

RE-LIVE WASTE- Improving innovation capacities of private and public actors for sustainable and profitable REcycling of LIVEstock WASTE

Project title and acronym	RE-LIVE WASTE
Work Package	WP3
Deliverable n. and title	D382 TRAINING MATERIALS, PRESENTATION
Responsible Partner	SERECO BIOTEST
Participating partners	ALL PARTNERS
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Dissemination level ¹	PARTNERSHIP

• ¹ Partnership; JS; Thematic community; MED community; Public

REL-LIVE WASTE
STEERING COMMITTEE MEETING

Limassol, 22nd October 2019

Sereco-Biotest - Italy

TRAINING
Part I Introductory issues

**Dr. Biologist Luca Poletti –
Dr. Agr. Roberto Poletti**

ENVIROTECH R&D CONSULTANCY HUB

50 PUBLICATIONS

7 PATENTS

PARTICIPATION TO FUNDED RESEARCH PROJECTS



SUSTAINABLE FERTILIZATION

NUE Nutrient Use Efficiency

STIMULATING RESTITUTION → fertilization not to be regarded a mere chemical supply of nutrients (mass balance) but as a stimulation practice of the plant biological functions

UPGRADING secondary raw materials and organic wastes

-Savings on

- Synthetic chemicals
- Administration reduction (low release fertilizers: SCU, Sulphur Coated Urea, Struvite; controlled release fertilizers: polymer coating)

ENVIRONMENTAL IMPACT REDUCTION → Circular  economy

- Run-off (NO_3) and volatilization (NH_3)
- Soil pollutant burden (eg: Cadmium)

CROP QUALITY

Organoleptic (es: dimension, homogeneity, fruit colour)

Technological (es: protein content of durum wheat, malting barley, ecc..)

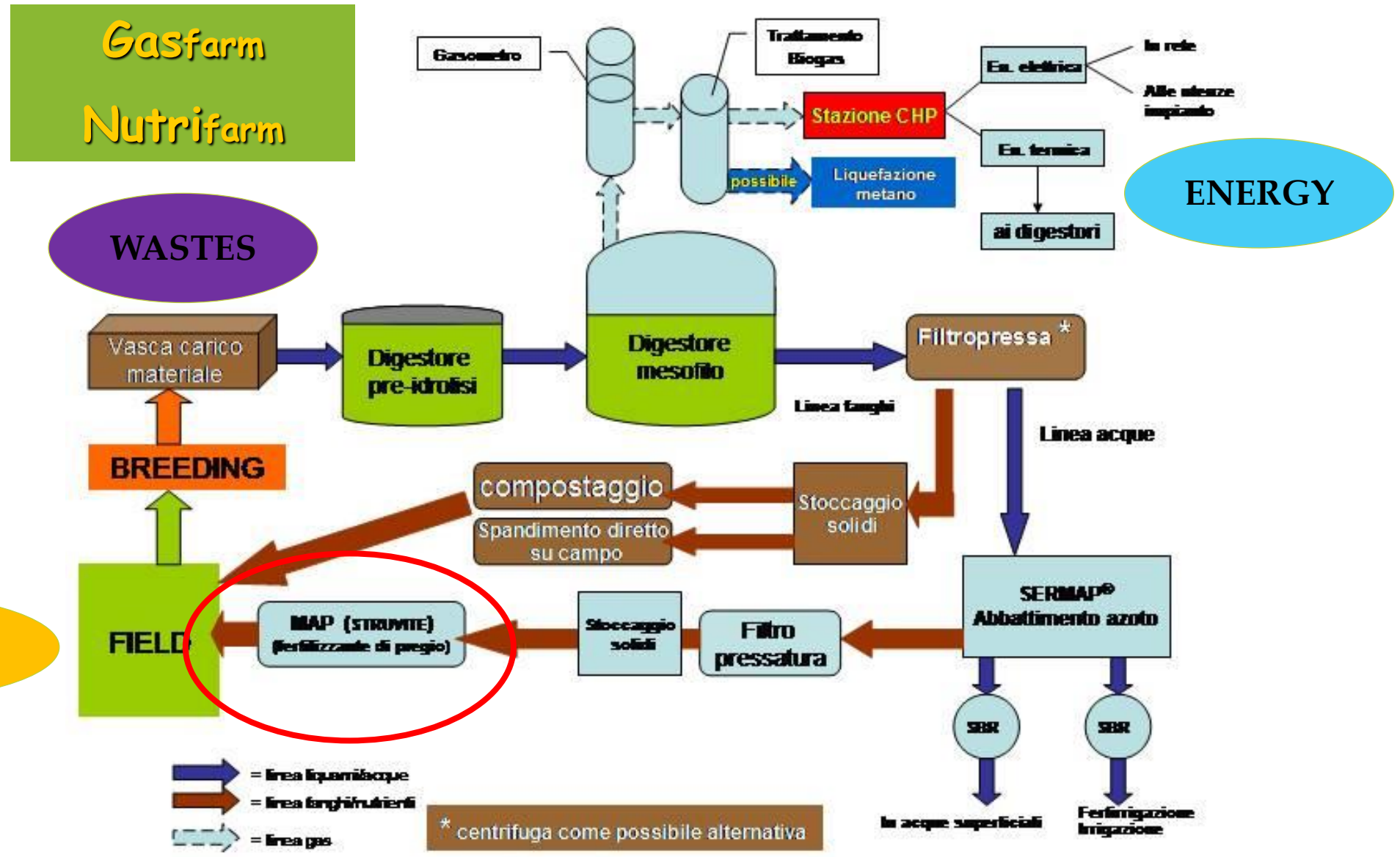
Nutritional (es: nitrate excess on leaf vegetables)

Green labelling



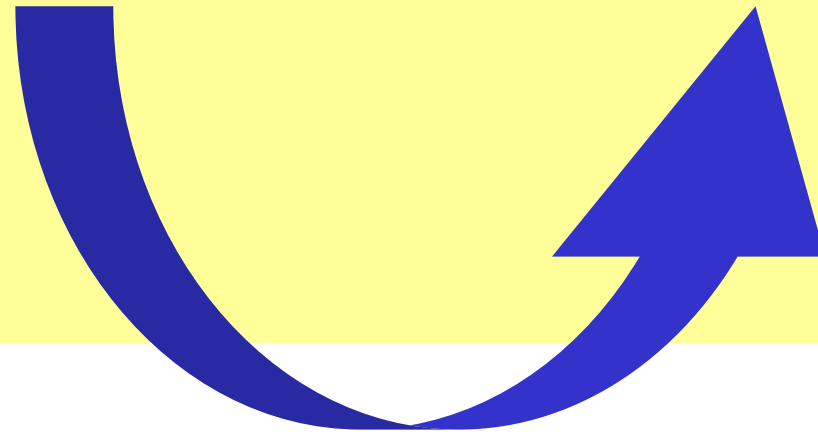
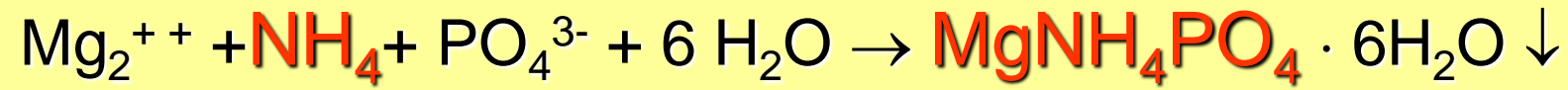
AN EXAMPLE OF CIRCULAR AGRO-ECONOMY

