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PILOT PLANT UNIT FOR A CROSS - FLOW MICROFILTRATION AND ULTRAFILTRATION OF FERMENTATION BROTHS*

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ABSTRACT

A detailed description of an automated pilot plant unit of our original design is given. The operating parameters such as transmembrane pressure drop, cross-flow velocity, permeate flow rate, temperature and pH can be controlled and continuously monitored during test runs. The pilot plant operation is controlled by an industrial programmable logic controller, connected to a personal computer. An original SCADA application was developed which enables a remote control of the pilot plant and data aquisition. The pilot plant can be run either by static or dynamic counterpressure of the permeate. The dynamic counterpressure of the permeate assures a uniform transmembrane pressure drop (UTP) along the whole filtration element. Three different modes of operation can be selected: a constant transmembrane pressure drop mode, a constant flux mode and a stand-by mode for startup and cleaning operations. The unit is succesfully used for the microfiltration and ultrafiltration of various fermentation broths. Some experimental results are shown and discussed.

INTRODUCTION

Membrane separation techniques such us microfiltration, ultrafiltration, nanofiltration and reverse osmosis have been in use for years by the Pharmaceutical and Chemical Company LEK. Cross-flow microfiltration is succesfully used for the filtration of various fermentation broths. Some of the advantages of the cross-flow

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*Dedicated to professor Drago Leskovšek on his 80th birthday.

microfiltration are high quality of the filtrate, continuous mode of operation, high degree of automation and simple scale-up [1, 2, 3, 4]. Due to the complex nature of fermentation broths, it is very difficult to predict their behaviour during filtration [5]. The only way of obtaining reliable design parameters for the production scale plant is to run pilot plant tests under different operating conditions and with different membranes. Important parameters such as transmembrane pressure drop, cross-flow velocity, permeate flow rate, temperature and pH need to be carefully controlled and continuously monitored during test runs. This requires an automated pilot plant unit, equipped with measuring sensors, capable of continuous data aquisition. To satisfay such requirements we designed our own pilot plant unit, which we use for small scale microfiltration or ultrafiltration studies and pilot plant production. The flexibility of the plant construction allows the use of various types of modules and membranes. Tests can be run either batch or continuous at constant transmembrane pressure drop or constant permeate flux.

A special feature of the pilot plant is to run microfiltration tests with a dynamic permeate counterpressure, using a permeate recirculation pump and specially designed filtration modules. The dynamic counterpressure of the permeate assures a uniform transmembrane pressure drop (UTP) along the whole filtration element [6, 7, 8, 9, 10]. The operation of the pilot plant is controlled by an industrial programable logic controller (PLC). Measured values are transferred from the PLC to the personal computer.

DESCRIPTION OF THE PILOT PLANT UNIT

The pilot plant unit has been constructed from two feed tanks, a high pressure feed pump, a retentate recirculation pump, a permeate recirculation pump, a filtration module (membrane modul), a heat exchanger, measuring sensors, regulating valves, an electro-cabinet, tubing and valves. Figure 1 shows a simplified flow-chart of the plant.

The feed is pumped from one of the stainless-steel feed-tanks to the retentate recirculation loop by the high pressure centrifugal pump P1 (Grundfos CRN 2-90, Q=2 m/h^3, H=65 m). The flow-rate of the feed is regulated by a manual ball valve V1. The retentate cross-flow is maintained with the recirculation pump P2 (Grundfos CRN 16-30, Q=16 m/h^3, H=34.4 m). Cross-flow velocity is regulated manually with the ball valve V2. The flow-rate of the retentate in the retentate recirculation loop is measured by an electromagnetic flowmeter FI 1 (Promag 30A, Endress+Hauser, Germany) which is built in the loop. The outlet pressure of the retentate is regulated by an electropneumatic regulating valve RV 1 (valve type 241, IP positioner type 4763, pneumatic actuator type 3271, valve K_{vs}=0.4, Samson, Germany). The retentate flow-rate is measured by a simple rotameter FI 3 (60-640 l/h, Gemü, Germany). The backpressure of the permeate or the permeate flow-rate is controlled by an electropneumatic regulating valve RV 2 (valve type 241, IP positioner type 4763, pneumatic actuator type 3271, valve K_{vs}=0.25, Samson, Germany) and the flow-rate of the permeate is measured by an electromagnetic flowmeter FI 2 (Tecmag Picomag II T DMI 6733, Endress+Hauser, Germany). When operating with dynamic permeate counterpressure the permeate is recirculated through the fitration module with the centrifugal pump P3 (Grundfos CRN 4-30, Q=4 m/h^3, H=23.1 m). The pressure drop of the permeate in the module is regulated manually by adjusting the flow-rate of the recirculating permeate with the ball valve V3. The retentate and permeate inlet and outlet pressures are measured with piezoelectric sensors PT1, PT2, PT3 and PT4 (Cerabar PMC 532 with local LC display Cerabar VU 130, Endress+Hauser, Germany). To cool the retentate, a tube and shell heat exchanger HEx is built in the retentate recirculation loop. The temperature of the retentate is regulated by a thermostat TC, coupled to a regulating valve RV 3 (thermostat type 2430, regulating valve type 2432, Samson, Germany) which controls the outflow of the cooling water from the heat exchanger. The temperature of the retentate is measured by the Pt100 sensor TT1 (Wikatronic, Wika, Austria). Both feed tanks are equipped with pH standard weld-in sockets (Ingold, Mettler Toledo, Switzerland) in which standard pH electrode housings (InFit 761-25BT/70, Mettler Toledo, Switzerland) can be mounted. The pH electrode (HA405-DPA-SC-S8/120, Mettler Toledo, Switzerland) is connected to a pH meter pHT (Liquisys CPM 252, Endress+Hauser, Germany).

