

LATEST INNOVATIONS IN LOW TEMPERATURE CONCENTRATION OF AQUEOUS SOLUTIONS

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Introduction

In the beverage industry the demand for high quality products at an acceptable price is forcing the juice processing industry towards new technologies to create a beverage that looks and tastes fresh. Maintaining the "natural" look and fresh taste generally creates a conflict with factors like shelf life, transportation and storage costs, etc.

Water is the focus of this problem. To combat the expenses of transportation, packaging and storage, a variety of concentration techniques have been developed.

The aim of concentration is to add value to the product. The gross added value is the difference in market value between the concentrated and non-concentrated product.

The added value can be created from:

- Cost savings due to volume reduction (transport/storage/packaging)
- Consumer convenience
- Reduction of drying cost
- Upgrading raw materials
- Increase if product stability
- Ingredient with low water content
- New product development

The net added value is the gross added value minus additional processing costs.

The main driving force to look for a concentration technology are often the economics however, the net added value is also influence by the quality of the concentrate product.

It is for that reason the industry is looking for processes that can maintain the original quality of the products at acceptable cost.

For concentration of food liquids basically three different processes are commercially available, being evaporation, reverses osmosis and freeze concentration. This paper will focus on freeze concentration.

Factors influencing the net added value

Selective physical losses

Freeze concentration is associated with the crystallization of aqueous solutions. Crystallization is a highly selective separation process in that crystallizes water into ice crystals without any inclusions in the ice crystals. The ice can be fully separated from the concentrated juice without any losses of volatile components or dissolved solids.

Physical changes

Next to the effect of the concentration itself, which is independent of the process, physical changes are affected by chemical reactions, including enzymatic reactions resulting in browning, vitamin destruction, reduction of sensory quality, etc.

Since these reactions are strongly influenced by temperature, it is obvious that due to its low processing temperature, freeze concentration slows down or even prevents such changes.

The concentration factor

The concentration factor has a major economic impact on the net added value.

The maximum concentration that can be obtained in the freeze concentration process is influenced by the viscosity. In freeze concentration only the viscosity at the equilibrium freezing temperature is relevant. With increasing concentration, the viscosity increases both as a result of the increasing concentration itself and the decreasing temperature.

For most products the typical achievable concentration level for freeze concentration is in the range of 35-50 Wt%.

Costs of concentration

The cost of freeze concentration is the main limiting factor for applying freeze concentration technology. It is therefore, that new developments have been focusing on reducing the capital and energy cost of this low temperature technology.

Conclusion: there are no selective physical losses or physical changes in freeze concentration. Maintaining quality is therefore the main reason for using freeze concentration technology, however, the cost of freeze concentration strongly influences the net added value of the product.

Freeze Concentration

Concentration by freezing dates back many years, in principle being applied in a very elementary form. By simple letting the product freeze in winter time, one did create low temperature concentration by decanting the ice.

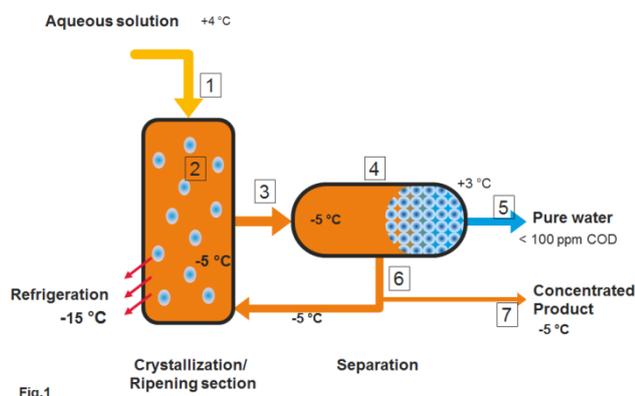
Without considering the cost of the equipment, the main obstacle to the development of an efficient technology has been the necessity of eliminating losses of concentrate and volatile aromas.

The late Professor Thijssen at the Eindhoven University of Technology has developed the foundation for freeze concentration technology while GEA has further commercialized the process.

Most food liquids consist of water and dissolved solids. In most cases the water content is 90% or more.

The principle of freeze concentration is to change part of the water into ice crystals (crystallization section) and then mechanically separating the ice from the mother liquid (wash column section) in a very selective way.

The Freeze Concentration Process



The fresh feed (1) is cooled to its freezing point in the crystallization section (2). At freezing point a part of the water is crystallized into small ice crystals. These ice crystals are too small for efficient separation. Conditions (residence time) are created for the ice crystals to grow. This results in larger, pure and spherical ice crystals (Fig 2).