Energy&Material Flow Optimization (EMFO) in agro-energetic cycles



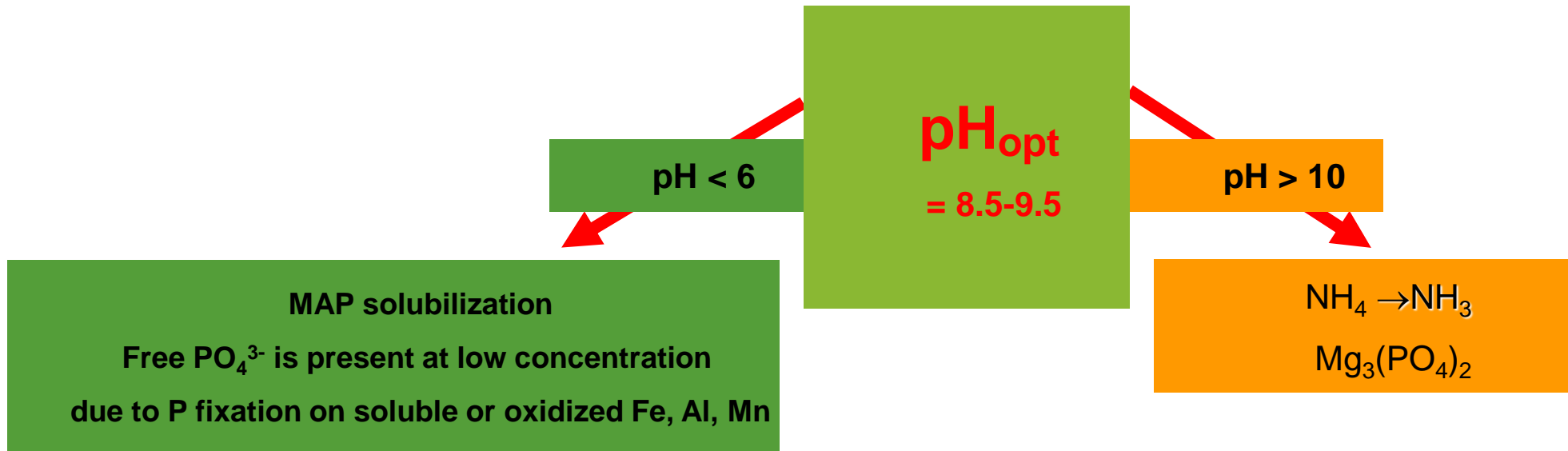
MAP: MAGNESIUM-AMMONIUM- PHOSPHATE PRECIPITATION

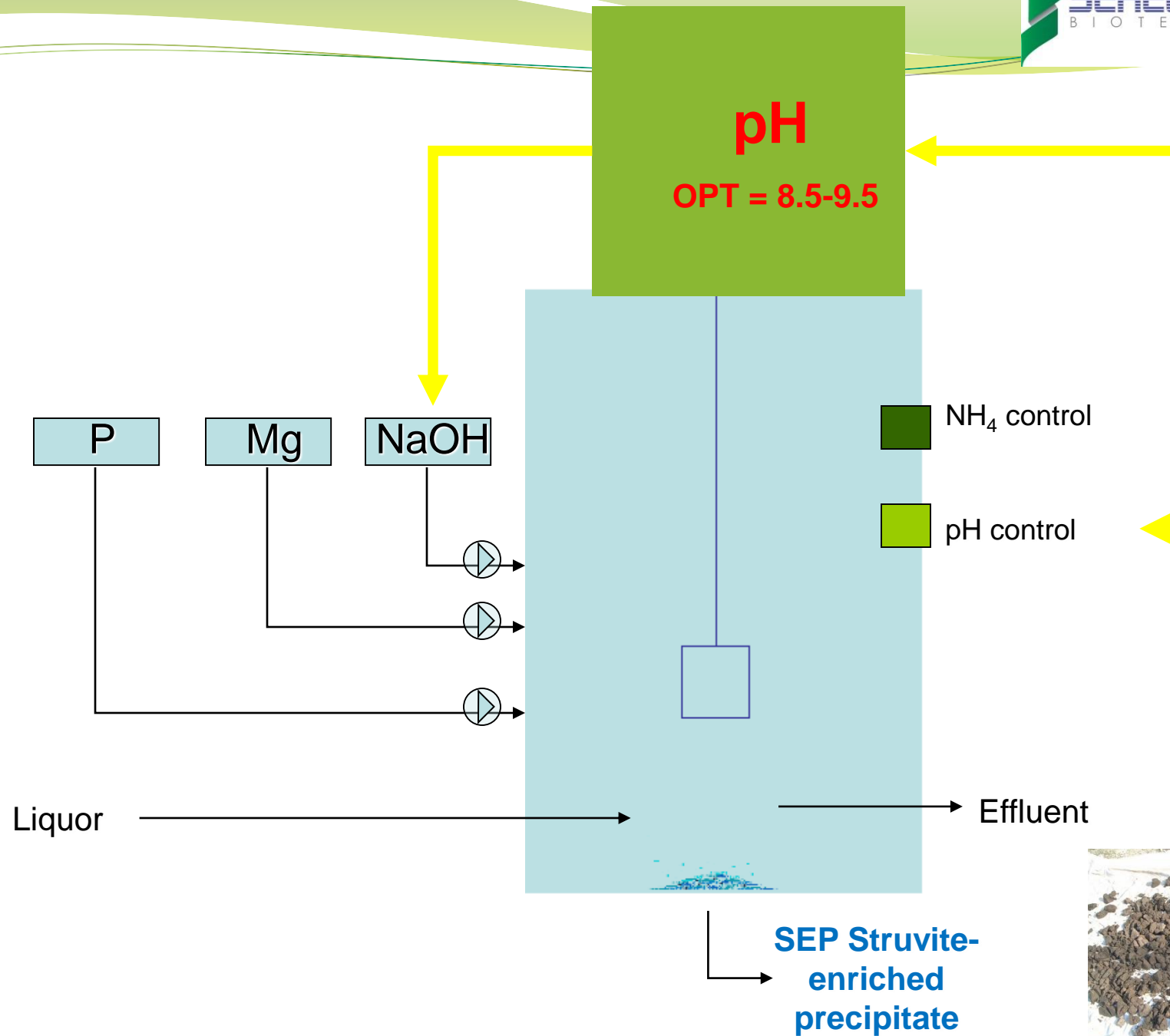
THE MAP PROCESS IS BASED ON THE PRECIPITATION OF THE AMMONIUM ION ACCORDING TO THE FOLLOWING REACTION



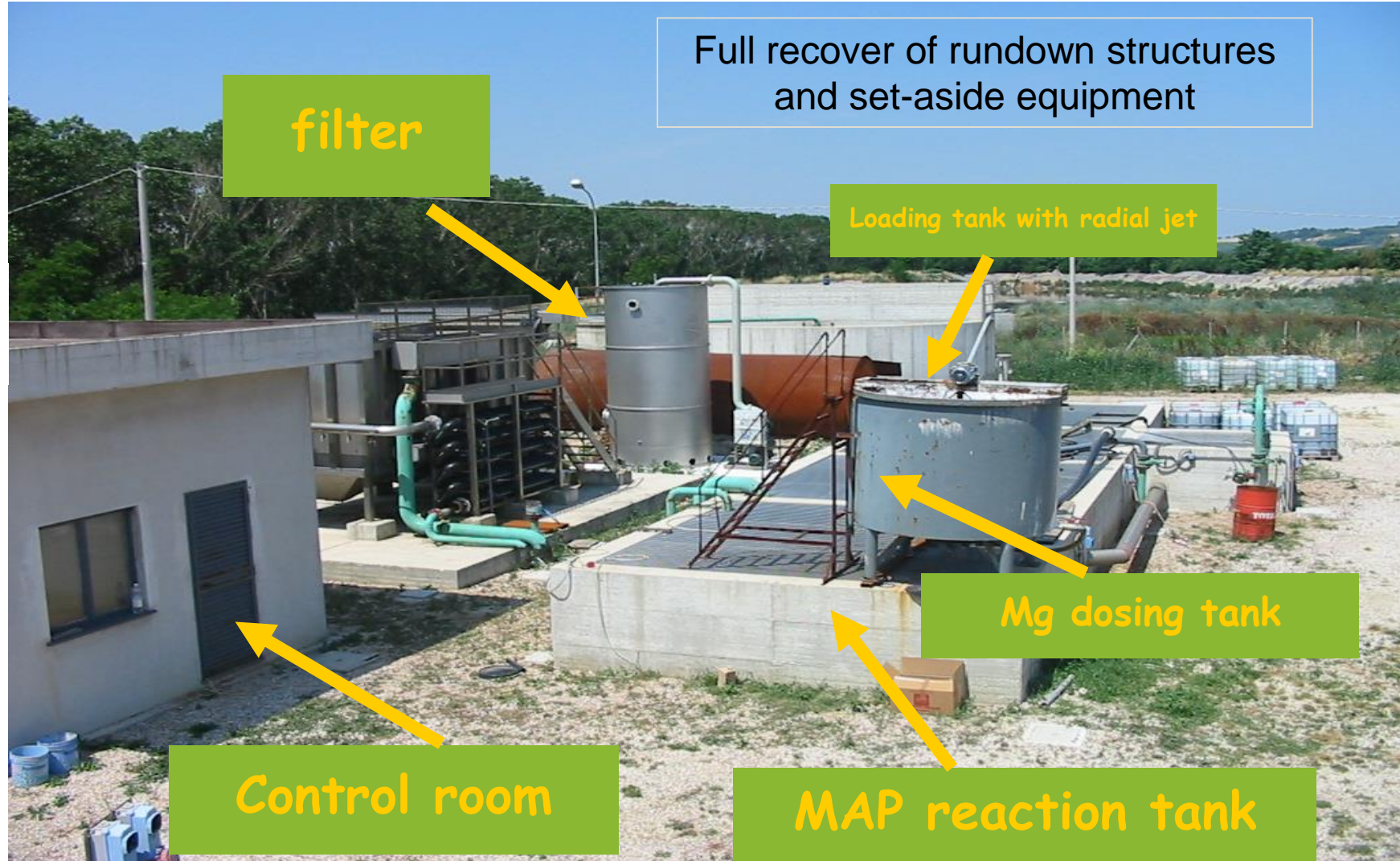
MAP: MAGNESIUM-AMMONIUM-PHOSPHATE PRECIPITATION

The crystalline precipitatin of struvite is strictly dependent on the reaction pH





EXPERIMENTAL PLANT AT THE AD DIGESTION STATION IN BETTONA (ASSISI)



Plant details



SENSORS



**Electronic
control unit**



Electrical control panel

MAP – mixed liquor (slurry)



Organic Struvite-
Enriched Precipitate
O-SEP



AGRONOMICAL CONSIDERATIONS

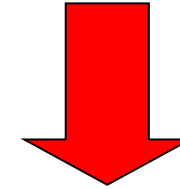


MAP (Magnesium Ammonium
Phosphate)
ENT (Essiccato Naturale Tecnico)



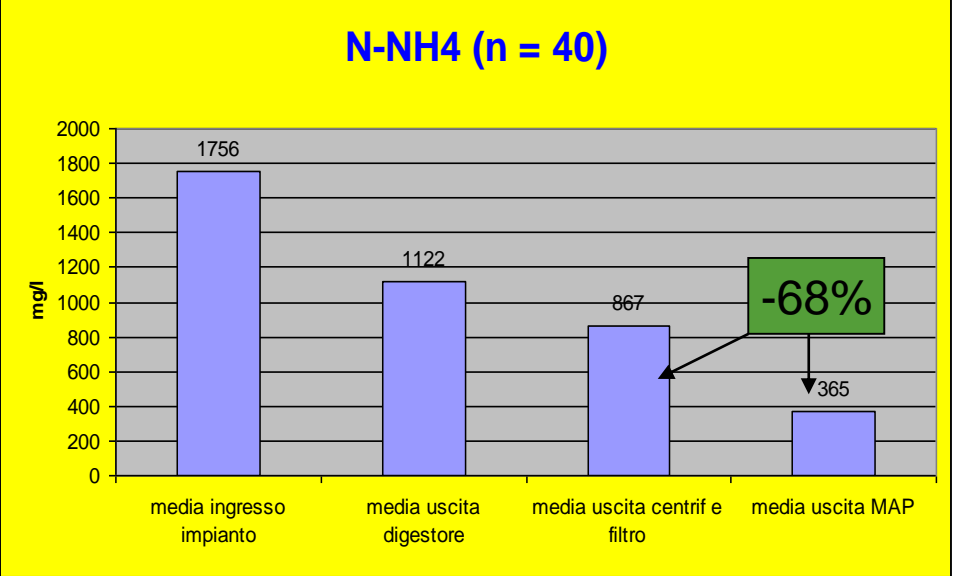
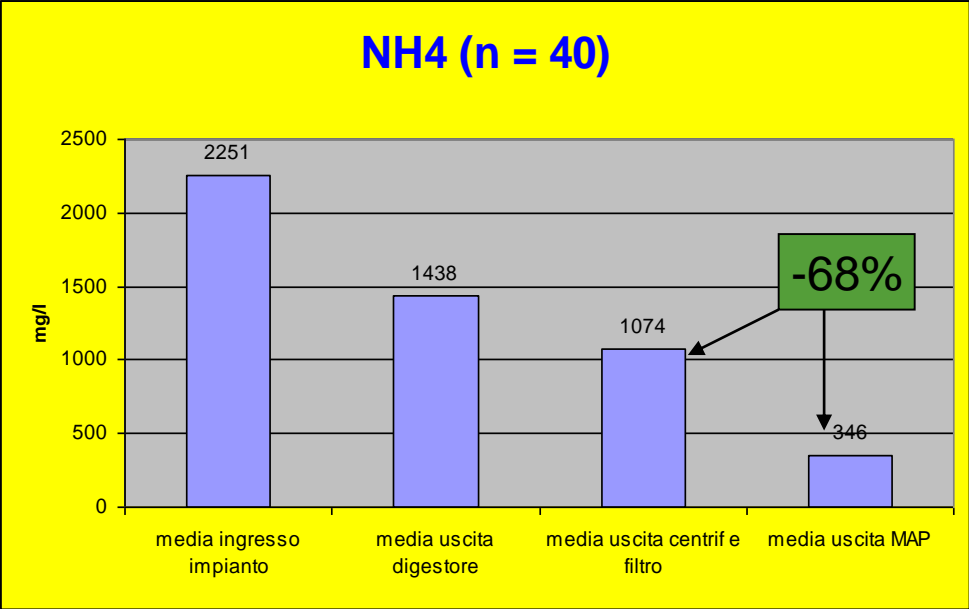
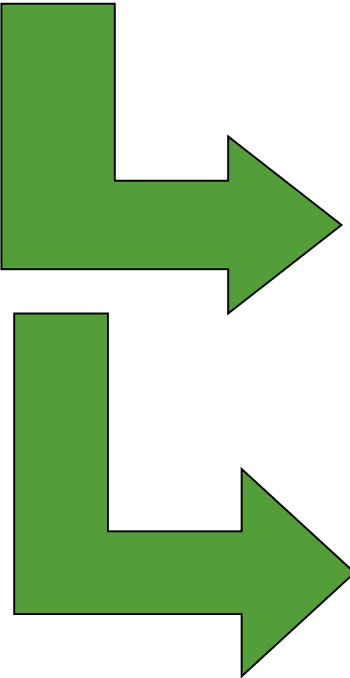
- High thermodynamical stability (weight loss 53% @ 200°C – DSC and TGA measurements)
- MAP water solubility is very low and pH-dependent. Below 10 C° is practically insoluble. **pKSO** (Conditional Solubility Product) varies from 5,4 @ pH 6,3 , to 7 @ pH 7 until 9,4 @ pH 9,5 .