The pilot plant has been designed to run microfiltration and ultrafiltration tests using Membralox® 1P19-40, 1P19-40 GL and 1P19-60 modules (US Filters, Societe

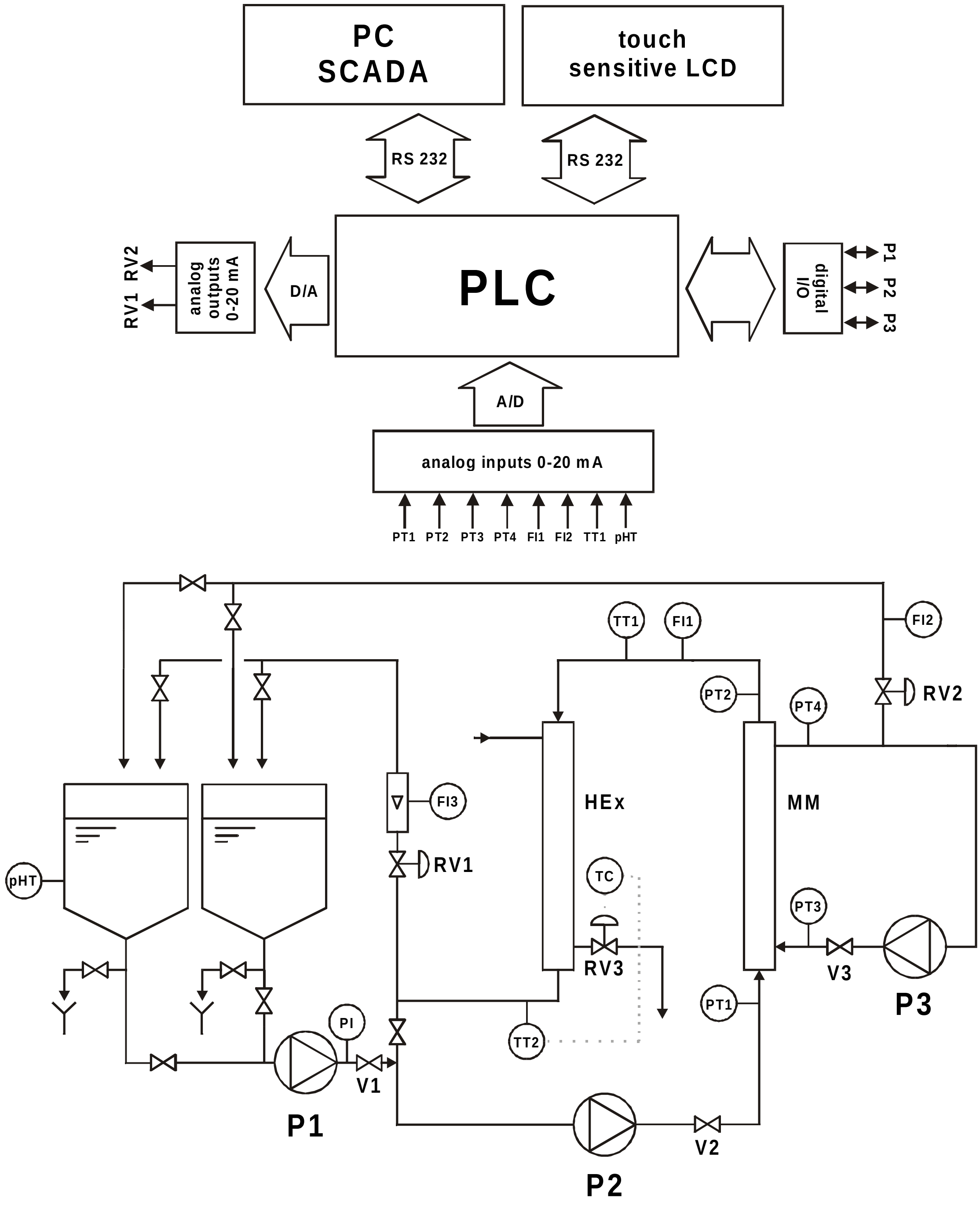


Figure 1. Simplified flow-chart of the microfiltration and ultrafiltration pilot plant unit. Legend: P1-high pressure feed pump, P2-retentate recirculation pump, P3-permeate recirculation pump, PI-feed pressure indicator, PT1-retentate inlet pressure sensor, PT2-retentate oulet pressure sensor, PT3-permeate inlet pressure sensor, PT4-permeate oulet pressure sensor, FI 1-retentate flow-meter, FI 2-permeate flow-meter, FI 3-retentate outlet flow-meter, TT1-Pt 100 temperature sensor, RV1-electropneumatic regulating valve for the retentate outlet pressure control, RV2-electropneumatic regulating valve for the permeate flow-rate or permeate back-pressure control, TT2-temperature sensor, TC-temperature regulator, RV3-cooling water outflow regulating valve, pHT-pH meter, PLC-programmable logic controller, HEx-tube and shell-heat exchanger, MM-membrane module.

des Ceramiques Techniques, France) fitted with various Membralox® ceramic membrane filter elements (P37-30 850, filtration area 0.3 m^2 with 3 mm dia. channels, P19-40 850, filtration area 0.2 m^2 with 4 mm dia. channels, P19-40 1020, filtration area 0.24 m^2 with 4 mm dia. channels and P19-60 850, filtration area 0.3 m^2 with 6 mm dia. channels). When using a dynamic counterpressure, the Membralox® modules have to be filled with polypropylene spheres (4 mm dia.) on the permeate side. The flexibility of the plant is achieved by using clamps which allow for mounting of any other module.

The pilot plant unit operation is controlled by an industrial programmable logic controller (Sysmac CQM1, CPU 45-V1, Omron, Japan). The PLC is connected through two RS232 ports with a touch sensitive colour LC display (NT 30C-ST141-E, Omron, Japan) and to a personal computer. The programmable logic controller has eight analog inputs (0-20 mA), two analog outputs (0-20 mA), eight digital inputs and eight digital outputs. The PLC is programmed to control three feedback control loops, i.e. retentate outlet pressure, transmembrane pressure drop and permeate flow rate (Figure 5). A simplified flow-chart of the pilot plant unit as well as the menu and the mode of operation keys are shown on the display. Pumps are switched on/off by pressing the touch sensitive pump symbols on the flow-chart. Similary, menus and modes of operation are chosen by pressing touch sensitive keys. The set points, low and high alarm limits, regulators constants and other parameters can be set in menus. A beeping sound and blinking light are turned on to signalize that the alarm limit, either low or high, is reached, while the alarm description is shown in the alarm history log.

