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Isolation and separation of food components by membrane processes

PART II

NANOFILTRATION AND DESALINATION ON THE FILTRATION UNIT TIA RO/NF SYSTEM WITH SPIRAL WOUND MEMBRANES

SAFETY INSTRUCTIONS

- **Be extra cautious during module cleaning! Hot Ultrasil 115 (sodium hydroxide) solution and Ultrasil 73 (acid) solution are used.**
- **During the filtration, check the pressure. The maximum pressure inside the module should not exceed 40 bar (4 MPa).**
- **Maximum temperature inside the module must not exceed 40 °C. Use water cooler to keep temperature constant.**
- **Do not discharge hot cleaning solutions into the sewage. You have to cool them down before discharging.**
- **The pump in the filtration module must not pump an air during the drainage; otherwise the pump could be destroyed. If you drain the module, observe the level of solution in the feed tank.**

NANOFILTRATION MODULE DESCRIPTION

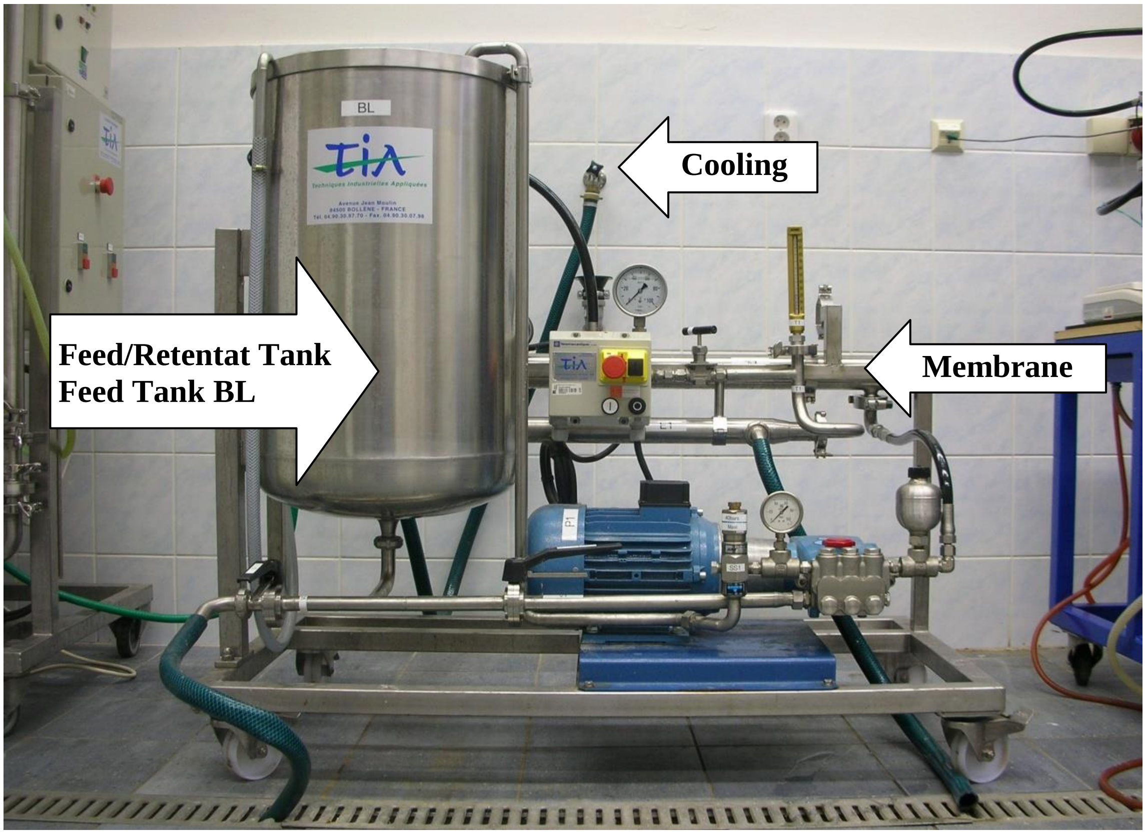

Fig. 1: Filtration unit TIA RO/NF System

Filtration unit TIA RO/NF System uses polymeric spiral wound nanofiltration and reverse osmosis membranes with a membrane area of 2.6 m^2.