NH₄ ABATEMENT EFFICIENCY



Run	Data	RM	NH4 ini	NH4 fin	Eff abbattimento
		NH4:Mg:PO4	mg/l	mg/l	%
1	23.12.04	A	930	450	51,6
2	18.01.05	B	1503	432	71,3
3	01.02.05	C	860	420	51,2
4	02.02.05	B	917	200	78,2
5	03.02.05	D	990	220	77,8
6	04.02.05	A	765	460	39,9
7	17.03.05	F	900	270	70,0
8	17.03.05	E	1560	325	79,2
9	18.03.05	E	1030	340	67,0
10	18.03.05	E	1288	350	72,8

**AVERAGE NH₄
 ABATEMENT
 RATE:
 68%**



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TRAINING

Part II How to improve the quality of struvite through seeding

**Dr. Biologist Luca Poletti –
Dr. Agr. Roberto Poletti**

A number of factors affects the QUALITY of a crystalline product

effectiveness of downstream processes → commercial value of the final product

Quality factors (crystal properties)	Processing characteristics
<ul style="list-style-type: none">• CSD → small crystals have a higher tendency to caking• Particle surface area• Crystal shape• Polymorphism• Mother liquor inclusions in the crystal (can lead to breakage)• Impurities uptake• Degree of agglomeration	<ul style="list-style-type: none">• Filtration• Drying rates• Particle flow-properties• Bulk density• Packing characteristics (e.g. propensity to cake)

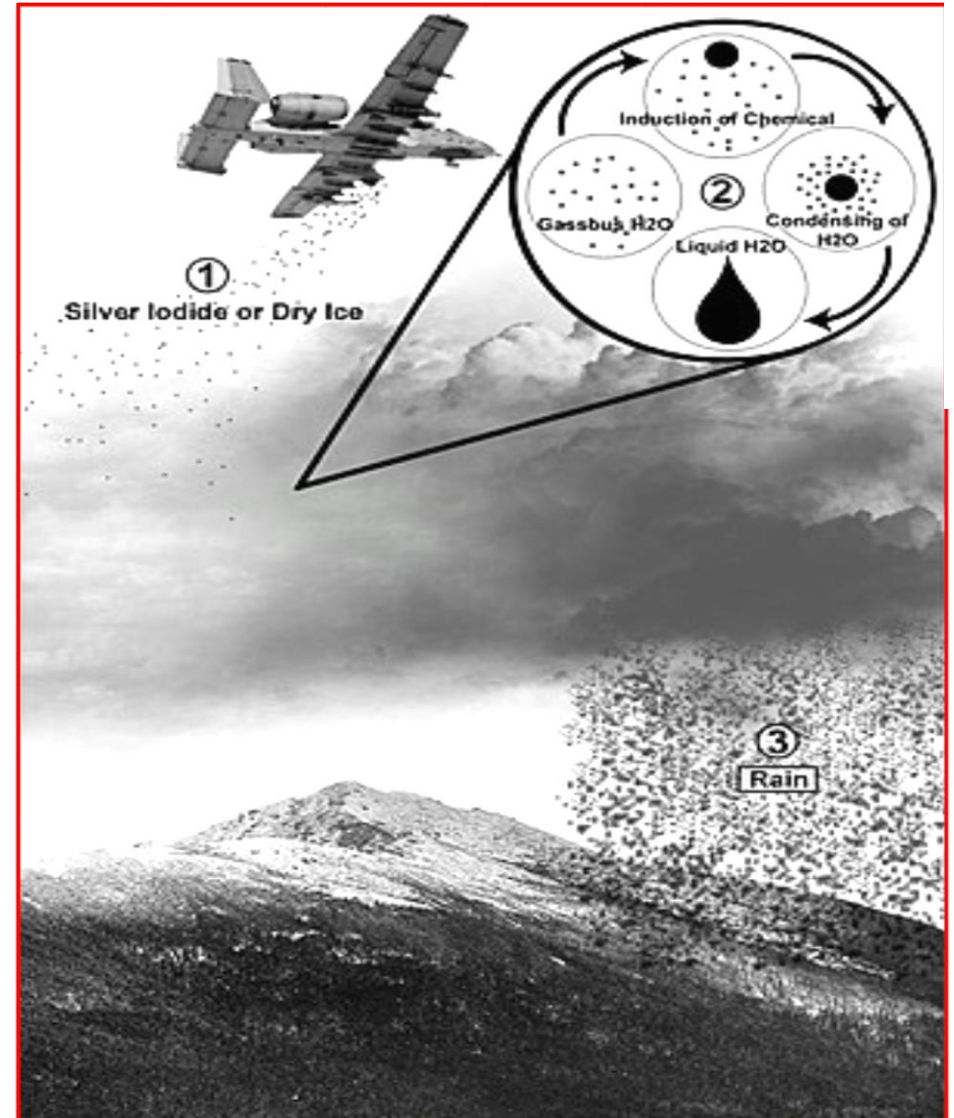
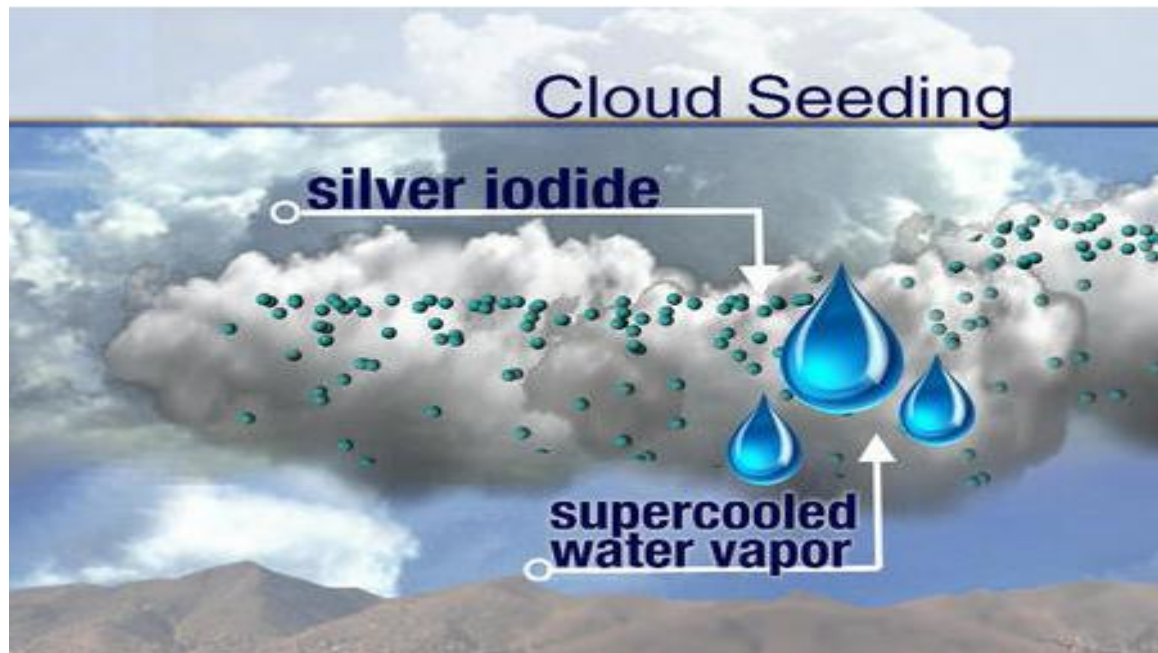
PARTICLE DESIGN



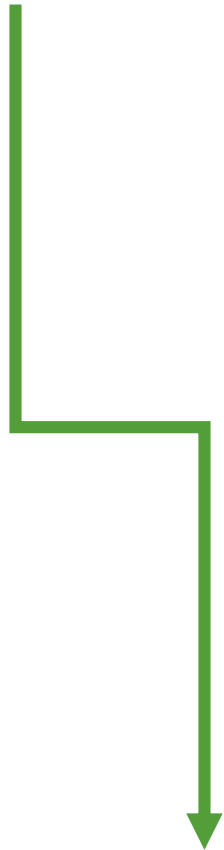
Ice-cream texture affected by crystallization

One way of driving the characteristics of crystals within a crystallization process is through SEEDING, that is by introducing foreign particles in the system to promote crystallization in the right way

CLOUD SEEDING FOR RAINMAKING



Goals of seeding

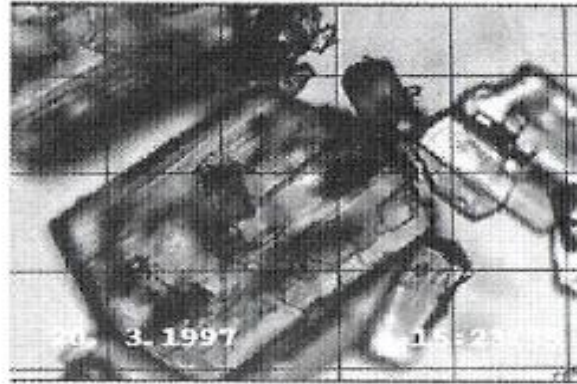
- To inhibit supersaturation avoiding the onset of an excessive proliferation of crystals bearing low mean dimensions
 - To control induction time (time elapsing btw. supersaturation and crystal growth)
 - To act as a «template» for the accumulation of crystallizing material
 - To control particle properties (polymorphism and CSD)
- 

SEEDING CAN OCCUR WITHOUT WE WANTING IT...