Measured values are transferred from the PLC to the personal computer where an Omron SYSMAC-SCS SCADA (Supervisory Control and Data Acquisition) software package is installed. An original SCADA application was developed by the author of this work D. Senica (Figures 2 and 3). The SCADA application allows for interactive remote control of the pilot plant from the personal computer. Pumps can be switched on/off by clicking pump symbols on the flow-chart and the regulating valves can be manually adjusted. Several modes of operation can be selected and set points, low and high alarm limits, regulator constants and other parameters can be changed from the application. Alarms and other events, such as set point and modes of operation changes are printed together with time and date marks on the printer to keep a pilot plant unit

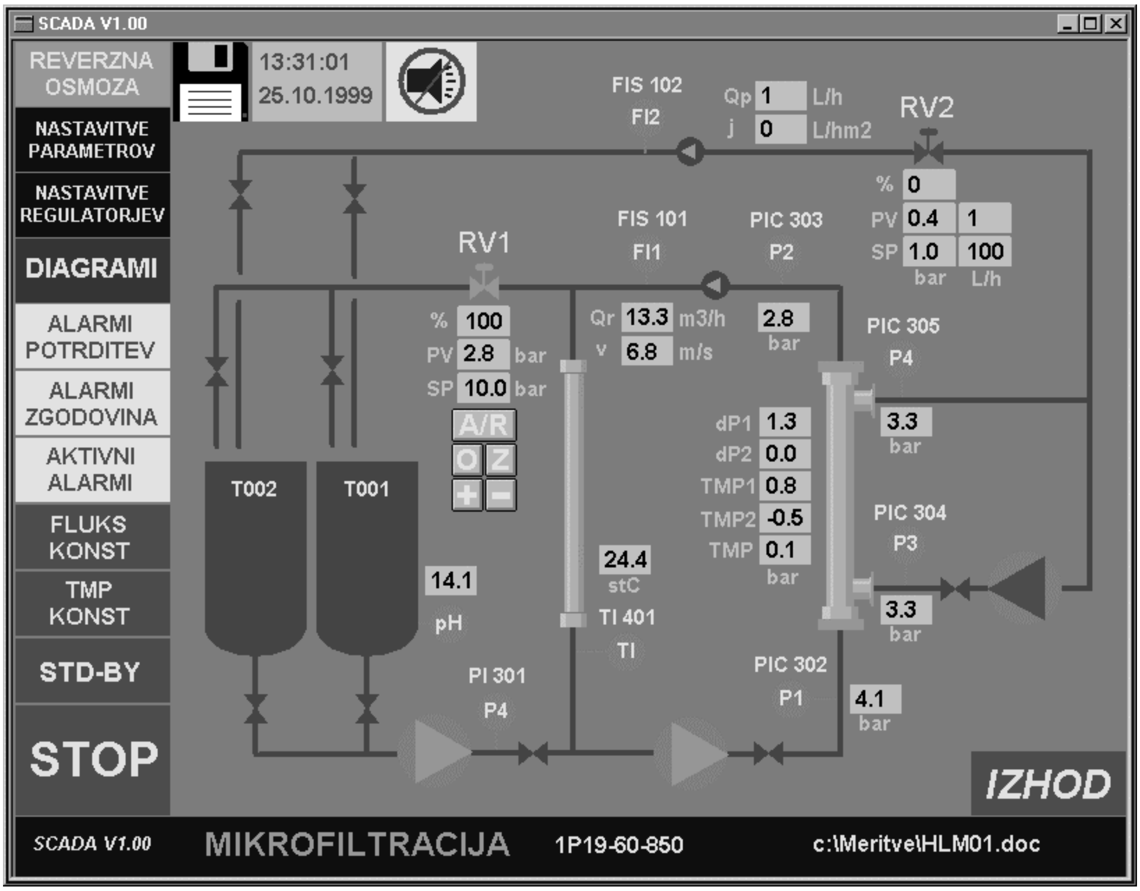


Figure 2. Simplified flow-chart of the pilot plant unit shown in the SCADA application, running on a personal computer. The application allows for an interactive remote control of the pilot plant unit and data acqusition.

history log. Measured values of permeate and retentate pressures, retentate and permeate flow-rates, temperature and pH can be wieved on a simplified flow-chart (Figure 2) or on real time charts (Figure 3). Values can be stored in a text file on a hard disc drive in regular time intervals set by the user. After the test run, stored data can be imported to a spreadsheet program for further evaluation.

RANGE OF OPERATING PARAMETERS

The pilot plant unit can operate in a relatively broad range of operating parameters. Volumes of the feed tanks are approximately 80 liters per tank. Since a dead volume of the retentate side is approximately 15 L, maximum volumetric concentration

