary of leading cases in European Environment law*

[http://europa.eu.int/comm/environment/law/leading\\_cases\\_en.pdf](http://europa.eu.int/comm/environment/law/leading_cases_en.pdf)

*Advocate General to the European Court conclusions concerning France's failure to adequately designate "Sensitive Areas", refs. C-280/02 Opinion 2004-03-25:*

<http://www.curia.eu.int/jurisp/cgi-bin/form.pl?lang=en&Submit=Submit&docrequire=alldocs&numaff=c-280/02>

## The Scope Newsletter

The SCOPE Newsletter is produced by the Centre Européen d'Etudes des Polyphosphates, the phosphate industry's research association and a sector group of CEFIC (the European Chemical Industry Council).

The SCOPE Newsletter seeks to promote the sustainable use of phosphates through recovery and recycling and a better understanding of the role of phosphates in the environment.

The SCOPE Newsletter is open to input from its readers and we welcome all comments or information. Contributions from readers are invited on all subjects concerning phosphates, detergents, sewage treatment and the environment. You are invited to submit scientific papers for review.

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