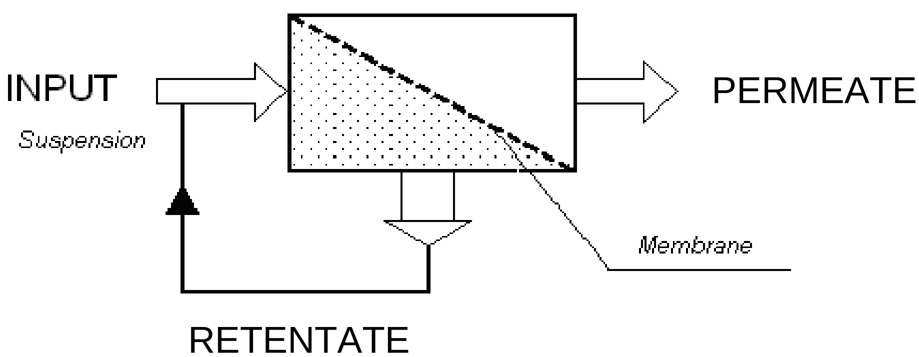

Fig. 2: Set-up of the filtration process.

BASIC TERMINOLOGY

- *Trans-membrane pressure* is the main driving force of the process.
- *Temperature* is a function of viscosity and characteristics of the flow. Higher temperature reduces viscosity, therefore the filtration velocity is higher too.
- *Concentration polarization* causes flux decline within a time and retards filtration.
- Characteristics of the suspension/solution is given by its texture, particle size distribution, concentration, density.
- *Retentate* is the concentrated stream.
- *Permeate* is the filtrate (the diluted stream).

THE GOALS OF THE COURSE

Nanofiltration of sucrose solution or cheese whey solution

- Prepare sucrose solution or cheese whey solution. The teaching assistant will tell you what solution you will need.
- Observe the change of the solution composition before and after the filtration using the refractometer
- Obtain a concentrate (retentate)
- Measure the filtration kinetics, the mass concentration factors.

PROCEDURE:

Preparation

1. Rinse the filtration module thoroughly using tap water. Then fill about half of the feed tank with demineralised water.

Measuring of pure water flow rate (water capacity)

2. Run the pump and let the system degas. Set the pressure to 10 bar. Once ready, open the permeate valves and let the permeate run out. Measure the volumetric flow of permeate (l/h) by collecting permeate into a graduated cylinder for 10 seconds or volumetric mass flow (kg/h) by measuring the weight of permeate for 10 seconds. Record current temperature and pressure.
3. Calculate the pure water flow rate using following formula:

$$Jw = \frac{P \cdot KT}{S \cdot \Delta p}$$

where J_W is a pure water flow rate (l/h.m^2) at pressure 1 bar and temperature 20 °C, P is volumetric flow of permeate (l/h), S is membrane area (2.6 m^2), Δp is average trans-membrane pressure (bar) calculated as upper pressure + lower pressure divided by two, and KT is a temperature coefficient (given for each temperature, see supplement tables).

4. Carry out the measurement several times and calculate the average pure water flow rate.
5. Repeat the same procedure after the filtration and module cleaning. Compare the values of pure water flow rate before and after the filtration. It they differ by more than 20 %, additional cleaning is necessary using stronger cleaning solution.

Filtration

6. Empty the filtration module. Close the drainage valve.
7. Either prepare 30 litres of a sugar solution or use the cheese whey permeate from previous ultrafiltration. The assistant will tell you which solution you will use for nanofiltration.
8. Measure the precise weight of feed solution.
9. Pour the suspension into the feed tank.
10. Start the pump. Set gently the pressure-difference of 20 bar and set the temperature 35°C.
11. Filtration proceeds in a mode with retentate recycling into the feed tank.
12. Open the cooling water valve.
13. Take sample of the feed solution.
14. Start filtration by opening the permeate valve on a membrane.
15. In regular intervals (every 3 minutes) during the filtration, measure the weight of permeate, record the temperature and pressure, eventually keep both parameters constant by regulation valves (pressure, temperature, permeate flow).
16. Filter until there is some retentate left in the feed tank. Watch the level in the feed tank carefully. When the feed tank is nearly empty, stop the filtration. **The pump must not run without any liquid!!!**
17. Take the sample of retentate. Measure the weight of permeate.
18. Empty the module into sewage using the pump.
19. Turn-off thermostat and cooling water.
20. Rinse the module several times with water. When the water in the feed tank is clear, you can do the chemical cleaning procedure.