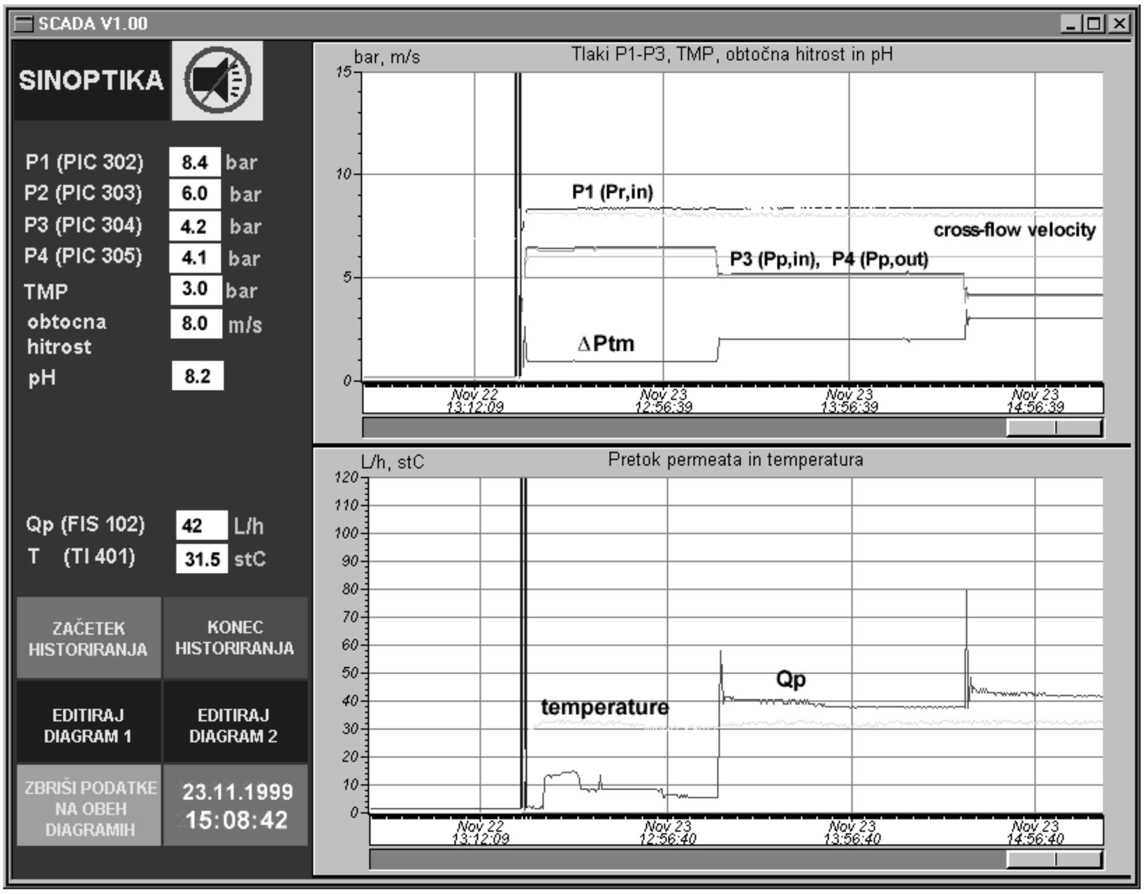


Figure 3. Measured values of permeate and retentate pressures, retentate and permeate flow-rates, temperature and pH are displayed on two real time charts in the SCADA application.Values can be stored in a text file on the hard disc drive in regular time intervals set by the user and after the test run imported to a spreadsheet program for further evaluation.

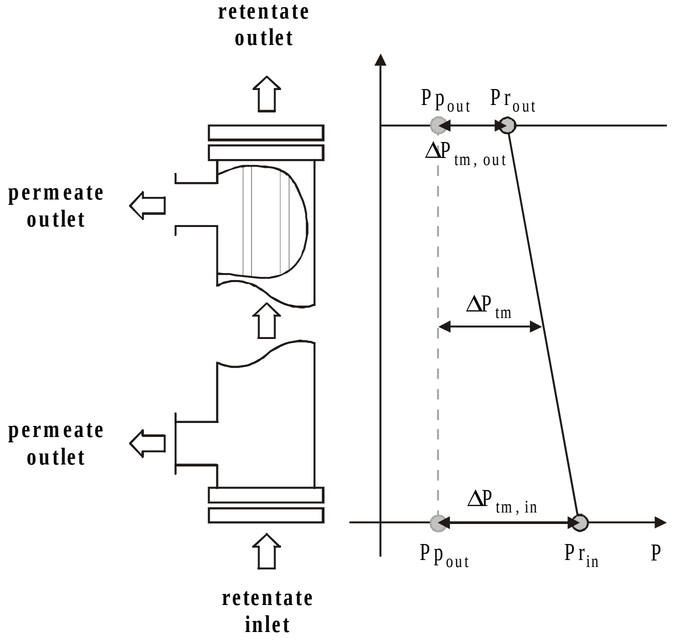
factors (VCF) of about 3 to 6 can be reached in a batch concentration mode. The filtering areas of the Membralox® filtration modules are 0.2 to 0.3 m^2, depending on the module and the filter element (P19-40, P19-40 GL or P19-60) used. The measuring range of the permeate flowmeter FI 2 is set to the maximum value of 100 l/h which corresponds to maximum flux of 500 L/hm^2 with P19-40 (0.2 m^2 of filtering area) and 330 L/hm^2 with P19-60 (0.3 m^2 of filtering area) filtration elements. The measuring range of the FI 2 can be changed to higher values if needed.

The maximum retentate inlet pressure is 11 to 12 bars and the maximum retentate outlet pressure approximately 10 bars, depending on the retentate pressure drop in the filtration module. The maximum transmembrane pressure drop is about 8-9 bars. It depends on the filtration element used (P19-40 or P19-60) and on the flow-rate of the recirculating retentate, i.e. on the pressure drop of the retentate in the filtration module. With Membralox® P19-60 filter elements, the maximum retentate flow-rate is 13 m^3/h at a pressure drop of 1.3 bars. The corresponding maximum cross-flow velocity is therefore aproximatelly 6.5 m/s. With Membralox® P19-40 filter elements the maximum retentate flow-rate is 7.6 m^3/h due to the higher pressure drop of the retentate in the module, approximately 2 bars. The maximum cross-flow velocity for P19-40 filter elements is approximatelly 8.5 m/s and is higher than for P19-60 filter elements. During batch concentration, the cross-flow velocity cannot be kept constant due to a high dependence of the retentate recirculation pump flow-curve on the viscosity of the retentate.

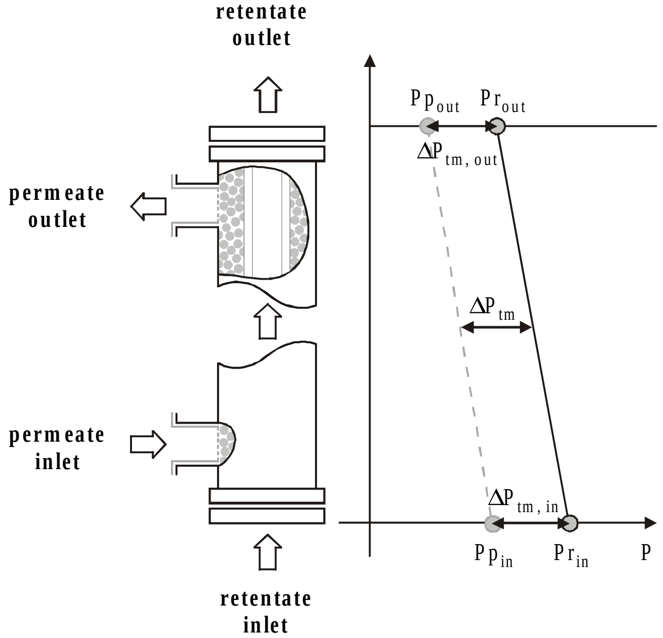
STATIC AND DYNAMIC PERMEATE COUNTER-PRESSURE

Cross-flow microfiltration operates at high cross-flow velocity which results in high pressure drops on the retentate side and therefore in high retentate inlet (Pr_{in}) and outlet pressures (Pr_{out}). To keep the transmembrane pressure drop (ΔP_{tm}) low when desired, the permeate must be under static or dynamic counterpressure. A disadvantage of the static permeate counter-pressure is an unequal transmembrane pressure drop along the filtration element, which is high at the inlet ($\Delta P_{tm,\ in}$) and low at the outlet ($\Delta P_{tm,\ out}$) from the filtration module (Figure 4). A uniform transmembrane pressure drop (UTP) along the whole filtration element can be assured by using dynamic permeate counter-pressure [6, 7, 8, 9, 10].

