Isolation and separation of food components by membrane processes

PART I

MICROFILTRATION AND ULTRAFILTRATION

SAFETY INSTRUCTIONS

- **Be extra cautious during module cleaning, when using hot acid and hydroxide solution!**
- **During the filtration, check the pressure regularly. The maximum pressure inside the module must not exceed 5 bar (0, 5 MPa).**
- **Maximum temperature inside the module must not exceed 80 °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.**
- **If you drain the module, use the alimentation pump only. Circulation pump must not run without any liquid inside the system!**

ULTRA- AND MICROFILTRATION MODULE DESCRIPTION

Filtration module T.I.A. (Bollene, France) is equipped with two tubular ceramic membranes Membralox of various a pore sizes. For the experiments we usually use membranes with cut-off 20 and 100 nm. Filtration area is 0.2 m^2 / one membrane (Fig. 1).

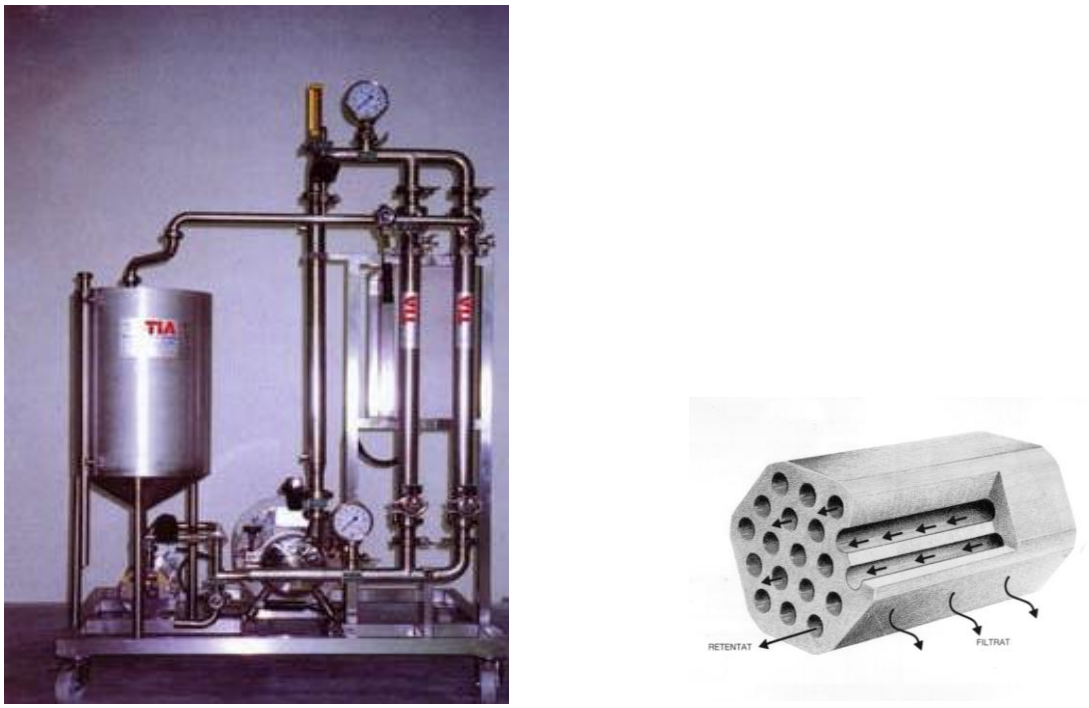

Fig. 1: Filtration unit for micro- and ultrafiltration TIA (left) and tubular ceramic membrane MEMBRALOX (right).

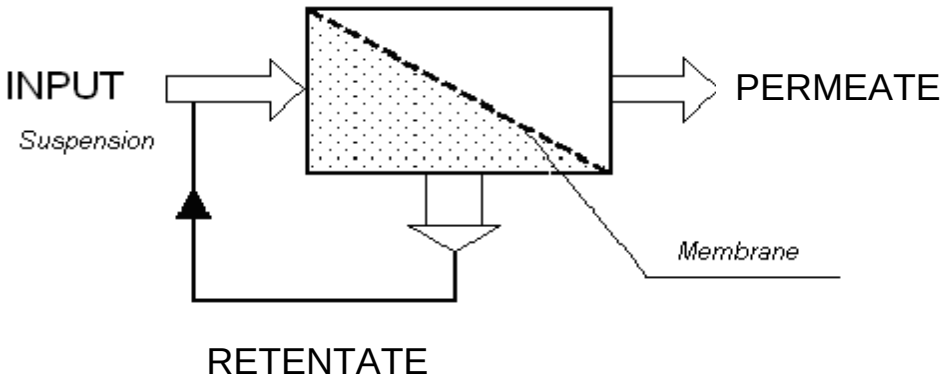

Fig. 2: Set-up of the filtration process.