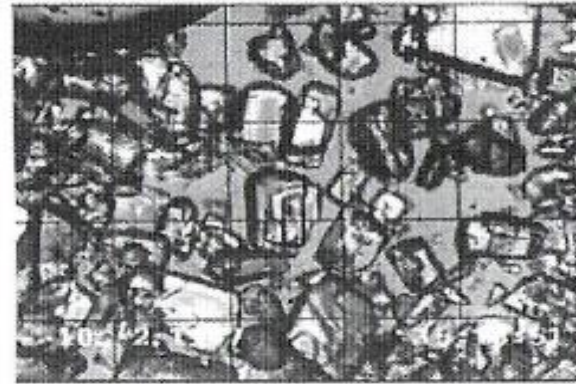
- The most natural form of seeding is the so-called «secondary nucleation» that occurs when the already-formed crystals coming up from the primary nucleation (**parent crystals**) break into new nuclei
- Seeding can also occur naturally for the unavoidable presence of foreign particles (dust, dirt, etc...) which is more than highly likely in manures, digestates and other solid-rich matrices. In this case we refer to «heterogeneous primary nucleation»

...BUT CAN BE TRIGGERED TECHNOLOGICALLY

- **Factors affecting technological seeding procedures:**
 - Number and size of seed particles
 - Surface area
 - Isomorphism
 - Seed purity and inertness to crystallizing liquor
 - Addition timing
 - Seed quality and form (e.g. micronized, milled)



Spontaneous nucleation
evaporative crystallization



Cooling crystallization
0.1% seeds



Cooling crystallization
0.5% seeds



Cooling crystallization
1% seeds

**Particle size of a crystalline final product as a result of the variation in seed amount
Seeding comes off if the particle number remains constant with particle size increasing →
crystal growth and no nucleation**

Different seed material used in struvite precipitation and their effects on recovery (1)

S. No.	Seed used	Source of struvite	Seed size (μm)	Effects on struvite production	Reference
1	Struvite	Synthetic liquor	1000	<ul style="list-style-type: none"> Production of struvite fine as product Seeding is insignificant (process appeared "self-seeding") 	Regy et al. (2002)
		Synthetic liquor and Sludge from wastewater plant	45-63	<ul style="list-style-type: none"> Crystals have similar shape with seed (no phase transformation during growth) 	Ali (2005)
		Landfill leachate	75-150	<ul style="list-style-type: none"> Effectiveness of seed requires consideration of pH (pH 9 being optimum) 	Kim et al. (2006)
		Synthetic liquor	NR	<ul style="list-style-type: none"> Enhancement of crystallization by 19% at low P concentration Increased crystal size, settle ability 	Liu et al. (2008)
		Synthetic liquor	250-500	<ul style="list-style-type: none"> No enhancement of P recovery and reduction in induction time 	Rahaman et al. (2008)
		Coking wastewater	NR	<ul style="list-style-type: none"> Increase in recovery by approximately 5% (at pH 9.5) No effect of overdosing of seed on recovery (pH 9.5) 	Zhang et al. (2009)
2	Coarse sand	Synthetic wastewaters	NR	<ul style="list-style-type: none"> Reduction in induction time upto 75 min depending upon super-saturation 	Liu et al. (2011)
		Synthetic liquor	30-50	<ul style="list-style-type: none"> Similar shape of struvite with seed 	Mehta and Batstone (2013)
		Fertilizer wastewater	NR	<ul style="list-style-type: none"> Increase in rate of crystallization (by 21%) and size of crystal (from 1.72 to 2.08 μm) 	Yu et al. (2013)
		Synthetic liquor	200-300	<ul style="list-style-type: none"> No fixation of struvite on sand surface 	Regy et al. (2002)
3	Fine sand	Synthetic liquor	150-200	<ul style="list-style-type: none"> Strong primary nucleation and formation of fine 	Regy et al. (2002)
4	Borosilicate glass	Synthetic liquor	45-63	<ul style="list-style-type: none"> Slower reaction rate compared to struvite seed 	Ali (2005)
5	Sand grain/quartz particle	Sludge liquor in wastewater plant	210-350	<ul style="list-style-type: none"> Recovery of 80% of P onto seed bed 	Battistoni et al. (2000)
		Synthetic liquor	45-63	<ul style="list-style-type: none"> Slower reaction rate compared to struvite seed 	Ali (2005)
6	Phosphate rock	Dairy effluent	NR	<ul style="list-style-type: none"> No effect mentioned on crystal 	Massey et al. (2007)
7	Stainless steel mesh	Synthetic liquor	1000 μm hole	<ul style="list-style-type: none"> No significant increase in crystallization Reduction in struvite fine particle 	Le Corre et al. (2007)
8	Pumice stone	Synthetic liquor	NR	<ul style="list-style-type: none"> No effect of seed dosing on recovery Co precipitation of Ca & silica on seed 	Pakdil and Filibeli (2008)



Different seed material used in struvite precipitation and their effects on recovery (2)

Types of seed	Seed	Size of seed (μm)	Seed dose (g/L)	Effects on P recovery	Source of P (P concentration)	Recovery products	Products size (μm)	HRT	Phosphate recovery potential	Reactor types	Development scale	References
Natural materials	Grain/quartz particle	210–350	≈ 12	Fixation of P on seed bed	Sludge liquor in wastewater plant (30–60 mg P/L)	MAP	–	About 100 min	Recovery of 80% of P onto seed bed	FBR	Laboratory-scale	Battistoni et al. (2000)
	Fine sand	150–200	–	Strong primary nucleation and formation of fine	Synthetic liquor (250–300 mg P/L)	MAP	–	Over 100 min	Strong primary nucleation and formation of fine	SR	Laboratory-scale	Regy et al. (2002)
	Struvite	–	–	Seed crystal inoculation increased the rate of MAP crystallization by 20.86%	Fertilizer wastewater (–)	MAP	30.35	About 30 min	Enhancement recovery rate by 5% and crystallization size compared to un-seeded conditions.	Batch MAP reactor	Laboratory-, pilot-, and full-scale	Yu et al. (2013)
	Struvite	53–297	–	Crystals have similar shape with seed; promote struvite crystallization	Synthetic wastewater (300 mg P/L)	Struvite	50–1700	9.5 h	High P-recovery rate up to 95.8%; low microcrystalline ratio to 3.11%	FBR	Laboratory-scale	Shih et al. (2017)
	Struvite	–	–	Self-seeding by auto-crystallization	Wastewater from the wastewater treatment plant of Girona (Spain) (368.5 ± 14.9 mg P/L)	struvite	80–1000	138.6 h	High P-recovery rate up to 95.4%	Air-lift reactors	Laboratory-scale	Tarragó et al. (2016)
	Calcite	150–210	30	Precipitating crystals on the surface of seeds; dissolving Ca^{2+}	Anaerobic supernatant (17.5–28.2 mg P/L)	HAP	26.32	35 min	Recovery efficiency as high as 95.82%	Series-coupled air-agitated crystallization reactors	laboratory- and pilot-scale	Dai et al. (2016)
	Snail shell	–	2	Providing the required molar concentration of Ca^{2+} and reducing cost of the chemicals for P crystallization	Synthetic solution (25–300 mg/L)	HAP	–	2 h	High sorption capacity of the SS for P (222.2 mg/g) and the P laden SS can be used as soil conditioner cum fertilizer in agricultural practices.	Batch reactor	Laboratory-scale	Oladoja et al. (2012)
	Cow bone powder	125–198	2	Dissolving Ca^{2+} , co-precipitation of Ca and P on seed	Synthetic solutions (3.5 mg P/L)	HAP	–	3 h	The recovery rate of phosphorus reached 89.8%	FBR	Pilot-scale	Gu et al. (2015)
Synthetic materials	Boehmite	600–1300	–	Deposition of Ca-P onto the seed material surface	Wastewater from the biological WWTP (10 mg P/L)	HAP	–	1 h	Generated crystallization products of up to 13% P-tot (30% P_2O_5)	Fixed bed column and SR	Laboratory- and pilot-scale	Berg et al. (2005)
	Converter slag	150–180	1.7	Used as a seed for Ca-P crystallization; strong affinity to P	Synthetic solution (25 mg/L P)	HAP	–	3 h	The removal efficiency of P up to 95% and generated crystallization products of up to 14.5% P-tot (33.2% P_2O_5)	Batch reactor	Laboratory-scale	Duan et al. (2010)
	Porous CSH	<75	4	Ca^{2+} and OH^- release from CSH for P	Synthetic P solution (100 mg P/L)	HAP	–	1 h	Phosphorus content of A-CSH reached 18.64%	Batch reactor	Laboratory-scale	Guan et al. (2013)

REL-LIVE WASTE
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Limassol, 22nd October 2019

Sereco-Biotest - Italy

TRAINING
**Part III Alternative Mg and P sources for struvite
precipitation**

**Dr. Biologist Luca Poletti –
Dr. Agr. Roberto Poletti**

Alternative Mg sources for struvite production

S. No.	Mg source	Source of struvite	Mg content	Mg:P	pH	Phosphate recovery (%)	Key findings	Cost	Reference
1	Bittern	Coke manufacturing wastewater	31,390 mg l ⁻¹	1:1	10.5	99	Similar recovery efficiency as MgCl ₂	NR	Shin and Lee (1997)
		Landfill leachate	9220–24,900 mg l ⁻¹	1:1	11	NR	Comparable struvite precipitation efficiency with MgCl ₂ & MgSO ₄	NR	Li and Zhao (2002)
		Biologically treated swine wastewater	32,000 mg l ⁻¹	1.3:1	9.6	76	Bittern is more effective in P recovery than NH ₄ ⁺ recovery	NR	Lee et al. (2003)
2	Sea water	Urine	27,500	1.1:1	NR	98	More cost effective in coastal areas	0.23 USD/kg struvite	Etter et al. (2011)
		Wastewater from coke manufacturing process	1136 mg l ⁻¹	1:1	10.5	95	Same P recovery efficiency as MgCl ₂	NR	Shin and Lee (1997)
		Side-stream of water treatment plant	1250 mg l ⁻¹	1.6:1 and 2.2:1	7.6–8.4	70	Higher Mg:PO ₄ ³⁻ (>1.5:1) necessary for more than 70% P recovery	NR	Matsumiya et al. (2000)
		Municipal Waste water stream from water treatment plant	1250 mg l ⁻¹	1.6–2.4	8.4–76		Higher Mg:PO ₄ ³⁻ (>1.5:1) necessary for stabilized and easy P recovery	0.55 USD/kg struvite	Kumashiro et al. (2001)
3	Thermally decomposed magnesite (MgO)	Biologically treated swine wastewater	1200 mg l ⁻¹	1.36:1	10	81	Similar recovery efficiency as MgCl ₂ (75%)	NR	Lee et al. (2003)
		Filtrate of wastewater sludge	676.7 g kg ⁻¹	2.5:1	8.5–8.8	90	Need higher Mg:PO ₄ ³⁻ molar ratio for effective recovery	NR	Quintana et al. (2004)
4	Brucite	Rare-earth wastewater	650 g kg ⁻¹	1:1	8.5–9.5	97	Brucite can be used as liquid, solid Mg source, Reuse of brucite is possible	NR	Huang et al. (2011a)
5	Magnesite	Landfill leachate	300 g kg ⁻¹	NR	8.6	NR	Acid dissolution of magnesite increase struvite recovery by 50%	18% cost reduction than using MgCl ₂	Gunay et al. (2008)
		Rare-earth wastewater	940 g kg ⁻¹	1.1:1	5–9.2	Up to 99.7	Thermal decomposed magnesite is cost effective than acid dissolved magnesite	34% cost reduction than using MgCl ₂	Huang et al. (2010a,b)
		Urine	244 g kg ⁻¹	1:1:1	NR	90	Cheaper than bittern and MgSO ₄	12 USD/kg struvite	Etter et al. (2011)
6	Struvite monohydrate	Piggery wastewater	530 g kg ⁻¹	2.5:1	8–8.5	96	Similar effects on recovery as with Mg salt	81% cost reduction than using MgCl ₂	Huang et al. (2011b)
7	Wood ash	Urine	34 g kg ⁻¹	2.7:1	8.8	99	Presence of impurity such as calcite, heavy metal in product	0.016–0.05 USD/kg struvite	Sakthivel et al. (2011)
8	Synthetic nanofiltration brine from seawater	Synthetic wastewater, Real wastewater	146 mmol l ⁻¹	1:1	8	99.5	Effective as Mg source but pH, organic matter influence purity of product	NR	Zewahn et al. (2012)
9	Desalinated reject water	Synthetic centrate	1555–2795 mg l ⁻¹	2:1 & 8:1	8–8.5	Up to 55	Presence of other ions (Ca, Na) in reject water reduces recovery efficiency	NR	Fattah and Ahmed (2013)
10	Mg(II) solution from seawater	Supernatant of a municipal-sludge	8000 mg l ⁻¹	1:1	7.57, 8.13	90	Higher dose of Mg:PO ₄ ³⁻ (>1:1) has no effect on recovery	25% cost reduction than using Mg chemical	Lahav et al. (2013)