To create a dynamic counter-pressure, the permeate has to be pumped through the bed of polypropylene spheres, which surrounds the filtration element, concurrently with the flow of the retentate (Figure 4). As can be seen from Figure 4, pressure drops of the permeate and retentate along the module should be equal. Desired pressure drop of the recirculating permeate along the module is set by adjusting its flow-rate. A relatively low permeate flow-rates are necessary because of the high flow resistance through the bed of spheres. For a precise control of very low transmembrane pressure drops, corrections need to be done because of the pressure drops between the pressure



static permeate counter-pressure



dynamic permeate counter-pressure

Figure 4. Retentate and permeate pressure gradients in the filtration module when operating with static or dynamic counter-pressure. Pressure gradient is constant along the module at the dynamic permeate counter-pressure operating conditions. Pressure drop profiles in the module are shown simplified only; pressure loss at the retentate/permeate inlets to the module and outlets from the module are not shown.

sensors and the retentate/permeate inlets to the module and outlets from the module [7, 9].

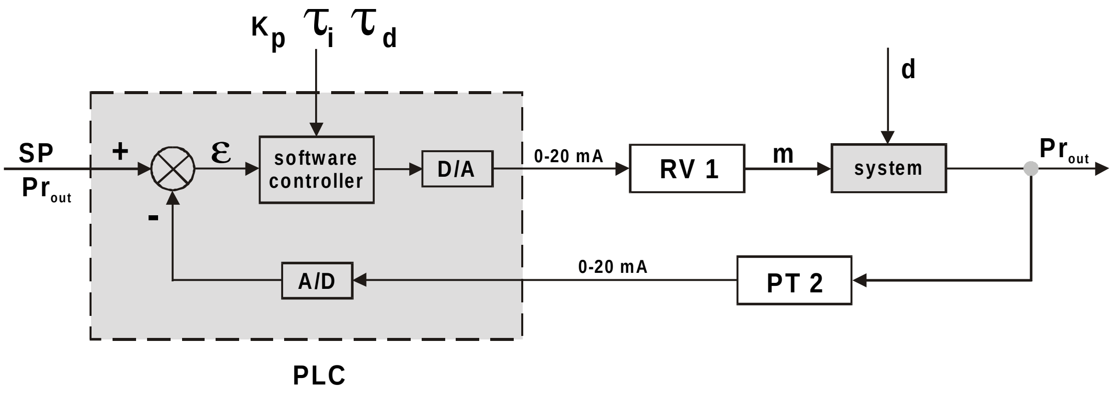
MODES OF OPERATION

The pilot plant was designed to run tests at the constant transmembrane pressure drop or at the constant flux. With the Membralox® ceramic microfiltration and ultrafiltration filter elements tests can be run with either static or dynamic permeate counter-pressure.

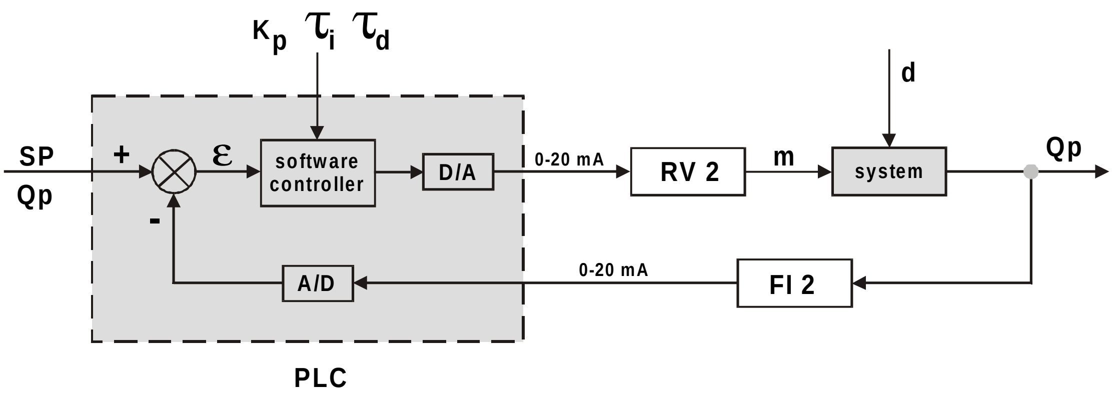
When filtration is performed with the **static permeate counter-pressure** and the *constant transmembrane pressure drop mode* is selected, either on the PLCs display or in the SCADA application, the control loops for transmembrane pressure drop and retentate outlet pressure are activated. The regulating valve RV 1 is closed until the preset pressure of the retentate Pr_{out} is reached. The preset transmembrane pressure drop is reached and kept constant by opening or closing the regulating valve RV 2 (Figures 1 and 5). Transmembrane pressure drop is calculated from measured retentate and permeate inlet and outlet pressures as follows from the equation $\Delta P_{tm}=(Pr_{in}+Pr_{out})/2 - (Pp_{in}+Pp_{out})/2$. Conversely, when the *constant flux mode* is selected, control loops for permeate flow-rate and retentate outlet pressure are activated. Regulating valve RV 1 is closed until the preset pressure of the retentate Pr_{out} is reached. The preset permeate flow-rate is reached and kept constant by opening or closing the regulating valve RV 2 (Figures 1 and 5). In both modes a desired cross-flow velocity of the retentate must be set manually by changing the flow-rate by adjusting the ballvalve V2.

When tests are run with the **dynamic permeate counter-pressure**, the permeate recirculation pump P3 has to be switched on. After the desired flow-rate of the retentate in the retentate recirculation loop has been set, the permeate presure drop has to be adjusted manually by changing the flow-rate of the permeate in the permeate recirculation loop with the ballvalve V3. After the desired permeate pressure drop is set, the filtration mode can be selected and control loops activated as described above.