Determination solids content in retentate:

21. Measure the content of sucrose in permeate, retentat and feed by refractometer (in °BRIX = *weigth %*) after the filtration and discuss the results.

Chemical cleaning of the filtration unit

22. The teaching assistant will decide what kind of chemical cleaning you will use.
23. Recirculate cleaning solution at least for 1 hour.
24. Then discharge the cleaning solution into sewage.
25. Rinse with water several times
26. Measure the pure water flux again to know how effective the cleaning procedure was by comparison with the pure water flux measured before the filtration.

The protocol and result presentation

27. Calculate the mass balance, i.e. mass of retentate. Calculated the mass concentration ratios and average permeate flow rate in $kg/h.m^2$.
28. Plot the time dependence of permeate flow rate and explain the shape of the curve.
29. Discuss the values of pure water flux before the filtration and after the membrane cleaning.

Isolation and separation of food components by membrane processes

PART III

NANOFILTRATION AND DESALINATION ON THE FILTRATION UNIT ARNO 600 USING FLAT MEMBRANES

SAFETY INSTRUCTIONS

- **Be extra cautious during module cleaning! Hot Ultrasil 115 (sodium hydroxide solution) and Ultrasil 73 (acid solution) are used.**
- **During the filtration, check the pressure. The maximum pressure inside the module should not exceed 30 bar (3 MPa).**
- **Maximum temperature inside the module must not exceed 40 °C. Use water cooler to keep temperature constant.**
- **Do not discharge hot cleaning solutions into the sewage. You have to cool them down before discharging.**
- **The pump in the filtration module must not pump an air during the drainage; otherwise the pump could be destroyed. If you drain the module, observe the level of solution in the feed tank.**

NANOFILTRATION MODULE DESCRIPTION

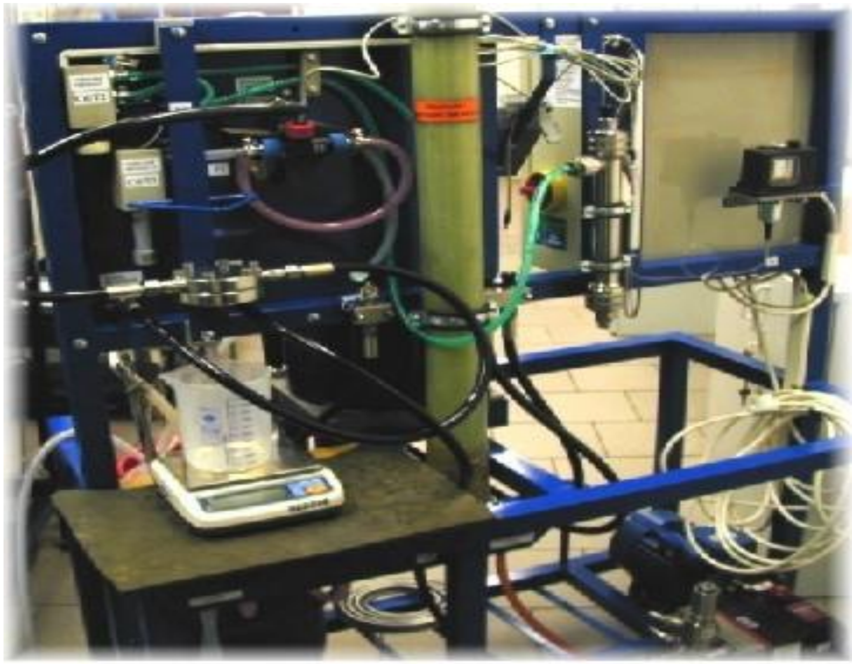

Fig. 1:Nanofiltration module in the filtration unit ARNO 600.

Nanofiltration dynamic cell is a part of the filtration unit ARNO 600 /Mikropur, Czech republic). Flat polymeric membranes are used for the filtration. The filtration area is 0.0044 m^2.

