PRODIAS
Processing Diluted Aqueous Systems

SPIRE PROJECT

Freeze Concentration for Thermal Sensitive Products

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Enzyme/Protein solutions

Enzymes and protein solutions in general are often temperature sensitive products. The processing to a dry and stable final product will have various steps where the reduction in activity and the lowest energy consumption need to be balanced. Initial tests with freeze concentration have shown that concentration at subzero temperatures will have little effect on the activity and is therefore considered as an essential building block in the downstream processing of enzymes and protein solutions.

Freeze Concentration

Freeze concentration is the removal of pure water in the form of ice crystals at subzero temperatures. Due to the low temperature processing there is **no thermal impact** on the product.

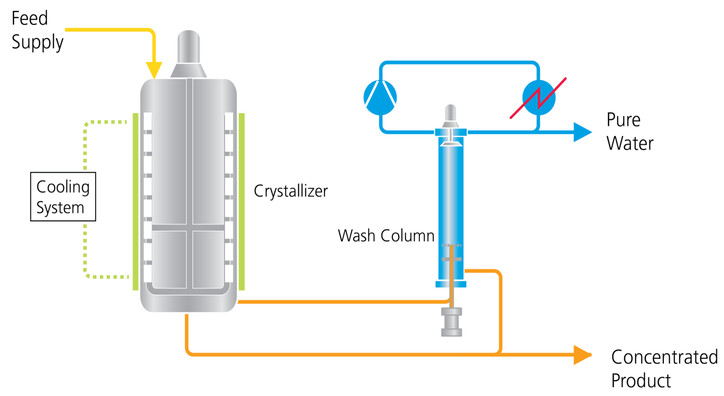


Figure: Single stage freeze concentration with piston wash column

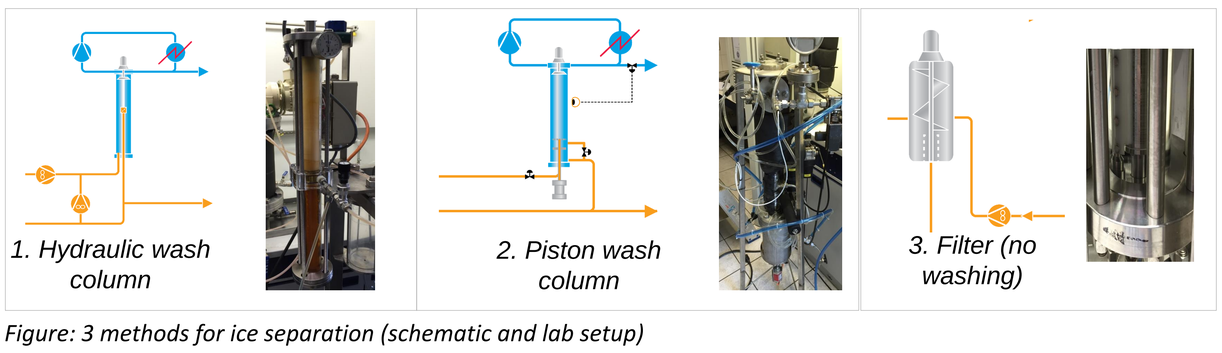


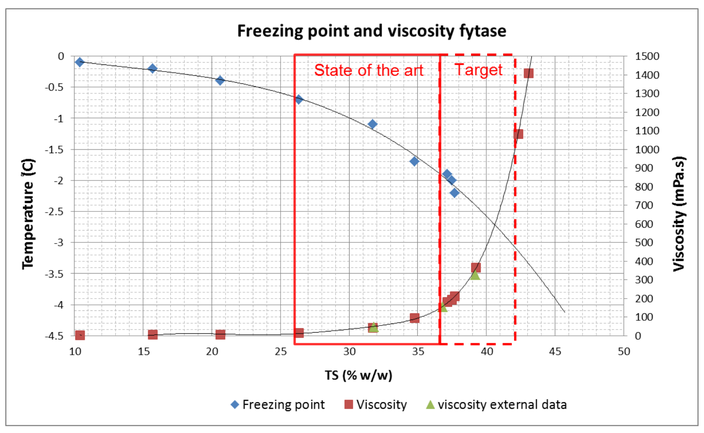
Figure: 3 methods for ice separation (schematic and lab setup)

Separability of the concentrate from the ice crystals is a function of viscosity, crystal size (residence time) and filter mesh. State of the art in freeze concentration allows separation up to ~150 mPa*s in multi-stage systems.