The large, pure ice crystals + product are moved (3) to the separation section (wash column, 4) where the ice crystals are separated from the liquid (6) by mechanical means in a way that no losses occur with the separated (ice) water (5).

The highly selective separation is possible by controlled counter current washing of the ice crystal surface. The product liquid (6) is recycled into the crystallization section. The side stream (7) from the concentrated liquid is taken for production. There is always a controlled volume balance and solid balance.

In the crystallization section, part of the water is converted into ice crystals (Fig 2) which are different in size but all crystals are spherical but above all, there are no inclusions of any sort inside the crystal.



Fig 2

Ice Crystal Growth

The first crystals that are formed are small. By creating residence time the crystals will grow.

This growing or ripening of ice crystals, referred to in literature as Gibbs-Thompson effect, is based on the thermodynamic phenomenon that the equilibrium temperature of a crystal depends slightly on its size or more specific on the curvature of its surface.

That means that larger crystals melt at a higher temperature than smaller ones. In the ripening vessel the mean bulk temperature will thus adjust itself between the extreme values of the various melting temperatures of crystals and nuclei (newly formed small crystals). All crystals bigger than the equilibrium crystal size will grow at the expense of the smaller nuclei, which melt. As the residence time in the ripening vessel increases a larger average crystal size results. Small crystals are thus the driving force for the ripening process. In the original design of freeze concentration equipment the production of small ice crystals is done separate from the ice growing zone, the so called “separate nucleation and growth process”.

Freeze Concentration Design

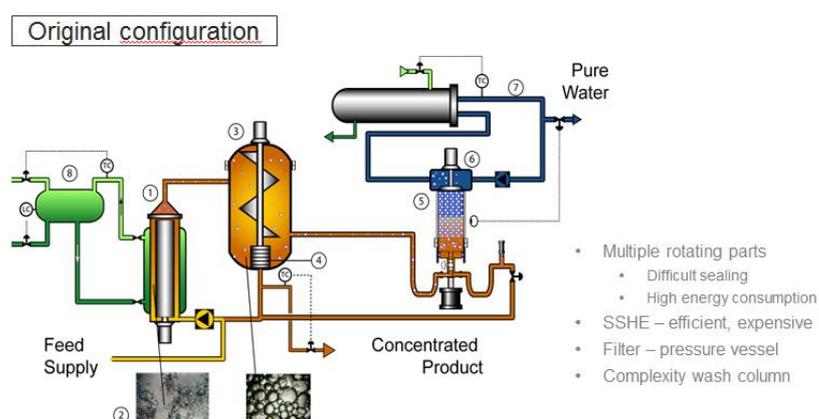


Fig.3.

In its simplest form (single stage design) the process works as illustrated in Figure 3. The fresh feed is pumped from a feed tank into a scraped surface heat exchanger (S.S.H.E.) where small crystals are formed instantly. The heat exchanger is specifically designed to allow for high flow rates and high ΔT 's and thus results in low residence times and a high undercooling. These conditions are required to continuously produce the small nuclei that are necessary for the application of the concept of separate nucleation and growth (SNG). The crystals are then subsequently fed to the re-crystallizer where they are mixed with larger crystals.



Fig.4
Wash column

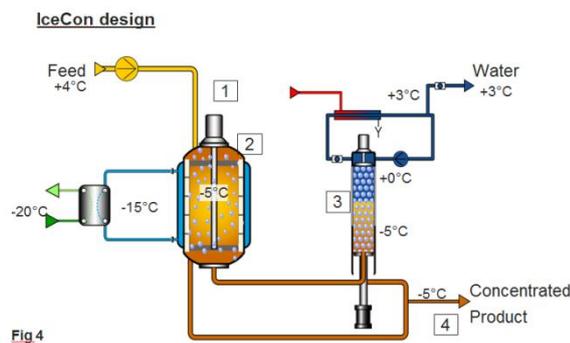
The slurry (concentrate and crystals) is transported from the recrystallizer to the wash column (5). In the wash column (Fig.4) the concentrate is "squeezed" through a filter at the bottom of the wash column. In this way a packed bed of ice crystals is formed. A packed bed is "pushed" upwards. At the top of the wash column, the ice is scraped off (6) and melted (7). Part of the melted ice is used to "wash" the packed bed. A sharp separation will be formed between the washed part of the bed (crystals and water) and the unwashed part of the bed (crystals and concentrate). This is called the wash front. The concentrate, which is still present in between the ice crystals, will be removed here. The water used for washing freezes onto the colder crystals and is carried with the crystal bed and removed at the top of the wash column with the rest of the ice. In this

separation process, the loss of soluble solids in the removed water is generally in the ppm or even ppb range. The final concentrate will be pumped to the storage vessel or to the next stage in the production process.