Composition of alternative Mg sources for struvite production

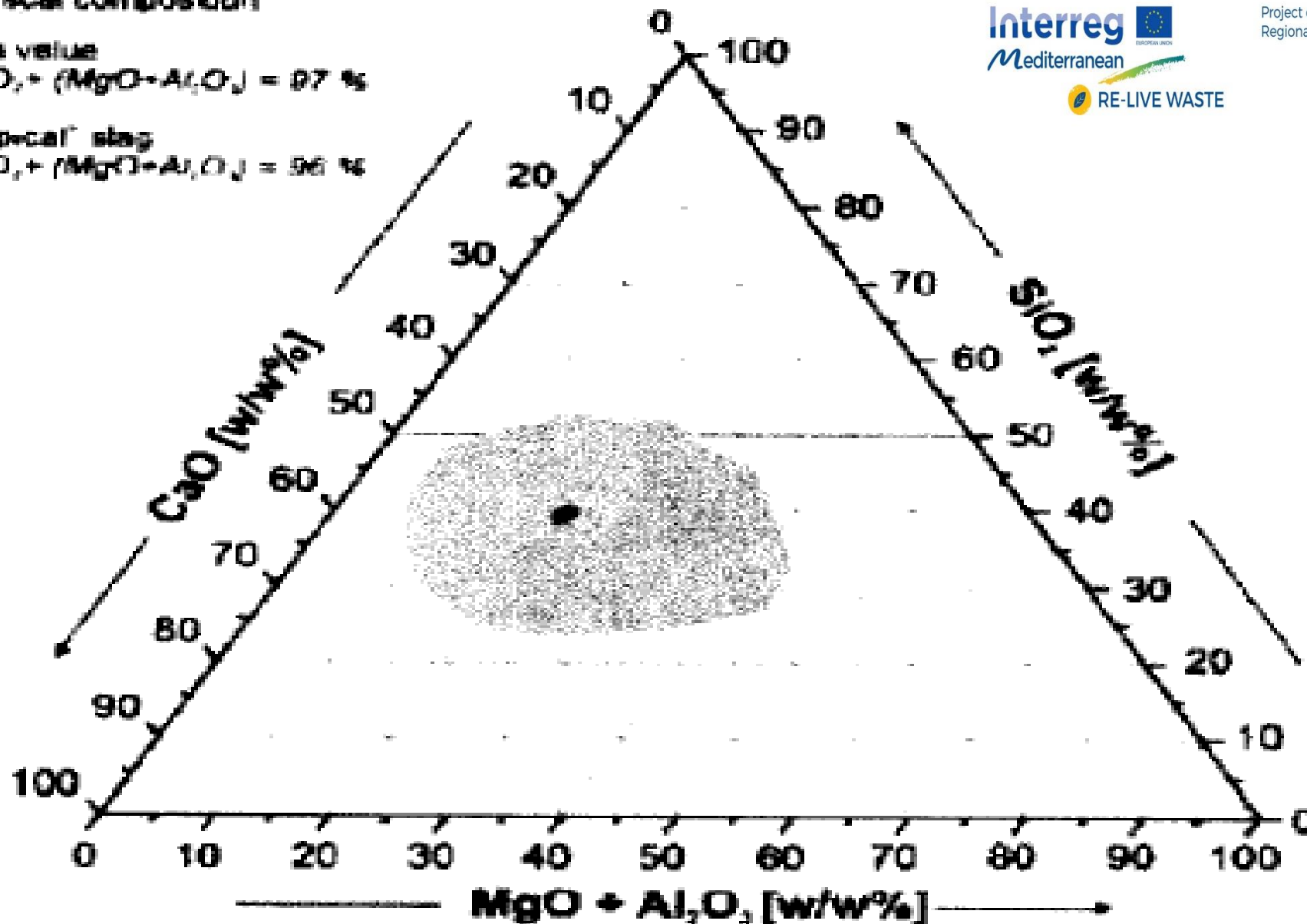
	Wood ash, ppm	Bittern, ppm	By-product of MgO production, ppm	Magnesite, ppm	Magnesia (MgO), ppm	Seawater, ppm
Mg	34,200	9220–44,000	676,700 ^a	940,000 ^b	898,000–634,000 ^a	1010
Ca	27,400	10–650	95,800 ^c	10,000–15,000 ^c	15,000–87,000 ^c	950
Al ₂ O ₃	10,800 ^d		3700	2000		
Fe ₂ O ₃	6090 ^e		26,300	3000–8000	24,000	
SO ₃	12,300 ^f	3300–60,000 ^f	39,500		38,000	
SiO ₂			26,000	7000–38,000	32,000	
K	74,600	1900–12,300				207
Mn	19,300					
Na	5160	3200–78,100				9658
Zn	2670					
Cr	1290					
Cu	1050					
Pd	590					
Ni	49					
Cd	28					
Cl ⁻		17,400–202,000				
Br ⁻		5300				
Reference	Sakthivel et al. (2011)	Li and Zhao (2002), Etter et al. (2011), Huang et al. (2014)	Quintana et al. (2008)	Gunay et al. (2008), Huang et al. (2010b)	Romero-Gilizaa et al. (2015)	Li and Zhao (2002)

^a As MgO.
^b As MgCO₃.
^c As CaO.
^d As Al³⁺.
^e As Fe.
^f As SO₄²⁻.

Ternary composition diagram for blast furnace slag

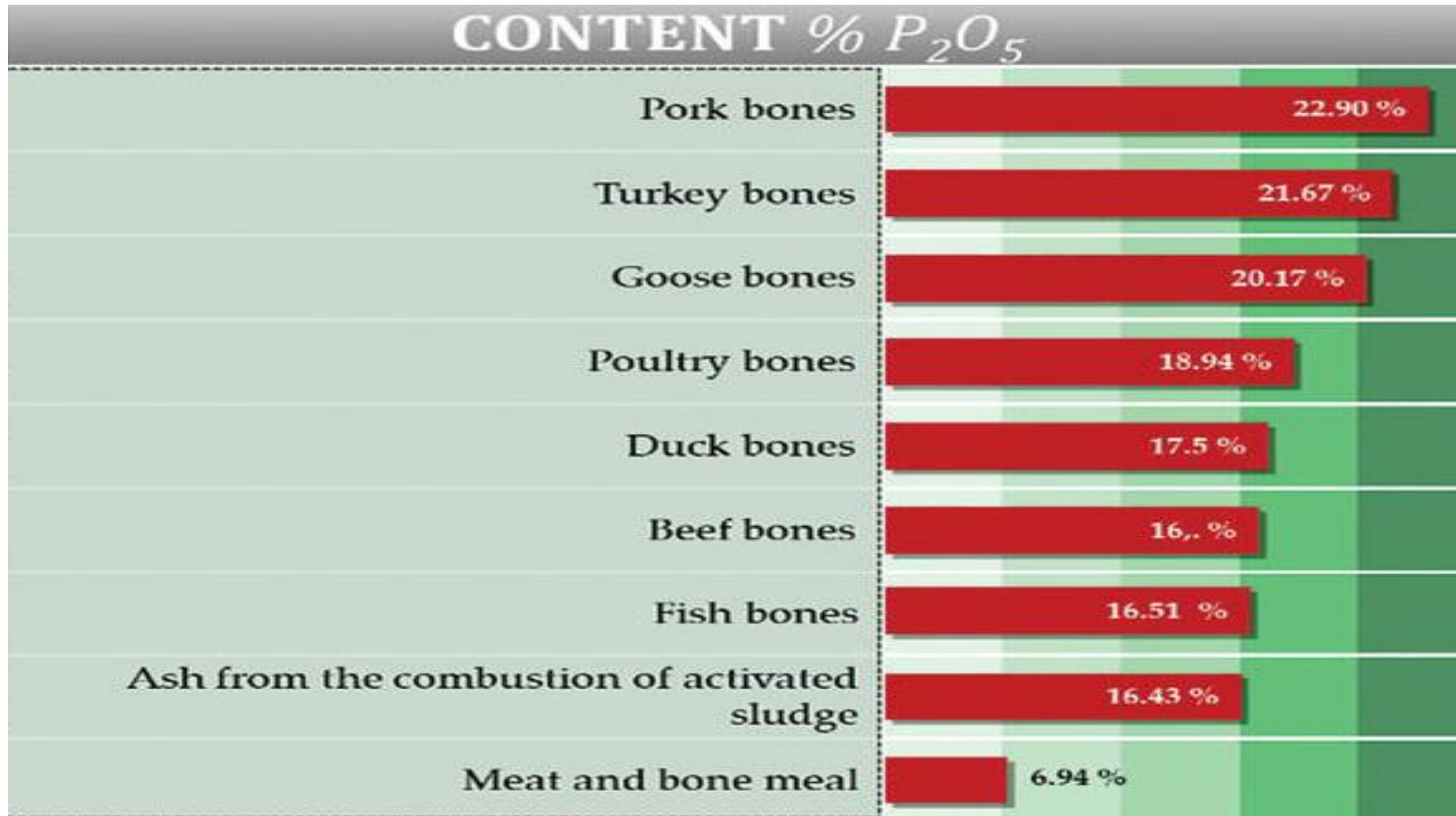
possible chemical composition

- typical mean value
 $\sum \text{CaO} + \text{SiO}_2 + (\text{MgO} + \text{Al}_2\text{O}_3) = 97 \%$
- selected „typical“ slag
 $\sum \text{CaO} + \text{SiO}_2 + (\text{MgO} + \text{Al}_2\text{O}_3) = 96 \%$