BASIC TERMINOLOGY

– *Trans-membrane pressure* is the main driving force of the process.
– *Temperature* is a function of viscosity and characteristics of the flow. Higher temperature reduces viscosity, therefore the filtration velocity is higher too.
– *Concentration polarization* causes flux decline within a time and retards filtration.
– The characteristics of the suspension/solution is given by its texture, particle size distribution, concentration, density.
– *Retentate* is the concentrated stream.
– *Permeate* is the filtrate (the diluted stream).

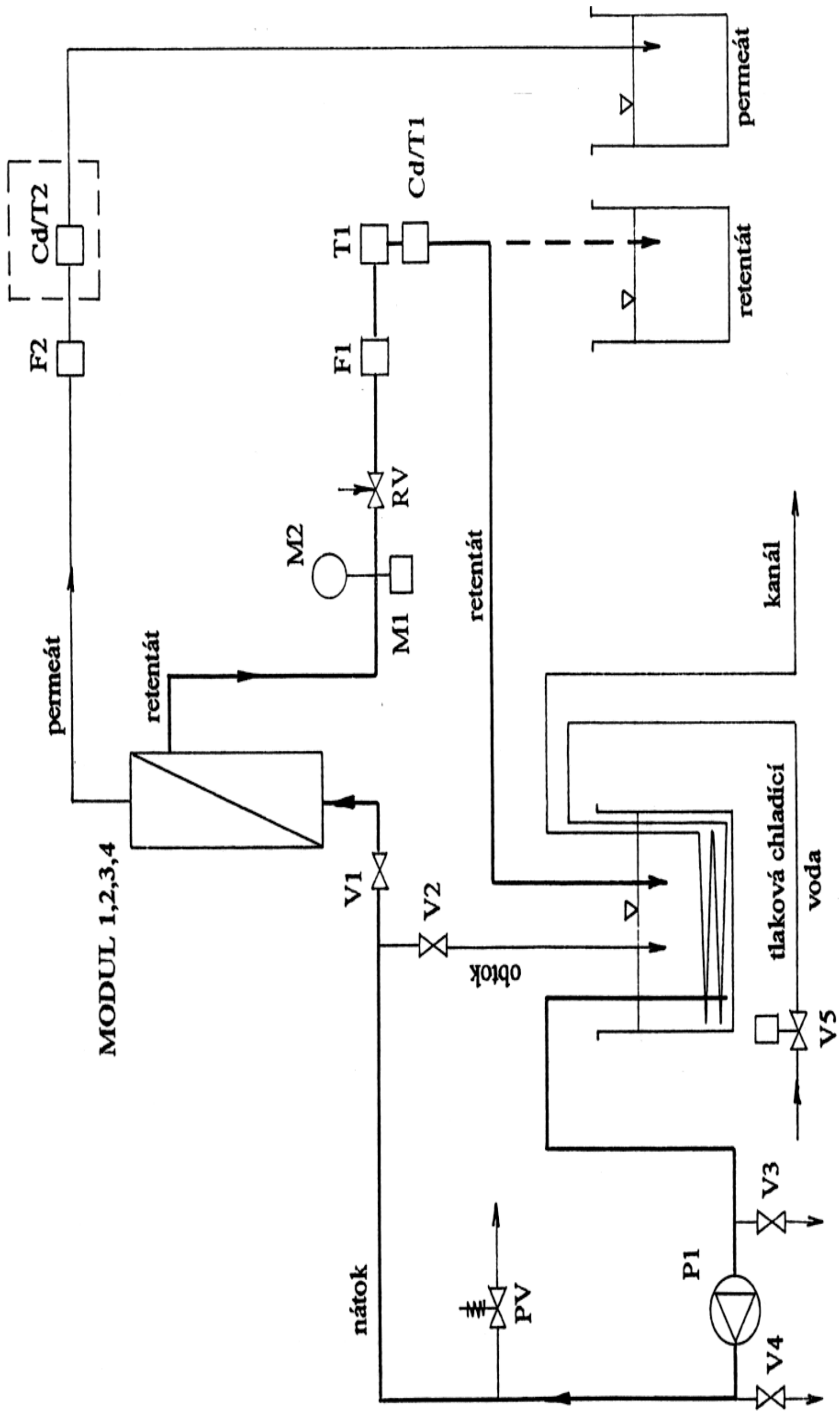

Fig. 2: The scheme of the nanofiltration in the filtration unit ARNO 600.