The recrystallizer or ripening vessel used in the SNG configuration represents a pressure vessel, which is equipped with a mixer and a scraped filter to allow for the crystal-free feed to the S.S.H.E.

Latest Developments

The high capital cost for both S.S.H.E. and recrystallizer, have been the incentive to evaluate other crystallization alternatives for the FC process. The major change in the freeze concentration design is the change from separate nucleation and growth into slurry crystallization. In slurry crystallization the production of ice crystals and ice crystal growth takes place in the same vessel design. This eliminates the need for pressure vessel and reduces the quantity of equipment. This latest development is called the IceCon.



In its simplest form (single stage design) the IceCon process works as illustrated in Figure 4. The fresh feed (1) is pumped from a combined crystallization/ripening vessel (2) where small crystals are formed instantly and ice crystal growth takes place. The mixer and scrapers is a combined design. The cooling of the vessel is via a secondary medium which is refrigerated by a traditional compressor system.

The slurry (concentrate and crystals) is transported from the recrystallizer to the wash column (3). This wash column is of similar design like in the traditional design in the wash column.

The final concentrate leaves (4) the system as side stream from the wash filtrate outlet.

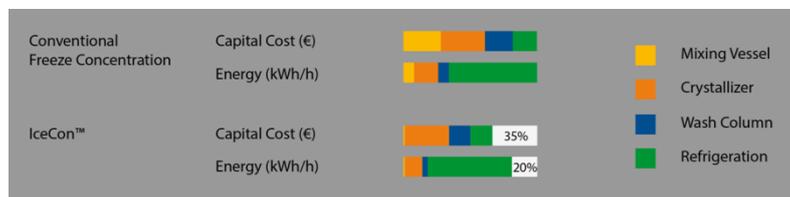
Advantage of IceCon System

Major differences between traditional design and latest IceCon design:

Due to the combined ice production and ice growth in one vessel

- No pressure vessels needed
- Elimination of internal filters
- Simplified design

- Standardization
- Lower maintenance due to less moving parts
- Better cleanability



This results in significant lower capital cost (30-40%) while also due to less moving equipment the energy consumption (15-20%) is reduced.

Conclusion

The present status of the freeze concentration technology is that this process has proven to be a reliable and viable unit operation. Recent innovations have given a significant improvement of the economic feasibility and is therefore, available for wider range of applications.

Applications

The freeze concentration process can be used for virtually every liquid with a low to medium viscosity. The main advantage of preventing quality loss makes the process ideal for high quality, heat-sensitive products. Prevention of solid losses and separation of pure water makes this process interesting for applications in the chemical industry, too.

Coffee industry

The first application on an industrial scale was in the instant coffee industry for freeze dried coffee. The purpose is to reduce the overall production cost without scarifying quality. Now also pre-concentration in combination with spray drying and production of coffee concentrate are industrial applications.

Fruit juice industry

The most interesting application is for heat sensitive, aroma containing juices. The gross added value of freeze concentrate is significantly higher compared to concentrates from conventional evaporators.

A compromise between costs and quality is the use of freeze concentrated juice for mixing with evaporated juices, where the freeze concentrated juice adds the fresh aromas to the aroma deficient evaporated product.

Quantity-wise smaller, but quality-wise very interesting, is the application for special fruit juices such as strawberry, pineapple, peach, blackberry, black- and red currant and cherry juices.

Besides taste, also prevention of colour changes makes the use of freeze concentration attractive.

The alcohol industry

The advantage of freeze concentration can especially be applied for alcoholic products such as beer, wine and cider.

Concentrations of 20-25% alcohol are feasible.

The volume can be reduced which leads to savings in distribution cost and opens the possibility to the post-mix concept.

Vinegar

Even a low priced product like vinegar is being freeze concentrated. Besides savings through volume reduction, concentrating vinegar is interesting for the pickling industry. Use of concentrate is much more economical in the processing and therefore, gives an added value.

No other concentration technique is capable of concentrating natural vinegar as efficiently as freeze concentration.