Experiments

Goal: Demonstrate feasibility of freeze concentration for enzyme/protein solutions in terms of activity preservation and maximize the final concentration.

Exemplary system: Phytase is used as additive for animal feed (pigs and poultry). It degrades phytate and is concentrated by membranes up to 30 wt%. At concentrations >40 wt%, the viscosity rapidly increases.



Approach: Determine the limits of ice separation for the three different separation systems and compare performance for different residence times and viscosities. Analyses of crystal photos should give insights in relation between separability and crystal size distribution. It is expected that optimized crystal growth conditions and improved filter design and process setup stretches the limits for ice separation.

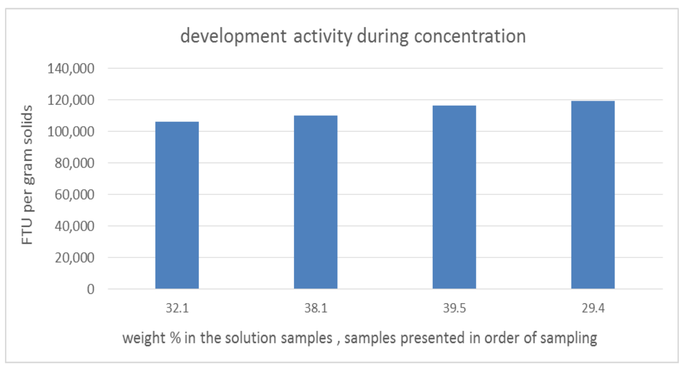
Experimental setup: A 7-liter steel crystallizer is constructed for ice production. The lab-scale separation systems can be connected to the crystallizer. A microscope with camera is installed to analyze the crystal size distribution.

Results:

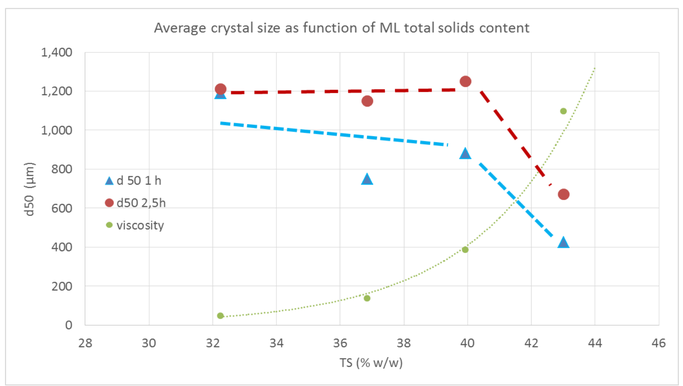
Concentration

	Max. concentration °Brix	Max. concentration wt%	Viscosity mPa*s
Piston wash column	46.5	39.5	400
Hydraulic wash column	45.6	38.9	320
Filter (no washing)	51.1	43.1	1400

Activity



CSD



Profitability

Combination with a spray drier
- Capex neutral
- Saving in energy
- Pay back time around 5 years for 400 kg/h and 2 years for 3,500 kg/h dewatering

Combination with freeze drying
- Capex savings
- Energy savings
- Pay back time around 1 year for 400 kg/h dewatering plant

The effect of increased activity (or quality in case of proteins) needs to be investigated in detail for the whole line. Due to the high prices of enzymes a low temperature in NF pre concentration might already lead to large savings and justification of freeze concentration technology.

Conclusions

- It is feasible to remove 30-45% of the water from pre concentrated solutions
- No decrease in activity observed
- Even at highest concentration still separable crystal sizes
- Focus from design of extra fine filters on crystallizers and mixing
- Optimum combination with membranes and activity in total chain to be investigated
- Acceptable pay back times in combination with spray drying for larger capacities and in combination with freeze concentration always

Next steps

- Optimize combinations with membranes (Hybrid System)
- Design and construct pilot plant to proof principle and optimize system on pilot scale

PRODIAS CONSORTIUM

BASF We create chemistry · Alfa Laval · GEA · Imperial College London · Cargill · XENDO Solutions for Life Sciences · enviplan · Technische Universität Kaiserslautern · UPM

ABOUT THE PROJECT

- Start date: 1st January 2015
- Duration 48 Months, until 31st December 2018
- Budget: 14 million €
- Project web site: www.spire2030.eu/prodias/

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