THE THEORITICAL BACKGROUNG

The aims of nanofiltration in whey processing are:

- Reduce the mineral salt content of whey, while the amount of valuable compounds (protein, lactose) remains constant.
- NF membranes have a low retention toward monovalent ions, but multivalent ions and disaccharides are highly retained.
- The separation mechanism is based on steric and electrical effects.
- NF membranes are mostly composed of a thin film composite layer and are negatively charged at neutral pH, therefore the charge of solution components and membrane affects the separation as well.

THE GOALS OF THE COURSE

The students will measure the kinetics of the process (i.e. dependence of flow permeate rate on time, temperature and working pressure relative to unit filtration area).in experimental work.

PROCEDURE:

Preparation

1. Soak the membrane at least for 30 minutes in demineralised water before the filtration.
2. Place the membrane into the module, tight all screws well.
3. Rinse the filtration module thoroughly using tap water. Close the drainage valve. Then fill about one third of the feed tank with demineralised water.

Measuring of pure water flow rate (water capacity)

4. Close the inlet valve to nanofiltration module. Open the pressure valve and recirculation (by-pass) valve.
5. Run the pump and let the system degas. Open the valve pumping solution into the module. Slowly close the by-pass valve until it is completely closed.
6. Set gently the pressure to 10 bar. Measure the mass flow of permeate (g/min) by collecting permeate into a flask placed on a balance for 60 seconds. Record current temperature and pressure.
7. Calculate the pure water flow rate using following formula:

$$Jw = \frac{P \cdot KT}{S \cdot \Delta p}$$

where J_W is a pure water flow rate (l/h.m^2) at pressure 1 bar and temperature 20 °C, P is volumetric flow of permeate (l/h), S is membrane area (0.0044 m^2), Δp is average trans-membrane pressure (bar), and KT is a temperature coefficient (given for each temperature, see supplement tables). Water density is 1000 kg/m^3.

8. Carry out the measurement several times and calculate the average pure water flow rate.
9. Repeat the same procedure after the filtration and module cleaning. Compare the values of pure water flow rate before and after the filtration. It they differ by more than 20 %, additional cleaning is necessary using stronger cleaning solution.

Filtration

10. Empty the filtration module. Close the drainage valve.
11. Either prepare 30 litres of a suspension of sweet whey powder in water of an approximate concentration 15 g/l or you will filter permeate from previous ultrafiltration. The assistant will tell you which solution you will use for nanofiltration.
12. Measure the precise weight of feed solution.
13. Pour the suspension into the feed tank.
14. Close the inlet valve to nanofiltration module. Open the pressure valve and recirculation (by-pass) valve.
15. Filtration proceeds in a mode with retentate recycling into the feed tank.
16. Open the cooling water valve. Set the temperature using ALMEMO control panel to 35 °C.
17. Run the pump.
18. Take a sample of the feed solution.
19. Start filtration by opening the inlet into a module and closing the by-pass valve.
20. Set gently the pressure-difference of 20 bar.
21. In regular intervals (every 3 minutes) during the filtration, measure the weight of permeate, record the temperature and pressure, eventually keep both parameters constant by regulation valves (pressure, temperature, permeate flow).
22. Filter until there is some retentate left in the feed tank. Watch the level in the feed tank carefully. When the feed tank is nearly empty, stop the filtration. **The pump must not run without any liquid!!!**

23. Take the sample of retentate.
24. Measure the mass of permeate.
25. Empty the module into sewage using the pump.
26. Turn-off thermostat and cooling water.
27. Rinse the module several times with tap water. When the water in the feed tank is clear, you can do the chemical cleaning procedure.

Chemical cleaning of the filtration unit

28. The teaching assistant will decide what kind of chemical cleaning you will use, if any.
29. Recirculate cleaning solution at least for 1 hour.
30. Then discharge the cleaning solution into sewage.
31. Rinse with water several times.
32. Measure the pure water flux again to know how effective the cleaning procedure was by comparison with the pure water flux measured before the filtration.

The protocol and result presentation

33. Calculate the mass balance, i.e. mass of retentate. Calculated the mass concentration ratios and average permeate flow rate in kg/h.m^2 if you know that sweet whey density is 1040 kg/m^3.
34. Plot the time dependence of permeate flow rate and explain the shape of the curve.
35. Discuss the values of pure water flux before the filtration and after the membrane cleaning.