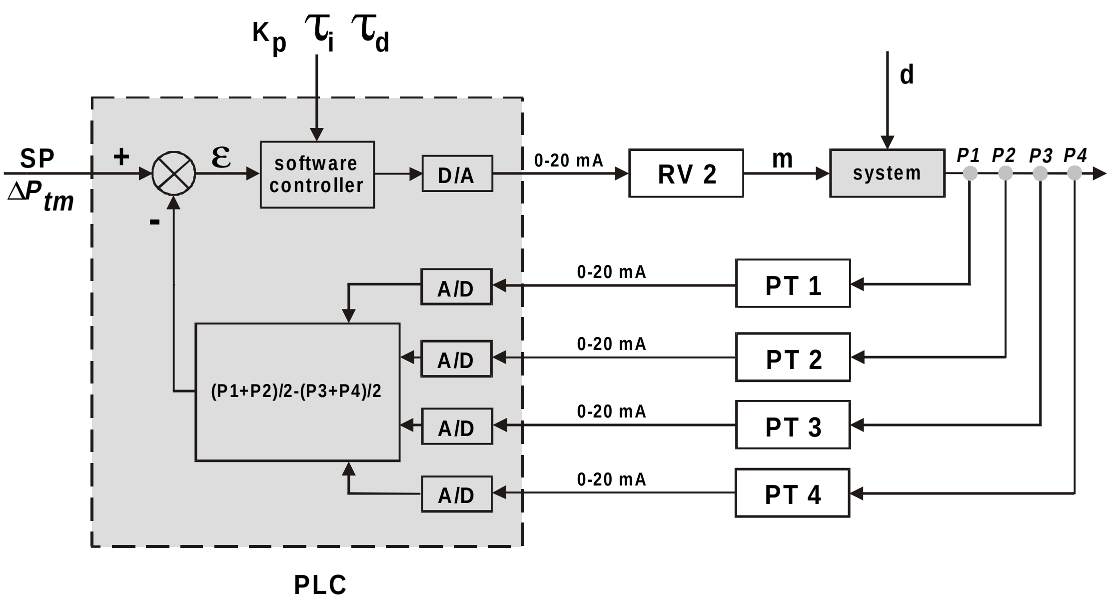
In the *stand-by* mode the regulating valve RV 1 is fully opened while the regulating valve RV 2 is fully closed and there is virtualy zero, or at least very low



retentate outlet pressure (Pr_{out}) feedback control loop



permeate flow rate (Qp) feedback control loop



transmembrane pressure (ΔP_{tm}) feedback control loop

Figure 5. Shematic presentation of the three feedback control loops designed for an automatic control of the retentate outlet pressure, the permeate flow-rate and the transmembrane pressure drop.

transmembrane pressure drop. This mode of operation is selected for the startup of the filtration and during cleaning operations.

SOME EXPERIMENTAL RESULTS

Some results of test runs are shown to illustrate experimental capabilities of the pilot plant unit.

The influence of the transmembrane pressure drop on the flux of the permeate during the microfiltration of the *Amycolatopsis orientalis* fermentation broth was studied using Membralox® ceramic microfiltration element P19-40 GL with 0.5 μm pore size, mounted in a special UTP module. The temperature, the cross-flow velocity and the concentration of the total solids were kept constant during the test run. The transmembrane pressure was changed gradually from 0.3 to 3 bars in regular time intervals. All parameters were continuously monitored. The time courses of the permeate flux, transmembrane pressure drop and the pressure difference ($\delta(\Delta P_{tm})= \Delta P_{tm,\ in} - \Delta P_{tm,\ out}$) between the transmembrane pressure drop at the inlet to the filtration module ($P_{tm,\ in}$) and the transmembrane pressure drop at the oulet from the filtration module ($P_{tm,\ out}$), during two test runs, are shown in the diagrams (Figures 6 and 7). The results clearly illustrate the advantages of the microfiltration with the dynamic permeate counter-pressure. The negligible pressure difference $\delta(\Delta P_{tm})$ confirms the uniformity of the transmembrane pressure drop along the whole filtration element. When the test was run with the static permeate counterpressure, the difference $\delta(\Delta P_{tm})$ was considerable.

The use of the constant flux mode is illustrated with the results of a study of a fouling phenomena during the microfiltration of *Micromonospora inyoensis* fermentation broth (Figure 8). The test was run with the static permeate counterpressure. Membralox® ceramic ultrafiltration element P19-60 with 0.1 μm pore size was used. The fermentation broth was first concentrated by batch filtration to the volumetric concentration factor of aproximatelly 2.3 and then the retentate and the permeate were recirculated to keep the total solids concentration constant. During the concentration step the cross-flow velocity dropped from 6.2 m/s to 5.8 m/s due to the rise of the