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.
- The characteristic of the suspension/solution is given by its texture, particle size distribution, concentration and density.
- *Retentate* is the concentrated stream.
- *Permeate* is the filtrate (the diluted stream).

THE GOALS OF THE COURSE

Students will study the kinetics of the process (i.e. the dependence of permeate flow rate on time, filtration area, temperature and working pressure) and study the changes in concentration of the initial solution during filtration.

Students will measure a permeate flow rate J (l/h.m^2.bar): Measure the volume of a permeate sample collected into a graduated cylinder for given period (e.g. 10, 20 or 30 seconds). Record the temperature and the pressure and converse the volumetric flow rate to the temperature of 20 °C and membrane area of 1 m^2 according to formula:

$$J = \frac{P \cdot KT}{S \cdot p}$$

where P is volumetric flow rate of permeate (l/h), S is membrane area (m^2), p is a pressure-difference (bar), and KT is a temperature coefficient for viscosity (see Tab. I).

PROCEDURE:

Preparation

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

Measuring of pure water flow rate (water capacity)

2. Run the alimentation pump and let the system degas. Increase the pressure slightly and run the circulation pump. Set the pressure. 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 – 30 seconds. Record the temperature and the pressure.
3. Calculate the pure water flow rate using following formula:

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

where J_W is a pure water flow rate (l/h.m^2) at pressure one bar and temperature 20 °C, P is volumetric flow of permeate (l/h), S is membrane area (m^2), p is trans-membrane pressure (bar) and KT is a temperature coefficient (given for each temperature, see supplement, Table I).

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. Prepare a suspension of sweet whey powder in water with the total volume of 30 litres and approximate concentration 15 g/l.
8. Pour the suspension into the feed tank.
9. Start the alimentation pump and let the system degas. Increase the pressure slightly and run the circulation pump. Set the pressure-difference (1 bar).
10. Let water go to the cooler.
11. Filtration proceeds in a mode with retentate recycling into the feed tank.
12. Start filtration by opening the permeate valve on a chosen membrane (the assistant will tell you which membrane you will use).
13. In regular intervals (2-3 minutes) during the filtration, take samples of retentate, record the temperature and pressure, eventually keep both parameters constant by

regulation valves (pressure, temperature, permeate flow). Also, measure the flow rate of permeate.

14. 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 circulation pump must not run without any solution!!!** Turning PA pump off will automatically switch the PC pump as well.
15. Empty the module into sewage using the alimentation pump.
16. Rinse the module several times with water. When the water in the feed tank is clear, you can do the chemical cleaning procedure.

Determination of solid content in retentate:

17. Determine solids content in the samples of retentate using direct oven drying method: Before drying, it is necessary to dry weighing dishes with polymeric microfilters in oven approximately for 20 minutes. Remove from the oven and let cool down in a desiccator. After cooling, weigh empty weighing dishes on an analytical balance.
18. Mix well the sample of retentate, pipette 10 ml of sample and filter over the polymeric microfiltration membrane taken out of the weighing dish. Place the microfilter back into the weighing dish and dry in the oven at the temperature 70 °C for 2 hours. Cool down the weighing dishes in a desiccator again and weigh.
19. Determine the dry solid content and express as mass of dry substances in 1 litre of retentate (g/l).

Chemical cleaning of the filtration unit

20. The teaching assistant will decide what kind of chemical cleaning you will use. It can be either alkaline or acid cleaning process:
    - Alkaline cleaning process: Wash with 2% NaOH at 70°C for 1 hour.
    - Acid cleaning process: Washing with 2% HNO_3 at 70°C for 1 hour.
21. During cleaning first turn off the cooler and let the filtration unit heat up by cleaning solution circulation. When the temperature reaches 70 °C, check the necessity to turn the cooling system on or if the temperature is stable you can let it without cooling.
22. Recirculate cleaning solution at least for 1 hour. Then turn the cooler on. **Do not discharge hot cleaning solutions into the sewage!** You have to cool them down before discharging.

23. When the temperature is less than 30 °C, you can discharge the solution.
24. Rinse with water several times.
25. 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

26. Plot the time dependence of permeate flow rate and explain the shape of the curve.
27. Discuss the values of pure water flux before the filtration and after the membrane cleaning.
28. Create a dependence of retentate concentration in time. Discuss the results.