Alternative P sources

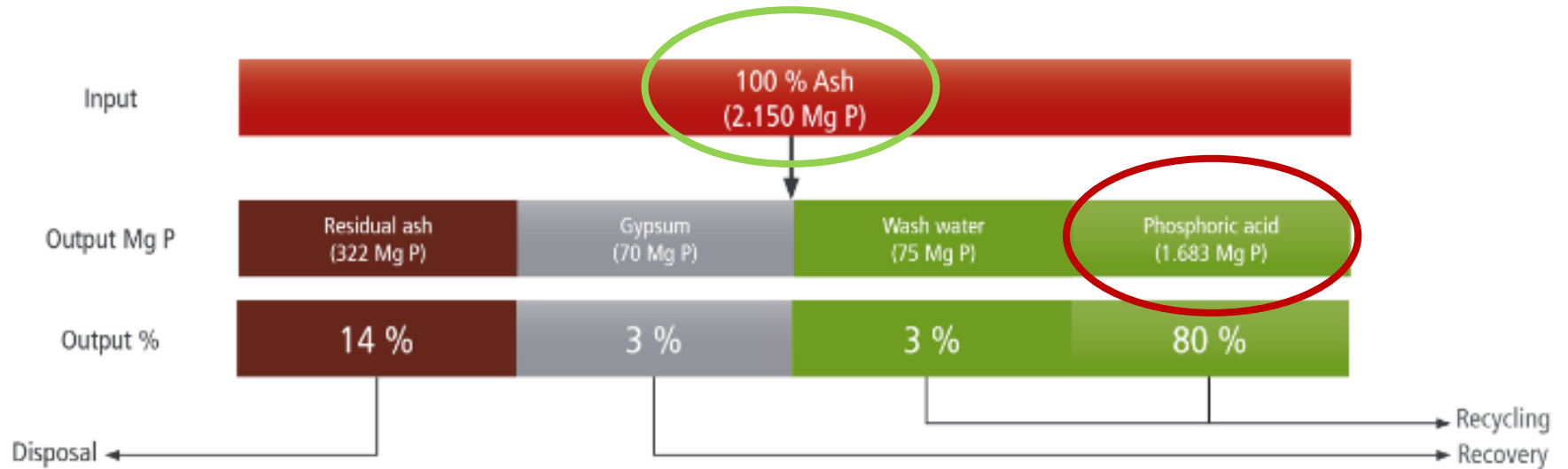
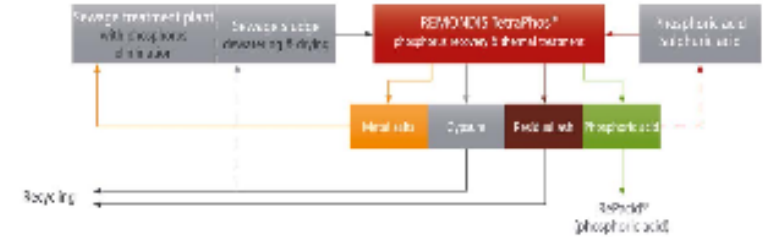
Bones from slaughterhouse activity



Alternative P sources

Ashes

REMONDIS TetraPhos® Mass balance phosphate

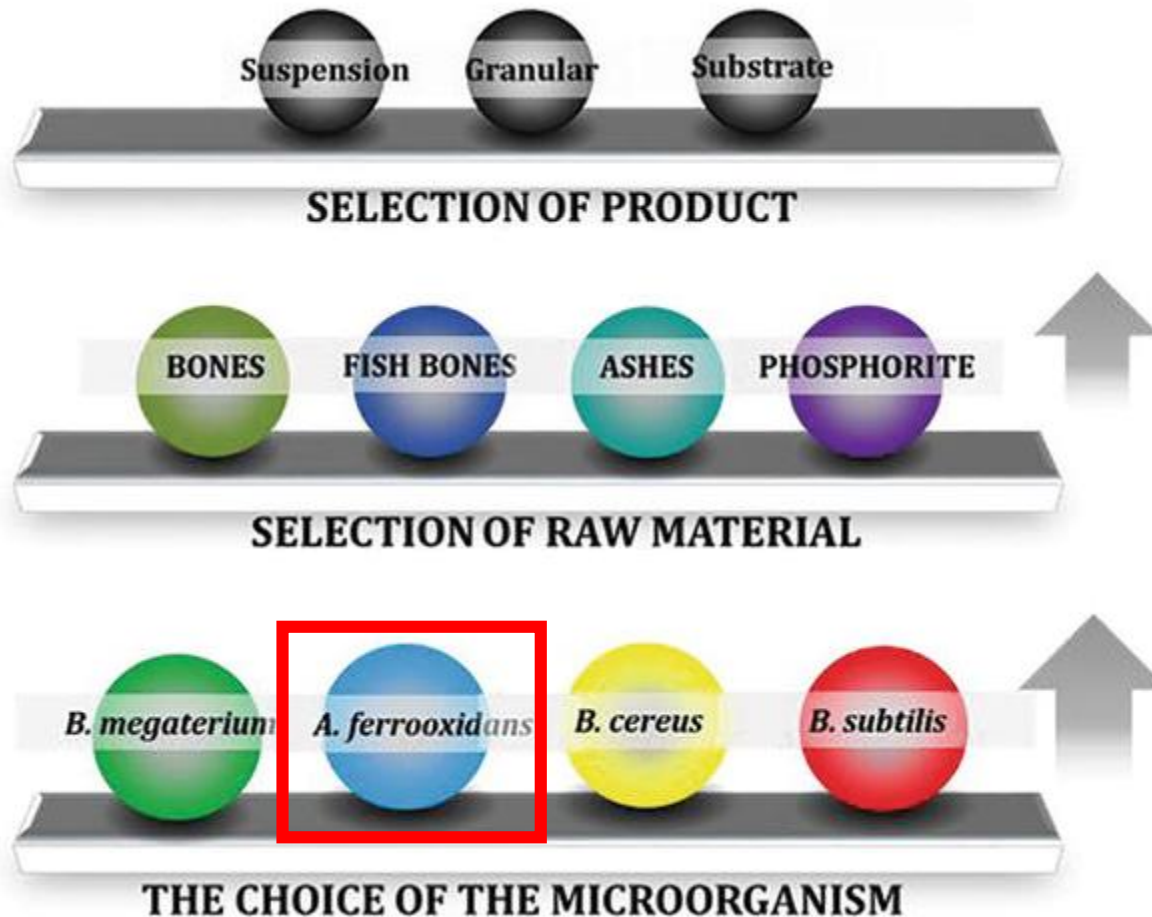


Pay attention to:

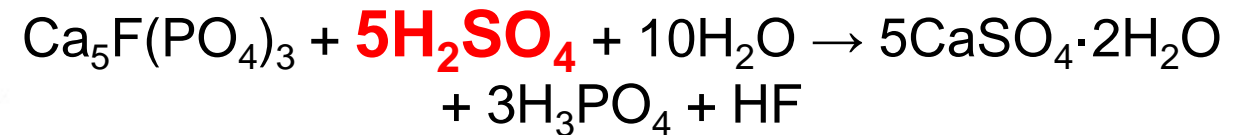
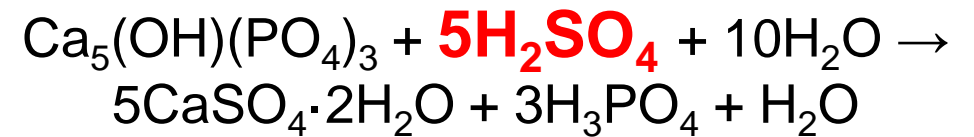
- Composition
- Contaminants
- Procedures of mineralization (acidification)

Alternative P sources

Microbiological P solubilization



Acidithiobacillus ferrooxidans is a chemolithotrophic and autotrophic PSB that oxidizes the ferrous ion thus inducing the chemical oxidation of S^0 and S^{2-} in FeS, CuS, FeS₂, and producing H₂SO₄ that, in turn, causes the bioleaching of P and metals (like As)



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TRAINING

Part IV Analytical methods for struvite characterization
An overview

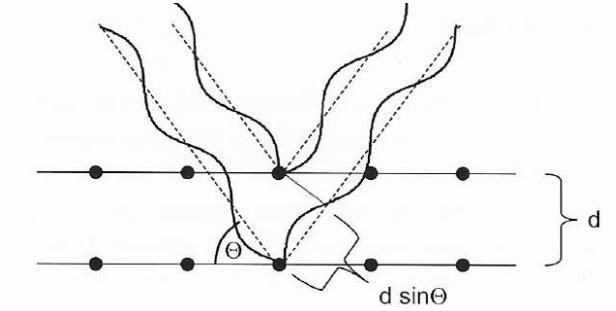
**Dr. Biologist Luca Poletti –
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Intrinsic properties of crystals

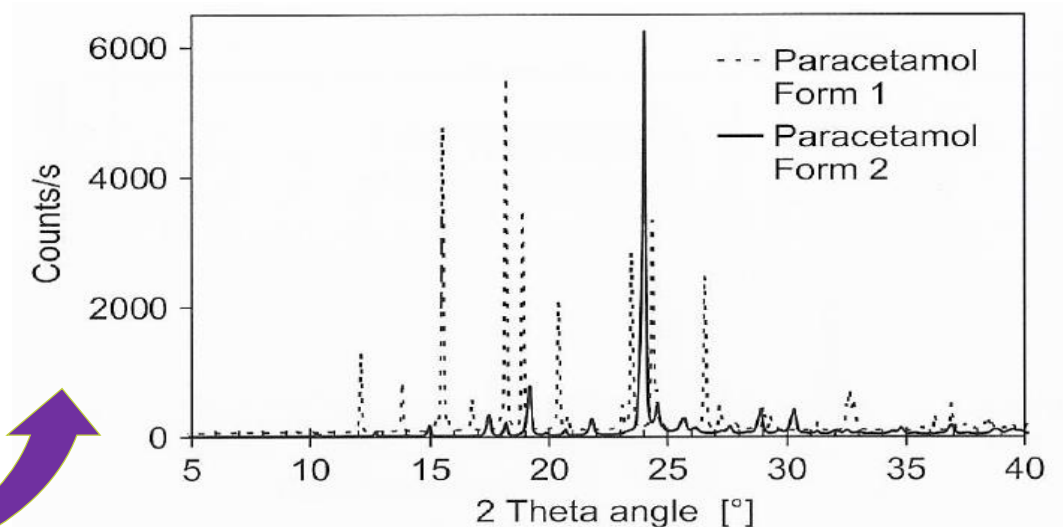
- Investigation of crystal structure

- X-Ray Powder Diffraction (XRPD)