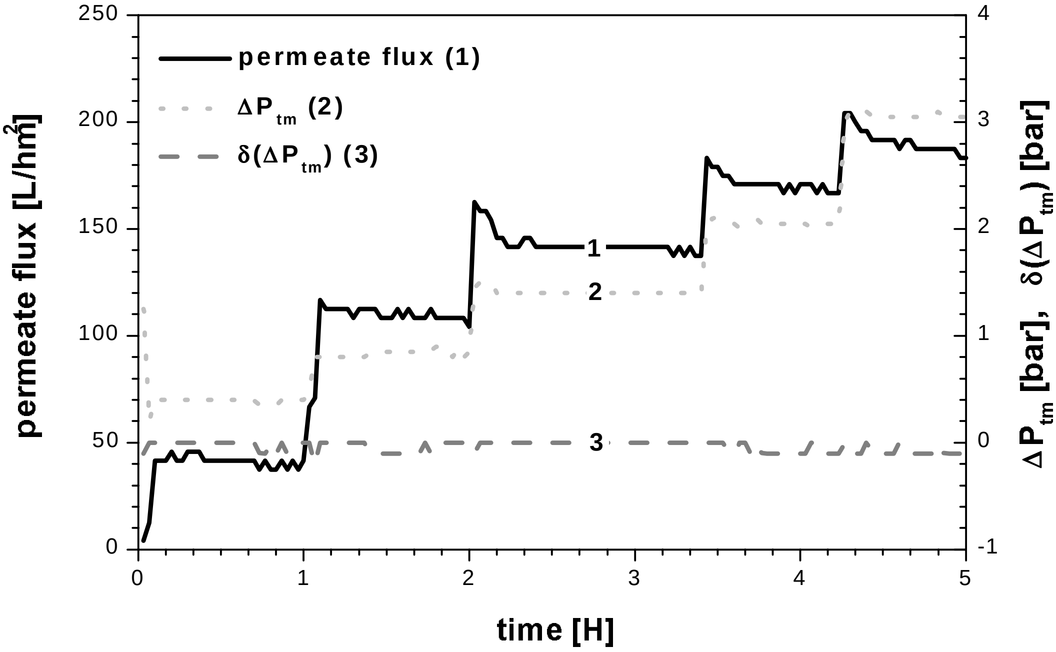


Figure 6. The influence of the transmembrane pressure drop (ΔP_{tm}) on the permeate flux during microfiltration of *Amycolatopsis orientalis* (ATCC 19759) fermentation broth (Membralox® P19-40 GL 0.5 μm, T=30±2°C, cross-flow velocity 6.8 m/s, volumetric concentration factor 1). The test was run with the dynamic permeate counter-pressure. As can be seen from the diagram, the difference $\delta(\Delta P_{tm})$ between the transmembrane pressure drop at the inlet to the filtration module ($\Delta P_{tm,\ in}$) and the transmembrane pressure drop at the oulet from the module ($\Delta P_{tm,\ out}$) is negligible.

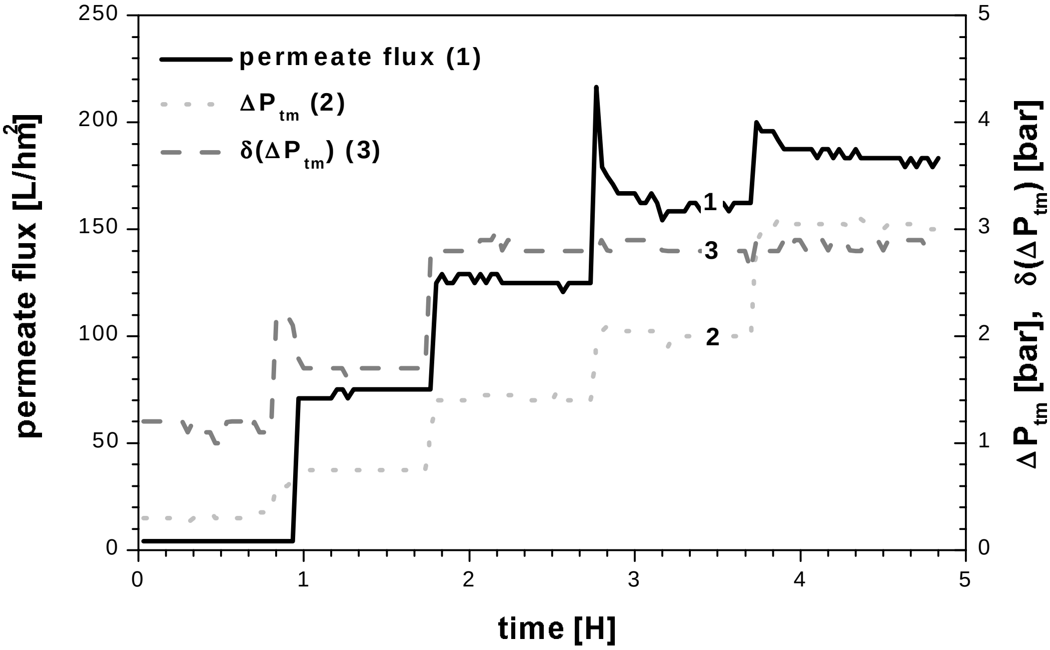


Figure 7. The influence of the transmembrane pressure drop (ΔP_{tm}) on the permeate flux during microfiltration of *Amycolatopsis orientalis* (ATCC 19759) fermentation broth (Membralox® P19-40 GL 0.5 μm, T=30±2°C, cross-flow velocity 6.8 m/s, volumetric concentration factor 1). The test was run with static permeate counter-pressure. A considerable difference $\delta(\Delta P_{tm})$ between the transmembrane pressure drop at the inlet to the filtration module ($\Delta P_{tm,\ in}$) and the transmembrane pressure drop at the oulet from the module ($\Delta P_{tm,\ out}$) can be seen from the diagram.

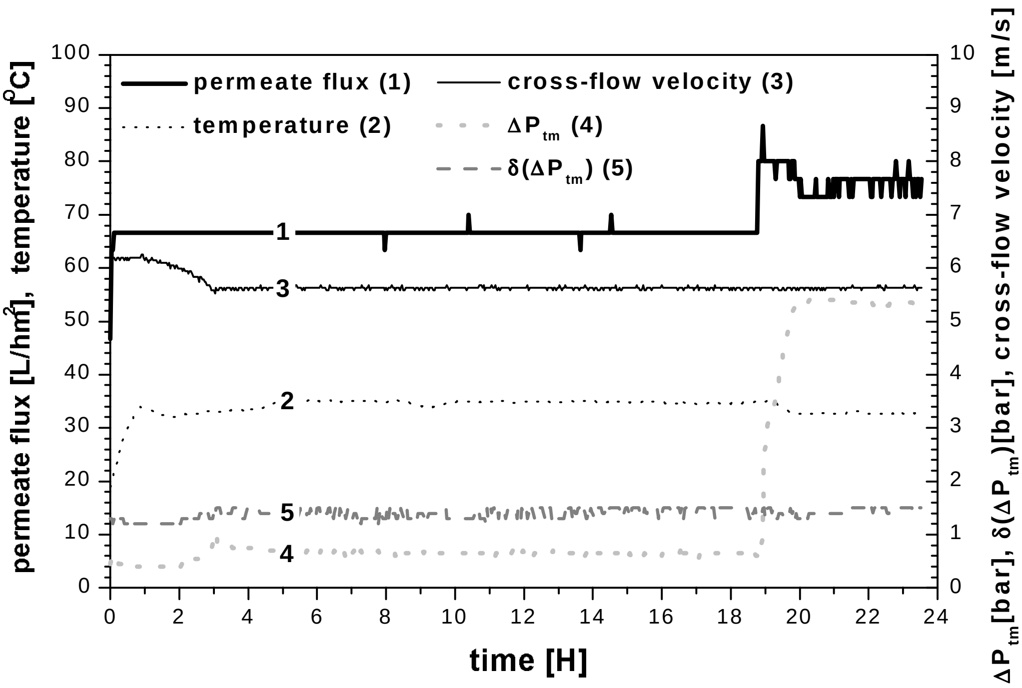


Figure 8. Study of a fouling phenomenon during the microfiltration of *Micromonospora inyoensis* fermentation broth with Membralox® P19-60 0.1 μm filtration element. The test was run with the static permeate counter-pressure. The fermentation broth was first concentrated by a batch filtration to the volumetric concentration factor of aproximatelly 2.3 and then the retentate and the permeate were recirculated to keep the total solids concentration constant.