Dairy industry

It has been proven that the taste of reconstituted freeze concentrated milk is equal to fresh milk, and it is certainly superior to conventionally concentrated- and dried milk products. This opens the way for production of:

- milk concentrates for industrial use
- milk concentrates for beverage use
- milk powders for beverage use
- value added dairy beverages
- retail milk concentrates for non-beverage use

The success of freeze concentration in the food industry is attributed directly to the distinct improvement in quality over existing products. Freeze concentrated products have developed their own market segment based on maintaining the original product quality in an economical and convenient concentrate.

On environmental issues freeze concentration is helpful by discharging pure water without the need for further waste treatment.

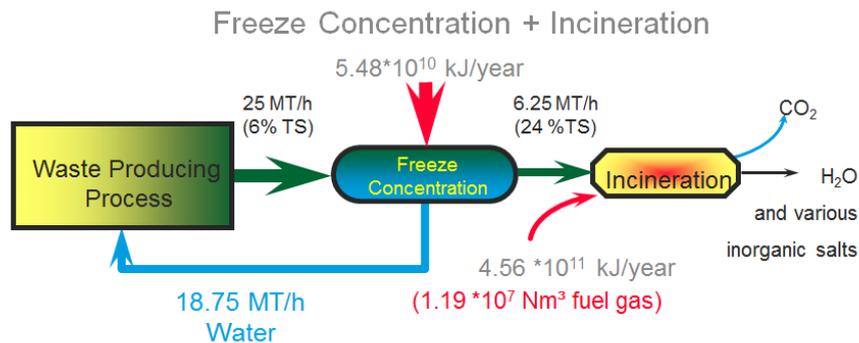
If beverages are distributed in concentrated form, significant savings can be obtained in the form of less fuel, less emission, savings on packaging material and thus saving on wood consumption etc., etc. Calculations indicate that considerable overall energy consumption will be saved if products are distributed as a concentrate.

Non-food industries

In the non-food industry the use of freeze concentration is driven by the high efficiency and purity of the separated water.

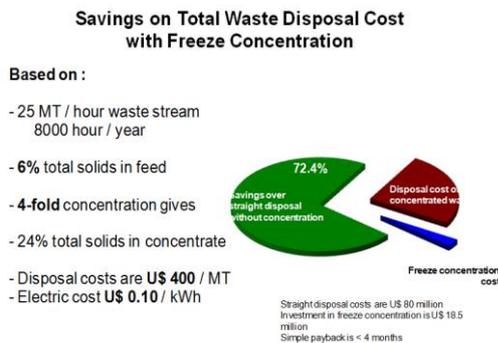
Waste water treatment

In the waste water industry processing the waste is a cost factor. Especially for processing of hazardous waste liquids freeze concentration is a serious alternative because freeze concentration gives in one single unit operation pure re-usable water while the concentrated can be processed in less volume and thus saving cost in the downstream processing.



Example:

In combination with incineration pre-concentration at 4 fold concentration and 25 MTon dewatering and 8000 operating hours gives a total savings of $1.75 \cdot 10^{12}$ kJ/year, 77% reduction of energy and 80% reduction in fuel gas consumption. The reduced fuel gas consumption also results in less CO₂ emission.



Thanks to the low temperature processing and the highly efficient separation of ice crystal freeze concentration technology is a potential interesting and feasible option for concentrating liquids.

References

1. Thijssen, H.A.C.-. Evaluation of concentration alternatives for liquid foods. *Int.J. of Food Techn. and Food Process Eng.* 34 (1983), 586.
2. Sheu, MJ., Wiley, R.C.: Preconcentration of apple juice by reverse osmosis, *J. Food Science* 48 (1983), 422.
3. Gekas, V., Hallstrom, B. and Tragardh, G.: Food and dairy, applications of membranes; the state of the art, *Desalination*, 53 (1985), 95.
4. Pepper, D., Orchard, A.C.J. and Merry, A.J.: Concentration of tomato juices and other fruit juices by reverse osmosis, *Desalination*, 53 (1985), 157.
5. Van Pelt, W., Roodenrijs, J.P.: Multi-stage counter current concentration system and method, U.S. patent 4,459,144, Jul. 10, 1984.
6. Thijssen, H.A.C., Van der Malen, B.G.M.: Continuous packed bed washcolumn, U.S. patent 4,332,599, Jun. 1, 1982.
7. Braddock, R.J.: Quality of freeze concentrated grapefruit juice: Proceeding XIX International Symposium of the Int. Fed. of Fruit Juice Producers, The Hague, May 12-15 (1986).
8. Thijssen, H.A.C.: The economics and potentials of freeze concentration for fruit juices, XIX International Symposium of the Int. Fed. of Fruit Juice Producers, The Hague, May 12-15 (1986).
9. GEA, Greco, Niro publications.