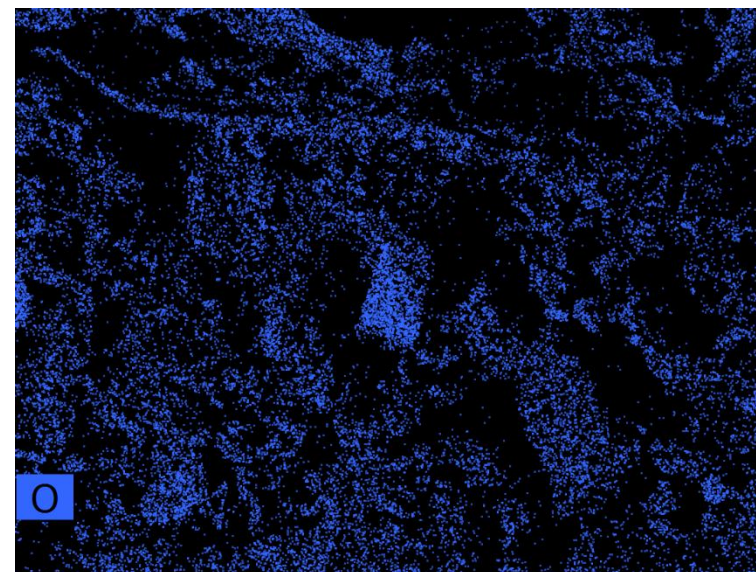
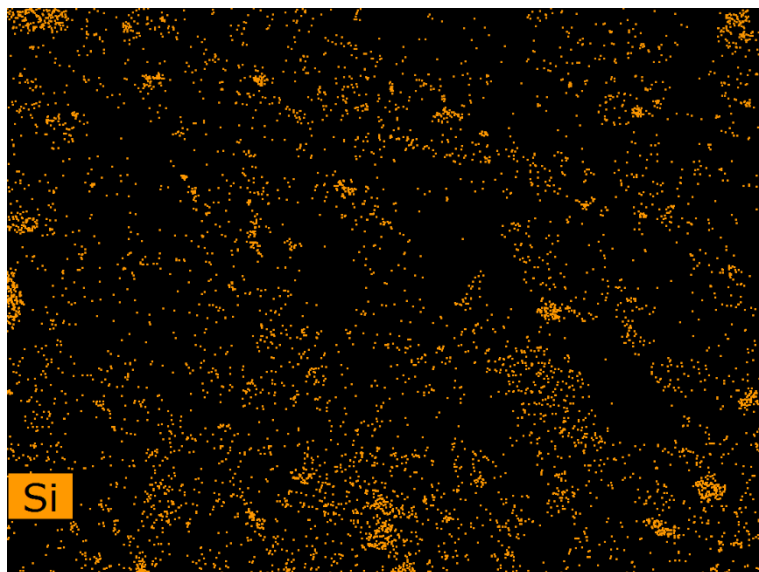
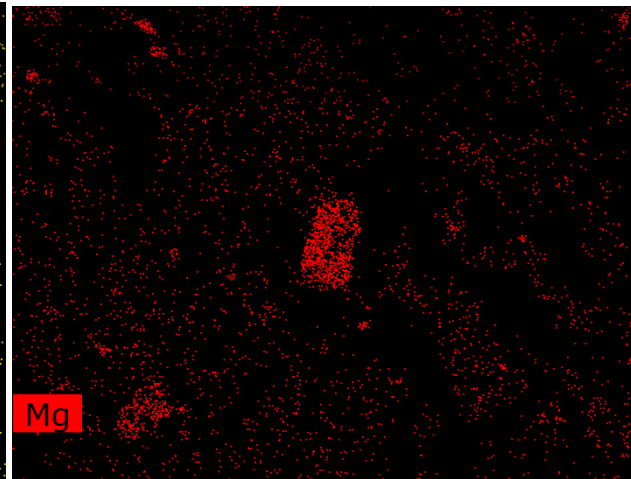
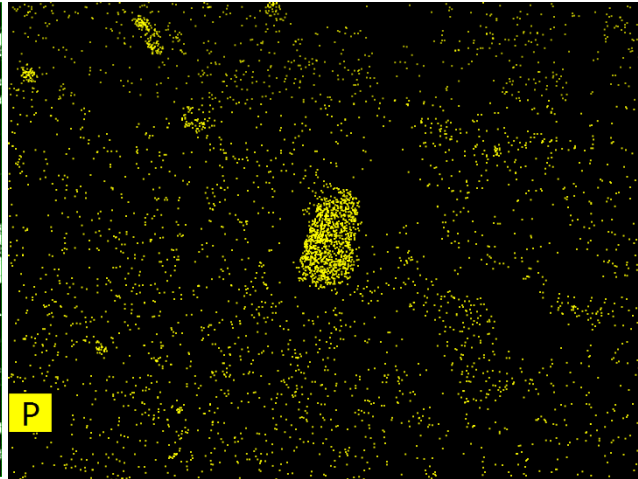
- Based on Bragg's law
- Effective in distinguishing polymorphs
- Problem with preferential orientation → if particles are nonspherical (e.g. plates or needles) and are not randomly oriented some lines in the XRPD may become greatly attenuated or even disappear
- Sample preparation critical



XRPD is able to differentiate between polymorphs



SEM-EDS

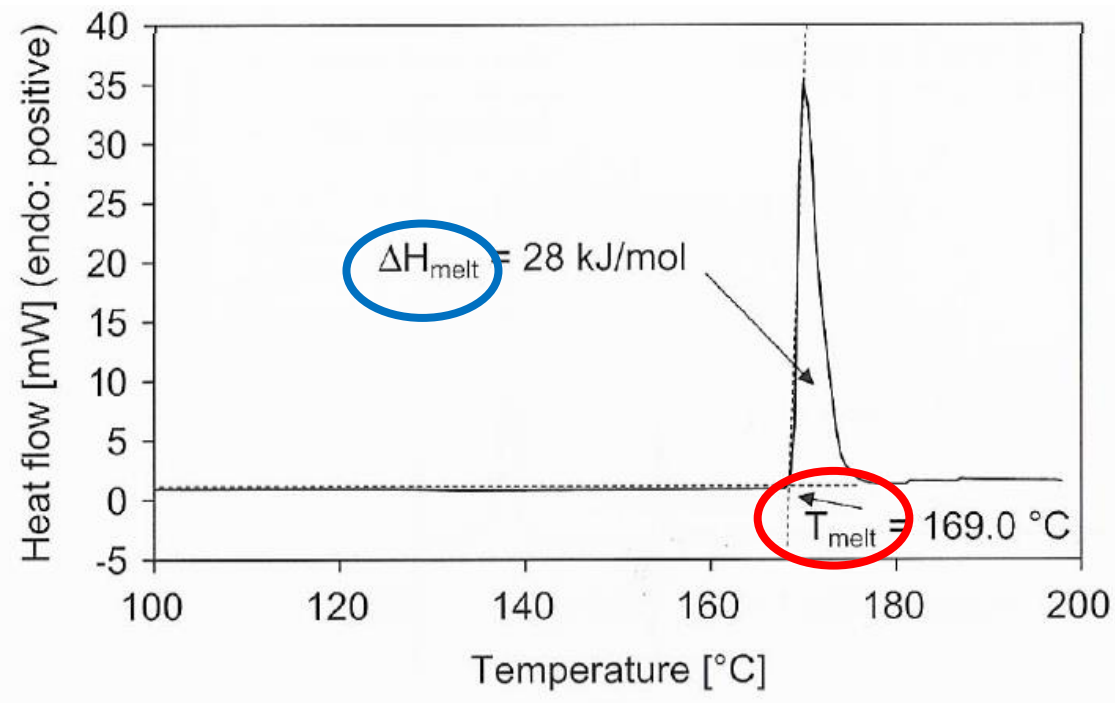


Intrinsic properties of crystals

- **Vibrational Spectroscopy** (Raman, IR, NIR)
 - With ATR-IR no sample preparation needed
 - Raman and NIR spectroscopy can be applied for online monitoring
- **Solid-state NMR** (ssNMR)
 - Powerful tool for R&D but not particularly suitable as a routine technique for QC for being time-consuming and expensive

Thermodynamical properties

- Differential Scanning Calorimetry (DSC)
 - **Melting point** and **enthalpy** are essential to describe a solid form



- Isothermal microcalorimetry
 - Useful in assessing chemical stability
 - Determination of amorphous content

Composition

- Thermogravimetry (TG, TG-FTIR, TG-MS)
- Dynamic Vapour Sorption (DVS) → hygroscopicity

Characterization of Particle Shape and Size

Microscopy (optical, electron)

Laser light diffraction (Fraunhofer)

Sieving

LLD and Sieving give similar results for spherical particles but differ significantly for rodlike and platelike

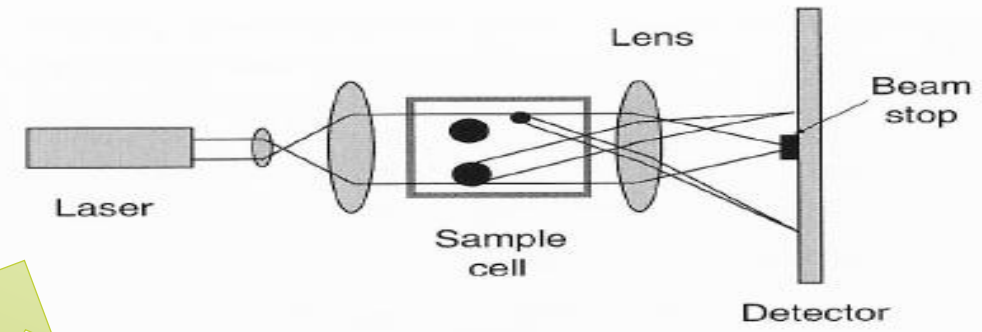
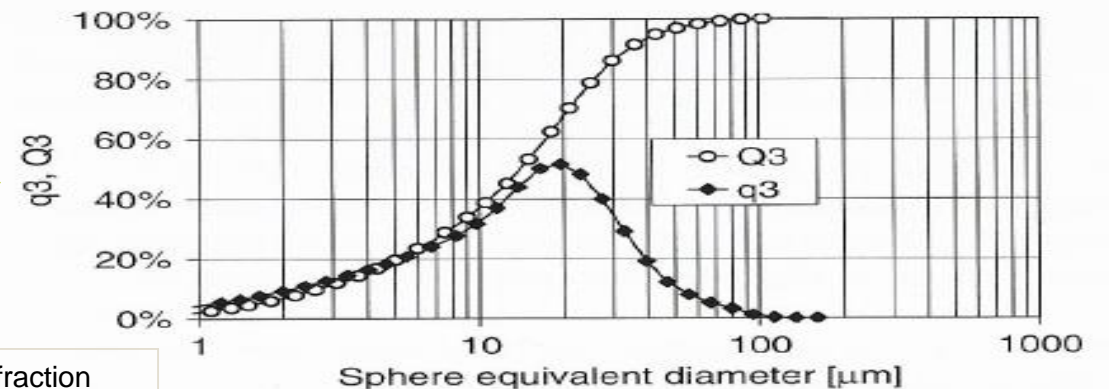


Figure 8.15 Principle of Fraunhofer diffraction instrument.



Typical result of a PSD obtained by laser light diffraction

Powder flow properties

Methods:

Hausner Ratio or compressibility index-→ Bulk volume and Tapped volume

$$\text{Hausner ratio} = \frac{V_0}{V_t}$$

$$\text{Compressibility index} = 100\% \frac{V_0 - V_t}{V_0}$$

Angle of response

$$\tan \alpha = \frac{2 \times \text{height of cone}}{\text{diameter of base of cone}}$$

Powder rheometry

In-process characterization

Real-time quantitative characterization
Through backscattered laser beam whose
Pulses determine a statistical distribution
Of the particle dimensions
(chord lengths distribution)