retentate viscosity. The temperature was kept at 34±2°C. The results show that no fouling occured and the flux was stable until the set point of the permeate flow rate was changed to a higher value. The transmembrane pressure drop raised from 0.6 bars to 5.4 bars, while the flux was not improved considerably. The reasons could be due to faster fouling and the compression of the secondary layer under higher transmembrane pressure drops. When the fouling phenomenon was studied with another batch of the *Micromonospora inyoensis* fermentation broth (Figure 9), the flux of the permeate dropped to half of its initial value and the transmembrane pressure drop raised from approximately 1 bar to more than 5 bars during batch filtration. After the recirculation of the permeate and the retentate was set, the flux remained stable until the end of the run.

CONCLUSIONS

An automated microfiltration and ultrafiltration pilot plant unit of our original

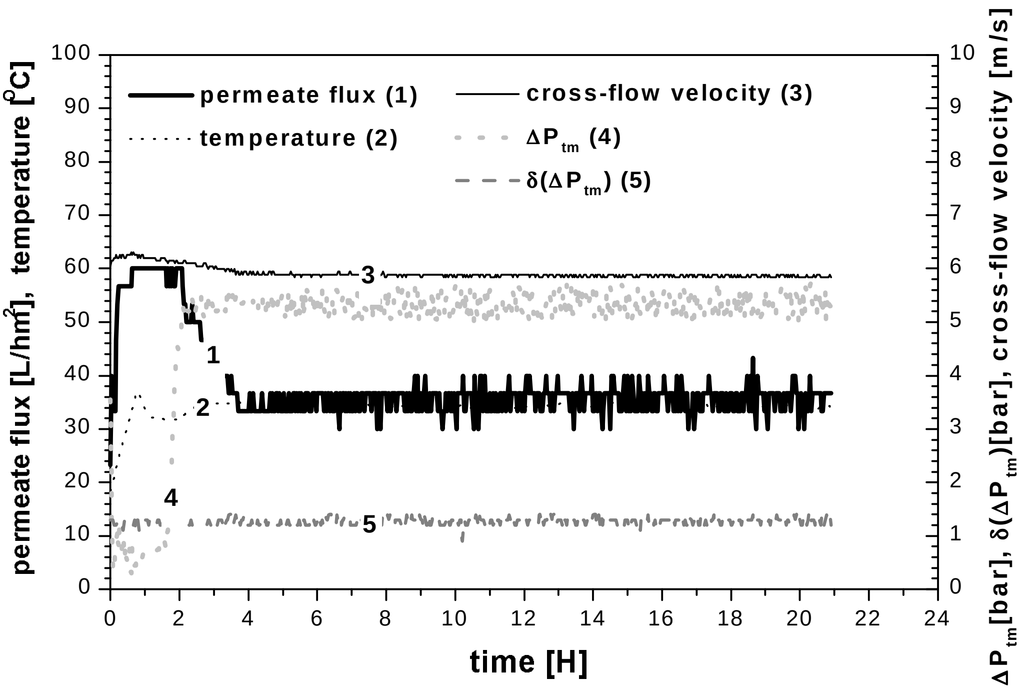


Figure 9. Study of the fouling phenomenon during the microfiltration of *Micromonospora inyoensis* fermentation broth with Membralox® P19-60 0.1 μm filtration element. The test was run with the static permeate counter-pressure. The fermentation broth was first concentrated by a batch filtration to the volumetric concentration factor of aproximatelly 2.3 and then the retentate and the permeate were recirculated to keep the total solids concentration constant. Flux of the permeate dropped to half of its initial value and the transmembrane pressure drop (ΔP_{tm}) raised from approximately 1 bar to more than 5 bars during batch filtration. After the recirculation velocity of the permeate and the retenate were set, the flux remained stable until the end of the run.

design with a broad range of possible operating conditions is described. The pilot plant unit is sucessfully used for the developement and the optimization of microfiltration and ultrafiltration steps in a downstreaming of various biotechnological products. Process parameters which are necessary for a full-scale microfiltration plant design were estimated and economical evaluation of the process was done on the basis of the results, obtained from the described pilot plant unit for clavulanic acid fermentation broth.

The pilot plant unit design enables the study of the effects of all important variables, (such as transmembrane pressure drop, retentate cross-flow velocity, temperature, pH, membrane types, static and dynamic permeate counter-pressure, fermentation broth properties) on the permeate flux and the permeate quality. Fouling and solute rejection phenomena can also be studied. The use of a dynamic permeate counter-pressure offers an excellent tool for cross-flow filtration studies to be done under carefully controled operating conditions.

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POVZETEK

Opisana je pilotna naprava za obtočno mikrofiltracijo in ultrafiltracijo, lastne konstrukcije, ki omogoča kontrolo in spremljanje pomembnih obratovalnih parametrov, kot so transmembranski pritisk, obtočna hitrost, pretok permeata, temperatura in pH. Obratovanje naprave kontrolira industrijski programabilni kontroler (PLC), ki je povezan z osebnim računalnikom. Razvita je bila lastna SCADA aplikacija, ki omogoča upravljanje naprave z osebnega računalnika ter avtomatski zajem in shranjevanje izmerjenih vrednosti. Naprava lahko obratuje ali s statičnim ali z dinamičnim protitlakom permeata. Dinamični protitlak permeata zagotavlja konstanten transmembranski tlak vzdolž celotnega filtracijskega elementa. Izbiramo lahko med obratovanjem s *konstantnim transmembranskim pritiskom,* med obratovanjem s *konstantnim pretokom permeata* ter med *stand-by* načinom, ki se uporablja na začetku filtracije in pri operacijah čiščenja membran. Naprava se uspešno uporablja za mikrofiltracijo in ultrafiltracijo različnih fermentacijskih brozg. Prikazanih in razloženih je tudi nekaj eksperimentalnih rezultatov, ki ilustrirajo uporabnost pilotne naprave.