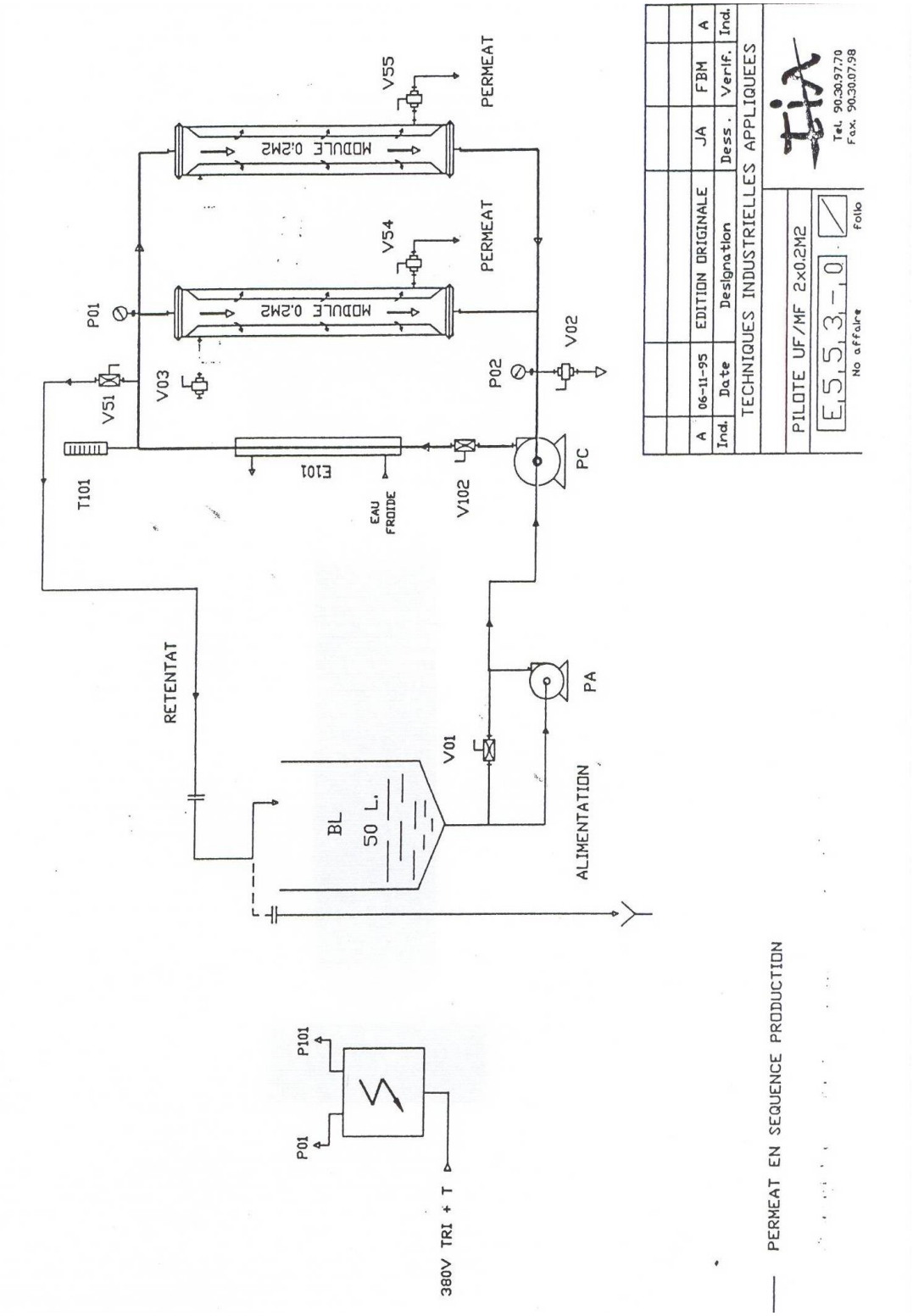

Fig. 3: Scheme of the filtration unit.

Tab. I: Dependence of the viscosity correlation coefficient on the temperature.

°C	KT	°C	KT
1	1,713	51	0,542
2	1,653	52	0,534
3	1,604	53	0,546
4	1,554	54	0,518
5	1,505	55	0,511
6	1,455	56	0,503
7	1,416	57	0,495
8	1,376	58	0,488
9	1,317	59	0,480
10	1,297	60	0,472
11	1,257	61	0,466
12	1,228	62	0,460
13	1,188	63	0,454
14	1,158	64	0,448
15	1,129	65	0,442
16	1,099	66	0,436
17	1,069	67	0,424
18	1,050	68	0,418
19	1,020	69	0,411
20	1,000	70	0,411
21	0,973	71	0,405
22	0,950	72	0,400
23	0,929	73	0,395
24	0,908	74	0,390
25	0,887	75	0,387
26	0,867	76	0,382
27	0,849	77	0,377
28	0,831	78	0,372
29	0,813	79	0,367
30	0,786	80	0,363
31	0,780	81	0,359
32	0,764	82	0,355
33	0,748	83	0,351
34	0,734	84	0,347
35	0,720	85	0,344
36	0,706	86	0,340
37	0,632	87	0,336
38	0,679	88	0,332
39	0,667	89	0,328
40	0,655	90	0,325
41	0,645	91	0,321
42	0,635	92	0,318
43	0,624	93	0,315
44	0,613	94	0,312
45	0,602	95	0,309
46	0,590	96	0,306
47	0,580	97	0,303
48	0,570	98	0,300
49	0,550	99	0,297
50	0,550	100	0.293