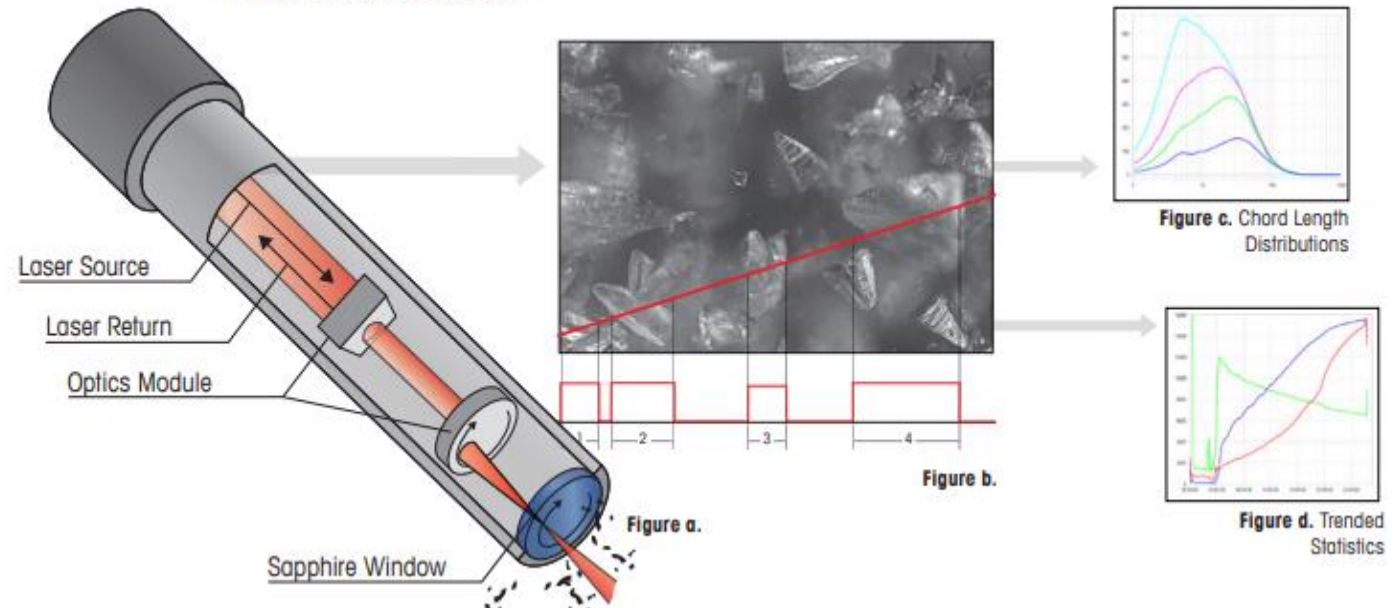
FBRM®

Highly suitable method to monitor
Seeded crystallization processes
Suitable for
QUANTITATIVE
MONITORING

Appendix A: ParticleTrack with FBRM® (Focused Beam Reflectance Measurement)

Measurement for optimization in real time – ParticleTrack is a precise and sensitive technology which tracks changes to particle dimension, particle shape, and particle count. Over a wide detection range from 0.5 to 2000 µm, measurements are acquired in real time while particles are forming and can still be modified enabling process optimization and control. No sampling or sample preparation is required – even in highly concentrated (70 % and higher) and opaque suspensions.

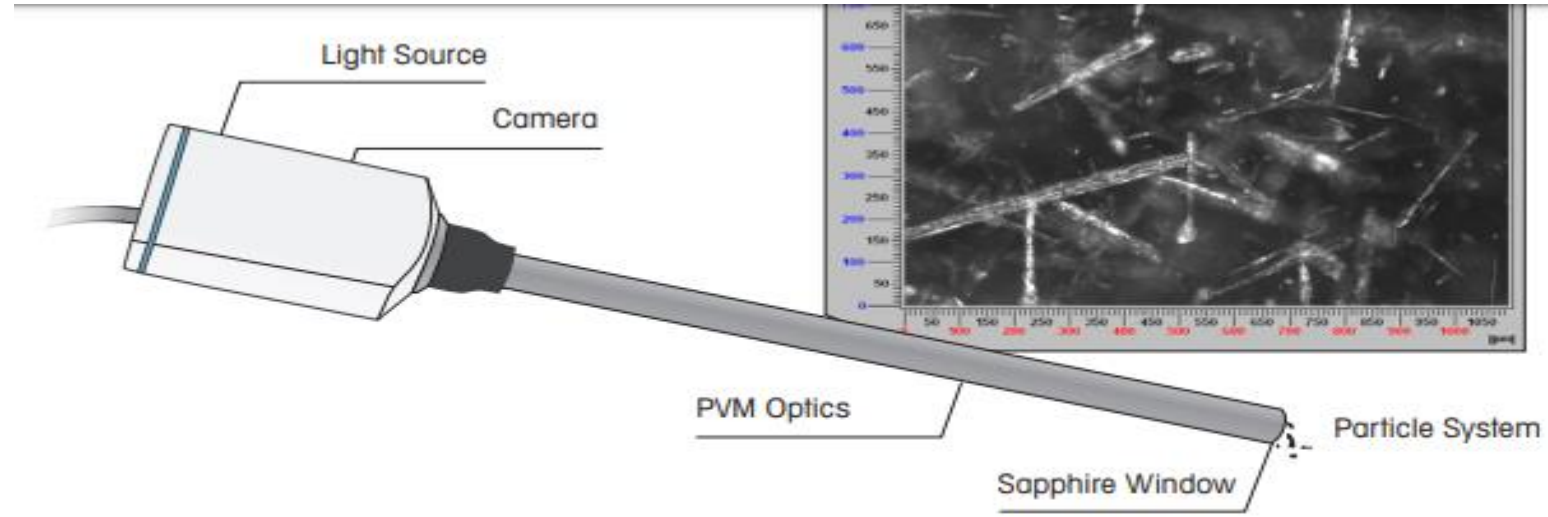
www.mt.com/ParticleTrack



In - process characterization

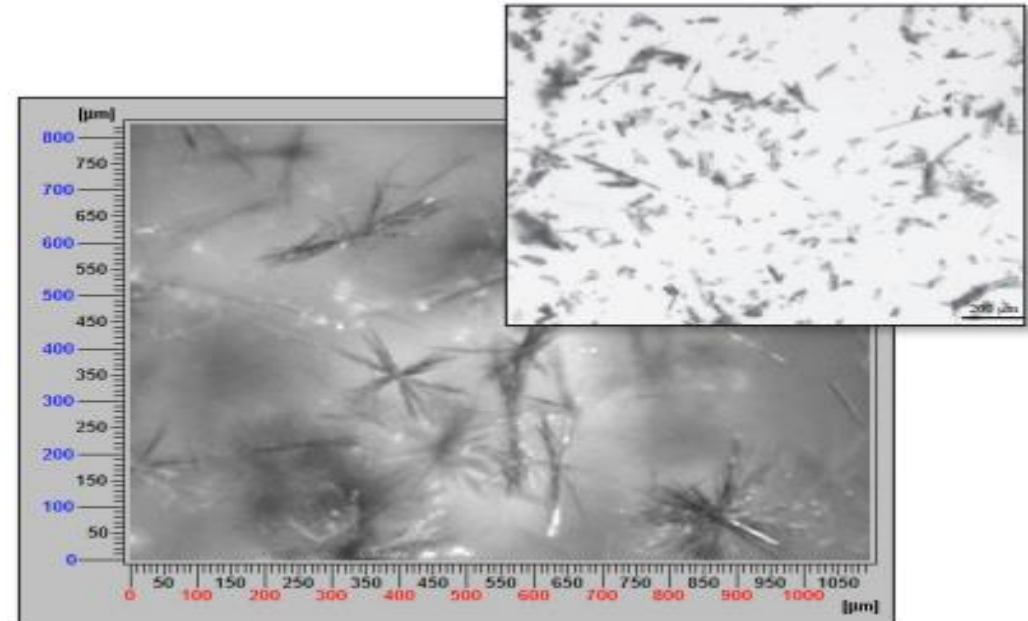
PVM®

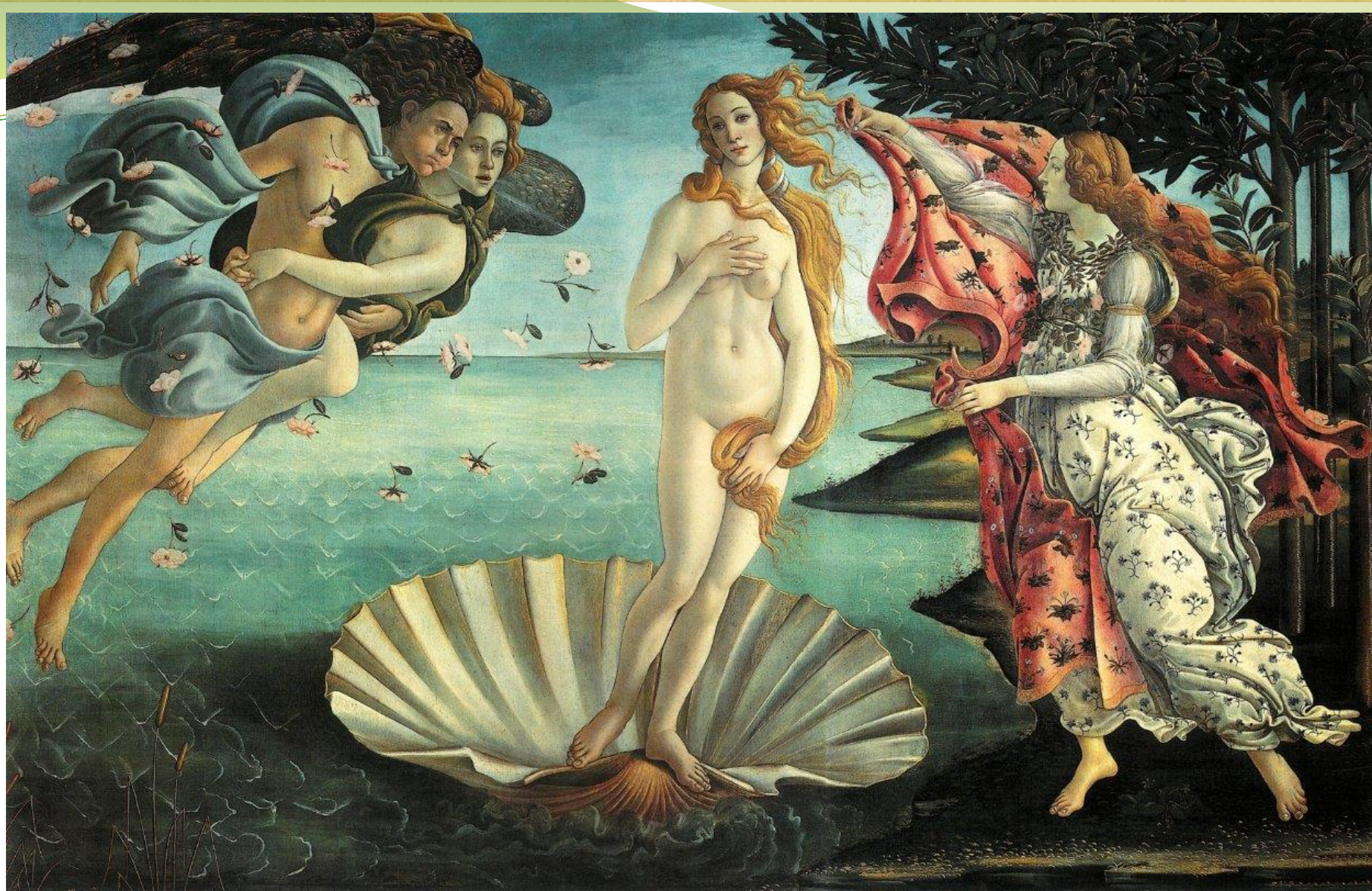
Qualitative monitoring



How does ParticleView work?

ParticleView uses a high resolution camera and internal illumination source to obtain high quality images even in dark and concentrated suspensions or emulsions. With no calibration needed and easy data interpretation, ParticleView quickly provides critical knowledge of crystal, particle, and droplet behavior.








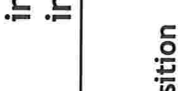





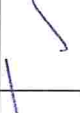


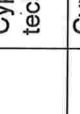
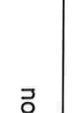


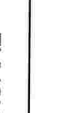
ευχαριστώ πολύ

Part A

Date: 22nd October 2019.

Place: Pilot plant facilities of CUT Partner: Cyprus University of
in Nicos Armenis & Sons Ltd Farm Technology
in Monagroulli

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